AUTHENTICATED
U.S. GOVERNMENT
INFORMATION
GPO

PART 136—GUIDELINES ESTABLISHING TEST PROCEDURES FOR THE ANALYSIS OF POLLUTANTS

Sec.
136.1 Applicability.
136.2 Definitions.
136.3 Identification of test procedures.
136.4 Application for alternate test procedures.
136.5 Approval of alternate test procedures.
APPENDIX A TO PART 136—METHODS FOR ORGANIC CHEMICAL ANALYSIS OF MUNICIPAL AND INDUSTRIAL WASTEWATER
APPENDIX B TO PART 136—DEFINITION AND PROCEDURE FOR THE DETERMINATION OF THE METHOD DETECTION LIMIT—REVISION 1.11
APPENDIX C TO PART 136—INDUCTIVELY COUPLED PLASMA—ATOMIC EMISSION SPECTROMETRIC METHOD FOR TRACE ELEMENT ANALYSIS OF WATER AND WASTES METHOD 200.7
APPENDIX D TO PART 136—PRECISION AND RECOVERY STATEMENTS FOR METHODS FOR MEASURING METALS

AUTHORITY: Secs. 301, 304(h), 307 and 501(a), Pub. L. 95–217, 91 Stat. 1566, et seq. (33 U.S.C. 1251, et seq.) (the Federal Water Pollution Control Act Amendments of 1972 as amended by the Clean Water Act of 1977).

§ 136.1 Applicability.

The procedures prescribed herein shall, except as noted in § 136.5, be used to perform the measurements indicated whenever the waste constituent specified is required to be measured for:

(a) An application submitted to the Administrator, or to a State having an approved NPDES program for a permit under section 402 of the Clean Water Act of 1977, as amended (CWA), and/or to reports required to be submitted under NPDES permits or other requests for quantitative or qualitative effluent data under parts 122 to 125 of title 40, and,

(b) Reports required to be submitted by discharges under the NPDES established by parts 124 and 125 of this chapter, and,

(c) Certifications issued by States pursuant to section 401 of the CWA, as amended.

[38 FR 28758, Oct. 16, 1973, as amended at 49 FR 43250, Oct. 26, 1984]

§ 136.2 Definitions.

As used in this part, the term:

(a) *Act* means the Clean Water Act of 1977, Pub. L. 95–217, 91 Stat. 1566, et seq. (33 U.S.C. 1251 et seq.) (The Federal Water Pollution Control Act Amendments of 1972 as amended by the Clean Water Act of 1977).

(b) *Administrator* means the Administrator of the U.S. Environmental Protection Agency.

(c) *Regional Administrator* means one of the EPA Regional Administrators.

(d) *Director* means the Director of the State Agency authorized to carry out an approved National Pollutant Discharge Elimination System Program under section 402 of the Act.

(e) *National Pollutant Discharge Elimination System (NPDES)* means the national system for the issuance of permits under section 402 of the Act and includes any State or interstate program which has been approved by the Administrator, in whole or in part, pursuant to section 402 of the Act.

(f) *Detection limit* means the minimum concentration of an analyte (substance) that can be measured and reported with a 99% confidence that the analyte concentration is greater than zero as determined by the procedure set forth at appendix B of this part.

[38 FR 28758, Oct. 16, 1973, as amended at 49 FR 43250, Oct. 26, 1984]

§ 136.3 Identification of test procedures.

(a) Parameters or pollutants, for which methods are approved, are listed together with test procedure descriptions and references in tables IA, IB, IC, ID, and IE. The full text of the referenced test procedures are incorporated by reference into tables IA, IB, IC, ID, and IE. The references and the sources from which they are available are given in paragraph (b) of this section. These test procedures are incorporated as they exist on the day of approval and a notice of any change in these test procedures will be published in the FEDERAL REGISTER. The discharge parameter values for which reports are required must be determined by one of the standard analytical test procedures incorporated by reference and described in tables IA, IB, IC, ID, and IE, or by any alternate test procedure which has been approved by the Administrator under the provisions of

paragraph (d) of this section and §§ 136.4 and 136.5 of this part 136. Under certain circumstances (§ 136.3 (b) or (c) or 40 CFR 401.13) other test procedures may be used that may be more advantageous when such other test procedures have been previously approved by the Regional Administrator of the Region in which the discharge will occur, and providing the Director of the State in which such discharge will occur does not object to the use of such alternate test procedure.

TABLE IA.—LIST OF APPROVED BIOLOGICAL METHODS

Parameter and units	Method [1]	EPA	Standard methods, 18th Ed.	ASTM	USGS
Bacteria:					
1. Coliform (fecal), number per 100 mL.	Most Probable Number (MPN), 5 tube	p. 132 [3]	9221C E [4]	.....	
	3 dilution, or Membrane filter (MF) [2], single step	p. 124 [3]	9222D [4]		B–0050–85 [5]
2. Coliform (fecal) in presence of chlorine, number per 100 mL.	MPN, 5 tube, 3 dilution, or	p. 132 [3]	9221C E [4]	.....	
	MF, single step [6]	p. 124 [3]	9222D [4]		
3. Coliform (total), number per 100 mL.	MPN, 5 tube, 3 dilution, or	p. 114 [3]	9221B [4]	.....	
	MF [2] single step or two step	p. 108 [3]	9222B [4]		B–0025–85 [5]
4. Coliform (total), in presence of chlorine, number per 100 mL.	MPN, 5 tube, 3 dilution, or	p. 114 [3]	9221B [4]	.....	
	MF [2] with enrichment	p. 111 [3]	9222(B+B.5c) [4]		
5. Fecal streptococci, number per 100 mL.	MPN, 5 tube, 3 dilution	p. 139 [3]	9230B [4]	.....	
	MF [2], or	p. 136 [3]	9230C [4]		B–0055–85 [5]
	Plate count	p. 143 [3]			
Aquatic Toxicity:					
6. Toxicity, acute, fresh water organisms, LC50, percent effluent.	Daphnia, Ceriodaphnia, Fathead Minnow, Rainbow Trout, Brook Trout, or Bannerfish Shiner mortality.	Sec. 9 [7]		.....	
7. Toxicity, acute, estuarine and marine organisms, LC50, percent effluent.	Mysid, Sheepshead Minnow, or Menidia spp. mortality	Sec. 9 [7]		.....	
8. Toxicity, chronic, fresh water organisms, NOEC or IC25, percent effluent.	Fathead minnow larval survival and growth	1000.0 [8]		.....	
	Fathead minnow embryo-larval survival and teratogenicity	1001.0 [8]			
	Ceriodaphnia survival and reproduction				
	Selenastrum growth	1002.0 [8]			
		1003.0 [8]			
9. Toxicity, chronic, estuarine and marine organisms, NOEC or IC25, percent effluent.	Sheepshead minnow larval survival and growth	1004.0 [9]		.....	
	Sheepshead minnow embryo-larval survival and teratogenicity	1005.0 [9]			
	Menidia beryllina larval and growth				
	Mysidopsis bahia survival, growth, and fecundity	1006.0 [9]			
	Arbacia punctulata fertilization	1007.0 [9]			
	Champia parvula reproduction	1008.0 [9]			
		1009.0 [9]			

Notes to Table IA:

[1] The method must be specified when results are reported.

[2] A 0.45 um membrane filter (MF) or other pore size certified by the manufacturer to fully retain organisms to be cultivated and to be free of extractables which could interfere with their growth.

[3] USEPA. 1978. Microbiological Methods for Monitoring the Environment, Water, and Wastes. Environmental Monitoring and Support Laboratory, U.S. Environmental Protection Agency, Cincinnati, Ohio. EPA/600/8–78/017.

[4] APHA. 1992. Standard Methods for the Examination of Water and Wastewater. American Public Health Association. 18th Edition. Amer. Publ. Hlth. Assoc., Washington, DC.

[5] USGS. 1989. U.S. Geological Survey Techniques of Water-Resources Investigations, Book 5, Laboratory Analysis, Chapter A4, Methods for Collection and Analysis of Aquatic Biological and Microbiological Samples, U.S. Geological Survey, U.S. Department of Interior, Reston, Virginia.

[6] Because the MF technique usually yields low and variable recovery from chlorinated wastewaters, the Most Probable Number method will be required to resolve any controversies.

[7] USEPA. 1993. Methods for Measuring the Acute Toxicity of Effluents to Freshwater and Marine Organisms. Fourth Edition. Environmental Monitoring Systems Laboratory, U.S. Environmental Protection Agency, Cincinnati, Ohio. August 1993, EPA/600/4–90/027F.

[8] USEPA. 1994. Short-term Methods for Estimating the Chronic Toxicity of Effluents and Receiving Waters to Freshwater Organisms. Third Edition. Environmental Monitoring Systems Laboratory, U.S. Environmental Protection Agency USEPA. 1994, Cincinnati, Ohio (July 1994, EPA/600/4–91/002).

[9] Short-term Methods for Estimating the Chronic Toxicity of Effluents and Receiving Waters to Marine and Estuarine Organisms. Second Edition. Environmental Monitoring Systems Laboratory, U.S. Environmental Protection Agency, Cincinnati, Ohio (July 1994, EPA/600/4–91/003). These methods do not apply to marine waters of the Pacific Ocean.

TABLE IB.—LIST OF APPROVED INORGANIC TEST PROCEDURES

Parameter, units and method	Reference (method number or page)				
	EPA [1,35]	STD methods 18th ed.	ASTM	USGS [2]	Other
1. Acidity, as $CaCO_3$, mg/L:					
Electrometric endpoint or phenolphthalein endpoint	305.1	2310 B(4a)	D1067–92		
2. Alkalinity, as $CaCO_3$, mg/L:					
Electrometric or Colorimetric titration to pH 4.5, manual or automated.	310.1	2320 B	D1067–92	I–1030–85	973.43.[3]
	310.2			I–2030–85	
3. Aluminum—Total,[4] mg/L; Digestion [4] followed by:					
AA direct aspiration [36]	202.1	3111 D		I–3051–85	
AA furnace	202.2	3113 B			
Inductively Coupled Plasma/Atomic Emission Spectrometry (ICP/AES) [36].	[5] 200.7	3120 B			
Direct Current Plasma (DCP) [36]			D4190–82(88)		Note 34.
Colorimetric (Eriochrome cyanine R)		3500–Al D			
4. Ammonia (as N), mg/L:					
Manual, distillation (at pH 9.5),[6] followed by	350.2	4500–NH_3 B			973.49.[3]
Nesslerization	350.2	4500–NH_3 C	D1426–93(A)	I–3520–85	973.49.[3]
Titration	350.2	4500–NH_3 E			
Electrode	350.3	4500–NH_3 F or G	D1426–93(B)		
Automated phenate, or	350.1	4500–NH_3 H		I–4523–85	
Automated electrode					Note 7.
5. Antimony-Total,[4] mg/L; Digestion [4] followed by:					
AA direct aspiration [36]	204.1	3111 B			
AA furnace	204.2	3113 B			
ICP/AES [36]	[5] 200.7	3120 B			
6. Arsenic-Total,[4] mg/L:					
Digestion [4] followed by	206.5				
AA gaseous hydride	206.3	3114 B 4.d	D2972–93(B)	I–3062–85	
AA furnace	206.2	3113 B	D2972–93(C)		
ICP/AES,[36] or	[5] 200.7	3120 B			
Colorimetric (SDDC)	206.4	3500–As C	D2972–93(A)	I–3060–85	
7. Barium—Total,[4] mg/L; Digestion [4] followed by:					
AA direct aspiration [36]	208.1	3111 D		I–3084–85	
AA furnace	208.2	3113 B	D4382–91		
ICP/AES [36]	[5] 200.7	3120 B			
DCP [36]					Note 34.
8. Beryllium—Total,[4] mg/L; Digestion [4] followed by:					
AA direct aspiration	210.1	3111 D	D3645–93(88)(A)	I–3095–85	
AA furnace	210.2	3113 B	D3645–93(88)(B)		
ICP/AES	[5] 200.7	3120 B			
DCP, or			D4190–82(88)		Note 34.

Colorimetric (aluminon)		3500–Be D			
9. Biochemical oxygen demand (BOD_5), mg/L:					
Dissolved Oxygen Depletion	405.1	5210 B		I–1578–78 [8]	973.44,[3] p. 17.[9]
10. Boron [37]—Total, mg/L:					
Colorimetric (curcumin)	212.3	4500–B B		I–3112–85	
ICP/AES, or	[5] 200.7	3120 B			
DCP			D4190–82(88)		Note 34
11. Bromide, mg/L:					
Titrimetric	320.1		D1246–82(88)(C)	I–1125–85	p. S44.[10]
12. Cadmium—Total,[4] mg/L; Digestion [4] followed by:					
AA direct aspiration [36]	213.1	3111 B or C	D3557–90(A or B)	I–3135–85 or I–3136–85	974.27,[3] p. 37.[9]
AA furnace	213.2	3113 B	D3557–90(D)		
ICP/AES [36]	[5] 200.7	3120 B		I–1472–85	
DCP [36]			D4190–82(88)		Note 34.
Voltametry,[11] or			D3557–90(C)		
Colorimetric (Dithizone)		3500–Cd D			
13. Calcium—Total,[4] mg/L; Digestion [4] followed by:					
AA direct aspiration	215.1	3111 B	D511–93(B)	I–3152–85	
ICP/AES	[5] 200.7	3120 B			
DCP, or					Note 34.
Titrimetric (EDTA)	215.2	3500–Ca D	D511–93(A)		
14. Carbonaceous biochemical oxygen demand ($CBOD_5$), mg/L [12]:					
Dissolved Oxygen Depletion with nitrification inhibitor.		5210 B			
15. Chemical oxygen demand (COD), mg/L; Titrimetric, or.	410.1	5220 C	D1252–88(A)	I–3560–85	973.46,[3] p. 17.[9]
	410.2			I–3562–85	
	410.3				
Spectrophotometric, manual or automated	410.4	5220 D	D1252–88(B)	I–3561–85	Notes 13 or 14.
16. Chloride, mg/L:					
Titrimetric (silver nitrate) or		4500–Cl^- B	D512–89(B)	I–1183–85	
(Mercuric nitrate)	325.3	4500–Cl^- C	D512–89(A)	I–1184–85	973.51.[3]
Colorimetric, manual or				I–1187–85	
Automated (Ferricyanide)	325.1 or 325.2	4500–Cl^- E		I–2187–85	
17. Chlorine—Total residual, mg/L; Titrimetric:					
Amperometric direct	330.1	4500–Cl D	D1253–86(92)		
Iodometric direct	330.3	4500–Cl B			
Back titration ether end- point [15] or	330.2	4500–Cl C			
DPD–FAS	330.4	4500–Cl F			
Spectrophotometric, DPD	330.5	4500–Cl G			
Or Electrode					Note 16.
18. Chromium VI dissolved, mg/L; 0.45 micron filtration followed by:					
AA chelation-extraction or	218.4	3111 C		I–1232–85	
Colorimetric (Diphenylcarbazide)		3500–Cr D	D1687–92(A)	I–1230–85	
19. Chromium—Total,[4] mg/L; Digestion [4] followed by:					
AA direct aspiration [36]	218.1	3111 B	D1687–92(B)	I–3236–85	974.27.[3]
AA chelation-extraction	218.3	3111 C			
AA furnace	218.2	3113 B	D1687–92(C)		

TABLE IB.—LIST OF APPROVED INORGANIC TEST PROCEDURES—Continued

Parameter, units and method	Reference (method number or page)				
	EPA [1,35]	STD methods 18th ed.	ASTM	USGS [2]	Other
ICP/AES [36]	[5] 200.7	3120 B			
DCP,[36] or			D4190–82(88)		Note 34.
Colorimetric (Diphenylcarbazide)		3500–Cr D			
20. Cobalt—Total,[4] mg/L; Digestion [4] followed by:					
AA direct aspiration	219.1	3111 B or C	D3558–90(A or B)	I–3239–85	p. 37.[9]
AA furnace	219.2	3113 B	D3558–90(C)		
ICP/AES	[5] 200.7	3120 B			
DCP			D4190–82(88)		Note 34.
21. Color platinum cobalt units or dominant wavelength, hue, luminance purity:					
Colorimetric (ADMI), or	110.1	2120 E			Note 18.
(Platinum cobalt), or	110.2	2120 B		I–1250–85	
Spectrophotometric	110.3	2120 C			
22. Copper—Total,[4] mg/L; Digestion [4] followed by:					
AA direct aspiration [36]	220.1	3111 B or C	D1688–90(A or B)	I–3270–85 or I3271–85	974.27 [3] p. 37.[9]
AA furnace	220.2	3113 B	D1688–90(C)		
ICP/AES [36]	[5] 200.7	3120 B			
DCP [36] or			D4190–82(88)		Note 34.
Colorimetric (Neocuproine) or		3500–Cu D			
(Bicinchoninate)		Or E			Note 19.
23. Cyanide—Total, mg/L:					
Manual distillation with $MgCl_2$ followed by		4500–CN C	D2036–91(A)		
Titrimetric, or		4500–CN D			p. 22.[9]
Spectrophotometric, manual or	[31] 335.2	4500–CN E	D2036–91(A)	I–3300–85	
Automated [20]	[31] 335.3				
24. Cyanide amenable to chlorination,mg/L:					
Manual distillation with $MgCl_2$ followed by titrimetric or Spectrophotometric.	335.1	4500–CN G	D2036–91(B)		
25. Fluoride—Total, mg/L:					
Manual distillation [6] followed by		4500–F B			
Electrode, manual or	340.2	4500–F C	D1179–93(B)		
Automated				I–4327–85	
Colorimetric (SPADNS)	340.1	4500–F D	D1179–93(A)		
Or Automated complexone	340.3	4500–F E			
26. Gold—Total,[4] mg/L; Digestion [4] followed by:					
AA direct aspiration	231.1	3111 B			
AA furnace, or	231.2				
DCP					Note 34.
27. Hardness—Total, as $CaCO_3$, mg/L					
Automated colorimetric,	130.1				
Titrimetric (EDTA), or Ca plus Mg as their carbonates, by inductively coupled plasma or AA direct aspiration. (See Parameters 13 and 33).	130.2	2340 B or C	D1126–86(92)	I–1338–85	973.52B.[3]
28. Hydrogen ion (pH), pH units					

Electrometric measurement, or	150.1	4500–H^+ B	D1293–84(90)(A or B)	I–1586–85	973.41.[3]
Automated electrode					Note 21.
29. Iridium—Total,[4] mg/L; Digestion[4] followed by:					
AA direct aspiration or	235.1	3111 B			
AA furnace	235.2				
30. Iron—Total,[4] mg/L; Digestion[4] followed by:					
AA direct aspiration[36]	236.1	3111 B or C	D1068–90(A or B)	I–3381–85	974.27.[3]
AA furnace	236.2	3113 B	D1068–90(C)		
ICP/AES[36]	[5]200.7	3120 B			
DCP[36] or			D4190–82(88)		Note 34.
Colorimetric (Phenanthroline)		3500–Fe D	D1068–90(D)		Note 22.
31. Kjeldahl Nitrogen—Total, (as N), mg/L:					
Digestion and distillation followed by:	351.3	4500–NH_3 B or C	D3590–89(A)		
Titration	351.3	4500–NH_3 E	D3590–89(A)		973.48[3].
Nesslerization	351.3	4500–NH_3 C	D3590–89(A)		
Electrode	351.3	4500–NH_3 F or G			
Automated phenate colorimetric	351.1			I–4551–78[8]	
Semi-automated block digester colorimetric	351.2		D3590–89(B)		
Manual or block digester potentiometric	351.4		D3590–89(A)		
Block Digester, followed by:.					
Auto distillation and Titration, or					Note 39.
Nesslerization					Note 40.
Flow injection gas diffusion					Note 41.
32. Lead—Total,[4] mg/L; Digestion[4] followed by:					
AA direct aspiration[36]	239.1	3111 B or C	D3559–90(A or B)	I–3399–85	974.27.[3]
AA furnace	239.2	3113 B	D3559–90(D)		
ICP/AES[36]	[5]200.7	3120 B			
DCP[36]			D4190–82(88)		Note 34.
Voltametry[11] or			D3559–90(C)		
Colorimetric (Dithizone)		3500–Pb D			
33. Magnesium—Total,[4] mg/L; Digestion[4] followed by:					
AA direct aspiration	242.1	3111 B	D511–93(B)	I–3447–85	974.27.[3]
ICP/AES	[5]200.7	3120 B			
DCP, or					Note 34.
Gravimetric		3500–Mg D			
34. Manganese—Total,[4] mg/L; Digestion[4] followed by:					
AA direct aspiration[36]	243.1	3111 B	D858–90(A or B)	I–3454–85	974.27.[3]
AA furnace	243.2	3113 B	D858–90(C)		
ICP/AES[36]	[5]200.7	3120 B			
DCP[36] or			D4190–82(88)		Note 34.
Colorimetric (Persulfate), or		3500–Mn D			920.203.[3]
(Periodate)					Note 23.
35. Mercury—Total,[4] mg/L:					
Cold vapor, manual or	245.1	3112 B	D3223–91	I–3462–85	977.22.[3]
Automated	245.2				
36. Molybdenum—Total,[4] mg/L; Digestion[4] followed by:					
AA direct aspiration	246.1	3111 D		I–3490–85	
AA furnace	246.2	3113 B			
ICP/AES	[5]200.7	3120 B			

TABLE IB.—LIST OF APPROVED INORGANIC TEST PROCEDURES—Continued

Parameter, units and method	Reference (method number or page)				
	EPA [1,35]	STD methods 18th ed.	ASTM	USGS [2]	Other
DCP					Note 34.
37. Nickel—Total,[4] mg/L; Digestion [4] followed by:					
AA direct aspiration [36]	249.1	3111 B or C	D1886–90(A or B)	I–3499–85	
AA furnace	249.2	3113 B	D1886–90(C)		
ICP/AES [36]	[5] 200.7	3120 B			
DCP [36], or			D4190–82(88)		Note 34.
Colorimetric (heptoxime)		3500–Ni D			
38. Nitrate (as N), mg/L:					
Colorimetric (Brucine sulfate), or Nitrate-nitrite N minus Nitrite N (See parameters 39 and 40).	352.1				973.50,[3] 419 D,[17] p. 28.[9]
39. Nitrate-nitrite (as N), mg/L:					
Cadmium reduction, Manual or	353.3	4500–NO_3^- E	D3867–90(B)		
Automated, or	353.2	4500–NO_3^- F	D3867–90(A)	I–4545–85	
Automated hydrazine	353.1	4500–NO_3^- H			
40. Nitrite (as N), mg/L; Spectrophotometric:					
Manual or	354.1	4500–NO_2^- B			Note 25.
Automated (Diazotization)				I–4540–85	
41. Oil and grease—Total recoverable, mg/L:					
Gravimetric (extraction)	413.1	5520 B [38]			
42. Organic carbon—Total (TOC), mg/L:					
Combustion or oxidation	415.1	5310 B, C, or D	D2579–93 (A or B)		973.47,[3] p. 14.[24]
43. Organic nitrogen (as N), mg/L:					
Total Kjeldahl N (Parameter 31) minus ammonia N (Parameter 4)					
44. Orthophosphate (as P), mg/L; Ascorbic acid method:					
Automated, or	365.1	4500–P F		I–4601–85	973.56.[3]
Manual single reagent	365.2	4500–P E	D515–88(A)		973.55 [3].
Manual two reagent	365.3				
45. Osmium—Total [4], mg/L; Digestion [4] followed by:					
AA direct aspiration, or	252.1	3111 D			
AA furnace	252.2				
46. Oxygen, dissolved, mg/L:					
Winkler (Azide modification), or	360.2	4500–O C	D888–92(A)	I–1575–78 [8]	973.45B.[3]
Electrode	360.1	4500–O G	D888–92(B)	I–1576–78 [8]	
47. Palladium—Total,[4] mg/L; Digestion [4] followed by:					
AA direct aspiration, or	253.1	3111 B			p. S27.[10]
AA furnace	253.2				p. S28.[10]
DCP					Note 34.
48. Phenols, mg/L:					
Manual distillation [26]	420.1				Note 27.
Followed by:					
Colorimetric (4AAP) manual, or	420.1				Note 27.
Automated [19]	420.2				
49. Phosphorus (elemental), mg/L:					

Gas-liquid chromatography					Note 28.
50. Phosphorus—Total, mg/L:					
Persulfate digestion followed by	365.2	4500–P B,5			973.55.[3]
Manual or	365.2 or 365.3	4500–P E	D515–88(A)		
Automated ascorbic acid reduction	365.1	4500–P F		I–4600–85	973.56.[3]
Semi-automated block digestor	365.4		D515–88(B)		
51. Platinum—Total,[4] mg/L; Digestion[4] followed by:					
AA direct aspiration	255.1	3111 B			
AA furnace	255.2				
DCP					Note 34.
52. Potassium—Total,[4] mg/L; Digestion[4] followed by:					
AA direct aspiration	258.1	3111 B		I–3630–85	973.53.[3]
ICP/AES	[5]200.7	3120 B			
Flame photometric, or		3500–K D			
Colorimetric					317 B.[17]
53. Residue—Total, mg/L:					
Gravimetric, 103–105°	160.3	2540 B		I–3750–85	
54. Residue—filterable, mg/L:					
Gravimetric, 180°	160.1	2540 C		I–1750–85	
55. Residue—nonfilterable (TSS), mg/L:					
Gravimetric, 103–105° post washing of residue	160.2	2540 D		I–3765–85	
56. Residue—settleable, mg/L:					
Volumetric, (Imhoff cone), or gravimetric	160.5	2540 F			
57. Residue—Volatile, mg/L:					
Gravimetric, 550°	160.4			I–3753–85	
58. Rhodium—Total,[4] mg/L; Digestion[4] followed by:					
AA direct aspiration, or	265.1	3111 B			
AA furnace	265.2				
59. Ruthenium—Total,[4] mg/L; Digestion[4] followed by:					
AA direct aspiration, or	267.1	3111 B			
AA furnace	267.2				
60. Selenium—Total,[4] mg/L; Digestion[4] followed by:					
AA furnace	270.2	3113 B	D3859–93(B)		
ICP/AES,[36] or	[5]200.7	3120 B			
AA gaseous hydride		3114 B	D3859–93(A)	I–3667–85	
61. Silica[37]—Dissolved, mg/L; 0.45 micron filtration followed by:					
Colorimetric, Manual or	370.1	4500–Si D	D859–88	I–1700–85	
Automated (Molybdosilicate), or				I–2700–85	
ICP	[5]200.7	3120 B			
62. Silver—Total,[4] mg/L; Digestion[4, 29] followed by:					
AA direct aspiration	272.1	3111 B or C		I–3720–85	974.27,[3] p. 37.[9]
AA furnace	272.2	3113 B			
ICP/AES	[5]200.7	3120 B			
DCP					Note 34.
63. Sodium—Total,[4] mg/L; Digestion[4] followed by:					
AA direct aspiration	273.1	3111 B		I–3735–85	973.54.[3]
ICP/AES	[5]200.7	3120 B			
DCP, or					Note 34.

TABLE IB.—LIST OF APPROVED INORGANIC TEST PROCEDURES—Continued

Parameter, units and method	Reference (method number or page)				
	EPA [1,35]	STD methods 18th ed.	ASTM	USGS [2]	Other
Flame photometric		3500 Na D			
64. Specific conductance, micromhos/cm at 25 °C:					
Wheatstone bridge	120.1	2510 B	D1125–91(A)	I–1780–85	973.40.[3]
65. Sulfate (as SO_4), mg/L:					
Automated colorimetric (barium chloranilate)	375.1				
Gravimetric	375.3	4500–SO_4^{-2} C or D			925.54.[3]
Turbidimetric, or	375.4		D516–90		426C.[30]
66. Sulfide (as S), mg/L:					
Titrimetric (iodine), or	376.1	4500–S^{-2} E		I–3840–85	
Colorimetric (methylene blue)	376.2	4500–S^{-2} D			
67. Sulfite (as SO_3), mg/L:					
Titrimetric (iodine-iodate)	377.1	4500–SO_3^{-2} B			
68. Surfactants, mg/L:					
Colorimetric (methylene blue)	425.1	5540 C	D2330–88		
69. Temperature, °C:					
Thermometric	170.1	2550 B			Note 32.
70. Thallium—Total,[4] mg/L; Digestion [4] followed by:					
AA direct aspiration	279.1	3111 B			
AA furnace	279.2				
ICP/AES, or	[5] 200.7	3120 B			
71. Tin—Total,[4] mg/L; Digestion [4] followed by:					
AA direct aspiration	282.1	3111 B		I–3850–78 [8]	
AA furnace, or	282.2	3113 B			
ICP/AES	[5] 200.7				
72. Titanium—Total,[4] mg/L; Digestion [4] followed by:					
AA direct aspiration	283.1	3111 D			
AA furnace	283.2				
DCP					Note 34.
73. Turbidity, NTU:					
Nephelometric	180.1	2130 B	D1889–88(A)	I–3860–85	
74. Vanadium—Total,[4] mg/L; Digestion [4] followed by:					
AA direct aspiration	286.1	3111 D			
AA furnace	286.2		D3373–93		
ICP/AES	[5] 200.7	3120 B			
DCP, or			D4190–82(88)		Note 34.
Colorimetric (Gallic acid)		3500–V D			
75. Zinc—Total,[4] mg/L; Digestion [4] followed by:					
AA direct aspiration [36]	289.1	3111 B or C	D1691–90 (A or B)	I–3900–85	974.27,[3] p. 37.[9]
AA furnace	289.2				
ICP/AES [36]	[5] 200.7	3120 B			
DCP,[36] or			D4190–82(88)		Note 34.
Colorimetric (Dithizone) or		3500–Zn E			

(Zincon)		3500–Zn F			Note 33.

Table IB Notes:

[1] "Methods for Chemical Analysis of Water and Wastes", Environmental Protection Agency, Environmental Monitoring Systems Laboratory-Cincinnati (EMSL-CI), EPA–600/4–79–020, Revised March 1983 and 1979 where applicable.

[2] Fishman, M.J., et al, "Methods for Analysis of Inorganic Substances in Water and Fluvial Sediments," U.S. Department of the Interior, Techniques of Water—Resource Investigations of the U.S. Geological Survey, Denver, CO, Revised 1989, unless otherwise stated.

[3] "Official Methods of Analysis of the Association of Official Analytical Chemists," methods manual, 15th ed. (1990).

[4] For the determination of total metals the sample is not filtered before processing. A digestion procedure is required to solubilize suspended material and to destroy possible organic-metal complexes. Two digestion procedures are given in "Methods for Chemical Analysis of Water and Wastes, 1979 and 1983". One (section 4.1.3), is a vigorous digestion using nitric acid. A less vigorous digestion using nitric and hydrochloric acids (section 4.1.4) is preferred; however, the analyst should be cautioned that this mild digestion may not suffice for all samples types. Particularly, if a colorimetric procedure is to be employed, it is necessary to ensure that all organo-metallic bonds be broken so that the metal is in a reactive state. In those situations, the vigorous digestion is to be preferred making certain that at no time does the sample go to dryness. Samples containing large amounts of organic materials may also benefit by this vigorous digestion, however, vigorous digestion with concentrated nitric acid will convert antimony and tin to insoluble oxides and render them unavailable for analysis. Use of ICP/AES as well as determinations for certain elements such as antimony, arsenic, the noble metals, mercury, selenium, silver, tin, and titanium require a modified sample digestion procedure and in all cases the method write-up should be consulted for specific instructions and/or cautions.

NOTE TO TABLE IB NOTE 4: If the digestion procedure for direct aspiration AA included in one of the other approved references is different than the above, the EPA procedure must be used.

Dissolved metals are defined as those constituents which will pass through a 0.45 micron membrane filter. Following filtration of the sample, the referenced procedure for total metals must be followed. Sample digestion of the filtrate for dissolved metals (or digestion of the original sample solution for total metals) may be omitted for AA (direct aspiration or graphite furnace) and ICP analyses, provided the sample solution to be analyzed meets the following criteria:

a. has a low COD (<20)
b. is visibly transparent with a turbidity measurement of 1 NTU or less
c. is colorless with no perceptible odor, and
d. is of one liquid phase and free of particulate or suspended matter following acidification.

[5] The full text of Method 200.7, "Inductively Coupled Plasma Atomic Emission Spectrometric Method for Trace Element Analysis of Water and Wastes," is given at Appendix C of this Part 136.

[6] Manual distillation is not required if comparability data on representative effluent samples are on company file to show that this preliminary distillation step is not necessary: however, manual distillation will be required to resolve any controversies.

[7] Ammonia, Automated Electrode Method, Industrial Method Number 379–75 WE, dated February 19, 1976, (Bran & Luebbe (Technicon) Auto Analyzer II, Bran & Luebbe Analyzing Technologies, Inc., Elmsford, NY 10523.

[8] The approved method is that cited in "Methods for Determination of Inorganic Substances in Water and Fluvial Sediments", USGS TWRI, Book 5, Chapter A1 (1979).

[9] American National Standard on Photographic Processing Effluents, Apr. 2, 1975. Available from ANSI, 1430 Broadway, New York, NY 10018.

[10] "Selected Analytical Methods Approved and Cited by the United States Environmental Protection Agency", Supplement to the Fifteenth Edition of Standard Methods for the Examination of Water and Wastewater (1981).

[11] The use of normal and differential pulse voltage ramps to increase sensitivity and resolution is acceptable.

[12] Carbonaceous biochemical oxygen demand ($CBOD_5$) must not be confused with the traditional BOD_5 test which measures "total BOD". The addition of the nitrification inhibitor is not a procedural option, but must be included to report the $CBOD_5$ parameter. A discharger whose permit requires reporting the traditional BOD_5 may not use a nitrification inhibitor in the procedure for reporting the results. Only when a discharger's permit specifically states $CBOD_5$ is required can the permittee report data using the nitrification inhibitor.

[13] OIC Chemical Oxygen Demand Method, Oceanography International Corporation, 1978, 512 West Loop, P.O. Box 2980, College Station, TX 77840.

[14] Chemical Oxygen Demand, Method 8000, Hach Handbook of Water Analysis, 1979, Hach Chemical Company, P.O. Box 389, Loveland, CO 80537.

[15] The back titration method will be used to resolve controversy.

[16] Orion Research Instruction Manual, Residual Chlorine Electrode Model 97–70, 1977, Orion Research Incorporated, 840 Memorial Drive, Cambridge, MA 02138. The calibration graph for the Orion residual chlorine method must be derived using a reagent blank and three standard solutions, containing 0.2, 1.0, and 5.0 ml 0.00281 N potassium iodate/100 ml solution, respectively.

[17] The approved method is that cited in Standard Methods for the Examination of Water and Wastewater, 14th Edition, 1976.

[18] National Council of the Paper Industry for Air and Stream Improvement, (Inc.) Technical Bulletin 253, December 1971.

[19] Copper, Biocinchoinate Method, Method 8506, Hach Handbook of Water Analysis, 1979, Hach Chemical Company, P.O. Box 389, Loveland, CO 80537.

[20] After the manual distillation is completed, the autoanalyzer manifolds in EPA Methods 335.3 (cyanide) or 420.2 (phenols) are simplified by connecting the re-sample line directly to the sampler. When using the mainfold setup shown in Method 335.3, the buffer 6.2 should be replaced with the buffer 7.6 found in Method 335.2.

[21] Hydrogen ion (pH) Automated Electrode Method, Industrial Method Number 378–75WA, October 1976, Bran & Luebbe (Technicon) Autoanalyzer II. Bran & Luebbe Analyzing Technologies, Inc., Elmsford, NY 10523.

[22] Iron, 1,10-Phenanthroline Method, Method 8008, 1980, Hach Chemical Company, P.O. Box 389, Loveland, CO 80537.

[23] Manganese, Periodate Oxidation Method, Method 8034, Hach Handbook of Wastewater Analysis, 1979, pages 2–113 and 2–117, Hach Chemical Company, Loveland, CO 80537.

[24] Wershaw, R.L., et al, "Methods for Analysis of Organic Substances in Water," Techniques of Water-Resources Investigation of the U.S. Geological Survey, Book 5, Chapter A3, (1972 Revised 1987) p. 14.

[25] Nitrogen, Nitrite, Method 8507, Hach Chemical Company, P.O. Box 389, Loveland, CO 80537.

[26] Just prior to distillation, adjust the sulfuric-acid-preserved sample to pH 4 with 1 + 9 NaOH.

[27] The approved method is cited in Standard Methods for the Examination of Water and Wastewater, 14th Edition. The colorimetric reaction is conducted at a pH of 10.0±0.2. The approved methods are given on pp 576–81 of the 14th Edition: Method 510A for distillation, Method 510B for the manual colorimetric procedure, or Method 510C for the manual spectophotometric procedure.

[28] R. F. Addison and R.G. Ackman, ''Direct Determination of Elemental Phosphorus by Gas-Liquid Chromatography,'' Journal of Chromatography, vol. 47, No. 3, pp. 421–426, 1970.

[29] Approved methods for the analysis of silver in industrial wastewaters at concentrations of 1 mg/L and above are inadequate where silver exists as an inorganic halide. Silver halides such as the bromide and chloride are relatively insoluble in reagents such as nitric acid but are readily soluble in an aqueous buffer of sodium thiosulfate and sodium hydroxide to pH of 12. Therefore, for levels of silver above 1 mg/L, 20 mL of sample should be diluted to 100 mL by adding 40 mL each of 2 M $Na_2S_2O_3$ and NaOH. Standards should be prepared in the same manner. For levels of silver below 1 mg/L the approved method is satisfactory.

[30] The approved method is that cited in Standard Methods for the Examination of Water and Wastewater, 15th Edition.

[31] EPA Methods 335.2 and 335.3 require the NaOH absorber solution final concentration to be adjusted to 0.25 N before colorimetric determination of total cyanide.

[32] Stevens, H.H., Ficke, J.F., and Smoot, G.F., ''Water Temperature—Influential Factors, Field Measurement and Data Presentation'', Techniques of Water-Resources Investigations of the U.S. Geological Survey, Book 1, Chapter D1, 1975.

[33] Zinc, Zincon Method, Method 8009, Hach Handbook of Water Analysis, 1979, pages 2–231 and 2–333, Hach Chemical Company, Loveland, CO 80537.

[34] ''Direct Current Plasma (DCP) Optical Emission Spectrometric Method for Trace Elemental Analysis of Water and Wastes, Method AES0029,'' 1986—Revised 1991, Fison Instruments, Inc., 32 Commerce Center, Cherry Hill Drive, Danvers, MA 01923.

[35] Precision and recovery statements for the atomic absorption direct aspiration and graphite furnace methods, and for the spectrophotometric SDDC method for arsenic are provided in Appendix D of this part titled, ''Precision and Recovery Statements for Methods for Measuring Metals''.

[36] ''Closed Vessel Microwave Digestion of Wastewater Samples for Determination of Metals'', CEM Corporation, P.O. Box 200, Matthews, NC 28106–0200, April 16, 1992. Available from the CEM Corporation.

[37] When determining boron and silica, only plastic, PTFE, or quartz laboratory ware may be used from start until completion of analysis.

[38] Only the trichlorofluoromethane extraction solvent is approved.

[39] Nitrogen, Total Kjeldahl, Method PAI–DK01 (Block Digestion, Steam Distillation, Titrimetric Detection), revised 12/22/94, Perstop Analytical Corporation.

[40] Nitrogen, Total Kjeldahl, Method PAI–DK02 (Block Digestion, Steam Distillation, Colorimetric Detection), revised 12/22/94, Perstop Analytical Corporation.

[41] Nitrogen, Total Kjeldahl, Method PAI–DK03 (Block Digestion, Automated FIA Gas Diffusion), revised 12/22/94, Perstop Analytical Corporation.

TABLE IC.—LIST OF APPROVED TEST PROCEDURES FOR NON-PESTICIDE ORGANIC COMPOUNDS

Parameter [1]	EPA method number [27]					
	GC	GC/MS	HPLC	Standard method 18th Ed.	ASTM	Other
1. Acenaphthene	610	625, 1625	610	6410 B, 6440 B	D4657–92	
2. Acenaphthylene	610	625, 1625	610	6410 B, 6440 B	D4657–92	
3. Acrolein	603	[4] 604, 1624	.....			
4. Acrylonitrile	603	[4] 624, 1624	610			
5. Anthracene	610	625, 1625	610	6410 B, 6440 B	D4657–92	
6. Benzene	602	624, 1624	.....	6210 B, 6220 B		
7. Benzidine	.....	[5] 625, 1625	605			Note 3, p.1.
8. Benzo(a)anthracene	610	625, 1625	610	6410 B, 6440 B	D4657–92	
9. Benzo(a)pyrene	610	625, 1625	610	6410 B, 6440 B	D4657–92	
10. Benzo(b)fluoranthene	610	625, 1625	610	6410 B, 6440 B	D4657–92	
11. Benzo(g, h, i)perylene	610	625, 1625	610	6410 B, 6440 B	D4657–92	
12. Benzo(k)fluoranthene	610	625, 1625	610	6410 B, 6440 B	D4657–92	
13. Benzyl chloride	.....	.....	.....			Note 3, p.130: Note 6, p. S102.
14. Benzyl butyl phthalate	606	625, 1625	.....	6410 B		
15. Bis(2-chloroethoxy) methane	611	625, 1625	.....	6410 B		
16. Bis(2-chloroethyl) ether	611	625, 1625	.....	6410 B		
17. Bis (2-ethylhexyl) phthalate	606	625, 1625	.....	6410 B, 6230 B		
18. Bromodichloromethane	601	624, 1624	.....	6210 B, 6230 B		
19. Bromoform	601	624, 1624	.....	6210 B, 6230 B		
20. Bromomethane	601	624, 1624	.....	6210 B, 6230 B		
21. 4-Bromophenylphenyl ether	611	625, 1625	.....	6410 B		

22. Carbon tetrachloride	601	624, 1624	.....	6230 B, 6410 B		Note 3, p.130.
23. 4-Chloro-3-methylphenol	604	625, 1625	.....	6410 B, 6420 B		
24. Chlorobenzene	601, 602	624, 1624	.....	6210 B, 6220 B 6230 B		Note 3, p.130.
25. Chloroethane	601	624, 1624	.....	6210 B, 6230 B		
26. 2-Chloroethylvinyl ether	601	624, 1624	.....	6210 B, 6230 B		
27. Chloraform	601	624, 1624	.....	6210 B, 6230 B		Note, p.130.
28. Chloromethane	601	624, 1624	.....	6210 B. 6230 B		
29. 2-Chloronaphthalene	612	625, 1625	.....	6410 B		
30. 2-Chlorophenol	604	625, 1625	.....	6410 B, 6420 B		
31. 4-Chlorophenylphenyl ether	611	625, 1625	.....	6410 B		
32. Chrysene	610	625, 1625	610	6410 B, 6440 B	D4657–92	
33. Dibenzo(a,h)anthracene	610	625, 1625	610	6410 B, 6440 B	D4657–92	
34. Dibromochloromethane	601	624, 1624	.....	6210 B, 6230 B		
35. 1, 2-Dichlorobenzene	601,602,612	624,625,1625	.....	6410 B, 6230 B, 6220 B		
36. 1, 3-Dichlorobenzene	601,602,612	624,625,1625	.....	6410 B, 6230 B, 6220 B		
37. 1,4-Dichlorobenzene	601, 602, 612	624, 625, 1625	.....	6410 B, 6220 B, 6230 B		
38. 3, 3-Dichlorobenzidine	.....	625, 1625	605	6410 B		
39. Dichlorodifluoromethane	601	.....	.....	6230 B		
40. 1, 1-Dichloroethane	601	624, 1624	.....	6230 B, 6210 B		
41. 1, 2-Dichloroethane	601	624, 1624	.....	6230 B, 6210 B		
42. 1, 1-Dichloroethene	601	624, 1624	.....	6230 B, 6210 B		
43. trans-1, 2-Dichloroethene	601	624, 1624	.....	6230 B, 6210 B		
44. 2, 4-Dichlorophenol	604	625, 1625	.....	6420 B, 6410 B		
45. 1, 2-Dichloropropane	601	624, 1624	.....	6230 B, 6210 B		
46. cis-1, 3-Dichloropropene	601	624, 1624	.....	6230 B, 6210 B		
47. trans-1, 3-Dichloropropene	601	624, 1624	.....	6230 B, 6210 B		
48. Diethyl phthalate	606	625, 1625	.....	6410 B		
49. 2, 4-Dimethylphenol	604	625, 1625	.....	6420 B, 6410 B		
50. Dimethyl phthalate	606	625, 1625	.....	6410 B		
51. Di-n-butyl phthalate	606	625, 1625	.....	6410 B		
52. Di-n-octyl phthalate	606	625, 1625	.....	6410 B		
53. 2, 3-Dinitrophenol	604	625, 1625	.....	6420 B, 6410 B		
54. 2,4-Dinitrotoluene	609	625, 1625	.....	6410 B		
55. 2, 6-Dinitrotoluene	609	625, 1625	.....	6410 B		
56. Epichlorohydrin	.....	.....	.....			Note 3, p.130 Note 6, p.S102.
57. Ethylbenzene	602	624, 1624	.....	6220 B, 6210 B		
58. Fluoranthene	610	625, 1625	610	6410 B, 6440 B	D4657–92	
59. Fluorene	610	625, 1625	610	6410 B, 6440 B	D4657–92	
60. Hexachlorobenzene	612	625, 1625	.....	6410 B		
61. Hexachlorobutadiene	612	625, 1625	.....	6410 B		
62. Hexachlorocyclopentadiene	612	[5]625, 1625	.....	6410 B		
63. Hexachloroethane	616	625, 1625	.....	6410 B		
64. Ideno(1,2,3-cd) pyrene	610	625, 1625	610	6410 B, 6440 B	D4657–92	
65. Isophorone	609	625, 1625	.....	6410 B		
66. Methylene chloride	601	624, 1624	.....	6230 B		Note 3, p.130.
67. 2-Methyl-4,6-dinitrophenol	604	625, 1625	.....	6420 B, 6410 B		
68. Naphthalene	610	625, 1625	610	6410 B, 6440 B		

TABLE IC.—LIST OF APPROVED TEST PROCEDURES FOR NON-PESTICIDE ORGANIC COMPOUNDS—Continued

Parameter [1]	EPA method number [2 7]					
	GC	GC/MS	HPLC	Standard method 18th Ed.	ASTM	Other
69. Nitrobenezene	609	625, 1625	.....	6410 B		
70. 2-Nitrophenol	604	625, 1625	.....	6410 B, 6420 B		
71. 4-Nitrophenol	604	625, 1625	.....	6410 B, 6420 B		
72. N-Nitrosodimethylamine	607	625, 1625	.....	6410 B		
73. N-Nitrosodi-n-propylamine	607	[5]625, 1625	.....	6410 B		
74. N-Nitrosodiphenylamine	607	[5]625, 1625	.....	6410 B		
75. 2,2-Oxybis(1-chloropropane)	611	625, 1625	.....	6410 B		
76. PCB–1016	608	625	.....	6410 B		Note 3, p. 43; note 8.
77. PCB–1221	608	625	.....	6410 B		Note 3, p. 43; note 8.
78. PCB–1232	608	625	.....	6410 B		Note 3, p. 43; note 8.
79. PCB–1242	608	625	.....	6410 B		Note 3, p. 43; note 8.
80. PCB–1248	608	625	.....			Note 3, p. 43; note 8.
81. PCB–1254	608	625	.....	6410 B		Note 3, p. 43; note 8.
82. PCB–1260	608	625	.....	6410 B, 6630 B		Note 3, p. 43; note 8.
83. Pentachlorophenol	604	625, 1625	.....	6410 B, 6630 B		Note 3, p.140.
84. Phenanthrene	610	625, 1625	610	6410 B, 6440 B	D4657–92	
85. Phenol	604	625, 1625	.....	6420 B, 6410 B		
86. Pyrene	610	625, 1625	610	6410 B, 6440 B	D4675–92	
87. 2,3,7,8-Tetrachlorodibenzo-p-dioxin	.....	[5a]613	.....			
88. 1,1,2,2-Tetrachloroethane	601	624, 1624	.....	6230 B, 6210 B		Note 3, p.130.
89. Tetrachloroethene	601	624, 1624	.....	6230 B, 6210 B		Note 3, p.130.
90. Toluene	602	624, 1624	.....	6210 B, 6220 B		
91. 1,2,4-Trichlorobenzene	612	625, 1625	.....	6410 B		Note 3, p.130.
92. 1,1,1-Trichloroethane	601	624, 1624	.....	6210 B, 6230 B		
93. 1,1,2-Trichloroethane	601	624, 1624	.....	6210 B, 6230 B		Note 3, p.130.
94. Trichloroethene	601	624, 1624	.....	6210 B, 6230 B		
95. Trichlorofluoromethane	601	624	.....	6210 B, 6230 B		
96. 2,4,6-Trichlorophenol	604	625, 1625	.....	6410 B, 6240 B		
97. Vinyl chloride	601	624, 1624	.....	6210 B, 6230 B		

Table 1C notes:

[1] All parameters are expressed in micrograms per liter (µg/L).

[2] The full text of Methods 601–613, 624, 625, 1624, and 1625, are given at appendix A, ''Test Procedures for Analysis of Organic Pollutants,'' of this part 136. The standardized test procedure to be used to determine the method detection limit (MDL) for these test procedures is given at appendix B, ''Definition and Procedure for the Determination of the Method Detection Limit'' of this part 136.

[3] ''Methods for Benzidine: Chlorinated Organic Compounds, Pentachlorophenol and Pesticides in Water and Wastewater,'' U.S. Environmental Protection Agency, September, 1978.

[4] Method 624 may be extended to screen samples for Acrolein and Acrylonitrile. However, when they are known to be present, the preferred method for these two compounds is Method 603 or Method 1624.

[5] Method 625 may be extended to include benzidine, hexachlorocyclopentadiene, N-nitrosodimethylamine, and N-nitrosodiphenylamine. However, when they are known to be present, Methods 605, 607, and 612, or Method 1625, are preferred methods for these compounds.

[5a] 625, Screening only.

[6] "Selected Analytical Methods Approved and Cited by the United States Environmental Protection Agency", Supplement to the Fifteenth Edition of Standard Methods for the Examination of Water and Wastewater (1981).

[7] Each Analyst must make an initial, one-time demonstration of their ability to generate acceptable precision and accuracy with Methods 601–603, 624, 625, 1624, and 1625 (See Appendix A of this Part 136) in accordance with procedures each in section 8.2 of each of these Methods. Additionally, each laboratory, on an on-going basis must spike and analyze 10% (5% for Methods 624 and 625 and 100% for methods 1624 and 1625) of all samples to monitor and evaluate laboratory data quality in accordance with sections 8.3 and 8.4 of these Methods. When the recovery of any parameter falls outside the warning limits, the analytical results for that parameter in the unspiked sample are suspect and cannot be reported to demonstrate regulatory compliance.

NOTE: These warning limits are promulgated as an "interim final action with a request for comments."

[8] "Organochlorine Pesticides and PCBs in Wastewater Using Empore TM Disk", 3M Corporation Revised 10/28/94.

TABLE ID.—LIST OF APPROVED TEST PROCEDURES FOR PESTICIDES [1]

Parameter	Method	EPA [2 7]	Standard methods 18th Ed.	ASTM	Other
1. Aldrin	GC	608	6630 B & C	D3086–90	Note 3, p. 7; note 4, p. 30; note 8.
	GC/MS	625	6410 B		
2. Ametryn	GC				Note 3, p. 83; Note 6, p. S68.
3. Aminocarb	TLC				Note 3, p. 94; Note 6, p. S16.
4. Atraton	GC				Note 3, p. 83; Note 6, p. S68.
5. Atrazine	GC				Note 3, p. 83; Note 6, p. S68.
6. Azinphos methyl	GC				Note 3, p. 25; Note 6, p. S51.
7. Barban	TLC				Note 3, p. 104; Note 6, p. S64.
8. α-BHC	GC	608	6630 B & C	D3086–90	Note 3, p. 7; note 8.
	GC/MS	[5] 625	6410 B		
9. β-BHC	GC	608	6630 C	D3086–90	Note 8.
	GC/MS	[5] 625	6410 B		
10. δ-BHC	GC	608	6630 C	D3086–90	Note 8.
	GC/MS	[5] 625	6410 B		
11. δ-BHC (Lindane)	GC	608	6630 B & C	D3086–90	Note 3, p. 7; note 4, p. 30; note 8.
	GC/MS	625	6410 B		
12. Captan	GC		6630 B	D3086–90	Note 3, p. 7.
13. Carbaryl	TLC				Note 3, p. 94: Note 6, p. S60.
14. Carbophenothion	GC				Note 4, p. 30; Note 6, p. S73.
15. Chlordane	GC	608	6630 B & C	D3086–90	Note 3, p. 7; note 8.
	GC/MS	625	6410 B		
16. Chloropropham	TLC				Note 3, p. 104; Note 6, p. S64.
17. 2,4–D	GC		6640 B		Note 3, p. 115; Note 4, p. 35.
18. 4,4′–DDD	GC	608	6630 B & C	D3086–90	Note 3, p. 7; note 4, p. 30; note 8.
	GC/MS	625	6410 B		
19. 4,4′–DDE	GC	608	6630 B & C	D3086–90	Note 3, p. 7; note 4, p. 30; note 8.
	GC/MS	625	6410 B		
20. 4,4′–DDT	GC	608	6630 B & C	D3086–90	Note 3, p. 7; note 4, p. 30; note 8.
	GC/MS	625	6410 B		
21. Demeton–O	GC				Note 3, p. 25; Note 6, p. S51.
22. Demeton-S	GC				Note 3, p. 25: Note 6, p. S51.

TABLE ID.—LIST OF APPROVED TEST PROCEDURES FOR PESTICIDES[1]—Continued

Parameter	Method	EPA[2 7]	Standard methods 18th Ed.	ASTM	Other
23. Diazinon	GC				Note 3, p. 25; Note 4, p. 30; Note 6, p. S51.
24. Dicamba	GC				Note 3, p. 115.
25. Dichlofenthion	GC				Note 4, p. 30; Note 6, p. S73.
26. Dichloran	GC		6630 B & C		Note 3, p. 7.
27. Dicofol	GC			D3086–90	
28. Dieldrin	GC	608	6630 B & C		Note 3, p. 7; note 4, p. 30; note 8.
	GC/MS	625	6410 B		
29. Dioxathion	GC				Note 4, p. 30; Note 6, p. S73.
30. Disulfoton	GC				Note 3, p. 25; Note 6, p. S51.
31. Diuron	TLC				Note 3, p. 104; Note 6, p. S64.
32. Endosulfan I	GC	608	6630 B & C	D3086–90	Note 3, p. 7; note 8.
	GC/MS	[5]625	6410 B		
33. Endosulfan II	GC	608	6630 B & C	D3086–90	Note 3, p. 7; note 8.
	GC/MS	[5]625	6410 B		
34. Endosulfan Sulfate	GC	608	6630 C		Note 8.
	GC/MS	625	6410 B		
35. Endrin	GC	608	6630 B & C	D3086–90	Note 3, p. 7; note 4, p. 30; note 8.
	GC/MS	[5]625	6410 B		
36. Endrin aldehyde	GC	608			Note 8.
	GC/MS	625			
37. Ethion	GC				Note 4, p. 30; Note 6, p. S73.
38. Fenuron	TLC				Note 3, p. 104; Note 6, p. S64.
39. Fenuron–TCA	TLC				Note 3, p. 104; Note 6, p. S64.
40. Heptachlor	GC	608	6630 B & C	D3086–90	Note 3, p. 7; note 4, p. 30; note 8.
	GC/MS	625	6410 B		
41. Heptachlor epoxide	GC	608	6630 B & C	D3086–90	Note 3, p. 7; note 4, p. 30; note 6, p. S73; note 8.
	GC/MS	625	6410 B		
42. Isodrin	GC				Note 4, p. 30; Note 6, p. S73.
43. Linuron	GC				Note 3, p. 104; Note 6, p. S64.
44. Malathion	GC		6630 C		Note 3, p. 25; Note 4, p. 30; Note 6, p. S51.
45. Methiocarb	TLC				Note 3, p. 94; Note 6, p. S60.
46. Methoxychlor	GC		6630 B & C	D3086–90	Note 3, p. 7; note 4, p. 30; note 8.
47. Mexacarbate	TLC				Note 3, p. 94; Note 6, p. S60.
48. Mirex	GC		6630 B & C		Note 3, p. 7.
49. Monuron	TLC				Note 3, p. 104; Note 6, p. S64.
50. Monuron	TLC				Note 3, p. 104; Note 6, p. S64.
51. Nuburon	TLC				Note 3, p. 104; Note 6, p. S64.
52. Parathion methyl	GC		6630 C		Note 3, p. 25; Note 4, p. 30.
53. Parathion ethyl	GC		6630 C		Note 3, p. 25.
54. PCNB	GC		6630 B & C		Note 3, p. 7.

55. Perthane	GC			D3086–90	
56. Prometron	GC				Note 3, p. 83; Note 6, p. S68.
57. Prometryn	GC				Note 3, p. 83; Note 6, p. S68.
58. Propazine	GC				Note 3, p. 83; Note 6, p. S68.
59. Propham	TLC				Note 3, p. 104; Note 6, p. S64.
60. Propoxur	TLC				Note 3, p. 94; Note 6, p. S60.
61. Secbumeton	TLC				Note 3, p. 83; Note 6, p. S68.
62. Siduron	TLC				Note 3, p. 104; Note 6, p. S64.
63. Simazine	GC				Note 3, p. 83; Note 6, p. S68.
64. Strobane	GC		6630 B & C		Note 3, p. 7.
65. Swep	TLC				Note 3, p. 104; Note 6, p. S64.
66. 2,4,5–T	GC		6640 B		Note 3, p. 115; Note 4, p. 35.
67. 2,4,5–TP (Silvex)	GC		6640 B		Note 3, p. 115
68. Terbuthylazine	GC				Note 3, p. 83; Note 6, p. S68.
69. Toxaphene	GC	608	6630 B & C	D3086–90	Note 3, p. 7; note 4, p. 30; note 8.
	GC/MS	625	6410 B		
70. Trifluralin	GC		6630 B		Note 3, p. 7.

Table ID notes:

[1] Pesticides are listed in this table by common name for the convenience of the reader. Additional pesticides may be found under Table 1C, where entries are listed by chemical name.

[2] The full text of Methods 608 and 625 are given at Appendix A. ''Test Procedures for Analysis of Organic Pollutants,'' of this Part 136. The standardized test procedure to be used to determine the method detection limit (MDL) for these test procedures is given at Appendix B. ''Definition and Procedure for the Determination of the Method Detection Limit'', of this Part 136.

[3] ''Methods for Benzidine, Chlorinated Organic Compounds, Pentachlorophenol and Pesticides in Water and Wastewater,'' U.S. Environmental Protection Agency, September, 1978. This EPA publication includes thin-layer chromatography (TLC) methods.

[4] ''Methods for Analysis of Organic Substances in Water and Fluvial Sediments,'' Techniques of Water-Resources Investigations of the U.S. Geological Survey, Book 5, Chapter A3 (1987).

[5] The method may be extended to include α-BHC, γ-BHC, endosulfan I, endosulfan II, and endrin. However, when they are known to exist, Method 608 is the preferred method.

[6] ''Selected Analytical Methods Approved and Cited by the United States Environmental Protection Agency.'' Supplement to the Fifteenth Edition of Standard Methods for the Examination of Water and Wastewater (1981).

[7] Each analyst must make an initial, one-time, demonstration of their ability to generate acceptable precision and accuracy with Methods 608 and 625 (See Appendix A of this Part 136) in accordance with procedures given in section 8.2 of each of these methods. Additionally, each laboratory, on an-going basis, must spike and analyze 10% of all samples analyzed with Method 608 or 5% of all samples analyzed with Method 625 to monitor and evaluate laboratory data quality in accordance with Sections 8.3 and 8.4 of these methods. When the recovery of any parameter falls outside the warning limits, the analytical results for that parameter in the unspiked sample are suspect and cannot be reported to demonstrate regulatory compliance. These quality control requirements also apply to the Standard Methods, ASTM Methods, and other Methods cited.

NOTE: These warning limits are promulgated as an ''Interim final action with a request for comments.''

[8] ''Organochlorine Pesticides and PCBs in Wastewater Using Empore™ Disk'', 3M Corporation, Revised 10/28/94.

TABLE IE.—LIST OF APPROVED RADIOLOGIC TEST PROCEDURES

Parameter and units	Method	Reference (method number or page)			
		EPA[1]	Standard methods 18th Ed.	ASTM	USGS[2]
1. Alpha-Total, pCi per liter	Proportional or scintillation counter	900	7110 B	D1943–90	pp. 75 and 78.[3]
2. Alpha-Counting error, pCi per liter	Proportional or scintillation counter	Appendix B	7110 B	D1943–90	P. 79.
3. Beta-Total, pCi per liter	Proportional counter	900.0	7110 B	D1890–90	pp. 75 and 78.[3]
4. Beta-Counting error, pCi	Proportional counter	Appendix B	7110 B	D1890–90	p. 79.
5. (a) Radium Total pCi per liter	Proportional counter	903.0	7500Ra B	D2460–90	
(b)Ra, pCi per liter	Scintillation counter	903.1	7500Ra C	D3454–91	p. 81.

Table IE notes:
[1] Prescribed Procedures for Measurement of Radioactivity in Drinking Water,'' EPA–600/4–80–032 (1980), U.S. Environmental Protection Agency, August 1980.
[2] Fishman, M.J. and Brown, Eugene,'' Selected Methods of the U.S. Geological Survey of Analysis of Wastewaters,'' U.S. Geological Survey, Open-File Report 76–177 (1976).
[3] The method found on p. 75 measures only the dissolved portion while the method on p. 78 measures only the suspended portion. Therefore, the two results must be added to obtain the ''total''.

(b) The full texts of the methods from the following references which are cited in Tables IA, IB, IC, ID, and IE are incorporated by reference into this regulation and may be obtained from the sources identified. All costs cited are subject to change and must be verified from the indicated sources. The full texts of all the test procedures cited are available for inspection at the Environmental Monitoring Systems Laboratory, Office of Research and Development, U.S. Environmental Protection Agency, 26 West Martin Luther King Dr., Cincinnati, OH 45268 and the Office of the Federal Register, room 8301, 1110 L Street, NW., Washington, DC 20408.

REFERENCES, SOURCES, COSTS, AND TABLE CITATIONS:

(1) The full text of Methods 601–613, 624, 625, 1624, and 1625 are printed in appendix A of this part 136. The full text for determining the method detection limit when using the test procedures is given in appendix B of this part 136. The full text of Method 200.7 is printed in appendix C of this part 136. Cited in: Table IB, Note 5; Table IC, Note 2; and Table ID, Note 2.

(2) USEPA. 1978. Microbiological Methods for Monitoring the Environment, Water, and Wastes. Environmental Monitoring and Support Laboratory, U.S. Environmental Protection Agency, Cincinnati, Ohio. EPA/600/8–78/017. Available from: National Technical Information Service, 5285 Port Royal Road, Springfield, Virginia 22161, Publ. No. PB–290329/AS. Cost: $36.95. Table IA, Note 3.

(3) ''Methods for Chemical Analysis of Water and Wastes,'' U.S. Environmental Protection Agency, EPA–600/4–79–020, March 1979, or ''Methods for Chemical Analysis of Water and Wastes,'' U.S. Environmental Protection Agency, EPA–600/4–79–020, Revised March 1983. Available from: ORD Publications, CERI, U.S. Environmental Protection Agency, Cincinnati, Ohio 45268, Table IB, Note 1.

(4) ''Methods for Benzidine, Chlorinated Organic Compounds, Pentachlorophenol and Pesticides in Water and Wastewater,'' U.S. Environmental Protection Agency, 1978. Available from: ORD Publications, CERI, U.S. Environmental Protection Agency, Cincinnati, Ohio 45268, Table IC, Note 3; Table D, Note 3.

(5) ''Prescribed Procedures for Measurement of Radioactivity in Drinking Water,'' U.S. Environmental Protection Agency, EPA–600/4–80–032, 1980. Available from: ORD Publications, CERI, U.S. Environmental Protection Agency, Cincinnati, Ohio 45268, Table IE, Note 1.

(6) American Public Health Association. 1992. Standard Methods for the Examination of Water and Wastewater. 18th Edition. Amer. Publ. Hlth. Assoc., 1015 15th Street NW, Washington, DC 20005. Cost: $160.00. Table IA, Note 4.

(7) Ibid, 15th Edition, 1980. Table IB, Note 30; Table ID.

(8) Ibid, 14th Edition, 1975. Table IB, Notes 17 and 27.

(9) ''Selected Analytical Methods Approved and Cited by the United States Environmental Protection Agency,'' Supplement to the 15th Edition of Standard Methods for the Examination of Water and Wastewater, 1981. Available from: American Public Health Association, 1015 Fifteenth Street NW., Washington, DC 20036. Cost available from publisher. Table IB, Note 10; Table IC, Note 6; Table ID, Note 6.

(10) Annual Book of ASTM Standards, Water and Environmental Technology, Section 11, Volumes 11.01 and 11.02, 1994 in 40 CFR 136.3, Tables IB, IC, ID and IE.

(11) USGS. 1989. U.S. Geological Survey Techniques of Water-Resources Investigations, Book 5, Laboratory Analysis, Chapter A4, Methods for Collection and Analysis of Aquatic Biological and Microbiological Samples, U.S. Geological Survey, U.S. Department of the Interior, Reston, Virginia. Available from: USGS Books and Open-File Reports Section, Federal Center, Box 25425, Denver, Colorado 80225. Cost: $18.00. Table IA, Note 5.

(12) ''Methods for Determination of Inorganic Substances in Water and Fluvial Sediments,'' by M.J. Fishman and Linda C. Friedman, Techniques of Water-Resources Investigations of the U.S. Geological Survey, Book 5 Chapter A1 (1989). Available from: U.S. Geological Survey, Denver Federal Center, Box 25425, Denver, CO 80225. Cost: $108.75 (subject to change). Table IB, Note 2.

(13) ''Methods for Determination of Inorganic Substances in Water and Fluvial Sediments,'' N.W. Skougstad and others, editors. Techniques of Water-Resources Investigations of the U.S. Geological Survey, Book 5, Chapter A1 (1979). Available from: U.S. Geological Survey, Denver Federal Center, Box 25425, Denver, CO 80225. Cost: $10.00 (subject to change), Table IB, Note 8.

(14) ''Methods for the Determination of Organic Substances in Water and Fluvial Sediments,'' Wershaw, R.L., et al, Techniques of Water-Resources Investigations of the U.S. Geological Survey, Book 5, Chapter A3 (1987). Available from: U.S. Geological Survey, Denver Federal Center, Box 25425, Denver, CO 80225. Cost: $0.90 (subject to change). Table IB, Note 24; Table ID, Note 4.

(15) ''Water Temperature—Influential Factors, Field Measurement and Data Presentation,'' by H.H. Stevens, Jr., J. Ficke, and G.F. Smoot, Techniques of Water-Resources Investigations of the U.S. Geological Survey, Book 1, Chapter D1, 1975. Available from: U.S. Geological Survey, Denver Federal Center, Box 25425, Denver, CO 80225. Cost: $1.60 (subject to change). Table IB, Note 32.

(16) ''Selected Methods of the U.S. Geological Survey of Analysis of Wastewaters,'' by M.J. Fishman and Eugene Brown; U.S. Geological Survey Open File Report 76–77 (1976). Available from: U.S. Geological Survey, Branch of Distribution, 1200 South Eads Street, Arlington, VA 22202. Cost: $13.50 (subject to change). Table IE, Note 2.

(17) ''Official Methods of Analysis of the Association of Official Analytical Chemicals'', Methods manual, 15th Edition (1990). Price: $240.00. Available from: The Association of Official Analytical Chemists, 2200 Wilson Boulevard, Suite 400, Arlington, VA 22201. Table IB, Note 3.

(18) ''American National Standard on Photographic Processing Effluents,'' April 2, 1975. Available from: American National Standards Institute, 1430 Broadway, New York, New York 10018. Table IB, Note 9.

(19) ''An Investigation of Improved Procedures for Measurement of Mill Effluent and Receiving Water Color,'' NCASI Technical Bulletin No. 253, December 1971. Available from: National Council of the Paper Industry for Air and Stream Improvements, Inc., 260 Madison Avenue, New York, NY 10016. Cost available from publisher. Table IB, Note 18.

(20) Ammonia, Automated Electrode Method, Industrial Method Number 379–75WE, dated February 19, 1976. Technicon Auto Analyzer II. Method and price available from Technicon Industrial Systems, Tarrytown, New York 10591. Table IB, Note 7.

(21) Chemical Oxygen Demand, Method 8000, Hach Handbook of Water Analysis, 1979. Method price available from Hach Chemical Company, P.O. Box 389, Loveland, Colorado 80537. Table IB, Note 14.

(22) OIC Chemical Oxygen Demand Method, 1978. Method and price available from Oceanography International Corporation, 512 West Loop, P.O. Box 2980, College Station, Texas 77840. Table IB, Note 13.

(23) ORION Research Instruction Manual, Residual Chlorine Electrode Model 97–70, 1977. Method and price available from ORION Research Incorporation, 840 Memorial Drive, Cambridge, Massachusetts 02138. Table IB, Note 16.

(24) Bicinchoninate Method for Copper. Method 8506, Hach Handbook of Water Analysis, 1979, Method and price available from Hach Chemical Company, P.O. Box 300, Loveland, Colorado 80537. Table IB, Note 19.

(25) Hydrogen Ion (pH) Automated Electrode Method, Industrial Method Number 378–75WA. October 1976. Bran & Luebbe (Technicon) Auto Analyzer II. Method and price available from Bran & Luebbe Analyzing Technologies, Inc. Elmsford, N.Y. 10523. Table IB, Note 21.

(26) 1,10-Phenanthroline Method using FerroVer Iron Reagent for Water, Hach Method 8008, 1980. Method and price available from Hach Chemical Company, P.O. Box 389 Loveland, Colorado 80537. Table IB, Note 22.

(27) Periodate Oxidation Method for Manganese, Method 8034, Hach Handbook for Water Analysis, 1979. Method and price available from Hach Chemical Company, P.O. Box 389, Loveland, Colorado 80537. Table IB, Note 23.

(28) Nitrogen, Nitrite—Low Range, Diazotization Method for Water and Wastewater, Hach Method 8507, 1979.

Method and price available from Hach Chemical Company, P.O. Box 389, Loveland, Colorado 80537. Table IB, Note 25.

(29) Zincon Method for Zinc, Method 8009. Hach Handbook for Water Analysis, 1979. Method and price available from Hach Chemical Company, P.O. Box 389, Loveland, Colorado 80537. Table IB, Note 33.

(30) ''Direct Determination of Elemental Phosphorus by Gas-Liquid Chromatography,'' by R.F. Addison and R.G. Ackman, Journal of Chromatography, Volume 47, No. 3, pp. 421–426, 1970. Available in most public libraries. Back volumes of the Journal of Chromatography are available from Elsevier/North-Holland, Inc., Journal Information Centre, 52 Vanderbilt Avenue, New York, NY 10164. Cost available from publisher. Table IB, Note 28.

(31) ''Direct Current Plasma (DCP) Optical Emission Spectrometric Method for Trace Elemental Analysis of Water and Wastes'', Method AES 0029, 1986-Revised 1991, Fison Instruments, Inc., 32 Commerce Center, Cherry Hill Drive, Danvers, MA 01923. Table B, Note 34.

(32) ''Closed Vessel Microwave Digestion of Wastewater Samples for Determination of Metals, CEM Corporation, P.O. Box 200, Matthews, North Carolina 28106–0200, April 16, 1992. Available from the CEM Corporation. Table IB, Note 36.

(33) ''Organochlorine Pesticides and PCBs in Wastewater Using Empore ™ Disk'' Test Method 3M 0222, Revised 10/28/94. 3M Corporation, 3M Center Building 220–9E–10, St. Paul, MN 55144–1000. Method available from 3M Corporation. Table IC, Note 8 and Table ID, Note 8.

(34) USEPA. 1993. Methods for Measuring the Acute Toxicity of Effluents to Freshwater and Marine Organisms. Fourth Edition, December 1993. Environmental Monitoring Systems Laboratory, U.S. Environmental Protection Agency, Cincinnati, Ohio (EPA/600/4–90/027F). Available from: National Technical Information Service, 5285 Port Royal Road, Springfield, Virginia 22161, Publ. No. PB–91–167650. Cost: $31.00. Table IA, Note 17. See changes in the manual, listed in Part V of this rule.

(35) ''Nitrogen, Total Kjeldahl, Method PAI–DK01 (Block Digestion, Steam Distillation, Titrimetric Detection)'', revised 12/22/94. Available from Perstorp Analytical Corporation, 9445 SW Ridder Rd., Suite 310, P.O. Box 648, Wilsonville, OK 97070. Table IB, Note 39.

(36) ''Nitrogen, Total Kjeldahl, Method PAI–DK02 (Block Digestion, Steam Distillation, Colorimetric Detection)'', revised 12/22/94. Available from Perstorp Analytical Corporation, 9445 SW Ridder Rd., Suite 310, P.O. Box 648, Wilsonville, OK 97070. Table IB, Note 40.

(37) ''Nitrogen, Total Kjeldahl, Method PAI–DK03 (Block Digestion, Automated FIA Gas Diffusion)'', revised 12/22/94. Available from Perstorp Analytical Corporation, 9445 SW Ridder Rd., Suite 310, P.O. Box 648, Wilsonville, OK 97070. Table IB, Note 41.

(38) USEPA. 1994. Short-term Methods for Estimating the Chronic Toxicity of Effluents and Receiving Waters to Freshwater Organisms. Third Edition. July 1994. Environmental Monitoring Systems Laboratory, U.S. Environmental Protection Agency, Cincinnati, Ohio. (EPA/600/4–91/002). Available from: National Technical Information Service, 5285 Port Royal Road, Springfield, Virginia 22161, Publ. No. PB–92–139492. Cost: $31.00. Table IA, Note 8.

(39) USEPA. 1994. Short-term Methods for Estimating the Chronic Toxicity of Effluents and Receiving Waters to Marine and Estuarine Organisms. Second Edition, July 1994. Environmental Monitoring Systems Laboratory, U.S. Environmental Protection Agency, Cincinnati, Ohio. EPA/600/4–91/003. Available from: National Technical Information Service, 5285 Port Royal Road, Springfield, Virginia 22161, Publ. No. PB–92–139484. Cost: $45.00. Table IA, Note 9.

(c) Under certain circumstances the Regional Administrator or the Director in the Region or State where the discharge will occur may determine for a particular discharge that additional parameters or pollutants must be reported. Under such circumstances, additional test procedures for analysis of pollutants may be specified by the Regional Administrator, or the Director

upon the recommendation of the Director of the Environmental Monitoring Systems Laboratory—Cincinnati.

(d) Under certain circumstances, the Administrator may approve, upon recommendation by the Director, Environmental Monitoring Systems Laboratory—Cincinnati, additional alternate test procedures for nationwide use.

(e) Sample preservation procedures, container materials, and maximum allowable holding times for parameters cited in Tables IA, IB, IC, ID, and IE are prescribed in Table II. Any person may apply for a variance from the prescribed preservation techniques, container materials, and maximum holding times applicable to samples taken from a specific discharge. Applications for variances may be made by letters to the Regional Administrator in the Region in which the discharge will occur. Sufficient data should be provided to assure such variance does not adversely affect the integrity of the sample. Such data will be forwarded, by the Regional Administrator, to the Director of the Environmental Monitoring Systems Laboratory—Cincinnati, Ohio for technical review and recommendations for action on the variance application. Upon receipt of the recommendations from the Director of the Environmental Monitoring Systems Laboratory, the Regional Administrator may grant a variance applicable to the specific charge to the applicant. A decision to approve or deny a variance will be made within 90 days of receipt of the application by the Regional Administrator.

TABLE II—REQUIRED CONTAINERS, PRESERVATION TECHNIQUES, AND HOLDING TIMES

Parameter No./name	Container [1]	Preservation [2,3]	Maximum holding time [4]
Table IA—Bacteria Tests:			
1–4 Coliform, fecal and total	P,G	Cool, 4C, 0.008% $Na_2S_2O_3$ [5]	6 hours.
5 Fecal streptococci	P,G	Cool, 4C, 0.008% $Na_2S_2O_3$ [5]	6 hours.
Table IA—Aquatic Toxicity Tests:			
6–10 Toxicity, acute and chronic	P,G	Cool, 4C [16]	6 hours.
Table IB—Inorganic Tests:			
1. Acidity	P, G	Cool, 4°C	14 days.
2. Alkalinity	P, G	.....do	Do.
4. Ammonia	P, G	Cool, 4°C, H_2SO_4 to pH<2	28 days.
9. Biochemical oxygen demand	P, G	Cool, 4°C	48 hours.
10. Boron	P, PFTE, or Quartz.	HNO_3 TO pH<2	6 months.
11. Bromide	P, G	None required	28 days.
14. Biochemical oxygen demand, carbonaceous	P, G	Cool, 4°C	48 hours.
15. Chemical oxygen demand	P, G	Cool, 4°C, H_2SO_4 to pH<2	28 days.
16. Chloride	P, G	None required	Do.
17. Chlorine, total residual	P, G	.....do	Analyze immediately.
21. Color	P, G	Cool, 4°C	48 hours.
23–24. Cyanide, total and amenable to chlorination.	P, G	Cool, 4°C, NaOH to pH>12, 0.6g ascorbic acid [5].	14 days.[6]
25. Fluoride	P	None required	28 days.
27. Hardness	P, G	HNO_3 to pH<2, H_2SO_4 to pH<2	6 months.
28. Hydrogen ion (pH)	P, G	None required	Analyze immediately.
31, 43. Kjeldahl and organic nitrogen	P, G	Cool, 4°C, H_2SO_4 to pH<2	28 days.
Metals:[7]			
18. Chromium VI	P, G	Cool, 4°C	24 hours.
35. Mercury	P, G	HNO_3 to pH<2	6 months.
3, 5–8, 12, 13, 19, 20, 22, 26, 29, 30, 32–34, 36, 37, 45, 47, 51, 52, 58–60, 62, 63, 70–72, 74, 75. Metals, except boron, chromium VI and mercury.	P, G	.....do	6 months.
38. Nitrate	P, G	Cool, 4°C	48 hours.
39. Nitrate-nitrite	P, G	Cool, 4°C, H_2SO_4 to pH<2	28 days.
40. Nitrite	P, G	Cool, 4°C	48 hours.
41. Oil and grease	G	Cool to 4°C, HCl or H_2SO_4 to pH<2.	28 days.
42. Organic Carbon	P, G	Cool to 4 °C HC1 or H_2SO4 or H_3PO4, to pH<2.	28 days.
44. Orthophosphate	P, G	Filter immediately, Cool, 4°C	48 hours.
46. Oxygen, Dissolved Probe	G Bottle and top.	None required	Analyze immediately.
47. Winkler	.....do	Fix on site and store in dark	8 hours.
48. Phenols	G only	Cool, 4°C, H_2SO_4 to pH<2	28 days.

TABLE II—REQUIRED CONTAINERS, PRESERVATION TECHNIQUES, AND HOLDING TIMES—Continued

Parameter No./name	Container [1]	Preservation [2,3]	Maximum holding time [4]
49. Phosphorus (elemental)	G	Cool, 4°C	48 hours.
50. Phosphorus, total	P, G	Cool, 4°C, H_2SO_4 to pH<2	28 days.
53. Residue, total	P, G	Cool, 4°C	7 days.
54. Residue, Filterable	P, G	.....do	7 days.
55. Residue, Nonfilterable (TSS)	P, G	.....do	7 days.
56. Residue, Settleable	P, G	.....do	48 hours.
57. Residue, volatile	P, G	.....do	7 days.
61. Silica	P, PFTE, or Quartz.	Cool, 4 °C	28 days.
64. Specific conductance	P, G	.....do	Do.
65. Sulfate	P, G	.....do	Do.
66. Sulfide	P, G	Cool, 4°C add zinc acetate plus sodium hydroxide to pH>9.	7 days.
67. Sulfite	P, G	None required	Analyze immediately.
68. Surfactants	P ,G	Cool, 4°C	48 hours.
69. Temperature	P, G	None required	Analyze.
73. Turbidity	P, G	Cool, 4°C	48 hours.
Table IC—Organic Tests.[8]			
13, 18–20, 22, 24–28, 34–37, 39–43, 45–47, 56, 66, 88, 89, 92–95, 97. Purgeable Halocarbons.	G, Telflon-lined septum.	Cool, 4°C, 0.008% $Na_2S_2O_3$.[5]	14 days.
6, 57, 90. Purgeable aromatic hydrocarbons	.....do	Cool, 4°C, 0.008% $Na_2S_2O_3$[5], HC1 to pH2[9].	Do.
3, 4, Acrolein and acrylonitrile	.....do	Cool, 4°C, 0.008% $Na_2S_2O_3$[5]; Adjust pH to 4–5 [10].	Do.
23, 30, 44, 49, 53, 67, 70, 71, 83, 85, 96. Phenols [11].	G, Teflon-lined cap.	Cool, 4°C, 0.008% $Na_2S_2O_3$[5]	7 days until extraction, 40 days after extraction.
7, 38. Benzidines[11]	.....do	.....do	7 days until extraction.[13]
14, 17, 48, 50–52. Phthalate esters[11]	.....do	Cool, 4°C	7 days until extraction; 40 days after extraction.
72–74. Nitrosamines[11,14]	.....do	Cool, 4°C, store in dark, 0.008% $Na_2S_2O_3$[5].	Do.
76–82. PCBs[11] acrylonitrile	.....do	Cool, 4°C	Do.
54, 55, 65, 69. Nitroaromatics and isophorone[11]	.....do	Cool, 4°C, 0.008% $Na_2S_2O_3$[5] store in dark.	Do.
1, 2, 5, 8–12, 32, 33, 58, 59, 64, 68, 84, 86. Polynuclear aromatic hydrocarbons.[11].	.....do	.....do	Do.
15, 16, 21, 31, 75. Haloethers[11]	.....do	Cool, 4°C, 0.008% $Na_2S_2O_3$[5]	Do.
29, 35–37, 60–63, 91. Chlorinated hydrocarbons [11].	.....do	Cool, 4°C	Do.
87. TCDD [11]	.....do	Cool, 4°C, 0.008% $Na_2S_2O_3$[5]	Do.
Table ID—Pesticides Tests:			
1–70. Pesticides [11]	.....do	Cool, 4°C, pH 5–9 [15]	Do.
Table IE—Radiological Tests:			
1–5. Alpha, beta and radium	P, G	HNO_3 to pH<2	6 months.

Table II Notes

[1] Polyethylene (P) or glass (G). For microbiology, plastic sample containers must be made of sterilizable materials (polypropylene or other autoclavable plastic).

[2] Sample preservation should be performed immediately upon sample collection. For composite chemical samples each aliquot should be preserved at the time of collection. When use of an automated sampler makes it impossible to preserve each aliquot, then chemical samples may be preserved by maintaining at 4°C until compositing and sample splitting is completed.

[3] When any sample is to be shipped by common carrier or sent through the United States Mails, it must comply with the Department of Transportation Hazardous Materials Regulations (49 CFR part 172). The person offering such material for transportation is responsible for ensuring such compliance. For the preservation requirements of Table II, the Office of Hazardous Materials, Materials Transportation Bureau, Department of Transportation has determined that the Hazardous Materials Regulations do not apply to the following materials: Hydrochloric acid (HCl) in water solutions at concentrations of 0.04% by weight or less (pH about 1.96 or greater); Nitric acid (HNO_3) in water solutions at concentrations of 0.15% by weight or less (pH about 1.62 or greater); Sulfuric acid (H_2SO_4) in water solutions at concentrations of 0.35% by weight or less (pH about 1.15 or greater); and Sodium hydroxide (NaOH) in water solutions at concentrations of 0.080% by weight or less (pH about 12.30 or less).

[4] Samples should be analyzed as soon as possible after collection. The times listed are the maximum times that samples may be held before analysis and still be considered valid. Samples may be held for longer periods only if the permittee, or monitoring laboratory, has data on file to show that for the specific types of samples under study, the analytes are stable for the longer time, and has received a variance from the Regional Administrator under § 136.3(e). Some samples may not be stable for the maximum time period given in the table. A permittee, or monitoring laboratory, is obligated to hold the sample for a shorter time if knowledge exists to show that this is necessary to maintain sample stability. See § 136.3(e) for details. The term ''analyze immediately'' usually means within 15 minutes or less of sample collection.

[5] Should only be used in the presence of residual chlorine.

[6] Maximum holding time is 24 hours when sulfide is present. Optionally all samples may be tested with lead acetate paper before pH adjustments in order to determine if sulfide is present. If sulfide is present, it can be removed by the addition of cadmium nitrate powder until a negative spot test is obtained. The sample is filtered and then NaOH is added to pH 12.

[7] Samples should be filtered immediately on-site before adding preservative for dissolved metals.

[8] Guidance applies to samples to be analyzed by GC, LC, or GC/MS for specific compounds.

[9] Sample receiving no pH adjustment must be analyzed within seven days of sampling.

[10] The pH adjustment is not required if acrolein will not be measured. Samples for acrolein receiving no pH adjustment must be analyzed within 3 days of sampling.

[11] When the extractable analytes of concern fall within a single chemical category, the specified preservative and maximum holding times should be observed for optimum safeguard of sample integrity. When the analytes of concern fall within two or more chemical categories, the sample may be preserved by cooling to 4°C, reducing residual chlorine with 0.008% sodium thiosulfate, storing in the dark, and adjusting the pH to 6–9; samples preserved in this manner may be held for seven days before extraction and for forty days after extraction. Exceptions to this optional preservation and holding time procedure are noted in footnote 5 (re the requirement for thiosulfate reduction of residual chlorine), and footnotes 12, 13 (re the analysis of benzidine).

[12] If 1,2-diphenylhydrazine is likely to be present, adjust the pH of the sample to 4.0±0.2 to prevent rearrangement to benzidine.

[13] Extracts may be stored up to 7 days before analysis if storage is conducted under an inert (oxidant-free) atmosphere.

[14] For the analysis of diphenylnitrosamine, add 0.008% $Na_2S_2O_3$ and adjust pH to 7–10 with NaOH within 24 hours of sampling.

[15] The pH adjustment may be performed upon receipt at the laboratory and may be omitted if the samples are extracted within 72 hours of collection. For the analysis of aldrin, add 0.008% $Na_2S_2O_3$.

[16] Sufficient ice should be placed with the samples in the shipping container to ensure that ice is still present when the samples arrive at the laboratory. However, even if ice is present when the samples arrive, it is necessary to immediately measure the temperature of the samples and confirm that the 4C temperature maximum has not been exceeded. In the isolated cases where it can be documented that this holding temperature can not be met, the permittee can be given the option of on-site testing or can request a variance. The request for a variance should include supportive data which show that the toxicity of the effluent samples is not reduced because of the increased holding temperature.

[38 FR 28758, Oct. 16, 1973, as amended at 41 FR 52781, Dec. 1, 1976; 49 FR 43251, 43258, 43259, Oct. 26, 1984; 50 FR 691, 692, 695, Jan. 4, 1985; 51 FR 23693, June 30, 1986; 52 FR 33543, Sept. 3, 1987; 55 FR 24534, June 15, 1990; 55 FR 33440, Aug. 15, 1990; 56 FR 50759, Oct. 8, 1991; 57 FR 41833, Sept. 11, 1992; 58 FR 4505, Jan. 31, 1994; 60 FR 17160, Apr. 4, 1995; 60 FR 39588, 39590, Aug. 2, 1995; 60 FR 44672, Aug. 28, 1995; 60 FR 53542, 53543, Oct. 16, 1995]

§ 136.4 Application for alternate test procedures.

(a) Any person may apply to the Regional Administrator in the Region where the discharge occurs for approval of an alternative test procedure.

(b) When the discharge for which an alternative test procedure is proposed occurs within a State having a permit program approved pursuant to section 402 of the Act, the applicant shall submit his application to the Regional Administrator through the Director of the State agency having responsibility for issuance of NPDES permits within such State.

(c) Unless and until printed application forms are made available, an application for an alternate test procedure may be made by letter in triplicate. Any application for an alternate test procedure under this paragraph (c) shall:

(1) Provide the name and address of the responsible person or firm making the discharge (if not the applicant) and the applicable ID number of the existing or pending permit, issuing agency, and type of permit for which the alternate test procedure is requested, and the discharge serial number.

(2) Identify the pollutant or parameter for which approval of an alternate testing procedure is being requested.

(3) Provide justification for using testing procedures other than those specified in Table I.

(4) Provide a detailed description of the proposed alternate test procedure, together with references to published studies of the applicability of the alternate test procedure to the effluents in question.

(d) An application for approval of an alternate test procedure for nationwide use may be made by letter in triplicate to the Director, Analytical Methods Staff, Office of Science and Technology (4303), Office of Water, U.S. Environmental Protection Agency, 401 M Street, SW., Washington, DC 20460. Any application for an alternate test procedure under this paragraph (d) shall:

(1) Provide the name and address of the responsible person or firm making the application.

(2) Identify the pollutant(s) or parameter(s) for which nationwide approval of an alternate testing procedure is being requested.

(3) Provide a detailed description of the proposed alternate procedure, together with references to published or other studies confirming the general applicability of the alternate test procedure to the pollutant(s) or parameter(s) in waste water discharges from representative and specified industrial or other categories.

(4) Provide comparability data for the performance of the proposed alternate test procedure compared to the performance of the approved test procedures.

[38 FR 28760, Oct. 16, 1973, as amended at 41 FR 52785, Dec. 1, 1976; 62 FR 30763, June 5, 1997]

§ 136.5 Approval of alternate test procedures.

(a) The Regional Administrator of the region in which the discharge will occur has final responsibility for approval of any alternate test procedure proposed by the responsible person or firm making the discharge.

(b) Within thirty days of receipt of an application, the Director will forward such application proposed by the responsible person or firm making the discharge, together with his recommendations, to the Regional Administrator. Where the Director recommends rejection of the application for scientific and technical reasons which he provides, the Regional Administrator shall deny the application, and shall forward a copy of the rejected application and his decision to the Director of the State Permit Program and to the Director of the Analytical Methods Staff, Washington, DC.

(c) Before approving any application for an alternate test procedure proposed by the responsible person or firm making the discharge, the Regional Administrator shall forward a copy of the application to the Director of the Analytical Methods Staff, Washington, DC.

(d) Within ninety days of receipt by the Regional Administrator of an application for an alternate test procedure, proposed by the responsible person or firm making the discharge, the Regional Administrator shall notify the applicant and the appropriate State agency of approval or rejection, or shall specify the additional information which is required to determine whether to approve the proposed test procedure. Prior to the expiration of such ninety day period, a recommendation providing the scientific and other technical basis for acceptance or rejection will be forwarded to the Regional Administrator by the Director of the Analytical Methods Staff, Washington, DC. A copy of all approval and rejection notifications will be forwarded to the Director, Analytical Methods Staff, Washington, DC, for the purposes of national coordination.

(e) *Approval for nationwide use.* (1) Within sixty days of the receipt by the Director of the Analytical Methods Staff, Washington, DC, of an application for an alternate test procedure for nationwide use, the Director of the Analytical Methods Staff shall notify the applicant in writing whether the application is complete. If the application is incomplete, the applicant shall be informed of the information necessary to make the application complete.

(2) Within ninety days of the receipt of a complete package, the Analytical Methods Staff shall perform any analysis necessary to determine whether the alternate method satisfies the applicable requirements of this part, and the Director of the Analytical Methods Staff shall recommend to the Administrator that he/she approve or reject the application and shall also notify the applicant of such recommendation.

(3) As expeditiously as practicable, an alternate method determined by the Administrator to satisfy the applicable requirements of this part shall be proposed by EPA for incorporation in subsection 136.3 of 40 CFR part 136. EPA shall make available for review all the factual bases for its proposal, including any performance data submitted by the applicant and any available EPA analysis of those data.

(4) Following a period of public comment, EPA shall, as expeditiously as practicable, publish in the FEDERAL REGISTER a final decision to approve or reject the alternate method.

[38 FR 28760, Oct. 16, 1973, as amended at 41 FR 52785, Dec. 1, 1976; 55 FR 33440, Aug. 15, 1990; 62 FR 30763, June 5, 1997]

APPENDIX A TO PART 136—METHODS FOR ORGANIC CHEMICAL ANALYSIS OF MUNICIPAL AND INDUSTRIAL WASTEWATER

METHOD 601—PURGEABLE HALOCARBONS

1. Scope and Application

1.1 This method covers the determination of 29 purgeable halocarbons.

The following parameters may be determined by this method:

Parameter	STORET No.	CAS No.
Bromodichloromethane	32101	75–27–4
Bromoform	32104	75–25–2
Bromomethane	34413	74–83–9
Carbon tetrachloride	32102	56–23–5
Chlorobenzene	34301	108–90–7
Chloroethane	34311	75–00–3
2-Chloroethylvinyl ether	34576	100–75–8
Chloroform	32106	67–66–3
Chloromethane	34418	74–87–3
Dibromochloromethane	32105	124–48–1
1,2-Dichlorobenzene	34536	95–50–1
1,3-Dichlorobenzene	34566	541–73–1
1,4-Dichlorobenzene	34571	106–46–7
Dichlorodifluoromethane	34668	75–71–8
1,1-Dichloroethane	34496	75–34–3
1,2-Dichloroethane	34531	107–06–2
1,1-Dichloroethane	34501	75–35–4
trans-1,2-Dichloroethene	34546	156–60–5
1,2-Dichloropropane	34541	78–87–5
cis-1,3-Dichloropropene	34704	10061–01–5
trans-1,3-Dichloropropene	34699	10061–02–6
Methylene chloride	34423	75–09–2
1,1,2,2-Tetrachloroethane	34516	79–34–5
Tetrachloroethene	34475	127–18–4
1,1,1-Trichloroethane	34506	71–55–6
1,1,2-Trichloroethane	34511	79–00–5
Tetrachloroethene	39180	79–01–6
Trichlorofluoromethane	34488	75–69–4
Vinyl chloride	39715	75–01–4

1.2 This is a purge and trap gas chromatographic (GC) method applicable to the determination of the compounds listed above in municipal and industrial discharges as provided under 40 CFR 136.1. When this method is used to analyze unfamiliar samples for any or all of the compounds above, compound identifications should be supported by at least one additional qualitative technique. This method describes analytical conditions for a second gas chromatographic column that can be used to confirm measurements made with the primary column. Method 624 provides gas chromatograph/mass spectrometer (GC/MS) conditions appropriate for the qualitative and quantitative confirmation of results for most of the parameters listed above.

1.3 The method detection limit (MDL, defined in Section 12.1)[1] for each parameter is listed in Table 1. The MDL for a specific wastewater may differ from those listed, depending upon the nature of interferences in the sample matrix.

1.4 Any modification of this method, beyond those expressly permitted, shall be considered as a major modification subject to application and approval of alternate test procedures under 40 CFR 136.4 and 136.5.

1.5 This method is restricted to use by or under the supervision of analysts experienced in the operation of a purge and trap system and a gas chromatograph and in the interpretation of gas chromatograms. Each analyst must demonstrate the ability to generate acceptable results with this method using the procedure described in Section 8.2.

2. Summary of Method

2.1 An inert gas is bubbled through a 5-mL water sample contained in a specially-designed purging chamber at ambient temperature. The halocarbons are efficiently transferred from the aqueous phase to the vapor phase. The vapor is swept through a sorbent trap where the halocarbons are trapped. After purging is completed, the trap is heated and backflushed with the inert gas to desorb the halocarbons onto a gas chromatographic column. The gas chromatograph is temperature programmed to separate the halocarbons which are then detected with a halide-specific detector.[2,3]

2.2 The method provides an optional gas chromatographic column that may be helpful in resolving the compounds of interest from interferences that may occur.

3. Interferences

3.1 Impurities in the purge gas and organic compounds outgassing from the plumbing ahead of the trap account for the majority of contamination problems. The analytical system must be demonstrated to be free from contamination under the conditions of the analysis by running laboratory reagent blanks as described in Section 8.1.3. The use of non-Teflon plastic tubing, non-Teflon thread sealants, or flow controllers with rubber components in the purge and trap system should be avoided.

3.2 Samples can be contaminated by diffusion of volatile organics (particularly fluorocarbons and methylene chloride) through the septum seal ilto the sample during shipment and storage. A field reagent blank prepared from reagent water and carried through the sampling and handling protocol can serve as a check on such contamination.

3.3 Contamination by carry-over can occur whenever high level and low level samples are sequentially analyzed. To reduce carry-over, the purging device and sample syringe must be rinsed with reagent water between sample analyses. Whenever an unusually concentrated sample is encountered, it should be followed by an analysis of reagent water to check for cross contamination. For samples containing large amounts of water-soluble materials, suspended solids, high boiling compounds or high organohalide levels, it may be necessary to wash out the purging device with a detergent solution, rinse it with distilled water, and then dry it in a 105°C oven between analyses. The trap and other parts of the system are also subject to contamination; therefore, frequent bakeout and purging of the entire system may be required.

4. Safety

4.1 The toxicity or carcinogenicity of each reagent used in this method has not

been precisely defined; however, each chemical compound should be treated as a potential health hazard. From this viewpoint, exposure to these chemicals must be reduced to the lowest possible level by whatever means available. The laboratory is responsible for maintaining a current awareness file of OSHA regulations regarding the safe handling of the chemicals specified in this method. A reference file of material data handling sheets should also be made available to all personnel involved in the chemical analysis. Additional references to laboratory safety are available and have been identified[4–6] for the information of the analyst.

4.2 The following parameters covered by this method have been tentatively classified as known or suspected, human or mammalian carcinogens: carbon tetrachloride, chloroform, 1,4-dichlorobenzene, and vinyl chloride. Primary standards of these toxic compounds should be prepared in a hood. A NIOSH/MESA approved toxic gas respirator should be worn when the analyst handles high concentrations of these toxic compounds.

5. Apparatus and Materials

5.1 Sampling equipment, for discrete sampling.

5.1.1 Vial—25-mL capacity or larger, equipped with a screw cap with a hole in the center (Pierce #13075 or equivalent). Detergent wash, rinse with tap and distilled water, and dry at 105 °C before use.

5.1.2 Septum—Teflon-faced silicone (Pierce #12722 or equivalent). Detergent wash, rinse with tap and distilled water, and dry at 105 °C for 1 h before use.

5.2 Purge and trap system—The purge and trap system consists of three separate pieces of equipment: a purging device, trap, and desorber. Several complete systems are now commercially available.

5.2.1 The purging device must be designed to accept 5-mL samples with a water column at least 3 cm deep. The gaseous head space between the water column and the trap must have a total volume of less than 15 mL. The purge gas must pass through the water column as finely divided bubbles with a diameter of less than 3 mm at the origin. The purge gas must be introduced no more than 5 mm from the base of the water column. The purging device illustrated in Figure 1 meets these design criteria.

5.2.2 The trap must be at least 25 cm long and have an inside diameter of at least 0.105 in. The trap must be packed to contain the following minimum lengths of adsorbents: 1.0 cm of methyl silicone coated packing (Section 6.3.3), 7.7 cm of 2,6-diphenylene oxide polymer (Section 6.3.2), 7.7 cm of silica gel (Section 6.3.4), 7.7 cm of coconut charcoal (Section 6.3.1). If it is not necessary to analyze for dichlorodifluoromethane, the charcoal can be eliminated, and the polymer section lengthened to 15 cm. The minimum specifications for the trap are illustrated in Figure 2.

5.2.3 The desorber must be capable of rapidly heating the trap to 180 °C. The polymer section of the trap should not be heated higher than 180 °C and the remaining sections should not exceed 200 °C. The desorber illustrated in Figure 2 meets these design criteria.

5.2.4 The purge and trap system may be assembled as a separate unit or be coupled to a gas chromatograph as illustrated in Figures 3 and 4.

5.3 Gas chromatograph—An analytical system complete with a temperature programmable gas chromatograph suitable for on-column injection and all required accessories including syringes, analytical columns, gases, detector, and strip-chart recorder. A data system is recommended for measuring peak areas.

5.3.1 Column 1—8 ft long x 0.1 in. ID stainless steel or glass, packed with 1% SP–1000 on Carbopack B (60/80 mesh) or equivalent. This column was used to develop the method performance statements in Section 12. Guidelines for the use of alternate column packings are provided in Section 10.1.

5.3.2 Column 2—6 ft long x 0.1 in. ID stainless steel or glass, packed with chemically bonded n-octane on Porasil-C (100/120 mesh) or equivalent.

5.3.3 Detector—Electrolytic conductivity or microcoulometric detector. These types of detectors have proven effective in the analysis of wastewaters for the parameters listed in the scope (Section 1.1). The electrolytic conductivity detector was used to develop the method performance statements in Section 12. Guidelines for the use of alternate detectors are provided in Section 10.1.

5.4 Syringes—5-mL glass hypodermic with Luerlok tip (two each), if applicable to the purging device.

5.5 Micro syringes—25-µL, 0.006 in. ID needle.

5.6 Syringe valve—2-way, with Luer ends (three each).

5.7 Syringe—5-mL, gas-tight with shut-off valve.

5.8 Bottle—15-mL, screw-cap, with Teflon cap liner.

5.9 Balance—Analytical, capable of accurately weighing 0.0001 g.

6. Reagents

6.1 Reagent water—Reagent water is defined as a water in which an interferent is not observed at the MDL of the parameters of interest.

6.1.1 Reagent water can be generated by passing tap water through a carbon filter bed containing about 1 lb of activated carbon (Filtrasorb-300, Calgon Corp., or equivalent).

6.1.2 A water purification system (Millipore Super-Q or equivalent) may be used to generate reagent water.

6.1.3 Reagent water may also be prepared by boiling water for 15 min. Subsequently, while maintaining the temperature at 90 °C, bubble a contaminant-free inert gas through the water for 1 h. While still hot, transfer the water to a narrow mouth screw-cap bottle and seal with a Teflon-lined septum and cap.

6.2 Sodium thiosulfate—(ACS) Granular.

6.3 Trap Materials:

6.3.1 Coconut charcoal—6/10 mesh sieved to 26 mesh, Barnabey Cheney, CA–580–26 lot # M–2649 or equivalent.

6.3.2 2,6-Diphenylene oxide polymer—Tenax, (60/80 mesh), chromatographic grade or equivalent.

6.3.3 Methyl silicone packing—3% OV–1 on Chromosorb-W (60/80 mesh) or equivalent.

6.3.4 Silica gel—35/60 mesh, Davison, grade-15 or equivalent.

6.4 Methanol—Pesticide quality or equivalent.

6.5 Stock standard solutions—Stock standard solutions may be prepared from pure standard materials or purchased as certified solutions. Prepare stock standard solutions in methanol using assayed liquids or gases as appropriate. Because of the toxicity of some of the organohalides, primary dilutions of these materials should be prepared in a hood. A NIOSH/MESA approved toxic gas respirator should be used when the analyst handles high concentrations of such materials.

6.5.1 Place about 9.8 mL of methanol into a 10-mL ground glass stoppered volumetric flask. Allow the flask to stand, unstoppered, for about 10 min or until all alcohol wetted surfaces have dried. Weigh the flask to the learest 0.1 mg.

6.5.2 Add the assayed reference material:

6.5.2.1 Liquid—Using a 100 µL syringe, immediately add two or more drops of assayed reference material to the flask, then reweigh. Be sure that the drops fall directly into the alcohol without contacting the neck of the flask.

6.5.2.2 Gases—To prepare standards for any of the six halocarbons that boil below 30°C (bromomethane, chloroethane, chloromethane, dichlorodifluoromethane, trichlorofluoromethane, vinyl chloride), fill a 5-mL valved gas-tight syringe with the reference standard to the 5.0-mL mark. Lower the needle to 5 mm above the methanol meniscus. Slowly introduce the reference standard above the surface of the liquid (the heavy gas will rapidly dissolve into the methanol).

6.5.3 Reweigh, dilute to volume, stopper, then mix by inverting the flask several times. Calculate the concentration in µg/µL from the net gain in weight. When compound purity is assayed to be 96% or greater, the weight can be used without correction to calculate the concentration of the stock standard. Commercially prepared stock standards can be used at any concentration if they are certified by the malufacturer or by an independent source.

6.5.4 Transfer the stock standard solution into a Teflon-sealed screw-cap bottle. Store, with minimal headspace, at −10 to −20 °C and protect from light.

6.5.5 Prepare fresh standards weekly for the six gases and 2-chloroethylvinyl ether. All other standards must be replaced after one month, or sooner if comparison with check standards indicates a problem.

6.6 Secondary dilution standards—Using stock standard solutions, prepare secondary dilution standards in methanol that contain the compounds of interest, either singly or mixed together. The secondary dilution standards should be prepared at concentrations such that the aqueous calibration standards prepared in Section 7.3.1 or 7.4.1 will bracket the working range of the analytical system. Secondary dilution standards should be stored with minimal headspace and should be checked frequently for signs of degradation or evaporation, especially just prior to preparing calibration standards from them.

6.7 Quality control check sample concentrate—See Section 8.2.1.

7. Calibration

7.1 Assemble a purge and trap system that meets the specifications in Section 5.2. Condition the trap overnight at 180 °C by backflushing with an inert gas flow of at least 20 mL/min. Condition the trap for 10 min once daily prior to use.

7.2 Connect the purge and trap system to a gas chromatograph. The gas chromatograph must be operated using temperature and flow rate conditions equivalent to those given in Table 1. Calibrate the purge and trap-gas chromatographic system using either the external standard technique (Section 7.3) or the internal standard technique (Section 7.4).

7.3 External standard calibration procedure:

7.3.1 Prepare calibration standards at a miminum of three concentration levels for each parameter by carefully adding 20.0 µL of one or more secondary dilution standards to 100, 500, or 1000 µL of reagent water. A 25-µL syringe with a 0.006 in. ID needle should be used for this operation. One of the external standards should be at a concentration near, but above, the MDL (Table 1) and the other concentrations should correspond to the expected range of concentrations found in real samples or should define the working range of the detector. These aqueous standards can be stored up to 24 h, if held in sealed vials with zero headspace as described in Section 9.2. If not so stored, they must be discarded after 1 h.

7.3.2 Analyze each calibration standard according to Section 10, and tabulate peak height or area responses versus the concentration in the standard. The results can be used to prepare a calibration curve for each compound. Alternatively, if the ratio of response to concentration (calibration factor) is a constant over the working range (<10% relative standard deviation, RSD), linearity through the origin can be assumed and the average ratio or calibration factor can be used in place of a calibration curve.

7.4 Internal standard calibration procedure—To use this approach, the analyst must select one or more internal standards that are similar in analytical behavior to the compounds of interest. The analyst must further demonstrate that the measurement of the internal standard is not affected by method or matrix interferences. Because of these limitations, no internal standard can be suggested that is applicable to all samples. The compounds recommended for use as surrogate spikes in Section 8.7 have been used successfully as internal standards, because of their generally unique retention times.

7.4.1 Prepare calibration standards at a minimum of three concentration levels for each parameter of interest as described in Section 7.3.1.

7.4.2 Prepare a spiking solution containing each of the internal standards using the procedures described in Sections 6.5 and 6.6. It is recommended that the secondary dilution standard be prepared at a concentration of 15 µg/mL of each internal standard compound. The addition of 10 µL of this standard to 5.0 mL of sample or calibration standard would be equivalent to 30 µg/L.

7.4.3 Analyze each calibration standard according to Section 10, adding 10 µL of internal standard spiking solution directly to the syringe (Section 10.4). Tabulate peak height or area responses against concentration for each compound and internal standard, and calculate response factors (RF) for each compound using Equation 1.

$$RF = \frac{(A_s)(C_{is})}{(A_{is})(C_s)}$$

Equation 1

where:

A_s=Response for the parameter to be measured.

A_{is}=Response for the internal standard.

C_{is}=Concentration of the internal standard.

C_s=Concentration of the parameter to be measured.

If the RF value over the working range is a constant (<10% RSD), the RF can be assumed to be invariant and the average RF can be used for calculations. Alternatively, the results can be used to plot a calibration curve of response ratios, A_s/A_{is}, vs. RF.

7.5 The working calibration curve, calibration factor, or RF must be verified on each working day by the measurement of a QC check sample.

7.5.1 Prepare the QC check sample as described in Section 8.2.2.

7.5.2 Analyze the QC check sample according to Section 10.

7.5.3 For each parameter, compare the response (Q) with the corresponding calibration acceptance criteria found in Table 2. If the responses for all parameters of interest fall within the designated ranges, analysis of actual samples can begin. If any individual Q falls outside the range, proceed according to Section 7.5.4.

NOTE: The large number of parameters in Table 2 present a substantial probability that one or more will not meet the calibration acceptance criteria when all parameters are analyzed.

7.5.4 Repeat the test only for those parameters that failed to meet the calibration acceptance criteria. If the response for a parameter does not fall within the range in this second test, a new calibration curve, calibration factor, or RF must be prepared for that parameter according to Section 7.3 or 7.4.

8. Quality Control

8.1 Each laboratory that uses this method is required to operate a formal quality control program. The minimum requirements of this program consist of an initial demonstration of laboratory capability and an ongoing analysis of spiked samples to evaluate and document data quality. The laboratory must maintain records to document the quality of data that is generated. Ongoing data quality checks are compared with established performance criteria to determine if the results of analyses meet the performance characteristics of the method. When results of sample spikes indicate atypical method performance, a quality control check standard must be analyzed to confirm that the measurements were performed in an in-control mode of operation.

8.1.1 The analyst must make an initial, one-time, demonstration of the ability to generate acceptable accuracy and precision with this method. This ability is established as described in Section 8.2.

8.1.2 In recognition of advances that are occurring in chromatography, the analyst is permitted certain options (detailed in Section 10.1) to improve the separations or lower the cost of measurements. Each time such a modification is made to the method, the analyst is required to repeat the procedure in Section 8.2.

8.1.3 Each day, the analyst must analyze a reagent water blank to demonstrate that interferences from the analytical system are under control.

8.1.4 The laboratory must, on an ongoing basis, spike and analyze a minimum of 10% of all samples to monitor and evaluate laboratory data quality. This procedure is described in Section 8.3.

8.1.5 The laboratory must, on an ongoing basis, demonstrate through the analyses of quality control check standards that the operation of the measurement system is in control. This procedure is described in Section 8.4. The frequency of the check standard analyses is equivalent to 10% of all samples analyzed but may be reduced if spike recoveries from samples (Section 8.3) meet all specified quality control criteria.

8.1.6 The laboratory must maintain performance records to document the quality of data that is generated. This procedure is described in Section 8.5.

8.2 To establish the ability to generate acceptable accuracy and precision, the analyst must perform the following operations.

8.2.1 A quality control (QC) check sample concentrate is required containing each parameter of interest at a concentration of 10 µg/mL in methanol. The QC check sample concentrate must be obtained from the U.S. Environmental Protection Agency, Environmental Monitoring and Support Laboratory in Cincinnati, Ohio, if available. If not available from that source, the QC check sample concentrate must be obtained from another external source. If not available from either source above, the QC check sample concentrate must be prepared by the laboratory using stock standards prepared independently from those used for calibration.

8.2.2 Prepare a QC check sample to contain 20 µg/L of each parameter by adding 200 µL of QC check sample concentrate to 100 mL of reagent water.

8.2.3 Analyze four 5-mL aliquots of the well-mixed QC check sample according to Section 10.

8.2.4 Calculate the average recovery ($\bar{X}$) in µg/L, and the standard deviation of the recovery (s) in µg/L, for each parameter of interest using the four results.

8.2.5 For each parameter compare s and $\bar{X}$ with the corresponding acceptance criteria for precision and accuracy, respectively, found in Table 2. If s and $\bar{X}$ for all parameters of interest meet the acceptance criteria, the system performance is acceptable and analysis of actual samples can begin. If any individual s exceeds the precision limit or any individual $\bar{X}$ falls outside the range for accuracy, then the system performance is unacceptable for that parameter.

NOTE: The large number of parameters in Table 2 present a substantial probability that one or more will fail at least one of the acceptance criteria when all parameters are analyzed.

8.2.6 When one or more of the parameters tested fail at least one of the acceptance criteria, the analyst must proceed according to Section 8.2.6.1 or 8.2.6.2.

8.2.6.1 Locate and correct the source of the problem and repeat the test for all parameters of interest beginning with Section 8.2.3.

8.2.6.2 Beginning with Section 8.2.3, repeat the test only for those parameters that failed to meet criteria. Repeated failure, however, will confirm a general problem with the measurement system. If this occurs, locate and correct the source of the problem and repeat the test for all compounds of interest beginning with Section 8.2.3.

8.3 The laboratory must, on an ongoing basis, spike at least 10% of the samples from each sample site being monitored to assess accuracy. For laboratories analyzing one to ten samples per month, at least one spiked sample per month is required.

8.3.1 The concentration of the spike in the sample should be determined as follows:

8.3.1.1 If, as in compliance monitoring, the concentration of a specific parameter in the sample is being checked against a regulatory concentration limit, the spike should be at that limit or 1 to 5 times higher than the background concentration determined in Section 8.3.2, whichever concentration would be larger.

8.3.1.2 If the concentration of a specific parameter in the sample is not being checked against a limit specific to that parameter, the spike should be at 20 µg/L or 1 to 5 times higher than the background concentration determined in Section 8.3.2, whichever concentration would be larger.

8.3.2 Analyze one 5-mL sample aliquot to determine the background concentration (B) of each parameter. If necessary, prepare a new QC check sample concentrate (Section 8.2.1) appropriate for the background concentrations in the sample. Spike a second 5-mL sample aliquot with 10 µL of the QC check sample concentrate and analyze it to determine the concentration after spiking (A) of each parameter. Calculate each percent recovery (P) as 100(A–B)%/T, where T is the known true value of the spike.

8.3.3 Compare the percent recovery (P) for each parameter with the corresponding QC acceptance criteria found in Table 2. These acceptance criteria were calculated to include an allowance for error in measurement of both the background and spike concentrations, assuming a spike to background ratio of 5:1. This error will be accounted for to the extent that the analyst's spike to background ratio approaches 5:1.[7] If spiking was performed at a concentration lower than 20 µg/L, the analyst must use either the QC acceptance criteria in Table 2, or optional QC acceptance criteria calculated for the specific spike concentration. To calculate optional acceptance criteria for the recovery of a parameter: (1) Calculate accuracy (X′) using the equation in Table 3, substituting

the spike concentration (T) for C; (2) calculate overall precision (S′) using the equation in Table 3, substituting X′ for $\bar{X}$; (3) calculate the range for recovery at the spike concentration as (100 X′/T)±2.44(100 S′/T)%.[7]

8.3.4 If any individual P falls outside the designated range for recovery, that parameter has failed the acceptance criteria. A check standard containing each parameter that failed the criteria must be analyzed as described in Section 8.4.

8.4 If any parameter fails the acceptance criteria for recovery in Section 8.3, a QC check standard containing each parameter that failed must be prepared and analyzed.

NOTE: The frequency for the required analysis of a QC check standard will depend upon the number of parameters being simultaneously tested, the complexity of the sample matrix, and the performance of the laboratory. If the entire list of parameters in Table 2 must be measured in the sample in Section 8.3, the probability that the analysis of a QC check standard will be required is high. In this case the QC check standard should be routinely analyzed with the spiked sample.

8.4.1 Prepare the QC check standard by adding 10 µL of QC check sample concentrate (Section 8.2.1 or 8.3.2) to 5 mL of reagent water. The QC check standard needs only to contain the parameters that failed criteria in the test in Section 8.3.

8.4.2 Analyze the QC check standard to determine the concentration measured (A) of each parameter. Calculate each percent recovery (P_s) as 100 (A/T)%, where T is the true value of the standard concentration.

8.4.3 Compare the percent recovery (P_s) for each parameter with the corresponding QC acceptance criteria found in Table 2. Only parameters that failed the test in Section 8.3 need to be compared with these criteria. If the recovery of any such parameter falls outside the designated range, the laboratory performance for that parameter is judged to be out of control, and the problem must be immediately identified and corrected. The analytical result for that parameter in the unspiked sample is suspect and may not be reported for regulatory compliance purposes.

8.5 As part of the QC program for the laboratory, method accuracy for wastewater samples must be assessed and records must be maintained. After the analysis of five spiked wastewater samples as in Section 8.3, calculate the average percent recovery ($\bar{P}$) and the standard deviation of the percent recovery (s_p). Express the accuracy assessment as a percent recovery interval from $\bar{P}-2s_p$ to $\bar{P}+2s_p$. If $\bar{p}$=90% and s_p=10%, for example, the accuracy interval is expressed as 70–110%. Update the accuracy assessment for each parameter on a regular basis (e.g. after each five to ten new accuracy measurements).

8.6 It is recommended that the laboratory adopt additional quality assurance practices for use with this method. The specific practices that are most productive depend upon the needs of the laboratory and the nature of the samples. Field duplicates may be analyzed to assess the precision of the environmental measurements. When doubt exists over the identification of a peak on the chromatogram, confirmatory techniques such as gas chromatography with a dissimilar column, specific element detector, or mass spectrometer must be used. Whenever possible, the laboratory should analyze standard reference materials and participate in relevant performance evaluation studies.

8.7 The analyst should monitor both the performance of the analytical system and the effectiveness of the method in dealing with each sample matrix by spiking each sample, standard, and reagent water blank with surrogate halocarbons. A combination of bromochloromethane, 2-bromo-1-chloropropane, and 1,4-dichlorobutane is recommended to encompass the range of the temperature program used in this method. From stock standard solutions prepared as in Section 6.5, add a volume to give 750 µg of each surrogate to 45 mL of reagent water contained in a 50-mL volumetric flask, mix and dilute to volume for a concentration of 15 ng/µL. Add 10 µL of this surrogate spiking solution directly into the 5-mL syringe with every sample and reference standard analyzed. Prepare a fresh surrogate spiking solution on a weekly basis. If the internal standard calibration procedure is being used, the surrogate compounds may be added directly to the internal standard spiking solution (Section 7.4.2).

9. Sample Collection, Preservation, and Handling

9.1 All samples must be iced or refrigerated from the time of collection until analysis. If the sample contains free or combined chlorine, add sodium thiosulfate preservative (10 mg/40 mL is sufficient for up to 5 ppm Cl_2) to the empty sample bottle just prior to shipping to the sampling site. EPA Methods 330.4 and 330.5 may be used for measurement of residual chlorine.[8] Field test kits are available for this purpose.

9.2 Grab samples must be collected in glass containers having a total volume of at least 25 mL. Fill the sample bottle just to overflowing in such a manner that no air bubbles pass through the sample as the bottle is being filled. Seal the bottle so that no air bubbles are entrapped in it. If preservative has been added, shake vigorously for 1 min. Maintain the hermetic seal on the sample bottle until time of analysis.

9.3 All samples must be analyzed within 14 days of collection.[3]

10. Procedure

10.1 Table 1 summarizes the recommended operating conditions for the gas chromatograph. Included in this table are estimated retention times and MDL that can be achieved under these conditions. An example of the separations achieved by Column 1 is shown in Figure 5. Other packed columns, chromatographic conditions, or detectors may be used if the requirements of Section 8.2 are met.

10.2 Calibrate the system daily as described in Section 7.

10.3 Adjust the purge gas (nitrogen or helium) flow rate to 40 mL/min. Attach the trap inlet to the purging device, and set the purge and trap system to purge (Figure 3). Open the syringe valve located on the purging device sample introduction needle.

10.4 Allow the sample to come to ambient temperature prior to introducing it to the syringe. Remove the plunger from a 5-mL syringe and attach a closed syringe valve. Open the sample bottle (or standard) and carefully pour the sample into the syringe barrel to just short of overflowing. Replace the syringe plunger and compress the sample. Open the syringe valve and vent any residual air while adjusting the sample volume to 5.0 mL. Since this process of taking an aliquot destroys the validity of the sample for future analysis, the analyst should fill a second syringe at this time to protect against possible loss of data. Add 10.0 µL of the surrogate spiking solution (Section 8.7) and 10.0 µL of the internal standard spiking solution (Section 7.4.2), if applicable, through the valve bore, then close the valve.

10.5 Attach the syringe-syringe valve assembly to the syringe valve on the purging device. Open the syringe valves and inject the sample into the purging chamber.

10.6 Close both valves and purge the sample for 11.0±0.1 min at ambient temperature.

10.7 After the 11-min purge time, attach the trap to the chromatograph, adjust the purge and trap system to the desorb mode (Figure 4), and begin to temperature program the gas chromatograph. Introduce the trapped materials to the GC column by rapidly heating the trap to 180 °C while backflushing the trap with an inert gas between 20 and 60 mL/min for 4 min. If rapid heating of the trap cannot be achieved, the GC column must be used as a secondary trap by cooling it to 30 °C (subambient temperature, if poor peak geometry or random retention time problems persist) instead of the initial program temperature of 45 °C

10.8 While the trap is being desorbed into the gas chromatograph, empty the purging chamber using the sample introduction syringe. Wash the chamber with two 5-mL flushes of reagent water.

10.9 After desorbing the sample for 4 min, recondition the trap by returning the purge and trap system to the purge mode. Wait 15 s then close the syringe valve on the purging device to begin gas flow through the trap. The trap temperature should be maintained at 180 °C After approximately 7 min, turn off the trap heater and open the syringe valve to stop the gas flow through the trap. When the trap is cool, the next sample can be analyzed.

10.10 Identify the parameters in the sample by comparing the retention times of the peaks in the sample chromatogram with those of the peaks in standard chromatograms. The width of the retention time window used to make identifications should be based upon measurements of actual retention time variations of standards over the course of a day. Three times the standard deviation of a retention time for a compound can be used to calculate a suggested window size; however, the experience of the analyst should weigh heavily in the interpretation of chromatograms.

10.11 If the response for a peak exceeds the working range of the system, prepare a dilution of the sample with reagent water from the aliquot in the second syringe and reanalyze.

11. Calculations

11.1 Determine the concentration of individual compounds in the sample.

11.1.1 If the external standard calibration procedure is used, calculate the concentration of the parameter being measured from the peak response using the calibration curve or calibration factor determined in Section 7.3.2.

11.1.2 If the internal standard calibration procedure is used, calculate the concentration in the sample using the response factor (RF) determined in Section 7.4.3 and Equation 2.

Equation 2

$$\text{Concentration } (\mu g/L) = \frac{(A_s)(C_{is})}{(A_{is})(RF)}$$

where:

A_s=Response for the parameter to be measured.

A_{is}=Response for the internal standard.

C_{is}=Concentration of the internal standard.

11.2 Report results in µg/L without correction for recovery data. All QC data obtained should be reported with the sample results.

12. Method Performance

12.1 The method detection limit (MDL) is defined as the minimum concentration of a substance that can be measured and reported with 99% confidence that the value is above zero.[1] The MDL concentration listed in

Table 1 were obtained using reagent water.[11]. Similar results were achieved using representative wastewaters. The MDL actually achieved in a given analysis will vary depending on instrument sensitivity and matrix effects.

12.2 This method is recommended for use in the concentration range from the MDL to 1000×MDL. Direct aqueous injection techniques should be used to measure concentration levels above 1000×MDL.

12.3 This method was tested by 20 laboratories using reagent water, drinking water, surface water, and three industrial wastewaters spiked at six concentrations over the range 8.0 to 500 µg/L.[9] Single operator precision, overall precision, and method accuracy were found to be directly related to the concentration of the parameter and essentially independent of the sample matrix. Linear equations to describe these relationships are presented in Table 3.

References

1. 40 CFR part 136, appendix B.

2. Bellar, T.A., and Lichtenberg, J.J. ''Determining Volatile Organics at Microgram-per-Litre-Levels by Gas Chromatography,'' *Journal of the American Water Works Association, 66,* 739 (1974).

3. Bellar, T.A., and Lichtenberg, J.J. ''Semi-Automated Headspace Analysis of Drinking Waters and Industrial Waters for Purgeable Volatile Organic Compounds,'' Proceedings from Symposium on Measurement of Organic Pollutants in Water and Wastewater, American Society for Testing and Materials, STP 686, C.E. Van Hall, editor, 1978.

4. ''Carcinogens—Working With Carcinogens,'' Department of Health, Education, and Welfare, Public Health Service, Center for Disease Control, National Institute for Occupational Safety and Health, Publication No. 77–206, August 1977.

5. ''OSHA Safety and Health Standards, General Industry'' (29 CFR part 1910), Occupational Safety and Health Administration, OSHA 2206 (Revised, January 1976).

6. ''Safety in Academic Chemistry Laboratories,'' American Chemical Society Publication, Committee on Chemical Safety, 3rd Edition, 1979.

7. Provost, L.P., and Elder, R.S. ''Interpretation of Percent Recovery Data,'' *American Laboratory, 15,* 58–63 (1983). (The value 2.44 used in the equation in Section 8.3.3 is two times the value 1.22 derived in this report.)

8. ''Methods 330.4 (Titrimetric, DPD–FAS) and 330.5 (Spectrophotometric, DPD) for Chlorine, Total Residual,'' Methods for Chemical Analysis of Water and Wastes, EPA 600/4–79–020, U.S. Environmental Protection Agency, Environmental Monitoring and Support Laboratory, Cincinnati, Ohio 45268, March 1979.

9. ''EPA Method Study 24, Method 601—Purgeable Halocarbons by the Purge and Trap Method,'' EPA 600/4–84–064, National Technical Information Service, PB84–212448, Springfield, Virginia 22161, July 1984.

10. ''Method Validation Data for EPA Method 601,'' Memorandum from B. Potter, U.S. Environmental Protection Agency, Environmental Monitoring and Support Laboratory, Cincinnati, Ohio 45268, November 10, 1983.

11. Bellar, T. A., Unpublished data, U.S. Environmental Protection Agency, Environmental Monitoring and Support Laboratory, Cincinnati, Ohio 45268, 1981.

TABLE 1—CHROMATOGRAPHIC CONDITIONS AND METHOD DETECTION LIMITS

Parameter	Retention time (min)		Method detection limit (µg/L)
	Column 1	Column 2	
Chloromethane	1.50	5.28	0.08
Bromomethane	2.17	7.05	1.18
Dichlorodifluoromethane	2.62	nd	1.81
Vinyl chloride	2.67	5.28	0.18
Chloroethane	3.33	8.68	0.52
Methylene chloride	5.25	10.1	0.25
Trichlorofluoromethane	7.18	nd	nd
1,1-Dichloroethene	7.93	7.72	0.13
1,1-Dichloroethane	9.30	12.6	0.07
trans-1,2-Dichloroethene	10.1	9.38	0.10
Chloroform	10.7	12.1	0.05
1,2-Dichloroethane	11.4	15.4	0.03
1,1,1-Trichloroethane	12.6	13.1	0.03
Carbon tetrachloride	13.0	14.4	0.12
Bromodichloromethane	13.7	14.6	0.10
1,2-Dichloropropane	14.9	16.6	0.04
cis-1,3-Dichloropropene	15.2	16.6	0.34
Trichloroethene	15.8	13.1	0.12
Dibromochloromethane	16.5	16.6	0.09
1,1,2-Trichloroethane	16.5	18.1	0.02
trans-1,3-Dichloropropene	16.5	18.0	0.20
2-Chloroethylvinyl ether	18.0	nd	0.13

TABLE 1—CHROMATOGRAPHIC CONDITIONS AND METHOD DETECTION LIMITS—Continued

Parameter	Retention time (min)		Method detection limit (µg/L)
	Column 1	Column 2	
Bromoform	19.2	19.2	0.20
1,1,2,2-Tetrachloroethane	21.6	nd	0.03
Tetrachloroethene	21.7	15.0	0.03
Chlorobenzene	24.2	18.8	0.25
1,3-Dichlorobenzene	34.0	22.4	0.32
1,2-Dichlorobenzene	34.9	23.5	0.15
1,4-Dichlorobenzene	35.4	22.3	0.24

Column 1 conditions: Carbopack B (60/80 mesh) coated with 1% SP–1000 packed in an 8 ft x 0.1 in. ID stainless steel or glass column with helium carrier gas at 40 mL/min flow rate. Column temperature held at 45 °C for 3 min then programmed at 8 °C/min to 220 °C and held for 15 min.

Column 2 conditions: Porisil-C (100/120 mesh) coated with n-octane packed in a 6 ft x 0.1 in. ID stainless steel or glass column with helium carrier gas at 40 mL/min flow rate. Column temperature held at 50 °C for 3 min then programmed at 6 °C/min to 170 °C and held for 4 min.

nd=not determined.

TABLE 2—CALIBRATION AND QC ACCEPTANCE CRITERIA—METHOD 601 [a]

Parameter	Range for Q (µg/L)	Limit for s (µg/L)	Range for $\bar{X}$ (µg/L)	Range P, P_s (%)
Bromodichloromethane	15.2–24.8	4.3	10.7–32.0	42–172
Bromoform	14.7–25.3	4.7	5.0–29.3	13–159
Bromomethane	11.7–28.3	7.6	3.4–24.5	D–144
Carbon tetrachloride	13.7–26.3	5.6	11.8–25.3	43–143
Chlorobenzene	14.4–25.6	5.0	10.2–27.4	38–150
Chloroethane	15.4–24.6	4.4	11.3–25.2	46–137
2-Chloroethylvinyl ether	12.0–28.0	8.3	4.5–35.5	14–186
Chloroform	15.0–25.0	4.5	12.4–24.0	49–133
Chloromethane	11.9–28.1	7.4	D–34.9	D–193
Dibromochloromethane	13.1–26.9	6.3	7.9–35.1	24–191
1,2-Dichlorobenzene	14.0–26.0	5.5	1.7–38.9	D–208
1,3-Dichlorobenzene	9.9–30.1	9.1	6.2–32.6	7–187
1,4-Dichlorobenzene	13.9–26.1	5.5	11.5–25.5	42–143
1,1-Dichloroethane	16.8–23.2	3.2	11.2–24.6	47–132
1,2-Dichloroethane	14.3–25.7	5.2	13.0–26.5	51–147
1,1-Dichloroethene	12.6–27.4	6.6	10.2–27.3	28–167
trans-1,2-Dichloroethene	12.8–27.2	6.4	11.4–27.1	38–155
1,2-Dichloropropane	14.8–25.2	5.2	10.1–29.9	44–156
cis-1,3-Dichloropropene	12.8–27.2	7.3	6.2–33.8	22–178
trans-1,3-Dichloropropene	12.8–27.2	7.3	6.2–33.8	22–178
Methylene chloride	15.5–24.5	4.0	7.0–27.6	25–162
1,1,2,2-Tetrachloroethane	9.8–30.2	9.2	6.6–31.8	8–184
Tetrachloroethene	14.0–26.0	5.4	8.1–29.6	26–162
1,1,1-Trichloroethane	14.2–25.8	4.9	10.8–24.8	41–138
1,1,2-Trichloroethane	15.7–24.3	3.9	9.6–25.4	39–136
Trichloroethene	15.4–24.6	4.2	9.2–26.6	35–146
Trichlorofluoromethane	13.3–26.7	6.0	7.4–28.1	21–156
Vinyl chloride	13.7–26.3	5.7	8.2–29.9	28–163

[a] Criteria were calculated assuming a QC check sample concentration of 20 µg/L.
Q=Concentration measured in QC check sample, in µg/L (Section 7.5.3).
s=Standard deviation of four recovery measurements, in µg/L (Section 8.2.4).
$\bar{X}$=Average recovery for four recovery measurements, in µg/L (Section 8.2.4).
P, P_s=Percent recovery measured (Section 8.3.2, Section 8.4.2).
D=Detected; result must be greater than zero.

NOTE: These criteria are based directly upon the method performance data in Table 3. Where necessary, the limits for recovery have been broadened to assure applicability of the limits to concentrations below those used to develop Table 3.

TABLE 3.—METHOD ACCURACY AND PRECISION AS FUNCTIONS OF CONCENTRATION—METHOD 601

Parameter	Accuracy, as recovery, X′ (µg/L)	Single analyst precision, s_r' (µg/L)	Overall precision, S′ (µg/L)
Bromodichloromethane	1.12C − 1.02	0.11$\bar{X}$+0.04	0.20$\bar{X}$+1.00
Bromoform	0.96C − 2.05	0.12$\bar{X}$+0.58	0.21$\bar{X}$+2.41
Bromomethane	0.76C − 1.27	0.28$\bar{X}$+0.27	0.36$\bar{X}$+0.94
Carbon tetrachloride	0.98C − 1.04	0.15$\bar{X}$+0.38	0.20$\bar{X}$+0.39
Chlorobenzene	1.00C − 1.23	0.15$\bar{X}$ − 0.02	0.18$\bar{X}$+1.21
Choroethane	0.99C − 1.53	0.14$\bar{X}$ − 0.13	0.17$\bar{X}$+0.63

TABLE 3.—METHOD ACCURACY AND PRECISION AS FUNCTIONS OF CONCENTRATION—METHOD 601—Continued

Parameter	Accuracy, as recovery, X′ (μg/L)	Single analyst precision, s_r' (μg/L)	Overall precision, S′ (μg/L)
2-Chloroethylvinyl ether [a]	1.00C	0.20$\bar{X}$	0.35$\bar{X}$
Chloroform	0.93C − 0.39	0.13$\bar{X}$+0.15	0.19$\bar{X}$ − 0.02
Chloromethane	0.77C+0.18	0.28$\bar{X}$ − 0.31	0.52$\bar{X}$+1.31
Dibromochloromethane	0.94C+2.72	0.11$\bar{X}$+1.10	0.24$\bar{X}$+1.68
1,2-Dichlorobenzene	0.93C+1.70	0.20$\bar{X}$+0.97	0.13$\bar{X}$+6.13
1,3-Dichlorobenzene	0.95C+0.43	0.14$\bar{X}$+2.33	0.26$\bar{X}$+2.34
1,4-Dichlorobenzene	0.93C − 0.09	0.15$\bar{X}$+0.29	0.20$\bar{X}$+0.41
1,1-Dichloroethane	0.95C − 1.08	0.09$\bar{X}$+0.17	0.14$\bar{X}$+0.94
1,2-Dichloroethane	1.04C − 1.06	0.11$\bar{X}$+0.70	0.15$\bar{X}$+0.94
1,1-Dichloroethene	0.98C − 0.87	0.21$\bar{X}$ − 0.23	0.29$\bar{X}$ − 0.40
trans-1,2-Dichloroethene	0.97C − 0.16	0.11$\bar{X}$+1.46	0.17$\bar{X}$+1.46
1,2-Dichloropropane [a]	1.00C	0.13$\bar{X}$	0.23$\bar{X}$
cis-1,3-Dichloropropene [a]	1.00C	0.18$\bar{X}$	0.32$\bar{X}$
trans-1,3-Dichloropropene [a]	1.00C	0.18$\bar{X}$	0.32$\bar{X}$
Methylene chloride	0.91C − 0.93	0.11$\bar{X}$+0.33	0.21$\bar{X}$+1.43
1,1,2,2-Tetrachloroethene	0.95C+0.19	0.14$\bar{X}$+2.41	0.23$\bar{X}$+2.79
Tetrachloroethene	0.94C+0.06	0.14$\bar{X}$+0.38	0.18$\bar{X}$+2.21
1,1,1-Trichloroethane	0.90C − 0.16	0.15$\bar{X}$+0.04	0.20$\bar{X}$+0.37
1,1,2-Trichloroethane	0.86C+0.30	0.13$\bar{X}$ − 0.14	0.19$\bar{X}$+0.67
Trichloroethene	0.87C+0.48	0.13$\bar{X}$ − 0.03	0.23$\bar{X}$+0.30
Trichlorofluoromethane	0.89C − 0.07	0.15$\bar{X}$+0.67	0.26$\bar{X}$+0.91
Vinyl chloride	0.97C − 0.36	0.13$\bar{X}$+0.65	0.27$\bar{X}$+0.40

$\bar{X}'$=Expected recovery for one or more measurements of a sample containing a concentration of C, in μg/L.
s_r'=Expected single analyst standard deviation of measurements at an average concentration found of $\bar{X}$, in μg/L.
S^1=Expected interlaboratory standard deviation of measurements at an average concentration found of X, in μg/L.
C=True value for the concentration, in μg/L.
$\bar{X}$=Average recovery found for measurements of samples containing a concentration of C, in μg/L.
[a] Estimates based upon the performance in a single laboratory.[10]

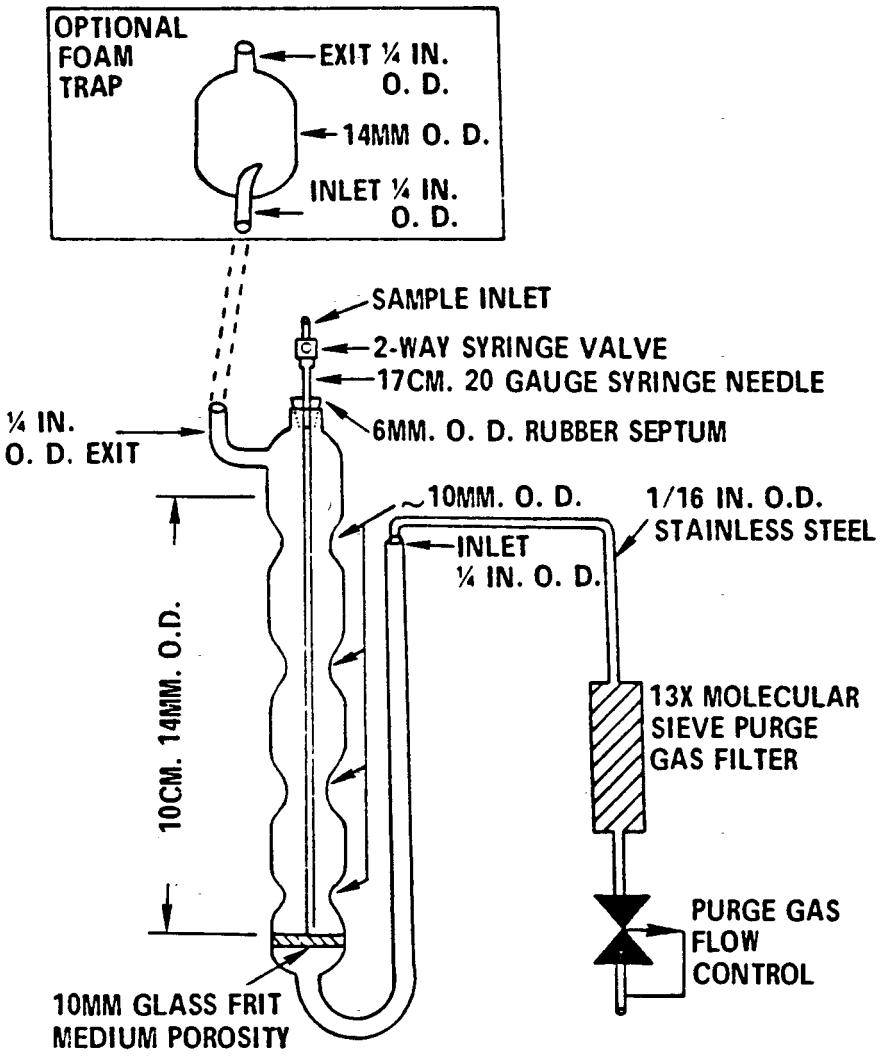


Figure 1. Purging device.

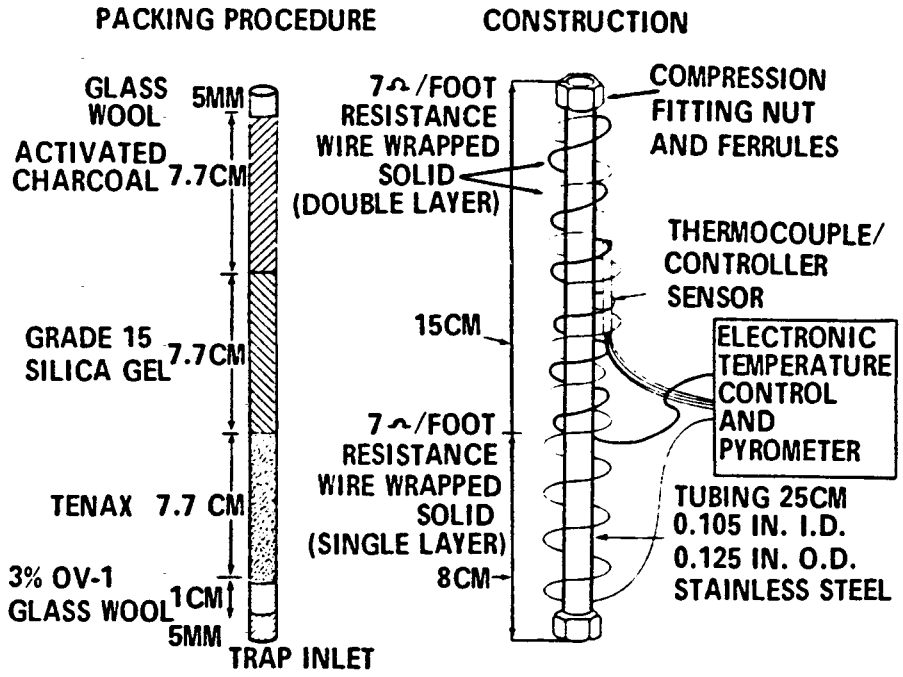


Figure 2. Trap packings and construction to include desorb capability

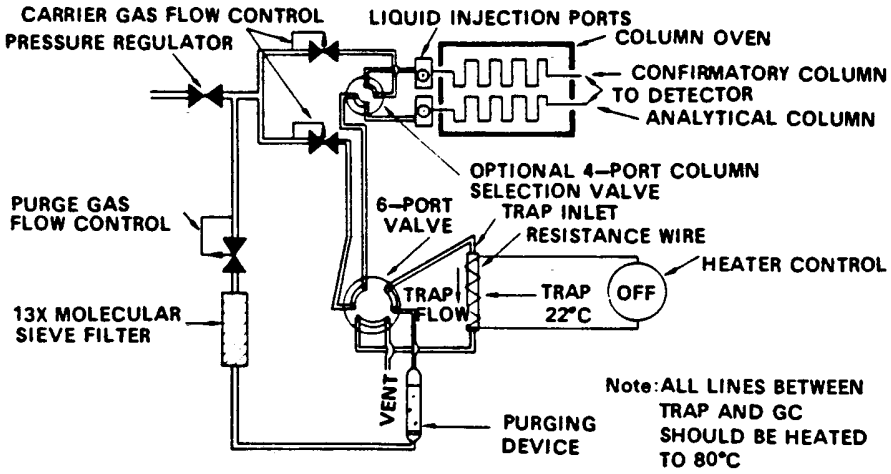


Figure 3. Purge and trap system-purge mode.

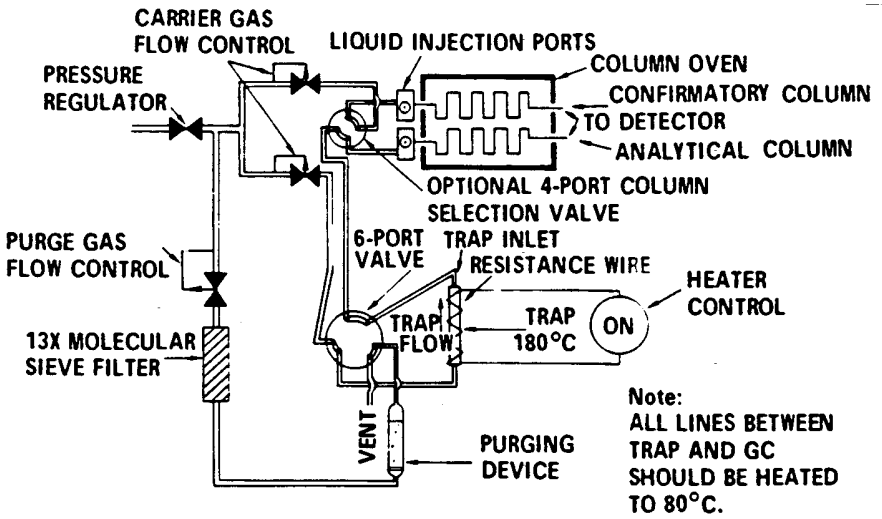


Figure 4. Purge and trap system - desorb mode.

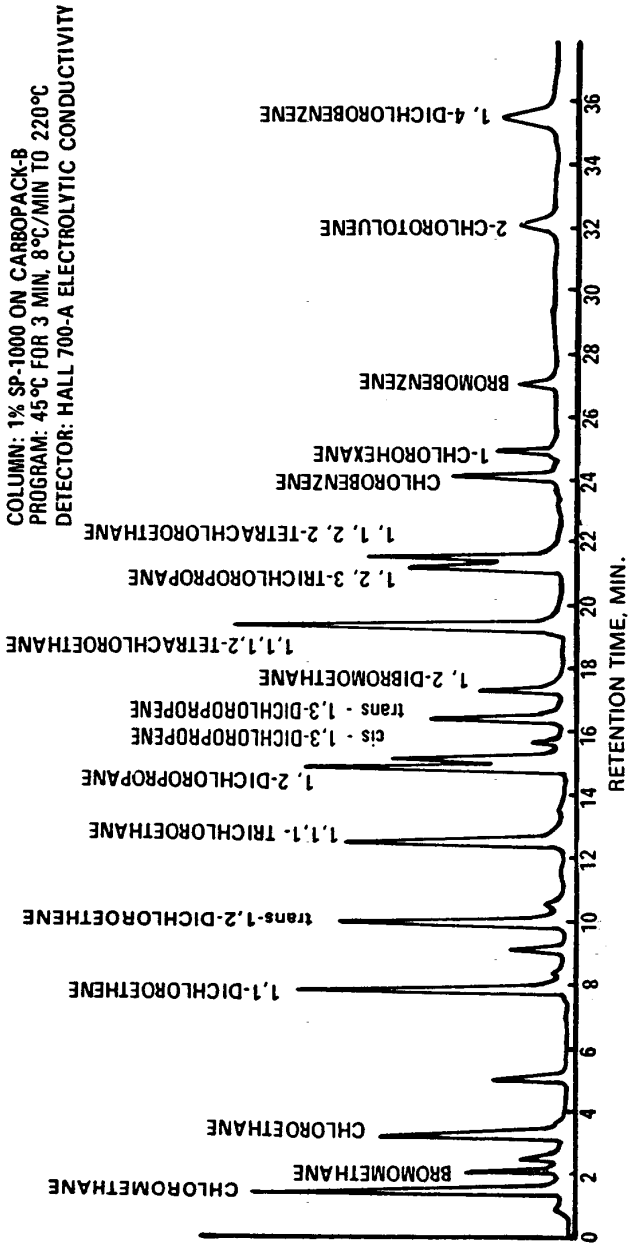


Figure 5. Gas chromatogram of purgeable halocarbons.

METHOD 602—PURGEABLE AROMATICS

1. Scope and Application

1.1 This method covers the determination of various purgeable aromatics. The following parameters may be determined by this method:

Parameter	STORET No.	CAS No.
Benzene	34030	71–43–2
Chlorobenzene	34301	108–90–7
1,2-Dichlorobenzene	34536	95–50–1
1,3-Dichlorobenzene	34566	541–73–1
1,4-Dichlorobenzene	34571	106–46–7
Ethylbenzene	34371	100–41–4
Toluene	34010	108–88–3

1.2 This is a purge and trap gas chromatographic (GC) method applicable to the determination of the compounds listed above in municipal and industrial discharges as provided under 40 CFR 136.1. When this method is used to analyze unfamiliar samples for any or all of the compounds above, compound identifications should be supported by at least one additional qualitative technique. This method describes analytical conditions for a second gas chromatographic column that can be used to confirm measurements made with the primary column. Method 624 provides gas chromatograph/mass spectrometer (GC/MS) conditions appropriate for the qualitative and quantitative confirmation of results for all of the parameters listed above.

1.3 The method detection limit (MDL, defined in Section 12.1)[1] for each parameter is listed in Table 1. The MDL for a specific wastewater may differ from those listed, depending upon the nature of interferences in the sample matrix.

1.4 Any modification of this method, beyond those expressly permitted, shall be considered as a major modification subject to application and approval of alternate test procedures under 40 CFR 136.4 and 136.5.

1.5 This method is restricted to use by or under the supervision of analysts experienced in the operation of a purge and trap system and a gas chromatograph and in the interpretation of gas chromatograms. Each analyst must demonstrate the ability to generate acceptable results with this method using the procedure described in Section 8.2.

2. Summary of Method

2.1 An inert gas is bubbled through a 5-mL water sample contained in a specially-designed purging chamber at ambient temperature. The aromatics are efficiently transferred from the aqueous phase to the vapor phase. The vapor is swept through a sorbent trap where the aromatics are trapped. After purging is completed, the trap is heated and backflushed with the inert gas to desorb the aromatics onto a gas chromatographic column. The gas chromatograph is temperature programmed to separate the aromatics which are then detected with a photoionization detector.[2,3]

2.2 The method provides an optional gas chromatographic column that may be helpful in resolving the compounds of interest from interferences that may occur.

3. Interferences

3.1 Impurities in the purge gas and organic compounds outgassing from the plumbing ahead of the trap account for the majority of contamination problems. The analytical system must be demonstrated to be free from contamination under the conditions of the analysis by running laboratory reagent blanks as described in Section 8.1.3. The use of non-Teflon plastic tubing, non-Teflon thread sealants, or flow controllers with rubber components in the purge and trap system should be avoided.

3.2 Samples can be contaminated by diffusion of volatile organics through the septum seal into the sample during shipment and storage. A field reagent blank prepared from reagent water and carried through the sampling and handling protocol can serve as a check on such contamination.

3.3 Contamination by carry-over can occur whenever high level and low level samples are sequentially analyzed. To reduce carry-over, the purging device and sample syringe must be rinsed with reagent water between sample analyses. Whenever an unusually concentrated sample is encountered, it should be followed by an analysis of reagent water to check for cross contamination. For samples containing large amounts of water-soluble materials, suspended solids, high boiling compounds or high aromatic levels, it may be necessary to wash the purging device with a detergent solution, rinse it with distilled water, and then dry it in an oven at 105 °C between analyses. The trap and other parts of the system are also subject to contamination; therefore, frequent bakeout and purging of the entire system may be required.

4. Safety

4.1 The toxicity or carcinogenicity of each reagent used in this method has not been precisely defined; however, each chemical compound should be treated as a potential health hazard. From this viewpoint, exposure to these chemicals must be reduced to the lowest possible level by whatever means available. The laboratory is responsible for maintaining a current awareness file of OSHA regulations regarding the safe handling of the chemicals specified in this method. A reference file of material data handling sheets should also be made available to all personnel involved in the chemical analysis. Additional references to laboratory safety

are available and have been identified[4–6] for the information of the analyst.

4.2 The following parameters covered by this method have been tentatively classified as known or suspected, human or mammalian carcinogens: benzene and 1,4-dichlorobenzene. Primary standards of these toxic compounds should be prepared in a hood. A NIOSH/MESA approved toxic gas respirator should be worn when the analyst handles high concentrations of these toxic compounds.

5. Apparatus and Materials

5.1 Sampling equipment, for discrete sampling.

5.1.1 Vial]25-mL capacity or larger, equipped with a screw cap with a hole in the center (Pierce #13075 or equivalent). Detergent wash, rinse with tap and distilled water, and dry at 105 °C before use.

5.1.2 Septum—Teflon-faced silicone (Pierce #12722 or equivalent). Detergent wash, rinse with tap and distilled water, and dry at 105°C for 1 h before use.

5.2 Purge and trap system—The purge and trap system consists of three separate pieces of equipment: A purging device, trap, and desorber. Several complete systems are now commercially available.

5.2.1 The purging device must be designed to accept 5-mL samples with a water column at least 3 cm deep. The gaseous head space between the water column and the trap must have a total volume of less than 15 mL. The purge gas must pass through the water column as finely divided bubbles with a diameter of less than 3 mm at the origin. The purge gas must be introduced no more than 5 mm from the base of the water column. The purging device illustrated in Figure 1 meets these design criteria.

5.2.2 The trap must be at least 25 cm long and have an inside diameter of at least 0.105 in.

5.2.2.1 The trap is packed with 1 cm of methyl silicone coated packing (Section 6.4.2) and 23 cm of 2,6-diphenylene oxide polymer (Section 6.4.1) as shown in Figure 2. This trap was used to develop the method performance statements in Section 12.

5.2.2.2 Alternatively, either of the two traps described in Method 601 may be used, although water vapor will preclude the measurement of low concentrations of benzene.

5.2.3 The desorber must be capable of rapidly heating the trap to 180 °C. The polymer section of the trap should not be heated higher than 180 °C and the remaining sections should not exceed 200 °C. The desorber illustrated in Figure 2 meets these design criteria.

5.2.4 The purge and trap system may be assembled as a separate unit or be coupled to a gas chromatograph as illustrated in Figures 3, 4, and 5.

5.3 Gas chromatograph—An analytical system complete with a temperature programmable gas chromatograph suitable for on-column injection and all required accessories including syringes, analytical columns, gases, detector, and strip-chart recorder. A data system is recommended for measuring peak areas.

5.3.1 Column 1—6 ft long x 0.082 in. ID stainless steel or glass, packed with 5% SP–1200 and 1.75% Bentone-34 on Supelcoport (100/120 mesh) or equivalent. This column was used to develop the method performance statements in Section 12. Guidelines for the use of alternate column packings are provided in Section 10.1.

5.3.2 Column 2—8 ft long x 0.1 in ID stainless steel or glass, packed with 5% 1,2,3-Tris(2-cyanoethoxy)propane on Chromosorb W–AW (60/80 mesh) or equivalent.

5.3.3 Detector—Photoionization detector (h-Nu Systems, Inc. Model PI–51–02 or equivalent). This type of detector has been proven effective in the analysis of wastewaters for the parameters listed in the scope (Section 1.1), and was used to develop the method performance statements in Section 12. Guidelines for the use of alternate detectors are provided in Section 10.1.

5.4 Syringes—5-mL glass hypodermic with Luerlok tip (two each), if applicable to the purging device.

5.5 Micro syringes—25-µL, 0.006 in. ID needle.

5.6 Syringe valve—2-way, with Luer ends (three each).

5.7 Bottle—15-mL, screw-cap, with Teflon cap liner.

5.8 Balance—Analytical, capable of accurately weighing 0.0001 g.

6. Reagents

6.1 Reagent water—Reagent water is defined as a water in which an interferent is not observed at the MDL of the parameters of interest.

6.1.1 Reagent water can be generated by passing tap water through a carbon filter bed containing about 1 lb of activated carbon (Filtrasorb-300, Calgon Corp., or equivalent).

6.1.2 A water purification system (Millipore Super-Q or equivalent) may be used to generate reagent water.

6.1.3 Reagent water may also be prepared by boiling water for 15 min. Subsequently, while maintaining the temperature at 90 °C, bubble a contaminant-free inert gas through the water for 1 h. While still hot, transfer the water to a narrow mouth screw-cap bottle and seal with a Teflon-lined septum and cap.

6.2 Sodium thiosulfate—(ACS) Granular.

6.3 Hydrochloric acid (1+1)—Add 50 mL of concentrated HCl (ACS) to 50 mL of reagent water.

6.4 Trap Materials:

6.4.1 2,6-Diphenylene oxide polymer—Tenax, (60/80 mesh), chromatographic grade or equivalent.

6.4.2 Methyl silicone packing—3% OV–1 on Chromosorb-W (60/80 mesh) or equivalent.

6.5 Methanol—Pesticide quality or equivalent.

6.6 Stock standard solutions—Stock standard solutions may be prepared from pure standard materials or purchased as certified solutions. Prepare stock standard solutions in methanol using assayed liquids. Because of the toxicity of benzene and 1,4-dichlorobenzene, primary dilutions of these materials should be prepared in a hood. A NIOSH/MESA approved toxic gas respirator should be used when the analyst handles high concentrations of such materials.

6.6.1 Place about 9.8 mL of methanol into a 10–mL ground glass stoppered volumetric flask. Allow the flask to stand, unstoppered, for about 10 min or until all alcohol wetted surfaces have dried. Weigh the flask to the nearest 0.1 mg.

6.6.2 Using a 100–µL syringe, immediately add two or more drops of assayed reference material to the flask, then reweigh. Be sure that the drops fall directly into the alcohol without contacting the neck of the flask.

6.6.3 Reweigh, dilute to volume, stopper, then mix by inverting the flask several times. Calculate the concentration in µg/µL from the net gain in weight. When compound purity is assayed to be 96% or greater, the weight can be used without correction to calculate the concentration of the stock standard. Commercially prepared stock standards can be used at any concentration if they are certified by the manufacturer or by an independent source.

6.6.4 Transfer the stock standard solution into a Teflon-sealed screw-cap bottle. Store at 4 °C and protect from light.

6.6.5 All standards must be replaced after one month, or sooner if comparison with check standards indicates a problem.

6.7 Secondary dilution standards—Using stock standard solutions, prepare secondary dilution standards in methanol that contain the compounds of interest, either singly or mixed together. The secondary dilution standards should be prepared at concentrations such that the aqueous calibration standards prepared in Section 7.3.1 or 7.4.1 will bracket the working range of the analytical system. Secondary solution standards must be stored with zero headspace and should be checked frequently for signs of degradation or evaporation, especially just prior to preparing calibration standards from them.

6.8 Quality control check sample concentrate—See Section 8.2.1.

7. Calibration

7.1 Assemble a purge and trap system that meets the specifications in Section 5.2. Condition the trap overnight at 180 °C by backflushing with an inert gas flow of at least 20 mL/min. Condition the trap for 10 min once daily prior to use.

7.2 Connect the purge and trap system to a gas chromatograph. The gas chromatograph must be operated using temperature and flow rate conditions equivalent to those given in Table 1. Calibrate the purge and trap-gas chromatographic system using either the external standard technique (Section 7.3) or the internal standard technique (Section 7.4).

7.3 External standard calibration procedure:

7.3.1 Prepare calibration standards at a minimum of three concentration levels for each parameter by carefully adding 20.0 µL of one or more secondary dilution standards to 100, 500, or 1000 mL of reagent water. A 25–µL syringe with a 0.006 in. ID needle should be used for this operation. One of the external standards should be at a concentration near, but above, the MDL (Table 1) and the other concentrations should correspond to the expected range of concentrations found in real samples or should define the working range of the detector. These aqueous standards must be prepared fresh daily.

7.3.2 Analyze each calibration standard according to Section 10, and tabulate peak height or area responses versus the concentration in the standard. The results can be used to prepare a calibration curve for each compound. Alternatively, if the ratio of response to concentration (calibration factor) is a constant over the working range (<10% relative standard deviation, RSD), linearity through the origin can be assumed and the average ratio or calibration factor can be used in place of a calibration curve.

7.4 Internal standard calibration procedure—To use this approach, the analyst must select one or more internal standards that are similar in analytical behavior to the compounds of interest. The analyst must further demonstrate that the measurement of the internal standard is not affected by method or matrix interferences. Because of these limitations, no internal standard can be suggested that is applicable to all samples. The compound, α,α,α,-trifluorotoluene, recommended as a surrogate spiking compound in Section 8.7 has been used successfully as an internal standard.

7.4.1 Prepare calibration standards at a minimum of three concentration levels for each parameter of interest as described in Section 7.3.1.

7.4.2 Prepare a spiking solution containing each of the internal standards using the procedures described in Sections 6.6 and 6.7. It is recommended that the secondary dilution standard be prepared at a concentration of 15 µg/mL of each internal standard compound. The addition of 10 µl of this

standard to 5.0 mL of sample or calibration standard would be equivalent to 30 µg/L.

7.4.3 Analyze each calibration standard according to Section 10, adding 10 µL of internal standard spiking solution directly to the syringe (Section 10.4). Tabulate peak height or area responses against concentration for each compound and internal standard, and calculate response factors (RF) for each compound using Equation 1.

$$RF = \frac{(A_s)(C_{is})}{(A_{is})(C_s)}$$

Equation 1

where:

A_s=Response for the parameter to be measured.

A_{is}=Response for the internal standard.

C_{is}=Concentration of the internal standard

C_s=Concentration of the parameter to be measured.

If the RF value over the working range is a constant (<10% RSD), the RF can be assumed to be invariant and the average RF can be used for calculations. Alternatively, the results can be used to plot a calibration curve of response ratios, A_s/A_{is}, vs. RF.

7.5 The working calibration curve, calibration factor, or RF must be verified on each working day by the measurement of a QC check sample.

7.5.1 Prepare the QC check sample as described in Section 8.2.2.

7.5.2 Analyze the QC check sample according to Section 10.

7.5.3 For each parameter, compare the response (Q) with the corresponding calibration acceptance criteria found in Table 2. If the responses for all parameters of interest fall within the designated ranges, analysis of actual samples can begin. If any individual Q falls outside the range, a new calibration curve, calibration factor, or RF must be prepared for that parameter according to Section 7.3 or 7.4.

8. Quality Control

8.1 Each laboratory that uses this method is required to operate a formal quality control program. The mimimum requirements of this program consist of an initial demonstration of laboratory capability and an ongoing analysis of spiked samples to evaluate and document data quality. The laboratory must maintain records to document the quality of data that is generated. Ongoing data quality checks are compared with established performance criteria to determine if the results of analyses meet the performance characteristics of the method. When results of sample spikes indicate atypical method performance, a quality control check standard must be analyzed to confirm that the measurements were performed in an in-control mode of operation.

8.1.1 The analyst must make an initial, one-time, demonstration of the ability to generate acceptable accuracy and precision with this method. This ability is established as described in Section 8.2.

8.1.2 In recognition of advances that are occurring in chromatography, the analyst is permitted certain options (detailed in Section 10.1) to improve the separations or lower the cost of measurements. Each time such a modification is made to the method, the analyst is required to repeat the procedure in Section 8.2.

8.1.3 Each day, the analyst must analyze a reagent water blank to demonstrate that interferences from the analytical system are under control.

8.1.4 The laboratory must, on an ongoing basis, spike and analyze a minimum of 10% of all samples to monitor and evaluate laboratory data quality. This procedure is described in Section 8.3.

8.1.5 The laboratory must, on an ongoing basis, demonstrate through the analyses of quality control check standards that the operation of the measurement system is in control. This procedure is described in Section 8.4. The frequency of the check standard analyses is equivalent to 10% of all samples analyzed but may be reduced if spike recoveries from samples (Section 8.3) meet all specified quality control criteria.

8.1.6 The laboratory must maintain performance records to document the quality of data that is generated. This procedure is described in Section 8.5.

8.2 To establish the ability to generate acceptable accuracy and precision, the analyst must perform the following operations.

8.2.1 A quality control (QC) check sample concentrate is required containing each parameter of interest at a concentration of 10 µg/mL in methanol. The QC check sample concentrate must be obtained from the U.S. Environmental Protection Agency, Environmental Monitoring and Support Laboratory in Cincinnati, Ohio, if available. If not available from that source, the QC check sample concentrate must be obtained from another external source. If not available from either source above, the QC check sample concentrate must be prepared by the laboratory using stock standards prepared independently from those used for calibration.

8.2.2 Prepare a QC check sample to contain 20 µg/L of each parameter by adding 200 µL of QC check sample concentrate to 100 mL of reagant water.

8.2.3 Analyze four 5-mL aliquots of the well-mixed QC check sample according to Section 10.

8.2.4 Calculate the average recovery ($\bar{X}$) in µg/L, and the standard deviation of the recovery (s) in µg/L, for each parameter of interest using the four results.

8.2.5 For each parameter compare s and $\bar{X}$ with the corresponding acceptance criteria

for precision and accuracy, respectively, found in Table 2. If s and $\bar{X}$ for all parameters of interest meet the acceptance criteria, the system performance is acceptable and analysis of actual samples can begin. If any individual s exceeds the precision limit or any individual $\bar{X}$ falls outside the range for accuracy, the system performance is unacceptable for that parameter.

NOTE: The large number of parameters in Table 2 present a substantial probability that one or more will fail at least one of the acceptance criteria when all parameters are analyzed.

8.2.6 When one or more of the parameters tested fail at least one of the acceptance criteria, the analyst must proceed according to Section 8.2.6.1 or 8.2.6.2.

8.2.6.1 Locate and correct the source of the problem and repeat the test for all parameters of interest beginning with Section 8.2.3.

8.2.6.2 Beginning with Section 8.2.3, repeat the test only for those parameters that failed to meet criteria. Repeated failure, however, will confirm a general problem with the measurement system. If this occurs, locate and correct the source of the problem and repeat the test for all compounds of interest beginning with Section 8.2.3.

8.3 The laboratory must, on an ongoing basis, spike at least 10% of the samples from each sample site being monitored to assess accuracy. For laboratories analyzing one to ten samples per month, at least one spiked sample per month is required.

8.3.1 The concentration of the spike in the sample should be determined as follows:

8.3.1.1 If, as in compliance monitoring, the concentration of a specific parameter in the sample is being checked against a regulatory concentration limit, the spike should be at that limit or 1 to 5 times higher than the background concentration determined in Section 8.3.2, whichever concentration would be larger.

8.3.1.2 If the concentration of a specific parameter in the sample is not being checked against a limit specific to that parameter, the spike should be at 20 µg/L or 1 to 5 times higher than the background concentration determined in Section 8.3.2, whichever concentration would be larger.

8.3.2 Analyze one 5-mL sample aliquot to determine the background concentration (B) of each parameter. If necessary, prepare a new QC check sample concentrate (Section 8.2.1) appropriate for the background concentrations in the sample. Spike a second 5-mL sample aliquot with 10 µL of the QC check sample concentrate and analyze it to determine the concentration after spiking (A) of each parameter. Calculate each percent recovery (P) as 100(A–B)%/T, where T is the known true value of the spike.

8.3.3 Compare the percent recovery (P) for each parameter with the corresponding QC acceptance criteria found in Table 2. These acceptance criteria were calculated to include an allowance for error in measurement of both the background and spike concentrations, assuming a spike to background ratio of 5:1. This error will be accounted for to the extent that the analyst's spike to background ratio approaches 5:1.[7] If spiking was performed at a concentration lower than 20 µg/L, the analyst must use either the QC acceptance criteria in Table 2, or optional QC acceptance criteria calculated for the specific spike concentration. To calculate optional acceptance criteria for the recovery of a parameter: (1) Calculate accuracy (X′) using the equation in Table 3, substituting the spike concentration (T) for C; (2) calculate overall precision (S′) using the equation in Table 3, substituting X′ for $\bar{X}$; (3) calculate the range for recovery at the spike concentration as (100 X′/T) ± 2.44(100 S′/T)%.[7]

8.3.4 If any individual P falls outside the designated range for recovery, that parameter has failed the acceptance criteria. A check standard containing each parameter that failed the criteria must be analyzed as described in Section 8.4.

8.4 If any parameter fails the acceptance criteria for recovery in Section 8.3, a QC check standard containing each parameter that failed must be prepared and analyzed.

NOTE: The frequency for the required analysis of a QC check standard will depend upon the number of parameters being simultaneously tested, the complexity of the sample matrix, and the performance of the laboratory.

8.4.1 Prepare the QC check standard by adding 10 µL of QC check sample concentrate (Section 8.2.1 or 8.3.2) to 5 mL of reagent water. The QC check standard needs only to contain the parameters that failed criteria in the test in Section 8.3.

8.4.2 Analyze the QC check standard to determine the concentration measured (A) of each parameter. Calculate each percent recovery (P_s) as 100 (A/T)%, where T is the true value of the standard concentration.

8.4.3 Compare the percent recovery (P_s) for each parameter with the corresponding QC acceptance criteria found in Table 2. Only parameters that failed the test in Section 8.3 need to be compared with these criteria. If the recovery of any such parameter falls outside the designated range, the laboratory performance for that parameter is judged to be out of control, and the problem must be immediately identified and corrected. The analytical result for that parameter in the unspiked sample is suspect and may not be reported for regulatory compliance purposes.

8.5 As part of the QC program for the laboratory, method accuracy for wastewater samples must be assessed and records must be maintained. After the analysis of five

spiked wastewater samples as in Section 8.3, calculate the average percent recovery (P) and the standard deviation of the percent recovery (s_p). Express the accuracy assessment as a percent recovery interval from $P-2s_p$ to $P+2s_p$. If P=90% and s_p=10%, for example, the accuracy interval is expressed as 70–110%. Update the accuracy assessment for each parameter on a regular basis (e.g. after each five to ten new accuracy measurements).

8.6 It is recommended that the laboratory adopt additional quality assurance practices for use with this method. The specific practices that are most productive depend upon the needs of the laboratory and the nature of the samples. Field duplicates may be analyzed to assess the precision of the environmental measurements. When doubt exists over the identification of a peak on the chromatogram, confirmatory techniques such as gas chromatography with a dissimilar column, specific element detector, or mass spectrometer must be used. Whenever possible, the laboratory should analyze standard reference materials and participate in relevant performance evaluation studies.

8.7 The analyst should monitor both the performance of the analytical system and the effectiveness of the method in dealing with each sample matrix by spiking each sample, standard, and reagent water blank with surrogate compounds (e.g. α, α, α,-trifluorotoluene) that encompass the range of the temperature program used in this method. From stock standard solutions prepared as in Section 6.6, add a volume to give 750 µg of each surrogate to 45 mL of reagent water contained in a 50-mL volumetric flask, mix and dilute to volume for a concentration of 15 mg/µL. Add 10 µL of this surrogate spiking solution directly into the 5-mL syringe with every sample and reference standard analyzed. Prepare a fresh surrogate spiking solution on a weekly basis. If the internal standard calibration procedure is being used, the surrogate compounds may be added directly to the internal standard spiking solution (Section 7.4.2).

9. Sample Collection, Preservation, and Handling

9.1 The samples must be iced or refrigerated from the time of collection until analysis. If the sample contains free or combined chlorine, add sodium thiosulfate preservative (10 mg/40 mL is sufficient for up to 5 ppm Cl_2) to the empty sample bottle just prior to shipping to the sampling site. EPA Method 330.4 or 330.5 may be used for measurement of residual chlorine.[8] Field test kits are available for this purpose.

9.2 Collect about 500 mL of sample in a clean container. Adjust the pH of the sample to about 2 by adding 1+1 HCl while stirring. Fill the sample bottle in such a manner that no air bubbles pass through the sample as the bottle is being filled. Seal the bottle so that no air bubbles are entrapped in it. Maintain the hermetic seal on the sample bottle until time of analysis.

9.3 All samples must be analyzed within 14 days of collection.[3]

10. Procedure

10.1 Table 1 summarizes the recommended operating conditions for the gas chromatograph. Included in this table are estimated retention times and MDL that can be achieved under these conditions. An example of the separations achieved by Column 1 is shown in Figure 6. Other packed columns, chromatographic conditions, or detectors may be used if the requirements of Section 8.2 are met.

10.2 Calibrate the system daily as described in Section 7.

10.3 Adjust the purge gas (nitrogen or helium) flow rate to 40 mL/min. Attach the trap inlet to the purging device, and set the purge and trap system to purge (Figure 3). Open the syringe valve located on the purging device sample introduction needle.

10.4 Allow the sample to come to ambient temperature prior to introducing it to the syringe. Remove the plunger from a 5-mL syringe and attach a closed syringe valve. Open the sample bottle (or standard) and carefully pour the sample into the syringe barrel to just short of overflowing. Replace the syringe plunger and compress the sample. Open the syringe valve and vent any residual air while adjusting the sample volume to 5.0 mL. Since this process of taking an aliquot destroys the validity of the sample for future analysis, the analyst should fill a second syringe at this time to protect against possible loss of data. Add 10.0 µL of the surrogate spiking solution (Section 8.7) and 10.0 µL of the internal standard spiking solution (Section 7.4.2), if applicable, through the valve bore, then close the valve.

10.5 Attach the syringe-syringe valve assembly to the syringe valve on the purging device. Open the syringe valves and inject the sample into the purging chamber.

10.6 Close both valves and purge the sample for 12.0±0.1 min at ambient temperature.

10.7 After the 12-min purge time, disconnect the purging device from the trap. Dry the trap by maintaining a flow of 40 mL/min of dry purge gas through it for 6 min (Figure 4). If the purging device has no provision for bypassing the purger for this step, a dry purger should be inserted into the device to minimize moisture in the gas. Attach the trap to the chromatograph, adjust the purge and trap system to the desorb mode (Figure 5), and begin to temperature program the gas chromatograph. Introduce the trapped materials to the GC column by rapidly heating the trap to 180 °C while backflushing the trap with an inert gas between 20 and 60 mL/min for 4 min. If rapid heating of the trap cannot be achieved, the GC column must be used as

a secondary trap by cooling it to 30 °C (sub-ambient temperature, if poor peak geometry and random retention time problems persist) instead of the initial program temperature of 50 °C.

10.8 While the trap is being desorbed into the gas chromatograph column, empty the purging chamber using the sample introduction syringe. Wash the chamber with two 5-mL flushes of reagent water.

10.9 After desorbing the sample for 4 min, recondition the trap by returning the purge and trap system to the purge mode. Wait 15 s, then close the syringe valve on the purging device to begin gas flow through the trap. The trap temperature should be maintained at 180 °C. After approximately 7 min, turn off the trap heater and open the syringe valve to stop the gas flow through the trap. When the trap is cool, the next sample can be analyzed.

10.10 Identify the parameters in the sample by comparing the retention times of the peaks in the sample chromatogram with those of the peaks in standard chromatograms. The width of the retention time window used to make identifications should be based upon measurements of actual retention time variations of standards over the course of a day. Three times the standard deviation of a retention time for a compound can be used to calculate a suggested window size; however, the experience of the analyst should weigh heavily in the interpretation of chromatograms.

10.11 If the response for a peak exceeds the working range of the system, prepare a dilution of the sample with reagent water from the aliquot in the second syringe and reanalyze.

11. Calculations

11.1 Determine the concentration of individual compounds in the sample.

11.1.1 If the external standard calibration procedure is used, calculate the concentration of the parameter being measured from the peak response using the calibration curve or calibration factor determined in Section 7.3.2.

11.1.2 If the internal standard calibration procedure is used, calculate the concentration in the sample using the response factor (RF) determined in Section 7.4.3 and Equation 2.

$$\text{Concentration } (\mu g/L) = \frac{(A_s)(C_{is})}{(A_{is})(RF)}$$

Equation 2

where:

A_s = Response for the parameter to be measured.

A_{is} = Response for the internal standard.

C_{is} = Concentration of the internal standard.

11.2 Report results in µg/L without correction for recovery data. All QC data obtained should be reported with the sample results.

12. Method Performance

12.1 The method detection limit (MDL) is defined as the minimum concentration of a substance that can be measured and reported with 99% confidence that the value is above zero.[1] The MDL concentrations listed in Table 1 were obtained using reagent water.[9] Similar results were achieved using representative wastewaters. The MDL actually achieved in a given analysis will vary depending on instrument sensitivity and matrix effects.

12.2 This method has been demonstrated to be applicable for the concentration range from the MDL to 100 X MDL.[9] Direct aqueous injection techniques should be used to measure concentration levels above 1000 x MDL.

12.3 This method was tested by 20 laboratories using reagent water, drinking water, surface water, and three industrial wastewaters spiked at six concentrations over the range 2.1 to 550 µg/L.[9] Single operator precision, overall precision, and method accuracy were found to be directly related to the concentration of the parameter and essentially independent of the sample matrix. Linear equations to describe these relationships are presented in Table 3.

References

1. 40 CFR part 136, appendix B.

2. Lichtenberg, J.J. ''Determining Volatile Organics at Microgram-per-Litre-Levels by Gas Chromatography,'' Journal American Water Works Association, *66,* 739 (1974).

3. Bellar, T.A., and Lichtenberg, J.J. ''Semi-Automated Headspace Analysis of Drinking Waters and Industrial Waters for Purgeable Volatile Organic Compounds,'' Proceedings of Symposium on Measurement of Organic Pollutants in Water and Wastewater. American Society for Testing and Materials, STP 686, C.E. Van Hall, editor, 1978.

4. ''Carcinogens—Working with Carcinogens,'' Department of Health, Education, and Welfare, Public Health Service, Center for Disease Control, National Institute for Occupational Safety and Health. Publication No. 77–206, August 1977.

5. ''OSHA Safety and Health Standards, General Industry,'' (29 CFR part 1910), Occupational Safety and Health Administration, OSHA 2206 (Revised, January 1976).

6. ''Safety in Academic Chemistry Laboratories,'' American Chemical Society Publication, Committee on Safety, 3rd Edition, 1979.

7. Provost, L.P., and Elder, R.S. ''Interpretation of Percent Recovery Data,'' *American Laboratory, 15,* 58-63 (1983). (The value 2.44

used in the equation in Section 8.3.3. is two times the value 1.22 derived in this report.)

8.''Methods 330.4 (Titrimetric, DPD–FAS) and 330.5 (Spectrophotometric, DPD) for Chlorine, Total Residual,'' Methods for Chemical Analysis of Water and Wastes, EPA–600/4–79–020, U.S. Environmental Protection Agency, Office of Research and Development, Environmental Monitoring and Support Laboratory, Cincinnati, Ohio 45268. March 1979.

9. ''EPA Method Study 25, Method 602, Purgeable Aromatics,'' EPA 600/4–84–042, National Technical Information Service, PB84–196682, Springfield, Virginia 22161, May 1984.

TABLE 1—CHROMATOGRAPHIC CONDITIONS AND METHOD DETECTION LIMITS

Parameter	Retention time (min)		Method detection limit (µg/L)
	Column 1	Column 2	
Benzene	3.33	2.75	0.2
Toluene	5.75	4.25	0.2
Ethylbenzene	8.25	6.25	0.2
Chlorobenzene	9.17	8.02	0.2
1,4-Dichlorobenzene	16.8	16.2	0.3
1,3-Dichlorobenzene	18.2	15.0	0.4
1,2-Dichlorobenzene	25.9	19.4	0.4

Column 1 conditions: Supelcoport (100/120 mesh) coated with 5% SP–1200/1.75% Bentone-34 packed in a 6 ft x 0.085 in. ID stainless steel column with helium carrier gas at 36 mL/min flow rate. Column temperature held at 50 °C for 2 min then programmed at 6 °C/min to 90 °C for a final hold.

Column 2 conditions: Chromosorb W–AW (60/80 mesh) coated with 5% 1,2,3-Tris(2-cyanoethyoxy)propane packed in a 6 ft x 0.085 in. ID stainless steel column with helium carrier gas at 30 mL/min flow rate. Column temperature held at 40 °C for 2 min then programmed at 2 °C/min to 100 °C for a final hold.

TABLE 2—CALIBRATION AND QC ACCEPTANCE CRITERIA—METHOD 602 [a]

Parameter	Range for Q (µg/L)	Limit for s (µg/L)	Range for $\bar{X}$ (µg/L)	Range for P, P_s (%)
Benzene	15.4–24.6	4.1	10.0–27.9	39–150
Chlorobenzene	16.1–23.9	3.5	12.7–25.4	55–135
1,2-Dichlorobenzene	13.6–26.4	5.8	10.6–27.6	37–154
1,3-Dichlorobenzene	14.5–25.5	5.0	12.8–25.5	50–141
1,4-Dichlorobenzene	13.9–26.1	5.5	11.6–25.5	42–143
Ethylbenzene	12.6–27.4	6.7	10.0–28.2	32–160
Toluene	15.5–24.5	4.0	11.2–27.7	46–148

Q=Concentration measured in QC check sample, in µg/L (Section 7.5.3).
s=Standard deviation of four recovery measurements, in µg/L (Section 8.2.4).
$\bar{X}$=Average recovery for four recovery measurements, in µg/L (Section 8.2.4).
P_s, P=Percent recovery measured (Section 8.3.2, Section 8.4.2).
[a] Criteria were calculated assuming a QC check sample concentration of 20 µg/L.

Note: These criteria are based directly upon the method performance data in Table 3. Where necessary, the limits for recovery have been broadened to assure applicability of the limits to concentrations below those used to develop Table 3.

TABLE 3—METHOD ACCURACY AND PRECISION AS FUNCTIONS OF CONCENTRATION—METHOD 602

Parameter	Accuracy, as recovery, X′ (µg/L)	Single analyst precision, s′ (µg/L)	Overall precision, S′ (µg/L)
Benzene	0.92C+0.57	$0.09\bar{X}+0.59$	$0.21\bar{X}+0.56$
Chlorobenzene	0.95C+0.02	$0.09\bar{X}+0.23$	$0.17\bar{X}+0.10$
1,2-Dichlorobenzene	0.93C+0.52	$0.17\bar{X}-0.04$	$0.22\bar{X}+0.53$
1,3-Dichlorobenzene	0.96C−0.05	$0.15\bar{X}-0.10$	$0.19\bar{X}+0.09$
1,4-Dichlorobenzene	0.93C−0.09	$0.15\bar{X}+0.28$	$0.20\bar{X}+0.41$
Ethylbenzene	0.94C+0.31	$0.17\bar{X}+0.46$	$0.26\bar{X}+0.23$
Toluene	0.94C+0.65	$0.09\bar{X}+0.48$	$0.18\bar{X}+0.71$

X′=Expected recovery for one or more measurements of a sample containing a concentration of C, in µg/L.
s′=Expected single analyst standard deviation of measurements at an average concentration found of $\bar{X}$, in µg/L.
S′=Expected interlaboratory standard deviation of measurements at an average concentration found of $\bar{X}$, in µg/L.
C=True value for the Concentration, in µg/L.
$\bar{X}$=Average recovery found for measurements of samples containing a concentration of C, in µg/L.

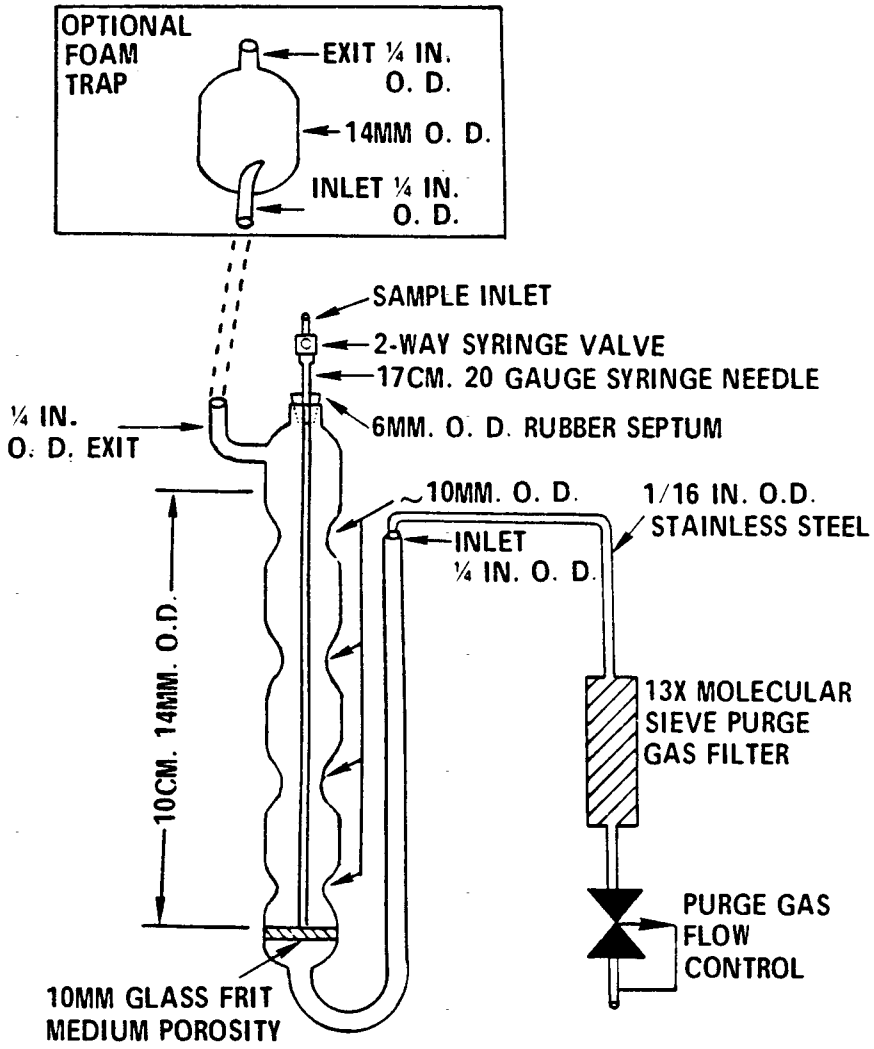


Figure 1. Purging device.

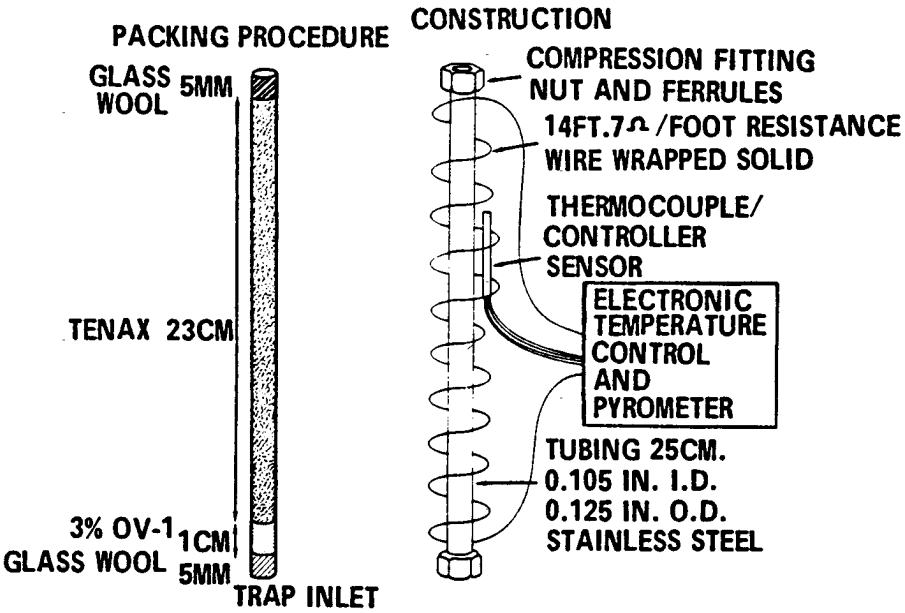


Figure 2. Trap packings and construction to include desorb capability.

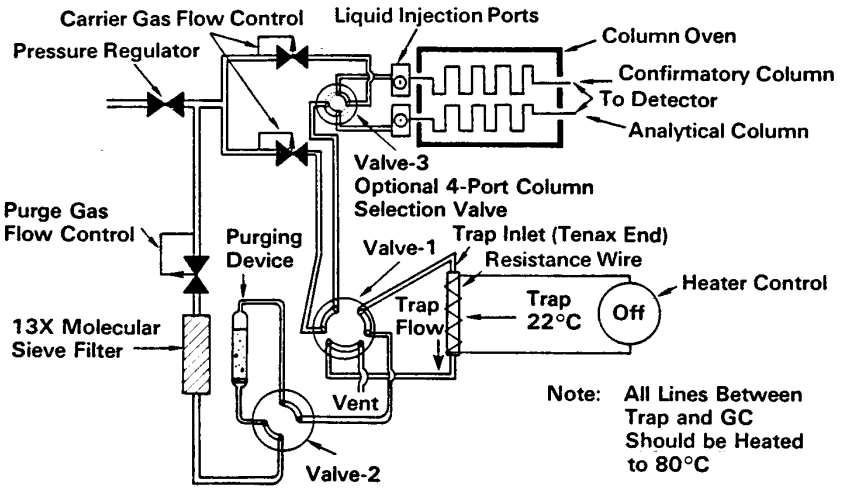


Figure 3. Purge and trap system - purge mode.

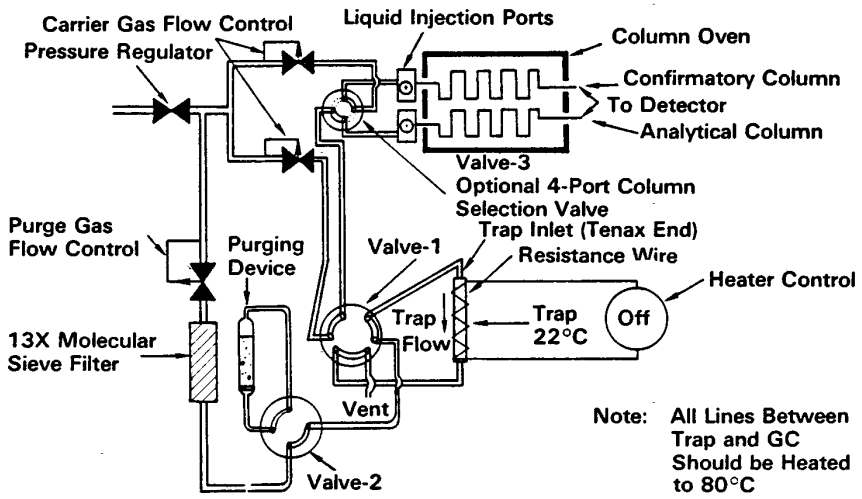


Figure 4. Purge and trap system-dry mode.

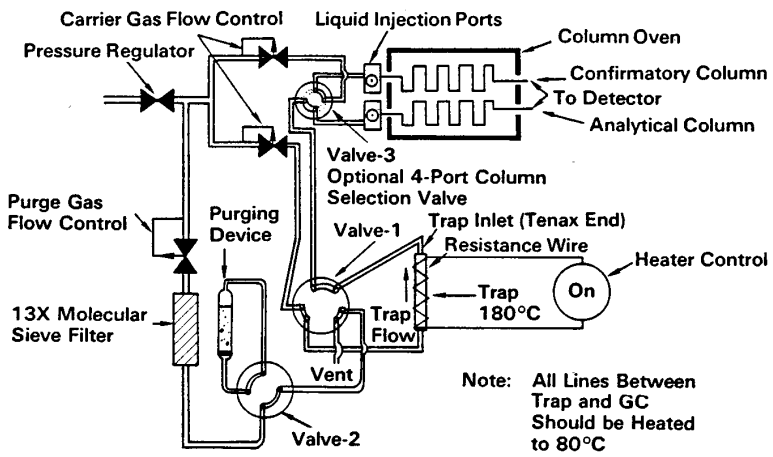


Figure 5. Purge and trap system-desorb mode.

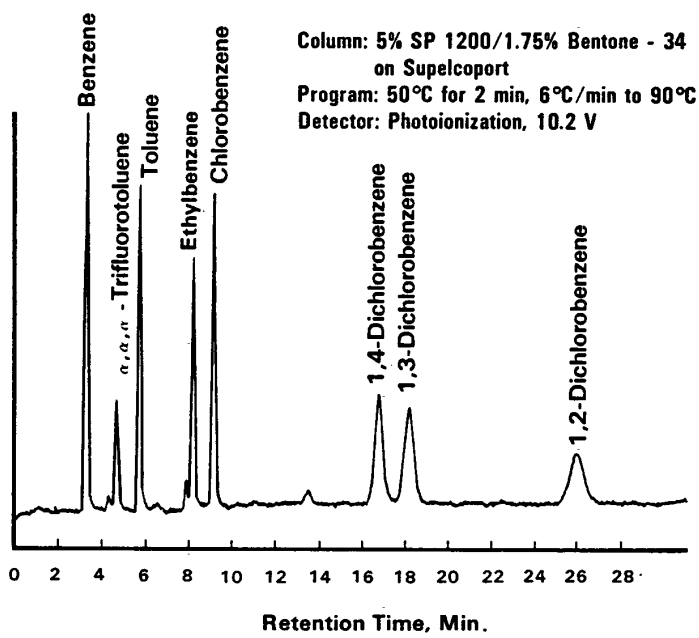


Figure 6. Gas chromatogram of purgeable aromatics.

METHOD 603—ACROLEIN AND ACRYLONITRILE

1. Scope and Application

1.1 This method covers the determination of acrolein and acrylonitrile. The following parameters may be determined by this method:

Parameter	STORET No.	CAS No.
Acrolein	34210	107–02–8
Acrylonitrile	34215	107–13–1

1.2 This is a purge and trap gas chromatographic (GC) method applicable to the determination of the compounds listed above in municipal and industrial discharges as provided under 40 CFR 136.1. When this method is used to analyze unfamiliar samples for either or both of the compounds above, compound identifications should be supported by at least one additional qualitative technique. This method describes analytical conditions for a second gas chromatographic column that can be used to confirm measurements made with the primary column. Method 624 provides gas chromatograph/mass spectrometer (GC/MS) conditions appropriate for the qualitative and quantitative confirmation of results for the parameters listed above, if used with the purge and trap conditions described in this method.

1.3 The method detection limit (MDL, defined in Section 12.1)[1] for each parameter is listed in Table 1. The MDL for a specific wastewater may differ from those listed, depending upon the nature of interferences in the sample matrix.

1.4 Any modification of this method, beyond those expressly permitted, shall be considered as a major modification subject to application and approval of alternate test procedures under 40 CFR 136.4 and 136.5.

1.5 This method is restricted to use by or under the supervision of analysts experienced in the operation of a purge and trap system and a gas chromatograph and in the interpretation of gas chromatograms. Each analyst must demonstrate the ability to generate acceptable results with this method using the procedure described in Section 8.2.

2. Summary of Method

2.1 An inert gas is bubbled through a 5-mL water sample contained in a heated purging chamber. Acrolein and acrylonitrile are transferred from the aqueous phase to the vapor phase. The vapor is swept through a sorbent trap where the analytes are trapped. After the purge is completed, the trap is heated and backflushed with the inert gas to desorb the compound onto a gas chromatographic column. The gas chromatograph is temperature programmed to separate the analytes which are then detected with a flame ionization detector.[2,3]

2.2 The method provides an optional gas chromatographic column that may be helpful in resolving the compounds of interest from the interferences that may occur.

3. Interferences

3.1 Impurities in the purge gas and organic compound outgassing from the plumbing of the trap account for the majority of contamination problems. The analytical system must be demonstrated to be free from contamination under the conditions of the analysis by running laboratory reagent blanks as described in Section 8.1.3. The use of non-Teflon plastic tubing, non-Teflon thread sealants, or flow controllers with rubber components in the purge and trap system should be avoided.

3.2 Samples can be contaminated by diffusion of volatile organics through the septum seal into the sample during shipment and storage. A field reagent blank prepared from reagent water and carried through the sampling and handling protocol can serve as a check on such contamination.

3.3 Contamination by carry-over can occur whenever high level and low level samples are sequentially analyzed. To reduce carry-over, the purging device and sample syringe must be rinsed between samples with reagent water. Whenever an unusually concentrated sample is encountered, it should be followed by an analysis of reagent water to check for cross contamination. For samples containing large amounts of water-soluble materials, suspended solids, high boiling compounds or high analyte levels, it may be necessary to wash the purging device with a detergent solution, rinse it with distilled water, and then dry it in an oven at 105°C between analyses. The trap and other parts of the system are also subject to contamination, therefore, frequent bakeout and purging of the entire system may be required.

4. Safety

4.1 The toxicity or carcinogenicity of each reagent used in this method has not been precisely defined; however, each chemical compound should be treated as a potential health hazard. From this view point, exposure to these chemicals must be reduced to the lowest possible level by whatever means available. The laboratory is responsible for maintaining a current awareness file of OSHA regulations regarding the safe handling of the chemicals specified in this method. A reference file of material data handling sheets should also be made available to all personnel involved in the chemical analysis. Additional references to laboratory safety are available and have been identified[4,6] for the information of the analyst.

5. Apparatus and Materials

5.1 Sampling equipment, for discrete sampling.

5.1.1 Vial—25-mL capacity or larger, equipped with a screw cap with a hole in the center (Pierce #13075 or equivalent). Detergent wash, rinse with tap and distilled water, and dry at 105 °C before use.

5.1.2 Septum—Teflon-faced silicone (Pierce #12722 or equivalent). Detergent wash, rinse with tap and distilled water and dry at 105 °C for 1 h before use.

5.2 Purge and trap system—The purge and trap system consists of three separate pieces of equipment: a purging device, trap, and desorber. Several complete systems are now commercially available.

5.2.1 The purging device must be designed to accept 5-mL, samples with a water column at least 3 cm deep. The gaseous head space between the water column and the trap must have a total volume of less than 15 mL. The purge gas must pass through the water column as finely divided bubbles with a diameter of less than 3 mm at the origin. The purge gas must be introduced no more than 5 mm from the base of the water column. The purging device must be capable of being heated to 85 °C within 3.0 min after transfer of the sample to the purging device and being held at 85 ±2 °C during the purge cycle. The entire water column in the purging device must be heated. Design of this modification to the standard purging device is optional, however, use of a water bath is suggested.

5.2.1.1 Heating mantle—To be used to heat water bath.

5.2.1.2 Temperature controller—Equipped with thermocouple/sensor to accurately control water bath temperature to ±2 °C. The purging device illustrated in Figure 1 meets these design criteria.

5.2.2 The trap must be at least 25 cm long and have an inside diameter of at least 0.105 in. The trap must be packed to contain 1.0 cm of methyl silicone coated packing (Section 6.5.2) and 23 cm of 2,6-diphenylene oxide polymer (Section 6.5.1). The minimum specifications for the trap are illustrated in Figure 2.

5.2.3 The desorber must be capable of rapidly heating the trap to 180 °C, The desorber illustrated in Figure 2 meets these design criteria.

5.2.4 The purge and trap system may be assembled as a separate unit as illustrated in Figure 3 or be coupled to a gas chromatograph.

5.3 pH paper—Narrow pH range, about 3.5 to 5.5 (Fisher Scientific Short Range Alkacid No. 2, #14–837–2 or equivalent).

5.4 Gas chromatograph—An analytical system complete with a temperature programmable gas chromatograph suitable for on-column injection and all required accessories including syringes, analytical columns, gases, detector, and strip-chart recorder. A data system is recommended for measuring peak areas.

5.4.1 Column 1—10 ft long x 2 mm ID glass or stainless steel, packed with Porapak-QS (80/100 mesh) or equivalent. This column was used to develop the method performance statements in Section 12. Guidelines for the use of alternate column packings are provided in Section 10.1.

5.4.2 Column 2—6 ft long x 0.1 in. ID glass or stainless steel, packed with Chromosorb 101 (60/80 mesh) or equivalent.

5.4.3 Detector—Flame ionization detector. This type of detector has proven effective in the analysis of wastewaters for the parameters listed in the scope (Section 1.1), and was used to develop the method performance statements in Section 12. Guidelines for the use of alternate detectors are provided in Section 10.1.

5.5 Syringes—5-mL, glass hypodermic with Luerlok tip (two each).

5.6 Micro syringes—25-µL, 0.006 in. ID needle.

5.7 Syringe valve—2-way, with Luer ends (three each).

5.8 Bottle—15-mL, screw-cap, with Teflon cap liner.

5.9 Balance—Analytical, capable of accurately weighing 0.0001 g.

6. Reagents

6.1 Reagent water—Reagent water is defined as a water in which an interferent is not observed at the MDL of the parameters of interest.

6.1.1 Reagent water can be generated by passing tap water through a carbon filter bed containing about 1 lb of activated carbon (Filtrasorb-300, Calgon Corp., or equivalent).

6.1.2 A water purification system (Millipore Super-Q or equivalent) may be used to generate reagent water.

6.1.3 Regent water may also be prepared by boiling water for 15 min. Subsequently, while maintaining the temperature at 90 °C, bubble a contaminant-free inert gas through the water for 1 h. While still hot, transfer the water to a narrow mouth screw-cap bottle and seal with a Teflon-lined septum and cap.

6.2 Sodium thiosulfate—(ACS) Granular.

6.3 Sodium hydroxide solution (10 N)—Dissolve 40 g of NaOH (ACS) in reagent water and dilute to 100 mL.

6.4 Hydrochloric acid (1+1)—Slowly, add 50 mL of concentrated HCl (ACS) to 50 mL of reagent water.

6.5 Trap Materials:

6.5.1 2,6-Diphenylene oxide polymer—Tenax (60/80 mesh), chromatographic grade or equivalent.

6.5.2 Methyl silicone packing—3% OV–1 on Chromosorb–W (60/80 mesh) or equivalent.

6.6 Stock standard solutions—Stock standard solutions may be prepared from pure standard materials or purchased as certified solutions. Prepare stock standard solutions in reagent water using assayed liquids. Since acrolein and acrylonitrile are lachrymators, primary dilutions of these compounds should be prepared in a hood. A NIOSH/MESA approved toxic gas respirator should be used when the analyst handles high concentrations of such materials.

6.6.1 Place about 9.8 mL of reagent water into a 10-mL ground glass stoppered volumetric flask. For acrolein standards the reagent water must be adjusted to pH 4 to 5. Weight the flask to the nearest 0.1 mg.

6.6.2 Using a 100-µL syringe, immediately add two or more drops of assayed reference material to the flask, then reweigh. Be sure that the drops fall directly into the water without contacting the neck of the flask.

6.6.3 Reweigh, dilute to volume, stopper, then mix by inverting the flask several times. Calculate the concentration in µg/µL from the net gain in weight. When compound purity is assayed to be 96% or greater, the weight can be used without correction to calculate the concentration of the stock staldard. Optionally, stock standard solutions may be prepared using the pure standard material by volumetrically measuring the appropriate amounts and determining the weight of the material using the density of the material. Commercially prepared stock standards may be used at any concentration if they are certified by the manufactaurer or by an independent source.

6.6.4 Transfer the stock standard solution into a Teflon-sealed screw-cap bottle. Store at 4 °C and protect from light.

6.6.5 Prepare fresh standards daily.

6.7 Secondary dilution standards—Using stock standard solutions, prepare secondary dilution standards in reagent water that contain the compounds of interest, either singly or mixed together. The secondary dilution standards should be prepared at concentrations such that the aqueous calibration standards prepared in Section 7.3.1 or 7.4.1 will bracket the working range of the analytical system. Secondary dilution standards should be prepared daily and stored at 4 °C.

6.8 Quality control check sample concentrate—See Section 8.2.1.

7. Calibration

7.1 Assemble a purge and trap system that meets the specifications in Section 5.2. Condition the trap overnight at 180 °C by backflushing with an inert gas flow of at least 20 mL/min. Condition the trap for 10 min once daily prior to use.

7.2 Connect the purge and trap system to a gas chromatograph. The gas chromatograph must be operated using temperature and flow rate conditions equivalent to those given in Table 1. Calibrate the purge and trap-gas chromatographic system using either the external standard technique (Section 7.3) or the internal standard technique (Section 7.4).

7.3 External standard calibration procedure:

7.3.1 Prepare calibration standards at a minimum of three concentration levels for each parameter by carefully adding 20.0 µL of one or more secondary dilution standards to 100, 500, or 1000 mL of reagent water. A 25-µL syringe with a 0.006 in. ID needle should be used for this operation. One of the external standards should be at a concentration near, but above, the MDL and the other concentrations should correspond to the expected range of concentrations found in real samples or should define the working range of the detector. These standards must be prepared fresh daily.

7.3.2 Analyze each calibration standard according to Section 10, and tabulate peak height or area responses versus the concentration of the standard. The results can be used to prepare a calibration curve for each compound. Alternatively, if the ratio of response to concentration (calibration factor) is a constant over the working range (< 10% relative standard deviation, RSD), linearity through the origin can be assumed and the average ratio or calibration factor can be used in place of a calibration curve.

7.4 Internal standard calibration procedure—To use this approach, the analyst must select one or more internal standards that are similar in analytical behavior to the compounds of interest. The analyst must further demonstrate that the measurement of the internal standard is not affected by method or matrix interferences. Because of these limitations, no internal standard can be suggested that is applicable to all samples.

7.4.1 Prepare calibration standards at a minimum of three concentration levels for each parameter of interest as described in Section 7.3.1.

7.4.2 Prepare a spiking solution containing each of the internal standards using the procedures described in Sections 6.6 and 6.7. It is recommended that the secondary dilution standard be prepared at a concentration of 15 µg/mL of each internal standard compound. The addition of 10 µL of this standard to 5.0 mL of sample or calibration standard would be equivalent to 30 µg/L.

7.4.3 Analyze each calibration standard according to Section 10, adding 10 µL of internal standard spiking solution directly to the syringe (Section 10.4). Tabulate peak height or area responses against concentration for each compound and internal standard, and calculate response factors (RF) for each compound using Equation 1.

$$RF = \frac{(A_s)(C_{is})}{(A_{is})(C_s)}$$

Equation 1

where:

A_s=Response for the parameter to be measured.

A_{is}=Response for the internal standard.

C_{is}=Concentration of the internal standard.

C_s=Concentration of the parameter to be measured.

If the RF value over the working range is a constant (<10% RSD), the RF can be assumed to be invariant and the average RF can be used for calculations. Alternatively, the results can be used to plot a calibration curve of response ratios, A_s/A_{is}, vs. RF.

7.5 The working calibration curve, calibration factor, or RF must be verified on each working day by the measurement of a QC check sample.

7.5.1 Prepare the QC check sample as described in Section 8.2.2.

7.5.2 Analyze the QC check sample according to Section 10.

7.5.3 For each parameter, compare the response (Q) with the corresponding calibration acceptance criteria found in Table 2. If the responses for all parameters of interest fall within the designated ranges, analysis of actual samples can begin. If any individual Q falls outside the range, a new calibration curve, calibration factor, or RF must be prepared for that parameter according to Section 7.3 or 7.4.

8. Quality Control

8.1 Each laboratory that uses this method is required to operate a formal quality control program. The minimum requirements of this program consist of an initial demonstration of laboratory capability and an ongoing analysis of spiked samples to evaluate and document data quality. The laboratory must maintain records to document the quality of data that is generated. Ongoing data quality checks are compared with established performance criteria to determine if the results of analyses meet the performance characteristics of the method. When results of sample spikes indicate atypical method performance, a quality control check standard must be analyzed to confirm that the measurements were performed in an in-control mode of operation.

8.1.1 The analyst must make an initial, one-time, demonstration of the ability to generate acceptable accuracy and precision with this method. This ability is established as described in Section 8.2.

8.1.2 In recognition of advances that are occurring in chromatography, the analyst is permitted certain options (detailed in Section 10.1) to improve the separations or lower the cost of measurements. Each time such a modification is made to the method, the analyst is required to repeat the procedure in Section 8.2.

8.1.3 Each day, the analyst must analyze a reagent water blank to demonstrate that interferences from the analytical system are under control.

8.1.4 The laboratory must, on an ongoing basis, spike and analyze a minimum of 10% of all samples to monitor and evaluate laboratory data quality. This procedure is described in Section 8.3.

8.1.5 The laboratory must, on an ongoing basis, demonstrate through the analyses of quality control check standards that the operation of the measurement system is in control. This procedure is described in Section 8.4. The frequency of the check standard analyses is equivalent to 10% of all samples analyzed but may be reduced if spike recoveries from samples (Section 8.3) meet all specified quality control criteria.

8.1.6 The laboratory must maintain performance records to document the quality of data that is generated. This procedure is described in Section 8.5.

8.2 To establish the ability to generate acceptable accuracy and precision, the analyst must perform the following operations.

8.2.1 A quality control (QC) check sample concentrate is required containing each parameter of interest at a concentration of 25 µg/mL in reagent water. The QC check sample concentrate must be obtained from the U.S. Environmental Protection Agency, Environmental Monitoring and Support Laboratory in Cincinnati, Ohio, if available. If not available from that source, the QC check sample concentrate must be obtained from another external source. If not available from either source above, the QC check sample concentrate must be prepared by the laboratory using stock standards prepared independently from those used for calibration.

8.2.2 Prepare a QC check sample to contain 50 µg/L of each parameter by adding 200 µL of QC check sample concentrate to 100 mL of reagent water.

8.2.3 Analyze four 5-mL aliquots of the well-mixed QC check sample according to Section 10.

8.2.4 Calculate the average recovery ($\bar{X}$) in µg/L, and the standard deviation of the recovery (s) in µg/L, for each parameter using the four results.

8.2.5 For each parameter compare s and $\bar{X}$ with the corresponding acceptance criteria for precision and accuracy, respectively, found in Table 3. If s and $\bar{X}$ for all parameters of interest meet the acceptance criteria, the system performance is acceptable and analysis of actual samples can begin. If either s exceeds the precision limit or $\bar{X}$ falls outside the range for accuracy, the system performance is unacceptable for that parameter. Locate and correct the source of the

problem and repeat the test for each compound of interest.

8.3 The laboratory must, on an ongoing basis, spike at least 10% of the samples from each sample site being monitored to assess accuracy. For laboratories analyzing one to ten samples per month, at least one spiked sample per month is required.

8.3.1 The concentration of the spike in the sample should be determined as follows:

8.3.1.1 If, as in compliance monitoring, the concentration of a specific parameter in the sample is being checked against a regulatory concentration limit, the spike should be at that limit or 1 to 5 times higher than the background concentration determined in Section 8.3.2, whichever concentration would be larger.

8.3.1.2 If the concentration of a specific parameter in the sample is not being checked against a limit specific to that parameter, the spike should be at 50 µg/L or 1 to 5 times higher than the background concentration determined in Section 8.3.2, whichever concentration would be larger.

8.3.2 Analyze one 5-mL sample aliquot to determine the background concentration (B) of each parameter. If necessary, prepare a new QC check sample concentrate (Section 8.2.1) appropriate for the background concentrations in the sample. Spike a second 5-mL sample aliquot with 10 µL of the QC check sample concentrate and analyze it to determine the concentration after spiking (A) of each parameter. Calculate each percent recovery (P) as 100(A–B)%/T, where T is the known true value of the spike.

8.3.3 Compare the percent recovery (P) for each parameter with the corresponding QC acceptance criteria found in Table 3. These acceptance criteria were calculated to include an allowance for error in measurement of both the background and spike concentrations, assuming a spike to background ratio of 5:1. This error will be accounted for to the extent that the analyst's spike to background ratio approaches 5:1.[7]

8.3.4 If any individual P falls outside the designated range for recovery, that parameter has failed the acceptance criteria. A check standard containing each parameter that failed the criteria must be analyzed as described in Section 8.4.

8.4 If any parameter fails the acceptance criteria for recovery in Section 8.3, a QC check standard containing each parameter that failed must be prepared and analyzed.

NOTE: The frequency for the required analysis of a QC check standard will depend upon the number of parameters being simultaneously tested, the complexity of the sample matrix, and the performance of the laboratory.

8.4.1 Prepare the QC check standard by adding 10 µL of QC check sample concentrate (Section 8.2.1 or 8.3.2) to 5 mL of reagent water. The QC check standard needs only to contain the parameters that failed criteria in the test in Section 8.3.

8.4.2 Analyze the QC check standard to determine the concentration measured (A) of each parameter. Calculate each percent recovery (P_s) as 100 (A/T)%, where T is the true value of the standard concentration.

8.4.3 Compare the percent recovery (P_s) for each parameter with the corresponding QC acceptance criteria found in Table 3. Only parameters that failed the test in Section 8.3 need to be compared with these criteria. If the recovery of any such parameter falls outside the designated range, the laboratory performance for that parameter is judged to be out of control, and the problem must be immediately identified and corrected. The analytical result for that parameter in the unspiked sample is suspect and may not be reported for regulatory compliance purposes.

8.5 As part of the QC program for the laboratory, method accuracy for wastewater samples must be assessed and records must be maintained. After the analysis of five spiked wastewater samples as in Section 8.3, calculate the average percent recovery ($\bar{P}$) and the standard deviation of the percent recovery (s_p). Express the accuracy assessment as a percent recovery interval from $\bar{P}-2s_p$ to $\bar{P}+2s_p$. If $\bar{P}$=90% and s_p=10%, for example, the accuracy interval is expressed as 70–110%. Update the accuracy assessment for each parameter on a regular basis (e.g. after each five to ten new accuracy measurements).

8.6 It is recommended that the laboratory adopt additional quality assurance practices for use with this method. The specific practices that are most productive depend upon the needs of the laboratory and the nature of the samples. Field duplicates may be analyzed to assess the precision of the environmental measurements. When doubt exists over the identification of a peak on the chromatogram, confirmatory techniques such as gas chromatography with a dissimilar column or mass spectrometer must be used. Whenever possible, the laboratory should analyze standard reference materials and participate in relevant performance evaluation studies.

9. Sample Collection, Preservation, and Handling

9.1 All samples must be iced or refrigerated from the time of collection until analysis. If the sample contains free or combined chlorine, add sodium thiosulfate preservative (10 mg/40 mL is sufficient for up to 5 ppm Cl_2) to the empty sample bottle just prior to shipping to the sampling site. EPA Methods 330.4 and 330.5 may be used for measurement of residual chlorine.[8] Field test kits are available for this purpose.

9.2 If acrolein is to be analyzed, collect about 500 mL of sample in a clean glass container. Adjust the pH of the sample to 4 to 5

using acid or base, measuring with narrow range pH paper. Samples for acrolein analysis receiving no pH adjustment must be analyzed within 3 days of sampling.

9.3 Grab samples must be collected in glass containers having a total volume of at least 25 mL. Fill the sample bottle just to overflowing in such a manner that no air bubbles pass through the sample as the bottle is being filled. Seal the bottle so that no air bubbles are entrapped in it. If preservative has been added, shake vigorously for 1 min. Maintain the hermetic seal on the sample bottle until time of analysis.

9.4 All samples must be analyzed within 14 days of collection.[3]

10. Procedure

10.1 Table 1 summarizes the recommended operating conditions for the gas chromatograph. Included in this table are estimated retention times and MDL that can be achieved under these conditions. An example of the separations achieved by Column 1 is shown in Figure 5. Other packed columns, chromatographic conditions, or detectors may be used if the requirements of Section 8.2 are met.

10.2 Calibrate the system daily as described in Section 7.

10.3 Adjust the purge gas (nitrogen or helium) flow rate to 20 mL-min. Attach the trap inlet to the purging device, and set the purge and trap system to purge (Figure 3). Open the syringe valve located on the purging device sample introduction needle.

10.4 Remove the plunger from a 5-mL syringe and attach a closed syringe valve. Open the sample bottle (or standard) and carefully pour the sample into the syringe barrel to just short of overflowing. Replace the syringe plunger and compress the sample. Open the syringe valve and vent any residual air while adjusting the sample volume to 5.0 mL. Since this process of taking an aliquot destroys the validity of the sample for future analysis, the analyst should fill a second syringe at this time to protect against possible loss of data. Add 10.0 µL of the internal standard spiking solution (Section 7.4.2), if applicable, through the valve bore then close the valve.

10.5 Attach the syringe-syringe valve assembly to the syringe valve on the purging device. Open the syringe valves and inject the sample into the purging chamber.

10.6 Close both valves and purge the sample for 15.0 ± 0.1 min while heating at 85 ± 2 °C.

10.7 After the 15-min purge time, attach the trap to the chromatograph, adjust the purge and trap system to the desorb mode (Figure 4), and begin to temperature program the gas chromatograph. Introduce the trapped materials to the GC column by rapidly heating the trap to 180 °C while backflushing the trap with an inert gas between 20 and 60 mL/min for 1.5 min.

10.8 While the trap is being desorbed into the gas chromatograph, empty the purging chamber using the sample introduction syringe. Wash the chamber with two 5-mL flushes of reagent water.

10.9 After desorbing the sample for 1.5 min, recondition the trap by returning the purge and trap system to the purge mode. Wait 15 s then close the syringe valve on the purging device to begin gas flow through the trap. The trap temperature should be maintained at 210 °C. After approximately 7 min, turn off the trap heater and open the syringe valve to stop the gas flow through the trap. When the trap is cool, the next sample can be analyzed.

10.10 Identify the parameters in the sample by comparing the retention times of the peaks in the sample chromatogram with those of the peaks in standard chromatograms. The width of the retention time window used to make identifications should be based upon measurements of actual retention time variations of standards over the course of a day. Three times the standard deviation of a retention time for a compound can be used to calculate a suggested window size; however, the experience of the analyst should weigh heavily in the interpretation of chromatograms.

11. Calculations

11.1 Determine the concentration of individual compounds in the sample.

11.1.1 If the external standard calibration procedure is used, calculate the concentration of the parameter being measured from the peak response using the calibration curve or calibration factor determined in Section 7.3.2.

11.1.2 If the internal standard calibration procedure is used, calculate the concentration in the sample using the response factor (RF) determined in Section 7.4.3 and Equation 2.

$$\text{Concentration } (\mu g/L) = \frac{(A_s)(C_{is})}{(A_{is})(RF)}$$

Equation 2

where:

A_s=Response for the parameter to be measured.

A_{is}=Response for the internal standard.

C_{is}=Concentration of the internal standard.

11.2 Report results in µg/L without correction for recovery data. All QC data obtained should be reported with the sample results.

12. Method Performance

12.1 The method detection limit (MDL) is defined as the minimum concentration of a substance that can be measured and reported

with 99% confidence that the value is above zero.[1] The MDL concentrations listed in Table 1 were obtained using reagent water.[9] The MDL actually achieved in a given analysis will vary depending on instrument sensitivity and matrix effects.

12.2 This method is recommended for the concentration range from the MDL to 1,000×MDL. Direct aqueous injection techniques should be used to measure concentration levels above 1,000×MDL.

12.3 In a single laboratory (Battelle-Columbus), the average recoveries and standard deviations presented in Table 2 were obtained.[9] Seven replicate samples were analyzed at each spike level.

References

1. 40 CFR part 136, appendix B.

2. Bellar, T.A., and Lichtenberg, J.J. ''Determining Volatile Organics at Microgram-per-Litre-Levels by Gas Chromatography,'' *Journal American Water Works Association, 66,* 739 (1974).

3. ''Evaluate Test Procedures for Acrolein and Acrylonitrile,'' Special letter report for EPA Project 4719–A, U.S. Environmental Protection Agency, Environmental Monitoring and Support Laboratory, Cincinnati, Ohio 45268, 27 June 1979.

4. ''Carcinogens—Working With Carcinogens,'' Department of Health, Education, and Welfare, Public Health Service, Center for Disease Control, National Institute for Occupational Safety and Health, Publication No. 77–206, August 1977.

5. ''OSHA Safety and Health Standards, General Industry,'' (29 CFR part 1910), Occupational Safety and Health Administration, OSHA 2206 (Revised, January 1976).

6. ''Safety in Academic Chemistry Laboratories,'' American Chemical Society Publication, Committee on Chemical Safety, 3rd Edition, 1979.

7. Provost, L.P., and Elder, R.S. ''Interpretation of Percent Recovery Data,'' *American Laboratory, 15,* 58–63 (1983).

8. ''Methods 330.4 (Titrimetric, DPD–FAS) and 330.5 (Spectrophotometric, DPD) for Chlorine, Total Residual,'' Methods for Chemical Analysis of Water and Wastes, EPA–600/4–79–020, U.S. Environmental Protection Agency, Environmental Monitoring and Support Laboratory, Cincinnati, Ohio 45268, March 1979.

9. ''Evaluation of Method 603 (Modified),'' EPA–600/4–84–ABC, National Technical Information Service, PB84–, Springfield, Virginia 22161, Nov. 1984.

TABLE 1—CHROMATOGRAPHIC CONDITIONS AND METHOD DETECTION LIMITS

Parameter	Retention time (min)		Method detection limit (µg/L)
	Column 1	Column 2	
Acrolein	10.6	8.2	0.7
Acrylonitrile	12.7	9.8	0.5

Column 1 conditions: Porapak-QS (80/100 mesh) packed in a 10 ft × 2 mm ID glass or stainless steel column with helium carrier gas at 30 mL/min flow rate. Column temperature held isothermal at 110 °C for 1.5 min (during desorption), then heated as rapidly as possible to 150 °C and held for 20 min; column bakeout at 190 °C for 10 min.[9]

Column 2 conditions: Chromosorb 101 (60/80 mesh) packed in a 6 ft. x 0.1 in. ID glass or stainless steel column with helium carrier gas at 40 mL/min flow rate. Column temperature held isothermal at 80°C for 4 min, then programmed at 50°C/min to 120°C and held for 12 min.

TABLE 2—SINGLE LABORATORY ACCURACY AND PRECISION—METHOD 603

Parameter	Sample matrix	Spike conc. (µg/L)	Average recovery (µg/L)	Standard deviation (µg/L)	Average percent recovery
Acrolein	RW	5.0	5.2	0.2	104
	RW	50.0	51.4	0.7	103
	POTW	5.0	4.0	0.2	80
	POTW	50.0	44.4	0.8	89
	IW	5.0	0.1	0.1	2
	IW	100.0	9.3	1.1	9
Acrylonitrile	RW	5.0	4.2	0.2	84
	RW	50.0	51.4	1.5	103
	POTW	20.0	20.1	0.8	100
	POTW	100.0	101.3	1.5	101
	IW	10.0	9.1	0.8	91
	IW	100.0	104.0	3.2	104

ARW=Reagent water.
APOTW=Prechlorination secondary effluent from a municipal sewage treatment plant.
AIW=Industrial wastewater containing an unidentified acrolein reactant.

TABLE 3—CALIBRATION AND QC ACCEPTANCE CRITERIA—METHOD 603 [a]

Parameter	Range for Q (μg/L)	Limit for S (μg/L)	Range for X (μg/L)	Range for P, P_s (%)
Acrolein	45.9–54.1	4.6	42.9–60.1	88–118
Acrylonitrile	41.2–58.8	9.9	33.1–69.9	71–135

[a]=Criteria were calculated assuming a QC check sample concentration of 50 □g/L.[9]

Q=Concentration measured in QC check sample, in μg/L (Section 7.5.3).

s=Standard deviation of four recovery measurements, in μg/L (Section 8.2.4).

X=Average recovery for four recovery measurements, in μg/L (Section 8.2.4).

P, P_s=Percent recovery measured (Section 8.3.2, Section 8.4.2).

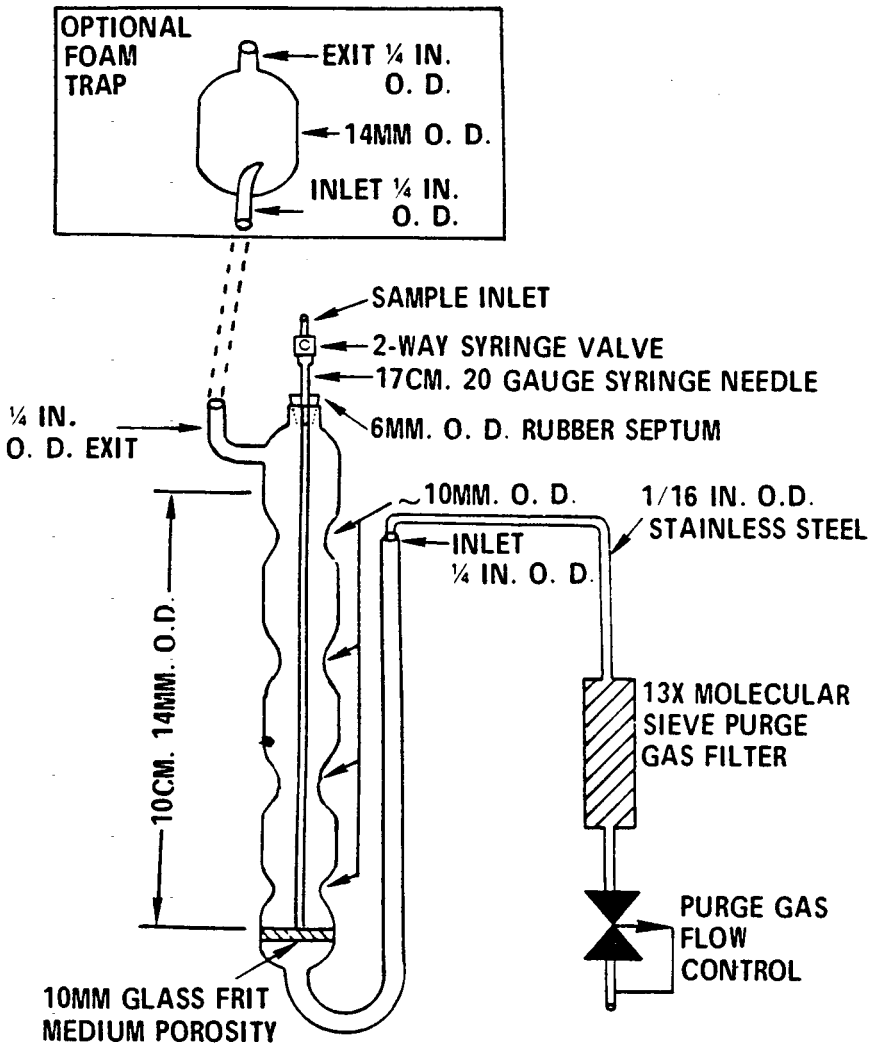


Figure 1. Purging device.

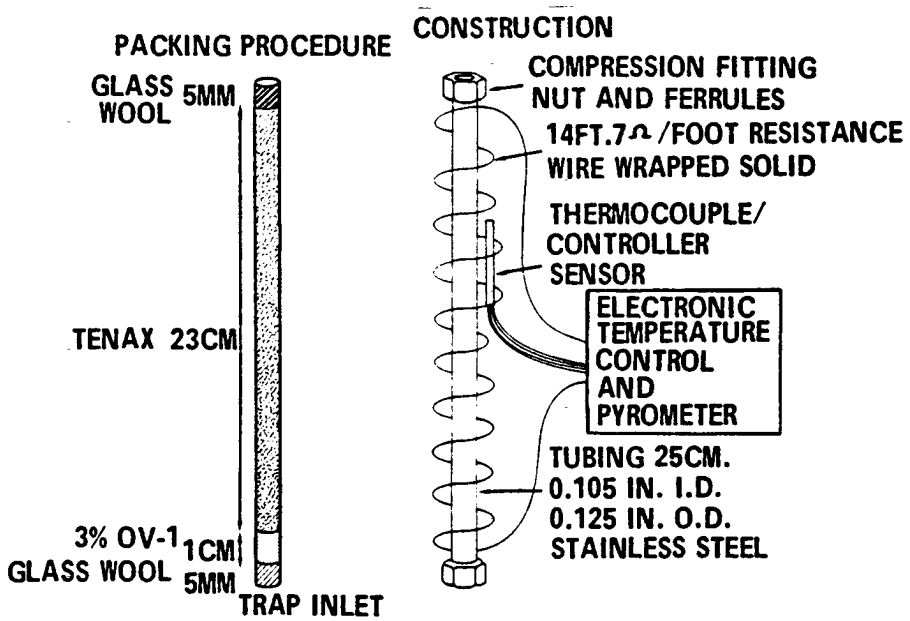


Figure 2. Trap packings and construction to include desorb capability.

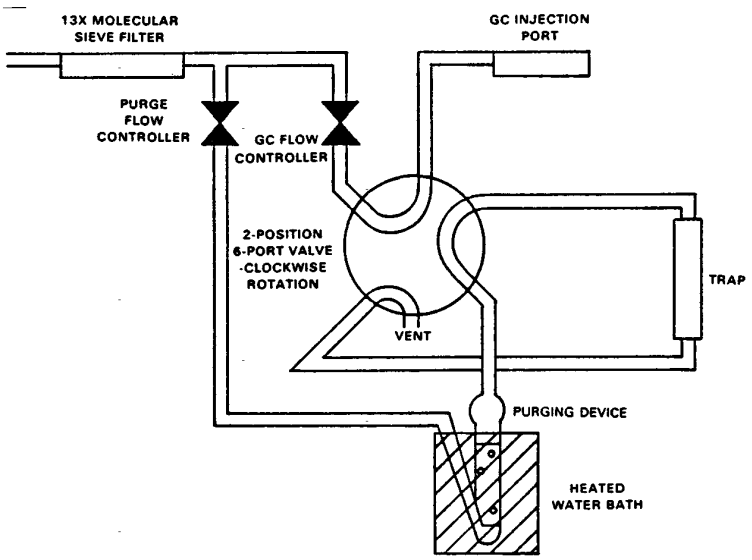


Figure 3. Purge and trap system-purge mode.

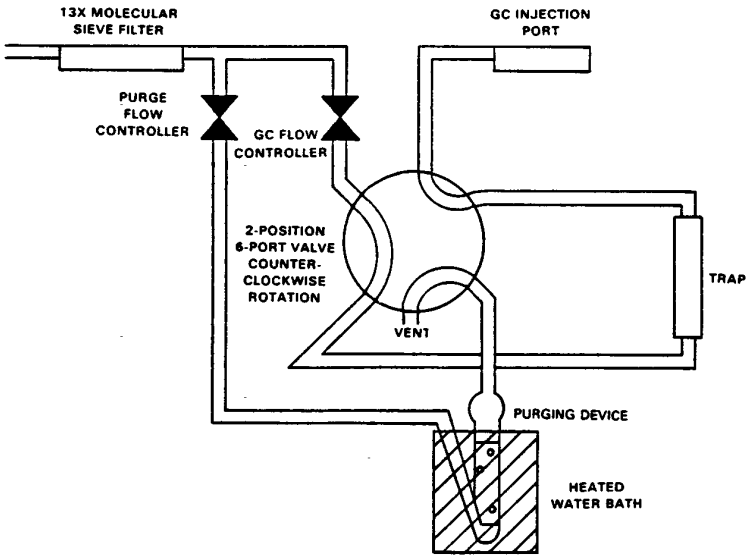


Figure 4. Purge and trap system-desorb mode.

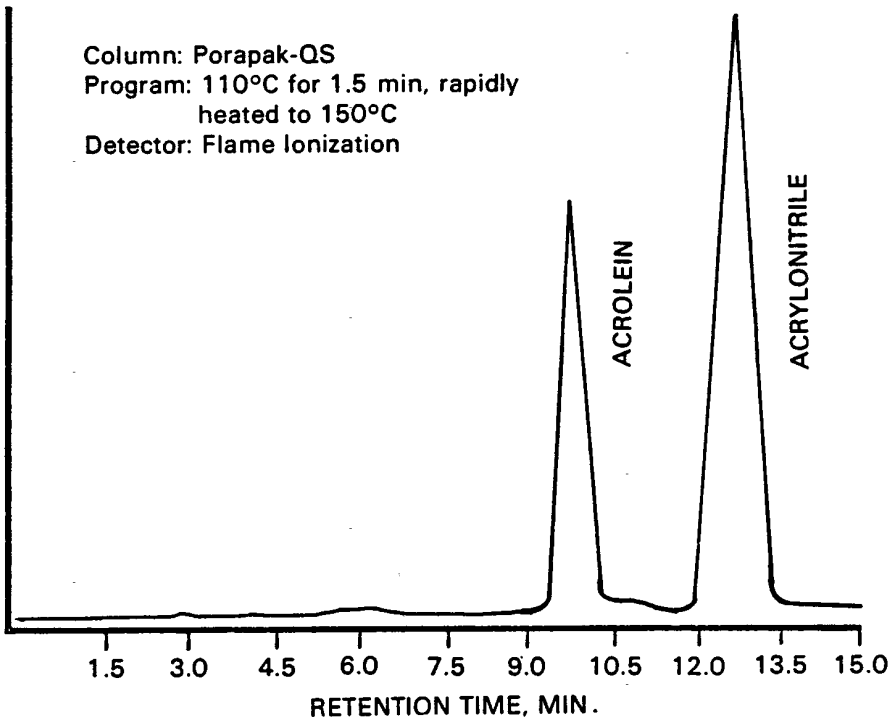


Figure 5. Gas chromatogram of acrolein and acrylonitrile.

METHOD 604—PHENOLS

1. Scope and Application

1.1 This method covers the determination of phenol and certain substituted phenols. The following parameters may be determined by this method:

Parameter	STORET No.	CAS No.
4-Chloro-3-methylphenol	34452	59–50–7
2–-Chlorophenol	34586	95–57–8
2,4-Dichlorophenol	34601	120–83–2
2,4-Dimethylphenol	34606	105–67–9
2,4-Dinitrophenol	34616	51–28–5
2-Methyl-4,6-dinitrophenol	34657	534–52–1
2-Nitrophenol	34591	88–75–5
4-Nitrophenol	34646	100–02–7
Pentachlorophenol	39032	87–86–5
Phenol	34694	108–95–2
2,4,6-Trichlorophenol	34621	88–06–2

1.2 This is a flame ionization detector gas chromatographic (FIDGC) method applicable to the determination of the compounds listed above in municipal and industrial discharges as provided under 40 CFR 136.1. When this method is used to analyze unfamiliar samples for any or all of the compounds above, compound identifications should be supported by at least one additional qualitative technique. This method describes analytical conditions for derivatization, cleanup, and electron capture detector gas chromatography (ECDGC) that can be used to confirm measurements made by FIDGC. Method 625 provides gas chromatograph/mass spectrometer (GC/MS) conditions appropriate for the qualitative and quantitative confirmation of results for all of the parameters listed above, using the extract produced by this method.

1.3 The method detection limit (MDL, defined in Section 14.1)[1] for each parameter is listed in Table 1. The MDL for a specific wastewater may differ from those listed, depending upon the nature of interferences in the sample matrix. The MDL listed in Table 1 for each parameter was achieved with a flame ionization detector (FID). The MDLs that were achieved when the derivatization cleanup and electron capture detector (ECD) were employed are presented in Table 2.

1.4 Any modification of this method, beyond those expressly permitted, shall be considered as a major modification subject to application and approval of alternate test procedures under 40 CFR 136.4 and 136.5.

1.5 This method is restricted to use by or under the supervision of analysts experienced in the use of a gas chromatograph and in the interpretation of gas chromatograms. Each analyst must demonstrate the ability to generate acceptable results with this method using the procedure described in Section 8.2.

2. Summary of Method

2.1 A measured volume of sample, approximately 1-L, is acidified and extracted with methylene chloride using a separatory funnel. The methylene chloride extract is dried and exchanged to 2-propanol during concentration to a volume of 10 mL or less. The extract is separated by gas chromatography and the phenols are then measured with an FID.[2]

2.2 A preliminary sample wash under basic conditions can be employed for samples having high general organic and organic base interferences.

2.3 The method also provides for a derivatization and column chromatography cleanup procedure to aid in the elimination of interferences.[2,3] The derivatives are analyzed by ECDGC.

3. Interferences

3.1 Method interferences may be caused by contaminants in solvents, reagents, glassware, and other sample processing hardware that lead to discrete artifacts and/or elevated baselines in gas chromatograms. All of these materials must be routinely demonstrated to be free from interferences under the conditions of the analysis by running laboratory reagent blanks as described in Section 8.1.3.

3.1.1 Glassware must be scrupulously cleaned.[4] Clean all glassware as soon as possible after use by rinsing with the last solvent used in it. Solvent rinsing should be followed by detergent washing with hot water, and rinses with tap water and distilled water. The glassware should then be drained dry, and heated in a muffle furnace at 400 °C for 15 to 30 min. Some thermally stable materials, such as PCBs, may not be eliminated by this treatment. Solvent rinses with acetone and pesticide quality hexane may be substituted for the muffle furnace heating. Thorough rinsing with such solvents usually eliminates PCB interference. Volumetric ware should not be heated in a muffle furnace. After drying and cooling, glassware should be sealed and stored in a clean environment to prevent any accumulation of dust or other contaminants. Store inverted or capped with aluminum foil.

3.1.2 The use of high purity reagents and solvents helps to minimize interference problems. Purification of solvents by distillation in all-glass systems may be required.

3.2 Matrix interferences may be caused by contaminants that are coextracted from the sample. The extent of matrix interferences will vary considerably from source to source, depending upon the nature and diversity of the industrial complex or municipality being sampled. The derivatization cleanup procedure in Section 12 can be used to overcome

many of these interferences, but unique samples may require additional cleanup approaches to achieve the MDL listed in Tables 1 and 2.

3.3 The basic sample wash (Section 10.2) may cause significantly reduced recovery of phenol and 2,4-dimethylphenol. The analyst must recognize that results obtained under these conditions are minimum concentrations.

4. Safety

4.1 The toxicity or carcinogenicity of each reagent used in this mothod has not been precisely defined; however, each chemical compound should be treated as a potential health hazard. From this viewpoint, exposure to these chemicals must be reduced to the lowest possible level by whatever means available. The laboratory is responsible for maintaining a current awareness file of OSHA regulations regarding the safe handling of the chemicals specified in this method. A reference file of material data handling sheets should also be made available to all personnel involved in the chemical analysis. Additional references to laboratory safety are available and have been identified[5—7] for the information of analyst.

4.2 Special care should be taken in handling pentafluorobenzyl bromide, which is a lachrymator, and 18-crown-6-ether, which is highly toxic.

5. Apparatus and Materials

5.1 Sampling equipment, for discrete or composite sampling.

5.1.1 Grab sample bottle—1–L or 1-qt, amber glass, fitted with a screw cap lined with Teflon. Foil may be substituted for Teflon if the sample is not corrosive. If amber bottles are not available, protect samples from light. The bottle and cap liner must be washed, rinsed with acetone or methylene chloride, and dried before use to minimize contamination.

5.1.2 Automatic sampler (optional)—The sampler must incorporate glass sample containers for the collection of a minimum of 250 mL of sample. Sample containers must be kept refrigerated at 4°C and protected from light during compositing. If the sampler uses a peristaltic pump, a minimum length of compressible silicone rubber tubing may be used. Before use, however, the compressible tubing should be thoroughly rinsed with methanol, followed by repeated rinsings with distilled water to minimize the potential for contamination of the sample. An integrating flow meter is required to collect flow proportional composites.

5.2 Glassware (All specifications are suggested. Catalog numbers are included for illustration only.):

5.2.1 Separatory funnel—2–L, with Teflon stopcock.

5.2.2 Drying column—Chromatographic column, 400 mm long x 19 mm ID, with coarse frit filter disc.

5.2.3 Chromatographic column—100 mm long x 10 mm ID, with Teflon stopcock.

5.2.4 Concentrator tube, Kuderna-Danish—10-mL, graduated (Kontes K–570050–1025 or equivalent). Calibration must be checked at the volumes employed in the test. Ground glass stopper is used to prevent evaporation of extracts.

5.2.5 Evaporative flask, Kuderna-Danish—500-mL (Kontes K–570001–0500 or equivalent). Attach to concentrator tube with springs.

5.2.6 Snyder column, Kuderna-Danish—Three-ball macro (Kontes K–503000–0121 or equivalent).

5.2.7 Snyder column, Kuderna-Danish—Two-ball micro (Kontes K–569001–0219 or equivalent).

5.2.8 Vials—10 to 15-mL, amber glass, with Teflon-lined screw cap.

5.2.9 Reaction flask—15 to 25-mL round bottom flask, with standard tapered joint, fitted with a water-cooled condenser and U-shaped drying tube containing granular calcium chloride.

5.3 Boiling chips—Approximately 10/40 mesh. Heat to 400 °C for 30 min or Soxhlet extract with methylene chloride.

5.4 Water bath—Heated, with concentric ring cover, capable of temperature control (±2°C). The bath should be used in a hood.

5.5 Balance—Analytical, capable of accurately weighting 0.0001 g.

5.6 Gas chromatograph—An analytical system complete with a temperature programmable gas chromatograph suitable for on-column injection and all required accessories including syringes, analytical columns, gases, detector, and strip-chart recorder. A data system is recommended for measuring peak areas.

5.6.1 Column for underivatized phenols—1.8 m long x 2 mm ID glass, packed with 1% SP–1240DA on Supelcoport (80/100 mesh) or equivalent. This column was used to develop the method performance statements in Section 14. Guidelines for the use of alternate column packings are provided in Section 11.1.

5.6.2 Column for derivatized phenols—1.8 m long x 2 mm ID glass, packed with 5% OV–17 on Chromosorb W–AW–DMCS (80/100 mesh) or equivalent. This column has proven effective in the analysis of wastewaters for derivatization products of the parameters listed in the scope (Section 1.1), and was used to develop the method performance statements in Section 14. Guidelines for the use of alternate column packings are provided in Section 11.1.

5.6.3 Detectors—Flame ionization and electron capture detectors. The FID is used when determining the parent phenols. The ECD is used when determining the derivatized phenols. Guidelines for the use of

alternatve detectors are provided in Section 11.1.

6. Reagents

6.1 Reagent water—Reagent water is defined as a water in which an interferent is not observed at the MDL of the parameters of interest.

6.2 Sodium hydroxide solution (10 N)—Dissolve 40 g of NaOH (ACS) in reagent water and dilute to 100 mL.

6.3 Sodium hydroxide solution (1 N)—Dissolve 4 g of NaOH (ACS) in reagent water and dilute to 100 mL.

6.4 Sodium sulfate—(ACS) Granular, anhydrous. Purify by heating at 400°C for 4 h in a shallow tray.

6.5 Sodium thiosulfate—(ACS) Granular.

6.6 Sulfuric acid (1+1)—Slowly, add 50 mL of H_2SO_4 (ACS, sp. gr. 1.84) to 50 mL of reagent water.

6.7 Sulfuric acid (1 N)—Slowly, add 58 mL of H_2SO_4 (ACS, sp. gr. 1.84) to reagent water and dilute to 1 L.

6.8 Potassium carbonate—(ACS) Powdered.

6.9 Pentafluorobenzyl bromide (α-Bromopentafluorotoluene)—97% minimum purity.

NOTE: This chemical is a lachrymator. (See Section 4.2.)

6.10 18-crown-6-ether (1,4,7,10,13,16-Hexaoxacyclooctadecane)—98% minimum purity.

NOTE: This chemical is highly toxic.

6.11 Derivatization reagent—Add 1 mL of pentafluorobenzyl bromide and 1 g of 18-crown-6-ether to a 50-mL volumetric flask and dilute to volume with 2-propanol. Prepare fresh weekly. This operation should be carried out in a hood. Store at 4 °C and protect from light.

6.12 Acetone, hexane, methanol, methylene chloride, 2-propanol, toluene—Pesticide quality or equivalent.

6.13 Silica gel—100/200 mesh, Davison, grade-923 or equivalent. Activate at 130 °C overnight and store in a desiccator.

6.14 Stock standard solutions (1.00 µg/µL)—Stock standard solutions may be prepared from pure standard materials or purchased as certified solutions.

6.14.1 Prepare stock standard solutions by accurately weighing about 0.0100 g of pure material. Dissolve the material in 2-propanol and dilute to volume in a 10-mL volumetric flask. Larger volumes can be used at the convenience of the analyst. When compound purity is assayed to be 96% or greater, the weight can be used without correction to calculate the concentration of the stock standard. Commercially prepared stock standards can be used at any concentration if they are certified by the manufacturer or by an independent source.

6.14.2 Transfer the stock standard solutions into Teflon-sealed screw-cap bottles. Store at 4 °C and protect from light. Stock standard solutions should be checked frequently for signs of degradation or evaporation, especially just prior to preparing calibration standards from them.

6.14.3 Stock standard solutions must be replaced after six months, or sooner if comparison with check standards indicates a problem.

6.15 Quality control check sample concentrate—See Section 8.2.1.

7. Calibration

7.1 To calibrate the FIDGC for the anaylsis of underivatized phenols, establish gas chromatographic operating conditions equivalent to those given in Table 1. The gas chromatographic system can be calibrated using the external standard technique (Section 7.2) or the internal standard technique (Section 7.3).

7.2 External standard calibration procedure for FIDGC:

7.2.1 Prepare calibration standards at a minimum of three concentration levels for each parameter of interest by adding volumes of one or more stock standards to a volumetric flask and diluting to volume with 2-propanol. One of the external standards should be at a concentration near, but above, the MDL (Table 1) and the other concentrations should correspond to the expected range of concentrations found in real samples or should define the working range of the detector.

7.2.2 Using injections of 2 to 5 µl, analyze each calibration standard according to Section 11 and tabulate peak height or area responses against the mass injected. The results can be used to prepare a calibration curve for each compound. Alternatively, if the ratio of response to amount injected (calibration factor) is a constant over the working range (<10% relative standard deviation, RSD), linearity through the origin can be assumed and the average ratio or calibration factor can be used in place of a calibration curve.

7.3 Internal standard calibration procedure for FIDGC—To use this approach, the analyst must select one or more internal standards that are similar in analytical behavior to the compounds of interest. The analyst must further demonstrate that the measurement of the internal standard is not affected by method or matrix interferences. Because of these limitations, no internal standard can be suggested that is applicable to all samples.

7.3.1 Prepare calibration standards at a minimum of three concentration levels for each parameter of interest by adding volumes of one or more stock standards to a volumetric flask. To each calibration standard, add a known constant amount of one or more internal standards, and dilute to volume with 2-propanol. One of the standards

should be at a concentration near, but above, the MDL and the other concentrations should correspond to the expected range of concentrations found in real samples or should define the working range of the detector.

7.3.2 Using injections of 2 to 5 µL, analyze each calibration standard according to Section 11 and tabulate peak height or area responses against concentration for each compound and internal standard. Calculate response factors (RF) for each compound using Equation 1.

$$RF = \quad (A_s)(C_{is}) \quad (A_{is})(C_s)$$

Equation 1

where:

A_s=Response for the parameter to be measured.

A_{is}=Response for the internal standard.

C_{is}=Concentration of the internal standard (µg/L).

C_s=Concentration of the parameter to be measured (µg/L).

If the RF value over the working range is a constant (<10% RSD), the RF can be assumed to be invariant and the average RF can be used for calculations. Alternatively, the results can be used to plot a calibration curve of response ratios, A_s/A_{is}, vs. RF.

7.4 The working calibration curve, calibration factor, or RF must be verified on each working day by the measurement of one or more calibration standards. If the response for any parameter varies from the predicted response by more than ±15%, a new calibration curve must be prepared for that compound.

7.5 To calibrate the ECDGC for the analysis of phenol derivatives, establish gas chromatographic operating conditions equivalent to those given in Table 2.

7.5.1 Prepare calibration standards at a minimum of three concentration levels for each parameter of interest by adding volumes of one or more stock standards to a volumetric flask and diluting to volume with 2-propanol. One of the external standards should be at a concentration near, but above, the MDL (Table 2) and the other concentrations should correspond to the expected range of concentrations found in real samples or should define the working range of the detector.

7.5.2 Each time samples are to be derivatized, simultaneously treat a 1-mL aliquot of each calibration standard as described in Section 12.

7.5.3 After derivatization, analyze 2 to 5 µL of each column eluate collected according to the method beginning in Section 12.8 and tabulate peak height or area responses against the calculated equivalent mass of underivatized phenol injected. The results can be used to prepare a calibration curve for each compound.

7.6 Before using any cleanup procedure, the analyst must process a series of calibration standards through the procedure to validate elution patterns and the absence of interferences from the reagents.

8. Quality Control

8.1 Each laboratory that uses this method is required to operate a formal quality control program. The minimum requirements of this program consist of an initial demonstration of laboratory capability and an ongoing analysis of spiked samples to evaluate and document data quality. The laboratory must maintain records to document the quality of data that is generated. Ongoing data quality checks are compared with established performance criteria to determine if the results of analyses meet the performance characteristics of the method. When results of sample spikes indicate atypical method performance, a quality control check standard must be analyzed to confirm that the measurements were performed in an in-control mode of operation.

8.1.1 The analyst must make an initial, one-time, demonstration of the ability to generate acceptable accuracy and precision with this method. This ability is established as described in Section 8.2.

8.1.2 In recognition of advances that are occurring in chromatography, the analyst is permitted certain options (detailed in Sections 10.6 and 11.1) to improve the separations or lower the cost of measurements. Each time such a modification is made to the method, the analyst is required to repeat the procedure in Section 8.2.

8.1.3 Before processing any samples the analyst must analyze a reagent water blank to demonstrate that interferences from the analytical system and glassware are under control. Each time a set of samples is extracted or reagents are changed a reagent water blank must be processed as a safeguard against laboratory contamination.

8.1.4 The laboratory must, on an ongoing basis, spike and analyze a minimum of 10% of all samples to monitor and evaluate laboratory data quality. This procedure is described in Section 8.3.

8.1.5 The laboratory must, on an ongoing basis, demonstrate through the analyses of quality control check standards that the operation of the measurement system is in control. This procedure is described in Section 8.4. The frequency of the check standard analyses is equivalent to 10% of all samples analyzed but may be reduced if spike recoveries from samples (Section 8.3) meet all specified quality control criteria.

8.1.6 The laboratory must maintain performance records to document the quality of data that is generated. This procedure is described in Section 8.5.

8.2 To establish the ability to generate acceptable accuracy and precision, the analyst must perform the following operations.

8.2.1 A quality control (QC) check sample concentrate is required containing each parameter of interest at a concentration of 100 µg/mL in 2-propanol. The QC check sample concentrate must be obtained from the U.S. Environmental Protection Agency, Environmental Monitoring and Support Laboratory in Cincinnati, Ohio, if available. If not available from that source, the QC check sample concentrate must be obtained from another external source. If not available from either source above, the QC check sample concentrate must be prepared by the laboratory using stock standards prepared independently from those used for calibration.

8.2.2 Using a pipet, prepare QC check samples at a concentration of 100 µg/L by adding 1.00 mL of QC check sample concentrate to each of four 1-L aliquots of reagent water.

8.2.3 Analyze the well-mixed QC check samples according to the method beginning in Section 10.

8.2.4 Calculate the average recovery ($\bar{X}$) in µg/L, and the standard deviation of the recovery (s) in µg/L, for each parameter using the four results.

8.2.5 For each parameter compare s and $\bar{X}$ with the corresponding acceptance criteria for precision and accuracy, respectively, found in Table 3. If s and $\bar{X}$ for all parameters of interest meet the acceptance criteria, the system performance is acceptable and analysis of actual samples can begin. If any individual s exceeds the precision limit or any individual $\bar{X}$ falls outside the range for accuracy, the system performance is unacceptable for that parameter.

NOTE: The large number of parameters in Talbe 3 present a substantial probability that one or more will fail at least one of the acceptance criteria when all parameters are analyzed.

8.2.6 When one or more of the parameters tested fail at least one of the acceptance criteria, the analyst must proceed according to Section 8.2.6.1 or 8.2.6.2.

8.2.6.1 Locate and correct the source of the problem and repeat the test for all parameters of interest beginning with Section 8.2.2.

8.2.6.2 Beginning with Section 8.2.2, repeat the test only for those parameters that failed to meet criteria. Repeated failure, however, will confirm a general problem with the measurement system. If this occurs, locate and correct the source of the problem and repeat the test for all compounds of interest beginning with Section 8.2.2.

8.3 The laboratory must, on an ongoing basis, spike at least 10% of the samples from each sample site being monitored to assess accuracy. For laboratories analyzing one to ten samples per month, at least one spiked sample per month is required.

8.3.1 The concentration of the spike in the sample should be determined as follows:

8.3.1.1 If, as in compliance monitoring, the concentration of a specific parameter in the sample is being checked against a regulatory concentration limit, the spike should be at that limit or 1 to 5 times higher than the background concentration determined in Section 8.3.2, whichever concentration would be larger.

8.3.1.2 If the concentration of a specific parameter in the sample is not being checked against a limit specific to that parameter, the spike should be at 100 µg/L or 1 to 5 times higher than the background concentration determined in Section 8.3.2, whichever concentration would be larger.

8.3.1.3 If it is impractical to determine background levels before spiking (e.g., maximum holding times will be exceeded), the spike concentration should be (1) the regulatory concentration limit, if any, or, if none, (2) the larger of either 5 times higher than the expected background concentration or 100 µg/L.

8.3.2 Analyze one sample aliquot to determine the background concentration (B) of each parameter. If necessary, prepare a new QC check sample concentrate (Section 8.2.1) appropriate for the background concentrations in the sample. Spike a second sample aliquot with 1.0 mL of the QC check sample concentrate and analyze it to determine the concentration after spiking (A) of each parameter. Calculate each percent recovery (P) as 100(A–B)%/T, where T is the known true value of the spike.

8.3.3 Compare the percent recovery (P) for each parameter with the corresponding QC acceptance criteria found in Table 3. These acceptance criteria were calculated to include an allowance for error in measurement of both the background and spike concentrations, assuming a spike to background ratio of 5:1. This error will be accounted for to the extent that the analyst's spike to background ratio approaches 5:1.[8] If spiking was performed at a concentration lower than 100 µg/L, the analyst must use either the QC acceptance criteria in Table 3, or optional QC acceptance criteria calculated for the specific spike concentration. To calculate optional acceptance criteria for the recovery of a parameter: (1) Calculate accuracy (X′) using the equation in Table 4, substituting the spike concentration (T) for C; (2) calculate overall precision (S′) using the equation in Table 4, substituting X′ for $\bar{X}$; (3) calculate the range for recovery at the spike concentration as (100 X′/T)±2.44(100 S′/T)%.[8]

8.3.4 If any individual P falls outside the designated range for recovery, that parameter has failed the acceptance criteria. A check standard containing each parameter that failed the criteria must be analyzed as described in Section 8.4.

8.4 If any parameter fails the acceptance criteria for recovery in Section 8.3, a QC check standard containing each parameter that failed must be prepared and analyzed.

NOTE: The frequency for the required analysis of a QC check standard will depend upon the number of parameters being simultaneously tested, the complexity of the sample matrix, and the performance of the laboratory.

8.4.1 Prepare the QC check standard by adding 1.0 mL of QC check sample concentrate (Section 8.2.1 or 8.3.2) to 1 L of reagent water. The QC check standard needs only to contain the parameters that failed criteria in the test in Section 8.3.

8.4.2 Analyze the QC check standard to determine the concentration measured (A) of each parameter. Calculate each percent recovery (P_s) as 100 (A/T)%, where T is the true value of the standard concentration.

8.4.3 Compare the percent recovery (P_s) for each parameter with the corresponding QC acceptance criteria found in Table 3. Only parameters that failed the test in Section 8.3 need to be compared with these criteria. If the recovery of any such parameter falls outside the designated range, the laboratory performance for that parameter is judged to be out of control, and the problem must be immediately identified and corrected. The analytical result for that parameter in the unspiked sample is suspect and may not be reported for regulatory compliance purposes.

8.5 As part of the QC program for the laboratory, method accuracy for wastewater samples must be assessed and records must be maintained. After the analysis of five spiked wastewater samples as in Section 8.3, calculate the average percent recovery ($\bar{P}$) and the standard deviation of the percent recovery (s_p). Express the accuracy assessment as a percent recovery interval from $\bar{P}-2s_p$ to $\bar{P}+2s_p$. If $\bar{P}$=90% and s_p=10%, for example, the accuracy interval is expressed as 70–110%. Update the accuracy assessment for each parameter on a regular basis (e.g. after each five to ten new accuracy measurements).

8.6. It is recommended that the laboratory adopt additional quality assurance practices for use with this method. The specific practices that are most productive depend upon the needs of the laboratory and the nature of the samples. Field duplicates may be analyzed to assess the precision of the environmental measurements. When doubt exists over the identification of a peak on the chromatogram, confirmatory techniques such as gas chromatography with a dissimilar column, specific element detector, or mass spectrometer must be used. Whenever possible, the laboratory should analyze standard reference materials and participate in relevant performance evaluation studies.

9. Sample Collection, Preservation, and Handling

9.1 Grab samples must be collected in glass containers. Conventional sampling practices[9] should be followed, except that the bottle must not be prerinsed with sample before collection. Composite samples should be collected in refrigerated glass containers in accordance with the requirements of the program. Automatic sampling equipment must be as free as possible of Tygon tubing and other potential sources of contamination.

9.2 All samples must be iced or refrigerated at 4 °C from the time of collection until extraction. Fill the sample bottles and, if residual chlorine is present, add 80 mg of sodium thiosulfate per liter of sample and mix well. EPA Methods 330.4 and 330.5 may be used for measurement of residual chlorine.[10] Field test kits are available for this purpose.

9.3 All samples must be extracted within 7 days of collection and completely analyzed within 40 days of extraction.[2]

10. Sample Extraction

10.1 Mark the water meniscus on the side of sample bottle for later determination of sample volume. Pour the entire sample into a 2-L separatory funnel.

10.2 For samples high in organic content, the analyst may solvent wash the sample at basic pH as prescribed in Sections 10.2.1 and 10.2.2 to remove potential method interferences. Prolonged or exhaustive contact with solvent during the wash may result in low recovery of some of the phenols, notably phenol and 2,4-dimethylphenol. For relatively clean samples, the wash should be omitted and the extraction, beginning with Section 10.3, should be followed.

10.2.1 Adjust the pH of the sample to 12.0 or greater with sodium hydroxide solution.

10.2.2 Add 60 mL of methylene chloride to the sample by shaking the funnel for 1 min with periodic venting to release excess pressure. Discard the solvent layer. The wash can be repeated up to two additional times if significant color is being removed.

10.3 Adjust the sample to a pH of 1 to 2 with sulfuric acid.

10.4 Add 60 mL of methylene chloride to the sample bottle, seal, and shake 30 s to rinse the inner surface. Transfer the solvent to the separatory funnel and extract the sample by shaking the funnel for 2 min. with periodic venting to release excess pressure. Allow the organic layer to separate from the water phase for a minimum of 10 min. If the emulsion interface between layers is more than one-third the volume of the solvent layer, the analyst must employ mechanical techniques to complete the phase separation. The optimum technique depends upon the sample, but may include stirring, filtration

of the emulsion through glass wool, centrifugation, or other physical methods. Collect the methylene chloride extract in a 250-mL Erlenmeyer flask.

10.5 Add a second 60-mL volume of methylene chloride to the sample bottle and repeat the extraction procedure a second time, combining the extracts in the Erlenmeyer flask. Perform a third extraction in the same manner.

10.6 Assemble a Kuderna-Danish (K–D) concentrator by attaching a 10-mL concentrator tube to a 500-mL evaporative flask. Other concentration devices or techniques may be used in place of the K–D concentrator if the requirements of Section 8.2 are met.

10.7 Pour the combined extract through a solvent-rinsed drying column containing about 10 cm of anhydrous sodium sulfate, and collect the extract in the K–D concentrator. Rinse the Erlenmeyer flask and column with 20 to 30 mL of methylene chloride to complete the quantitative transfer.

10.8 Add one or two clean boiling chips to the evaporative flask and attach a three-ball Snyder column. Prewet the Snyder column by adding about 1 mL of methylene chloride to the top. Place the K–D apparatus on a hot water bath (60 to 65 °C) so that the concentrator tube is partially immersed in the hot water, and the entire lower rounded surface of the flask is bathed with hot vapor. Adjust the vertical position of the apparatus and the water temperature as required to complete the concentration in 15 to 20 min. At the proper rate of distillation the balls of the column will actively chatter but the chambers will not flood with condensed solvent. When the apparent volume of liquid reaches 1 mL, remove the K–D apparatus and allow it to drain and cool for at least 10 min.

10.9 Increase the temperature of the hot water bath to 95 to 100 °C. Remove the Synder column and rinse the flask and its lower joint into the concentrator tube with 1 to 2 mL of 2-propanol. A 5-mL syringe is recommended for this operation. Attach a two-ball micro-Snyder column to the concentrator tube and prewet the column by adding about 0.5 mL of 2-propanol to the top. Place the micro-K–D apparatus on the water bath so that the concentrator tube is partially immersed in the hot water. Adjust the vertical position of the apparatus and the water temperature as required to complete concentration in 5 to 10 min. At the proper rate of distillation the balls of the column will actively chatter but the chambers will not flood. When the apparent volume of liquid reaches 2.5 mL, remove the K–D apparatus and allow it to drain and cool for at least 10 min. Add an additional 2 mL of 2-propanol through the top of the micro-Snyder column and resume concentrating as before. When the apparent volume of liquid reaches 0.5 mL, remove the K–D apparatus and allow it to drain and cool for at least 10 min.

10.10 Remove the micro-Snyder column and rinse its lower joint into the concentrator tube with a minimum amount of 2-propanol. Adjust the extract volume to 1.0 mL. Stopper the concentrator tube and store refrigerated at 4 °C if further processing will not be performed immediately. If the extract will be stored longer than two days, it should be transferred to a Teflon-sealed screw-cap vial. If the sample extract requires no further cleanup, proceed with FIDGC analysis (Section 11). If the sample requires further cleanup, proceed to Section 12.

10.11 Determine the original sample volume by refilling the sample bottle to the mark and transferring the liquid to a 1000-mL graduated cylinder. Record the sample volume to the nearest 5 mL.

11. Flame Ionization Detector Gas Chromatography

11.1 Table 1 summarizes the recommended operating conditions for the gas chromatograph. Included in this table are retention times and MDL that can be achieved under these conditions. An example of the separations achieved by this column is shown in Figure 1. Other packed or capillary (open-tubular) columns, chromatographic conditions, or detectors may be used if the requirements of Section 8.2 are met.

11.2 Calibrate the system daily as described in Section 7.

11.3 If the internal standard calibration procedure is used, the internal standard must be added to the sample extract and mixed thoroughly immediately before injection into the gas chromatograph.

11.4 Inject 2 to 5 µL of the sample extract or standard into the gas chromatograph using the solvent-flush technique.[11] Smaller (1.0 µL) volumes may be injected if automatic devices are employed. Record the volume injected to the nearest 0.05 µL, and the resulting peak size in area or peak height units.

11.5 Identify the parameters in the sample by comparing the retention times of the peaks in the sample chromatogram with those of the peaks in standard chromatograms. The width of the retention time window used to make identifications should be based upon measurements of actual retention time variations of standards over the course of a day. Three times the standard deviation of a retention time for a compound may be used to calculate a suggested window size; however, the experience of the analyst should weigh heavily in the interpretation of chromatograms.

11.6 If the response for a peak exceeds the working range of the system, dilute the extract and reanalyze.

11.7 If the measurement of the peak response is prevented by the presence of interferences, an alternative gas chromatographic procedure is required. Section 12 describes a derivatization and column chromatographic procedure which has been tested and found to be a practical means of analyzing phenols in complex extracts.

12. Derivatization and Electron Capture Detector Gas Chromatography

12.1 Pipet a 1.0-mL aliquot of the 2-propanol solution of standard or sample extract into a glass reaction vial. Add 1.0 mL of derivatizing reagent (Section 6.11). This amount of reagent is sufficient to derivatize a solution whose total phenolic content does not exceed 0.3 mg/mL.

12.2 Add about 3 mg of potassium carbonate to the solution and shake gently.

12.3 Cap the mixture and heat it for 4 h at 80 °C in a hot water bath.

12.4 Remove the solution from the hot water bath and allow it to cool.

12.5 Add 10 mL of hexane to the reaction flask and shake vigorously for 1 min. Add 3.0 mL of distilled, deionized water to the reaction flask and shake for 2 min. Decant a portion of the organic layer into a concentrator tube and cap with a glass stopper.

12.6 Place 4.0 g of silica gel into a chromatographic column. Tap the column to settle the silica gel and add about 2 g of anhydrous sodium sulfate to the top.

12.7 Preelute the column with 6 mL of hexane. Discard the eluate and just prior to exposure of the sodium sulfate layer to the air, pipet onto the column 2.0 mL of the hexane solution (Section 12.5) that contains the derivatized sample or standard. Elute the column with 10.0 mL of hexane and discard the eluate. Elute the column, in order, with: 10.0 mL of 15% toluene in hexane (Fraction 1); 10.0 mL of 40% toluene in hexane (Fraction 2); 10.0 mL of 75% toluene in hexane (Fraction 3); and 10.0 mL of 15% 2-propanol in toluene (Fraction 4). All elution mixtures are prepared on a volume: volume basis. Elution patterns for the phenolic derivatives are shown in Table 2. Fractions may be combined as desired, depending upon the specific phenols of interest or level of interferences.

12.8 Analyze the fractions by ECDGC. Table 2 summarizes the recommended operating conditions for the gas chromatograph. Included in this table are retention times and MDL that can be achieved under these conditions. An example of the separations achieved by this column is shown in Figure 2.

12.9 Calibrate the system daily with a minimum of three aliquots of calibration standards, containing each of the phenols of interest that are derivatized according to Section 7.5.

12.10 Inject 2 to 5 µL of the column fractions into the gas chromatograph using the solvent-flush technique. Smaller (1.0 µL) volumes can be injected if automatic devices are employed. Record the volume injected to the nearest 0.05 µL, and the resulting peak size in area or peak height units. If the peak response exceeds the linear range of the system, dilute the extract and reanalyze.

13. Calculations

13.1 Determine the concentration of individual compounds in the sample analyzed by FIDGC (without derivatization) as indicated below.

13.1.1 If the external standard calibration procedure is used, calculate the amount of material injected from the peak response using the calibration curve or calibration factor determined in Section 7.2.2. The concentration in the sample can be calculated from Equation 2.

$$\text{Concentration } (\mu g/L) = \frac{(A)(V_t)}{(V_i)(V_s)}$$

Equation 2

where:

A=Amount of material injected (ng).

V_i=Volume of extract injected (µL).

V_t=Volume of total extract (µL).

V_s=Volume of water extracted (mL).

13.1.2 If the internal standard calibration procedure is used, calculate the concentration in the sample using the response factor (RF) determined in Section 7.3.2 and Equation 3.

$$\text{Concentration } (\mu g/L) = \frac{(A_s)(I_s)}{(A_{is})(RF)(V_o)}$$

Equation 3

where:

A_s=Response for the parameter to be measured.

A_{is}=Response for the internal standard.

I_s=Amount of internal standard added to each extract (µg).

V_o=Volume of water extracted (L).

13.2 Determine the concentration of individual compounds in the sample analyzed by derivatization and ECDGC according to Equation 4.

$$\text{Concentration } (\mu g/L) = \frac{(A)(V_t)(B)(D)}{(V_i)(V_s)(C)(E)}$$

Equation 4

where:

A=Mass of underivatized phenol represented by area of peak in sample chromatogram, determined from calibration curve in Section 7.5.3 (ng).

V_i=Volume of eluate injected (µL).

V_t=Total volume of column eluate or combined fractions from which V_i was taken (µL).

V_s=Volume of water extracted in Section 10.10 (mL).

B=Total volume of hexane added in Section 12.5 (mL).

C=Volume of hexane sample solution added to cleanup column in Section 12.7 (mL).

D=Total volume of 2-propanol extract prior to derivatization (mL).

E=Volume of 2-propanol extract carried through derivatization in Section 12.1 (mL).

13.3 Report results in µg/L without correction for recovery data. All QC data obtained should be reported with the sample results.

14. Method Performance

14.1 The method detection limit (MDL) is defined as the minimum concentration of a substance that can be measured and reported with 99% confidence that the value is above zero.[1] The MDL concentrations listed in Tables 1 and 2 were obtained using reagent water.[12] Similar results were achieved using representative wastewaters. The MDL actually achieved in a given analysis will vary depending on instrument sensitivity and matrix effects.

14.2 This method was tested by 20 laboratories using reagent water, drinking water, surface water, and three industrial wastewaters spiked as six concentrations over the range 12 to 450 µg/L.[13] Single operator precision, overall precision, and method accuracy were found to be directly related to the concentration of the parameter and essentially independent of the sample matrix. Linear equations to describe these relationships for a flame ionization detector are presented in Table 4.

References

1. 40 CFR part 136, appendix B.

2. ''Determination of Phenols in Industrial and Municipal Wastewaters,'' EPA 600/4–84–ABC, National Technical Information Service, PBXYZ, Springfield, Virginia 22161, November 1984.

3. Kawahara, F. K. ''Microdetermination of Derivatives of Phenols and Mercaptans by Means of Electron Capture Gas Chromatography,'' *Analytical Chemistry, 40,* 1009 (1968).

4. ASTM Annual Book of Standards, Part 31, D3694–78. ''Standard Practices for Preparation of Sample Containers and for Preservation of Organic Constituents,'' American Society for Testing and Materials, Philadelphia.

5. ''Carcinogens—Working With Carcinogens,'' Department of Health, Education, and Welfare, Public Health Service, Center for Disease Control, National Institute for Occupational Safety and Health, Publication No. 77–206, August 1977.

6. ''OSHA Safety and Health Standards, General Industry,'' (29 CFR part 1910), Occupational Safety and Health Administration, OSHA 2206 (Revised, January 1976).

7. ''Safety in Academic Chemistry Laboratories,'' American Chemical Society Publication, Committee on Chemical Safety, 3rd Edition, 1979.

8. Provost, L. P., and Elder, R. S. ''Interpretation of Percent Recovery Data,'' *American Laboratory, 15,* 58–63 (1983). (The value 2.44 used in the equation in Section 8.3.3 is two times the value 1.22 derived in this report.)

9. ASTM Annual Book of Standards, Part 31, D3370–76. ''Standard Practices for Sampling Water,'' American Society for Testing and Materials, Philadelphia.

10. ''Methods 330.4 (Titrimetric, DPD–FAS) and 330.5 (Spectrophotometric, DPD) for Chlorine, Total Residual,'' Methmds for Chemical Analysis of Water and Wastes, EPA–600/4–79–020, U.S. Environmental Protection Agency, Environmental Monitoring and Support Laboratory, Cincinnati, Ohio 45268, March 1979.

11. Burke, J. A. ''Gas Chromatography for Pesticide Residue Analysis; Some Practical Aspects,'' *Journal of the Association of Official Analytical Chemists, 48,* 1037 (1965).

12. ''Development of Detection Limits, EPA Method 604, Phenols,'' Special letter report for EPA Contract 68–03–2625, U.S. Environmental Protection Agency, Environmental Monitoring and Support Laboratory, Cincinnati, Ohio 45268.

13. ''EPA Method Study 14 Method 604-Phenols,'' EPA 600/4–84–044, National Technical Information Service, PB84–196211, Springfield, Virginia 22161, May 1984.

TABLE 1—CHROMATOGRAPHIC CONDITIONS AND METHOD DETECTION LIMITS

Parameter	Retention time (min)	Method detection limit (µg/L)
2-Chlorophenol	1.70	0.31
2-Nitrophenol	2.00	0.45
Phenol	3.01	0.14
2,4-Dimethylphenol	4.03	0.32
2,4-Dichlorophenol	4.30	0.39
2,4,6-Trichlorophenol	6.05	0.64
4-Chloro-3-methylphenol	7.50	0.36
2,4-Dinitrophenol	10.00	13.0
2-Methyl-4,6-dinitrophenol	10.24	16.0
Pentachlorophenol	12.42	7.4
4-Nitrophenol	24.25	2.8

Column conditions: Supelcoport (80/100 mesh) coated with 1% SP–1240DA packed in a 1.8 m long x 2 mm ID glass column with nitrogen carrier gas at 30 mL/min flow rate. Column temperature was 80 °C at injection, programmed immediately at 8 °C/min to 150 °C final temperature. MDL were determined with an FID.

TABLE 2—SILICA GEL FRACTIONATION AND ELECTRON CAPTURE GAS CHROMATOGRAPHY OF PFBB DERIVATIVES

Parent compound	Percent recovery by fraction [a]				Retention time (min)	Method detection limit (µg/L)
	1	2	3	4		
2-Chlorophenol	.....	90	1	.....	3.3	0.58
2-Nitrophenol	.....	.....	9	90	9.1	0.77
Phenol	.....	90	10	.....	1.8	2.2
2,4-Dimethylphenol	.....	95	7	.....	2.9	0.63
2,4-Dichlorophenol	.....	95	1	.....	5.8	0.68
2,4,6-Trichlorophenol	50	50	.....	.....	7.0	0.58
4-Chloro-3-methylphenol	.....	84	14	.....	4.8	1.8
Pentachlorophenol	75	20	.....	.....	28.8	0.59
4-Nitrophenol	.....	.....	1	90	14.0	0.70

Column conditions: Chromosorb W–AW–DMCS (80/100 mesh) coated with 5% OV–17 packed in a 1.8 m long x 2.0 mm ID glass column with 5% methane/95% argon carrier gas at 30 mL/min flow rate. Column temperature held isothermal at 200 °C. MDL were determined with an ECD.

[a] Eluant composition:
Fraction 1—15% toluene in hexane.
Fraction 2—40% toluene in hexane.
Fraction 3—75% toluene in hexane.
Fraction 4—15% 2-propanol in toluene.

TABLE 3—QC ACCEPTANCE CRITERIA—METHOD 604

Parameter	Test conc. (µg/L)	Limit for s (µg/L)	Range for $\bar{X}$ (µg/L)	Range for P, P_s (percent)
4-Chloro-3-methylphenol	100	16.6	56.7–113.4	49–122
2-Chlorophenol	100	27.0	54.1–110.2	38–126
2,4-Dichlorophenol	100	25.1	59.7–103.3	44–119
2,4-Dimethylphenol	100	33.3	50.4–100.0	24–118
4,6-Dinitro-2-methylphenol	100	25.0	42.4–123.6	30–136
2,4-Dinitrophenol	100	36.0	31.7–125.1	12–145
2-Nitrophenol	100	22.5	56.6–103.8	43–117
4-Nitrophenol	100	19.0	22.7–100.0	13–110
Pentachlorophenol	100	32.4	56.7–113.5	36–134
Phenol	100	14.1	32.4–100.0	23–108
2,4,6-Trichlorophenol	100	16.6	60.8–110.4	53–119

s—Standard deviation of four recovery measurements, in µg/L (Section 8.2.4).
$\bar{X}$—Average recovery for four recovery measurements, in µg/L (Section 8.2.4).
P, P_s—Percent recovery measured (Section 8.3.2, Section 8.4.2).

NOTE: These criteria are based directly upon the method performance data in Table 4. Where necessary, the limits for recovery have been broadened to assure applicability of the limits to concentrations below those used to develop Table 4.

TABLE 4—METHOD ACCURACY AND PRECISION AS FUNCTIONS OF CONCENTRATION—METHOD 604

Parameter	Accuracy, as recovery, X′ (µg/L)	Single Analyst precision, s_r' (µg/L)	Overall precision, S′ (µg/L)
4-Chloro-3-methylphenol	0.87C–1.97	$0.11\bar{X}$–0.21	$0.16\bar{X}$+1.41
2-Chlorophenol	0.83C–0.84	$0.18\bar{X}$+0.20	$0.21\bar{X}$+0.75
2,4-Dichlorophenol	0.81C+0.48	$0.17\bar{X}$–0.02	$0.18\bar{X}$+0.62
2,4-Dimethylphenol	0.62C–1.64	$0.30\bar{X}$–0.89	$0.25\bar{X}$+0.48
4,6-Dinitro-2-methylphenol	0.84C–1.01	$0.15\bar{X}$+1.25	$0.19\bar{X}$+5.85
2,4-Dinitrophenol	0.80C–1.58	$0.27\bar{X}$–1.15	$0.29\bar{X}$+4.51
2-Nitrophenol	0.81C–0.76	$0.15\bar{X}$+0.44	$0.14\bar{X}$+3.84
4-Nitrophenol	0.46C+0.18	$0.17\bar{X}$+2.43	$0.19\bar{X}$+4.79
Pentachlorophenol	0.83C+2.07	$0.22\bar{X}$–0.58	$0.23\bar{X}$+0.57
Phenol	0.43C+0.11	$0.20\bar{X}$–0.88	$0.17\bar{X}$+0.77
2,4,6-Trichlorophenol	0.86C–0.40	$0.10\bar{X}$+0.53	$0.13\bar{X}$+2.40

X′=Expected recovery for one or more measurements of a sample containing a concentration of C, in µg/L.
s_r'=Expected single analyst standard deviation of measurements at an average concentration found of $\bar{X}$, in µg/L.
S′=Expected interlaboratory standard deviation of measurements at an average concentration found of $\bar{X}$, in µg/L.
C=True value for the concentration, in µg/L.
$\bar{X}$=Average recovery found for measurements of samples containing a concentration of C, in µg/L.

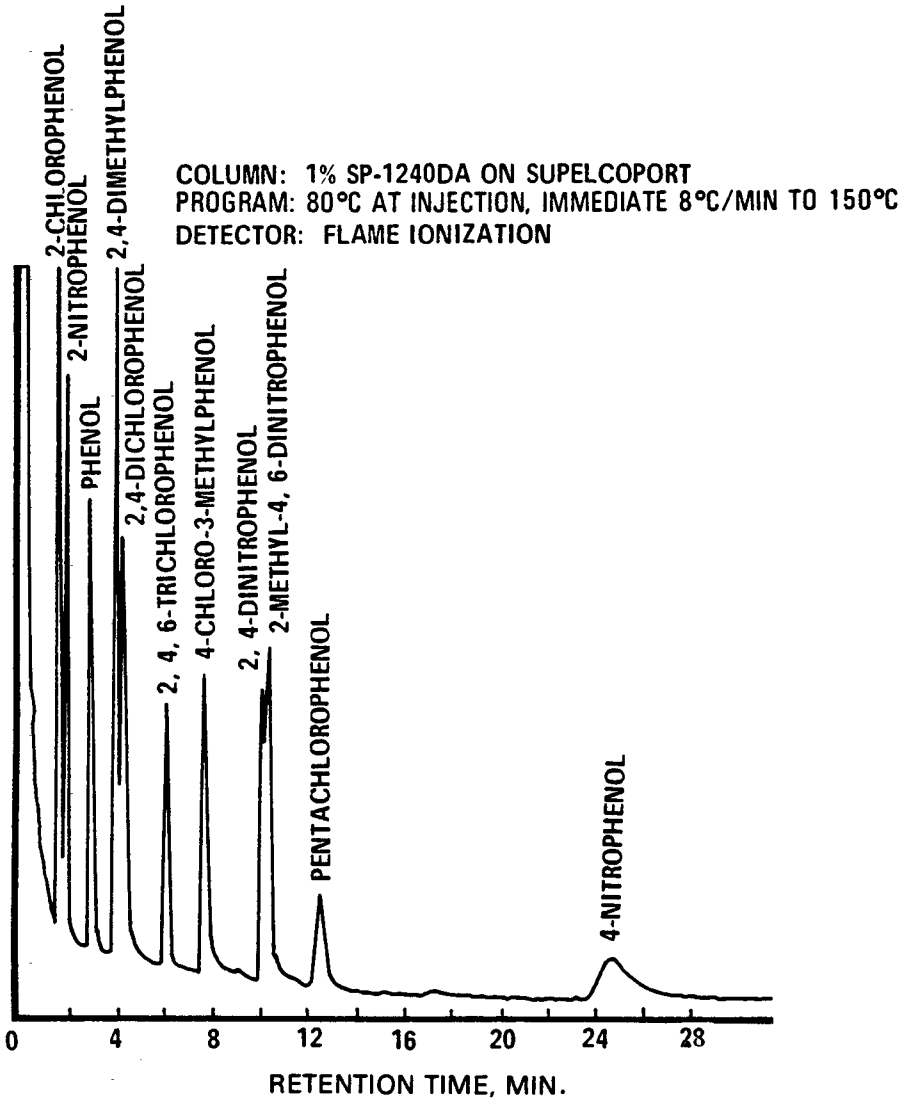


Figure 1. Gas chromatogram of phenols.

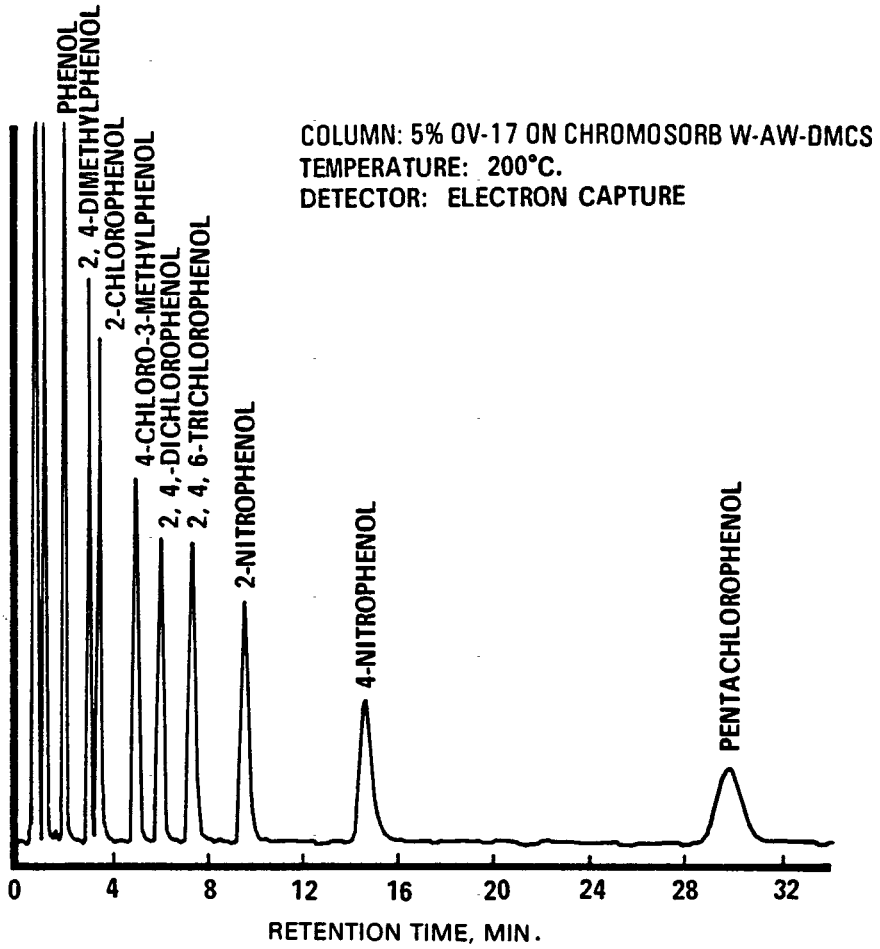


Figure 2. Gas chromatogram of PFB derivatives of phenols.

METHOD 605—BENZIDINES

1. Scope and Application

1.1 This method covers the determination of certain benzidines. The following parameters can be determined by this method:

Parameter	Storet No	CAS No.
Benzidine	39120	92–87–5
3,3′-Dichlorobenzidine	34631	91–94–1

1.2 This is a high performance liquid chromatography (HPLC) method applicable to the determination of the compounds listed above in municipal and industrial discharges as provided under 40 CFR 136.1. When this method is used to analyze unfamiliar samples for the compounds above, identifications should be supported by at least one additional qualitative technique. This method describes electrochemical conditions at a second potential which can be used to confirm measurements made with this method. Method 625 provides gas chromatograph/mass spectrometer (GC/MS) conditions appropriate for the qualitative and quantitative confirmation of results for the parameters listed above, using the extract produced by this method.

1.3 The method detection limit (MDL, defined in Section 14.1)[1] for each parameter is listed in Table 1. The MDL for a specific wastewater may differ from those listed, depending upon the nature of the interferences in the sample matrix.

1.4 Any modification of this method, beyond those expressly permitted, shall be considered as a major modification subject to application and approval of alternate test procedures under 40 CFR 136.4 and 136.5.

1.5 This method is restricted to use by or under the supervision of analysts experienced in the use of HPLC instrumentation and in the interpretation of liquid chromatograms. Each analyst must demonstrate the ability to generate acceptable results with this method using the procedure described in Section 8.2.

2. Summary of Method

2.1 A measured volume of sample, approximately 1–L, is extracted with chloroform using liquid-liquid extractions in a separatory funnel. The chloroform extract is extracted with acid. The acid extract is then neutralized and extracted with chloroform. The final chloroform extract is exchanged to methanol while being concentrated using a rotary evaporator. The extract is mixed with buffer and separated by HPLC. The benzidine compounds are measured with an electrochemical detector.[2]

2.2 The acid back-extraction acts as a general purpose cleanup to aid in the elimination of interferences.

3. Interferences

3.1 Method interferences may be caused by contaminants in solvents, reagents, glassware, and other sample processing hardware that lead to discrete artifacts and/or elevated baselines in chromatograms. All of these materials must be routinely demonstrated to be free from interferences under the conditions of the analysis by running laboratory reagent blanks as described in Section 8.1.3.

3.1.1 Glassware must be scrupulously cleaned.[3] Clean all glassware as soon as possible after use by rinsing with the last solvent used in it. Solvent rinsing should be followed by detergent washing with hot water, and rinses with tap water and distilled water. The glassware should then be drained dry, and heated in a muffle furnace at 400 °C for 15 to 30 min. Some thermally stable materials may not be eliminated by this treatment. Solvent rinses with acetone and pesticide quality hexane may be substituted for the muffle furnace heating. Volumetric ware should not be heated in a muffle furnace. After drying and cooling, glassware should be sealed and stored in a clean environment to prevent any accumulation of dust or other contaminants. Store inverted or capped with aluminum foil.

3.1.2 The use of high purity reagents and solvents helps to minimize interference problems. Purification of solvents by distillation in all-glass systems may be required.

3.2 Matrix interferences may be caused by contaminants that are co-extracted from the sample. The extent of matrix interferences will vary considerably from source to source, depending upon the nature and diversity of the industrial complex or municipality being sampled. The cleanup procedures that are inherent in the extraction step are used to overcome many of these interferences, but unique samples may require additional cleanup approaches to achieve the MDL listed in Table 1.

3.3 Some dye plant effluents contain large amounts of components with retention times closed to benzidine. In these cases, it has been found useful to reduce the electrode potential in order to eliminate interferences and still detect benzidine. (See Section 12.7.)

4. Safety

4.1 The toxicity or carcinogenicity of each reagent used in this method has not been precisely defined; however, each chemical compound should be treated as a potential health harzard. From this viewpoint, exposure to these chemicals must be reduced to the lowest possible level by whatever means available. The laboratory is responsible for maintaining a current awareness file of OSHA regulations regarding the safe handling of the chemicals specified in this method. A reference file of material data handling

sheets should also be made available to all personnel involved in the chemical analysis. Additional references to laboratory safety are available and have been identified [4–6] for the information of the analyst.

4.2 The following parameters covered by this method have been tentatively classified as known or suspected, human or mammalian carcinogens: benzidine and 3,3′-dichlorobenzidine. Primary standards of these toxic compounds should be prepared in a hood. A NIOSH/MESA approved toxic gas respirator should be worn when the analyst handles high concentrations of these toxic compounds.

4.3 Exposure to chloroform should be minimized by performing all extractions and extract concentrations in a hood or other well-ventiliated area.

5. Apparatus and Materials

5.1 Sampling equipment, for discrete or composite sampling.

5.1.1 Grab sample bottle—1–L or 1-qt, amber glass, fitted with a screw cap lined with Teflon. Foil may be substituted for Teflon if the sample is not corrosive. If amber bottles are not available, protect samples from light. The bottle and cap liner must be washed, rinsed with acetone or methylene chloride, and dried before use to minimize contamination.

5.1.2 Automatic sampler (optional)—The sampler must incorporate glass sample containers for the collection of a minimum of 250 mL of sample. Sample containers must be kept refrigerated at 4°C and protected from light during compositing. If the sampler uses a peristaltic pump, a minimum length of compressible silicone rubber tubing may be used. Before use, however, the compressible tubing should be thoroughly rinsed with methanol, followed by repeated rinsings with distilled water to minimize the potential for contamination of the sample. An integrating flow meter is required to collect flow proportional composites.

5.2 Glassware (All specifications are suggested):

5.2.1 Separatory funnels—2000, 1000, and 250-mL, with Teflon stopcock.

5.2.2 Vials—10 to 15-mL, amber glass, with Teflon-lined screw cap.

5.2.3 Rotary evaporator.

5.2.4 Flasks—Round bottom, 100–mL, with 24/40 joints.

5.2.5 Centrifuge tubes—Conical, graduated, with Teflon-lined screw caps.

5.2.6 Pipettes—Pasteur, with bulbs.

5.3 Balance—Analytical, capable of accurately weighing 0.0001 g.

5.4 High performance liquid chromatograph (HPLC)—An analytical system complete with column supplies, high pressure syringes, detector, and compatible recorder. A data system is recommended for measuring peak areas and retention times.

5.4.1 Solvent delivery system—With pulse damper, Altex 110A or equivalent.

5.4.2 Injection valve (optional)—Waters U6K or equivalent.

5.4.3 Electrochemical detector—Bioanalytical Systems LC–2A with glassy carbon electrode, or equivalent. This detector has proven effective in the analysis of wastewaters for the parameters listed in the scope (Section 1.1), and was used to develop the method performance statements in Section 14. Guidelines for the use of alternate detectors are provided in Section 12.1.

5.4.4 Electrode polishing kit—Princeton Applied Research Model 9320 or equivalent.

5.4.5 Column—Lichrosorb RP–2, 5 micron particle diameter, in a 25 cm × 4.6 mm ID stainless steel column. This column was used to develop the method performance statements in Section 14. Guidelines for the use of alternate column packings are provided in Section 12.1.

6. Reagents

6.1 Reagent water—Reagent water is defined as a water in which an interferent is not observed at the MDL of the parameters of interest.

6.2 Sodium hydroxide solution (5 N)—Dissolve 20 g of NaOH (ACS) in reagent water and dilute to 100 mL.

6.3 Sodium hydroxide solution (1 M)—Dissolve 40 g of NaOH (ACS) in reagent water and dilute to 1 L.

6.4 Sodium thiosulfate—(ACS) Granular.

6.5 Sodium tribasic phosphate (0.4 M)—Dissolve 160 g of trisodium phosphate decahydrate (ACS) in reagent water and dilute to 1 L.

6.6 Sulfuric acid (1+1)—Slowly, add 50 mL of H_2SO_4 (ACS, sp. gr. 1.84) to 50 mL of reagent water.

6.7 Sulfuric acid (1 M)—Slowly, add 58 mL of H_2SO_4 (ACS, sp. gr. 1.84) to reagent water and dilute to 1 L.

6.8 Acetate buffer (0.1 M, pH 4.7)—Dissolve 5.8 mL of glacial acetic acid (ACS) and 13.6 g of sodium acetate trihydrate (ACS) in reagent water which has been purified by filtration through a RO–4 Millipore System or equivalent and dilute to 1 L.

6.9 Acetonitrile, chloroform (preserved with 1% ethanol), methanol—Pesticide quality or equivalent.

6.10 Mobile phase—Place equal volumes of filtered acetonitrile (Millipore type FH filter or equivalent) and filtered acetate buffer (Millipore type GS filter or equivalent) in a narrow-mouth, glass container and mix thoroughly. Prepare fresh weekly. Degas daily by sonicating under vacuum, by heating an stirring, or by purging with helium.

6.11 Stock standard solutions (1.00 µg/µL)—Stock standard solutions may be prepared from pure standard materials or purchased as certified solutions.

6.11.1 Prepare stock standard solutions by accurately weighing about 0.0100 g of pure material. Dissolve the material in methanol and dilute to volume in a 10–mL volumetric flask. Larger volumes can be used at the convenience of the analyst. When compound purity is assayed to be 96% or greater, the weight can be used without correction to calculate the concentration of the stock standard. Commercially prepared stock standards can be used at any concentration if they are certified by the manufacturer or by an independent source.

6.11.2 Transfer the stock standard solutions into Teflon-sealed screw-cap bottles. Store at 4 °C and protect from light. Stock standard solutions should be checked frequently for signs of degradation or evaporation, especially just prior to preparing calibration standards from them.

6.11.3 Stock standard solutions must be replaced after six months, or sooner if comparison with check standards indicates a problem.

6.12 Quality control check sample concentrate—See Section 8.2.1.

7. Calibration

7.1 Establish chromatographic operating conditions equivalent to those given in Table 1. The HPLC system can be calibrated using the external standard technique (Section 7.2) or the internal standard technique (Section 7.3).

7.2 External standard calibration procedure:

7.2.1 Prepare calibration standards at a minimum of three concentration levels for each parameter of interest by adding volumes of one or more stock standards to a volumetric flask and diluting to volume with mobile phase. One of the external standards should be at a concentration near, but above, the MDL (Table 1) and the other concentrations should correspond to the expected range of concentrations found in real samples or should define the working range of the detector.

7.2.2 Using syringe injections of 5 to 25 µL or a constant volume injection loop, analyze each calibration standard according to Section 12 and tabulate peak height or area responses against the mass injected. The results can be used to prepare a calibration curve for each compound. Alternatively, if the ratio of response to amount injected (calibration factor) is a constant over the working range (<10% relative standard deviation, RSD), linearity through the origin can be assumed and the average ratio or calibration factor can be used in place of a calibration curve.

7.3 Internal standard calibration procedure—To use this approach, the analyst must select one or more internal standards that are similar in analytical behavior to the compounds of interest. The analyst must further demonstrate that the measurement of the internal standard is not affected by method or matrix interferences. Because of these limitations, no internal standard can be suggested that is applicable to all samples.

7.3.1 Prepare calibration standards at a minimum of three concentration levels for each parameter of interest by adding volumes of one or more stock standards to a volumetric flask. To each calibration standard, add a known constant amount of one or more internal standards, and dilute to volume with mobile phase. One of the standards should be at a concentration near, but above, the MDL and the other concentrations should correspond to the expected range of concentrations found in real samples or should define the working range of the detector.

7.3.2 Using syringe injections of 5 to 25 µL or a constant volume injection loop, analyze each calibration standard according to Section 12 and tabulate peak height or area responses against concentration for each compound and internal standard. Calculate response factors (RF) for each compound using Equation 1.

$$RF = \quad (A_s)(C_{is}) \quad (A_{is})(C_s)$$

Equation 1

where:

A_s=Response for the parameter to be measured.

A_{is}=Response for the internal standard.

C_{is}=Concentration of the internal standard (µg/L).

C_s=Concentration of the parameter to be measured (µg/L).

If the RF value over the working range is a constant (<10% RSD), the RF can be assumed to be invariant and the average RF can be used for calculations. Alternatively, the results can be used to plot a calibration curve of response ratios, A_s/A_{is}, vs. RF.

7.4 The working calibration curve, calibration factor, or RF must be verified on each working day by the measurement of one or more calibration standards. If the response for any parameter varies from the predicted response by more than ±15%, a new calibration curve must be prepared for that compound. If serious loss of response occurs, polish the electrode and recalibrate.

7.5 Before using any cleanup procedure, the analyst must process a series of calibration standards through the procedure to validate elution patterns and the absence of interferences from the reagents.

8. Quality Control

8.1 Each laboratory that uses this method is required to operate a formal quality control program. The minimum requirements of

this program consist of an initial demonstration of laboratory capability and an ongoing analysis of spiked samples to evaluate and document data quality. The laboratory must maintain records to document the quality of data that is generated. Ongoing data quality checks are compared with established performance criteria to determine if the results of analyses meet the performance characteristics of the method. When results of sample spikes indicate atypical method performance, a quality control check standard must be analyzed to confirm that the measurements were performed in an in-control mode of operation.

8.1.1 The analyst must make an initial, one-time, demonstration of the ability to generate acceptable accuracy and precision with this method. This ability is established as described in Section 8.2.

8.1.2 In recognition of advances that are occurring in chromatography, the analyst is permitted certain options (detailed in Sections 10.9, 11.1, and 12.1) to improve the separations or lower the cost of measurements. Each time such a modification is made to the method, the analyst is required to repeat the procedure in Section 8.2.

8.1.3 Before processing any samples, the analyst must analyze a reagent water blank to demonstrate that interferences from the analytical system and glassware are under control. Each time a set of samples is extracted or reagents are changed, a reagent water blank must be processed as a safeguard against laboratory contamination.

8.1.4 The laboratory must, on an ongoing basis, spike and analyze a minimum of 10% of all samples to monitor and evaluate laboratory data quality. This procedure is described in Section 8.3.

8.1.5 The laboratory must, on an ongoing basis, demonstrate through the analyses of quality control check standards that the operation of the measurement system is in control. This procedure is described in Section 8.4. The frequency of the check standard analyses is equivalent to 10% of all samples analyzed but may be reduced if spike recoveries from samples (Section 8.3) meet all specified quality control criteria.

8.1.6 The laboratory must maintain performance records to document the quality of data that is generated. This procedure is described in Section 8.5.

8.2 To establish the ability to generate acceptable accuracy and precision, the analyst must perform the following operations.

8.2.1 A quality control (QC) check sample concentrate is required containing benzidine and/or 3,3′-dichlorobenzidine at a concentration of 50 µg/mL each in methanol. The QC check sample concentrate must be obtained from the U.S. Environmental Protection Agency, Environmental Monitoring and Support Laboratory in Cincinnati, Ohio, if available. If not available from that source, the QC check sample concentrate must be obtained from another external source. If not available from either source above, the QC check sample concentrate must be prepared by the laboratory using stock standards prepared independently from those used for calibration.

8.2.2 Using a pipet, prepare QC check samples at a concentration of 50 µg/L by adding 1.00 mL of QC check sample concentrate to each of four 1–L-L aliquots of reagent water.

8.2.3 Analyze the well-mixed QC check samples according to the method beginning in Section 10.

8.2.4 Calculate the average recovery ($\bar{X}$) in µg/L, and the standard deviation of the recovery (s) in µg/L, for each parameter using the four results.

8.2.5 For each parameter compare s and $\bar{X}$ with the corresponding acceptance criteria for precision and accuracy, respectively, found in Table 2. If s and $\bar{X}$ for all parameters of interest meet the acceptance criteria, the system performance is acceptable and analysis of actual samples can begin. If any individual s exceeds the precision limit or any individual $\bar{X}$ falls outside the range for accuracy, the system performance is unacceptable for that parameter. Locate and correct the source of the problem and repeat the test for all parameters of interest beginning with Section 8.2.2.

8.3 The laboratory must, on an ongoing basis, spike at least 10% of the samples from each sample site being monitored to assess accuracy. For laboratories analyzing one to ten samples per month, at least one spiked sample per month is required.

8.3.1 The concentration of the spike in the sample should be determined as follows:

8.3.1.1 If, as in compliance monitoring, the concentration of a specific parameter in the sample is being checked against a regulatory concentration limit, the spike should be at that limit or 1 to 5 times higher than the background concentration determined in Section 8.3.2, whichever concentration would be larger.

8.3.1.2 If the concentration of a specific parameter in the sample is not being checked against a limit specific to that parameter, the spike should be at 50 µg/L or 1 to 5 times higher than the background concentration determined in Section 8.3.2, whichever concentration would be larger.

8.3.1.3 If it is impractical to determine background levels before spiking (e.g., maximum holding times will be exceeded), the spike concentration should be (1) the regulatory concentration limit, if any; or, if none (2) the larger of either 5 times higher than the expected background concentration or 50 µg/L.

8.3.2 Analyze one sample aliquot to determine the background concentration (B) of each parameter. If necessary, prepare a new QC check sample concentrate (Section 8.2.1)

appropriate for the background concentrations in the sample. Spike a second sample aliquot with 1.0 mL of the QC check sample concentrate and analyze it to determine the concentration after spiking (A) of each parameter. Calculate each percent recovery (P) as 100(A–B)%/T, where T is the known true value of the spike.

8.3.3 Compare the percent recovery (P) for each parameter with the corresponding QC acceptance criteria found in Table 2. These acceptance criteria were calculated to include an allowance for error in measurement of both the background and spike concentrations, assuming a spike to background ratio of 5:1. This error will be accounted for to the extent that the analyst's spike to background ratio approaches 5:1.[7] If spiking was performed at a concentration lower than 50 µg/L, the analyst must use either the QC acceptance criteria in Table 2, or optional QC acceptance criteria calculated for the specific spike concentration. To calculate optional acceptance criteria for the recovery of a parameter: (1) Calculate accuracy (X′) using the equation in Table 3, substituting the spike concentration (T) for C; (2) calculate overall precision (S′) using the equation in Table 3, substituting X′ for X; (3) calculate the range for recovery at the spike concentration as (100 X′/T)±2.44(100 S′/T)%.[7]

8.3.4 If any individual P falls outside the designated range for recovery, that parameter has failed the acceptance criteria. A check standard containing each parameter that failed the criteria must be analyzed as described in Section 8.4.

8.4 If any parameter fails the acceptance criteria for recovery in Section 8.3, a QC check standard containing each parameter that failed must be prepared and analyzed.

NOTE: The frequency for the required analysis of a QC check standard will depend upon the number of parameters being simultaneously tested, the complexity of the sample matrix, and the performance of the laboratory.

8.4.1 Prepare the QC check standard by adding 1.0 mL of QC check sample concentrate (Sections 8.2.1 or 8.3.2) to 1 L of reagent water. The QC check standard needs only to contain the parameters that failed criteria in the test in Section 8.3.

8.4.2 Analyze the QC check standard to determine the concentration measured (A) of each parameter. Calculate each percent recovery (P_s) as 100 (A/T)%, where T is the true value of the standard concentration.

8.4.3 Compare the percent recovery (P_s) for each parameter with the corresponding QC acceptance criteria found in Table 2. Only parameters that failed the test in Section 8.3 need to be compared with these criteria. If the recovery of any such parameter falls outside the designated range, the laboratory performance for that parameter is judged to be out of control, and the problem must be immediately identified and corrected. The analytical result for that parameter in the unspiked sample is suspect and may not be reported for regulatory compliance purposes.

8.5 As part of the QC program for the laboratory, method accuracy for wastewater samples must be assessed and records must be maintained. After the analysis of five spiked wastewater samples as in Section 8.3, calculate the average percent recovery ($\bar{P}$) and the standard deviation of the percent recovery (s_p). Express the accuracy assessment as a percent recovery interval from $\bar{P}-2s_p$ to $\bar{P}+2s_p$. If $\bar{P}$=90% and s_p=10%, for example, the accuracy interval is expressed as 70–110%. Update the accuracy assessment for each parameter on a regular basis (e.g. after each five to ten new accuracy measurements).

8.6 It is recommended that the laboratory adopt additional quality assurance practices for use with this method. The specific practices that are most productive depend upon the needs of the laboratory and the nature of the samples. Field duplicates may be analyzed to assess the precision of the environmental measurements. When doubt exists over the identification of a peak on the chromatogram, confirmatory techniques such as HPLC with a dissimilar column, gas chromatography, or mass spectrometer must be used. Whenever possible, the laboratory should analyze standard reference materials and participate in relevant performance evaluation studies.

9. Sample Collection, Preservation, and Handling

9.1 Grab samples must be collected in glass containers. Conventional sampling practices[8] should be followed, except that the bottle must not be prerinsed with sample before collection. Composite samples should be collected in refrigerated glass containers in accordance with the requirements of the program. Automatic sampling equipment must be as free as possible of Tygon tubing and other potential sources of contamination.

9.2 All samples must be iced or refrigerated at 4°C and stored in the dark from the time of collection until extraction. Both benzidine and 3,3′-dichlorobenzidine are easily oxidized. Fill the sample bottles and, if residual chlorine is present, add 80 mg of sodium thiosulfate per liter of sample and mix well. EPA Methods 330.4 and 330.5 may be used for measurement of residual chlorine.[9] Field test kits are available for this purpose. After mixing, adjust the pH of the sample to a range of 2 to 7 with sulfuric acid.

9.3 If 1,2-diphenylhydrazine is likely to be present, adjust the pH of the sample to 4.0± 0.2 to prevent rearrangement to benzidine.

9.4 All samples must be extracted within 7 days of collection. Extracts may be held up to 7 days before analysis, if stored under an inert (oxidant free) atmosphere.[2] The extract should be protected from light.

10. Sample Extraction

10.1 Mark the water meniscus on the side of the sample bottle for later determination of sample volume. Pour the entire sample into a 2–L separatory funnel. Check the pH of the sample with wide-range pH paper and adjust to within the range of 6.5 to 7.5 with sodium hydroxide solution or sulfuric acid.

10.2 Add 100 mL of chloroform to the sample bottle, seal, and shake 30 s to rinse the inner surface. (Caution: Handle chloroform in a well ventilated area.) Transfer the solvent to the separatory funnel and extract the sample by shaking the funnel for 2 min with periodic venting to release excess pressure. Allow the organic layer to separate from the water phase for a minimum of 10 min. If the emulsion interface between layers is more than one-third the volume of the solvent layer, the analyst must employ mechanical techniques to complete the phase separation. The optimum technique depends upon the sample, but may include stirring, filtration of the emulsion through glass wool, centrifugation, or other physical methods. Collect the chloroform extract in a 250-mL separatory funnel.

10.3 Add a 50-mL volume of chloroform to the sample bottle and repeat the extraction procedure a second time, combining the extracts in the separatory funnel. Perform a third extraction in the same manner.

10.4 Separate and discard any aqueous layer remaining in the 250-mL separatory funnel after combining the organic extracts. Add 25 mL of 1 M sulfuric acid and extract the sample by shaking the funnel for 2 min. Transfer the aqueous layer to a 250-mL beaker. Extract with two additional 25-mL portions of 1 M sulfuric acid and combine the acid extracts in the beaker.

10.5 Place a stirbar in the 250-mL beaker and stir the acid extract while carefully adding 5 mL of 0.4 M sodium tribasic phosphate. While monitoring with a pH meter, neutralize the extract to a pH between 6 and 7 by dropwise addition of 5 N sodium hydroxide solution while stirring the solution vigorously. Approximately 25 to 30 mL of 5 N sodium hydroxide solution will be required and it should be added over at least a 2-min period. Do not allow the sample pH to exceed 8.

10.6 Transfer the neutralized extract into a 250-mL separatory funnel. Add 30 mL of chloroform and shake the funnel for 2 min. Allow the phases to separate, and transfer the organic layer to a second 250-mL separatory funnel.

10.7 Extract the aqueous layer with two additional 20-mL aliquots of chloroform as before. Combine the extracts in the 250-mL separatory funnel.

10.8 Add 20 mL of reagent water to the combined organic layers and shake for 30 s.

10.9 Transfer the organic extract into a 100-mL round bottom flask. Add 20 mL of methanol and concentrate to 5 mL with a rotary evaporator at reduced pressure and 35 °C. An aspirator is recommended for use as the source of vacuum. Chill the receiver with ice. This operation requires approximately 10 min. Other concentration techniques may be used if the requirements of Section 8.2 are met.

10.10 Using a 9-in. Pasteur pipette, transfer the extract to a 15-mL, conical, screw-cap centrifuge tube. Rinse the flask, including the entire side wall, with 2-mL portions of methanol and combine with the original extract.

10.11 Carefully concentrate the extract to 0.5 mL using a gentle stream of nitrogen while heating in a 30 °C water bath. Dilute to 2 mL with methanol, reconcentrate to 1 mL, and dilute to 5 mL with acetate buffer. Mix the extract thoroughly. Cap the centrifuge tube and store refrigerated and protected from light if further processing will not be performed immediately. If the extract will be stored longer than two days, it should be transferred to a Teflon-sealed screw-cap vial. If the sample extract requires no further cleanup, proceed with HPLC analysis (Section 12). If the sample requires further cleanup, proceed to Section 11.

10.12 Determine the original sample volume by refilling the sample bottle to the mark and transferring the liquid to a 1,000-mL graduated cylinder. Record the sample volume to the nearest 5 mL.

11. Cleanup and Separation

11.1 Cleanup procedures may not be necessary for a relatively clean sample matrix. If particular circumstances demand the use of a cleanup procedure, the analyst first must demonstrate that the requirements of Section 8.2 can be met using the method as revised to incorporate the cleanup procedure.

12. High Performance Liquid Chromatography

12.1 Table 1 summarizes the recommended operating conditions for the HPLC. Included in this table are retention times, capacity factors, and MDL that can be achieved under these conditions. An example of the separations achieved by this HPLC column is shown in Figure 1. Other HPLC columns, chromatographic conditions, or detectors may be used if the requirements of Section 8.2 are met. When the HPLC is idle, it is advisable to maintain a 0.1 mL/min flow through the column to prolong column life.

12.2 Calibrate the system daily as described in Section 7.

12.3 If the internal standard calibration procedure is being used, the internal standard must be added to the sample extract and mixed thoroughly immediately before injection into the instrument.

12.4 Inject 5 to 25 µL of the sample extract or standard into the HPLC. If constant volume injection loops are not used, record the volume injected to the nearest 0.05 µL, and the resulting peak size in area or peak height units.

12.5 Identify the parameters in the sample by comparing the retention times of the peaks in the sample chromatogram with those of the peaks in standard chromatograms. The width of the retention time window used to make identifications should be based upon measurements of actual retention time variations of standards over the course of a day. Three times the standard deviation of a retention time for a compound can be used to calculate a suggested window size; however, the experience of the analyst should weigh heavily in the interpretation of chromatograms.

12.6 If the response for a peak exceeds the working range of the system, dilute the extract with mobile phase and reanalyze.

12.7 If the measurement of the peak response for benzidine is prevented by the presence of interferences, reduce the electrode potential to +0.6 V and reanalyze. If the benzidine peak is still obscured by interferences, further cleanup is required.

13. Calculations

13.1 Determine the concentration of individual compounds in the sample.

13.1.1 If the external standard calibration procedure is used, calculate the amount of material injected from the peak response using the calibration curve or calibration factor determined in Section 7.2.2. The concentration in the sample can be calculated from Equation 2.

$$\text{Concentration } (\mu g/L) = \frac{(A)(V_t)}{(V_i)(V_s)}$$

Equation 2

where:

A=Amount of material injected (ng).
V_i=Volume of extract injected (µL).
V_t=Volume of total extract (µL).
V_s=Volume of water extracted (mL).

13.1.2 If the internal standard calibration procedure is used, calculate the concentration in the sample using the response factor (RF) determined in Section 7.3.2 and Equation 3.

$$\text{Concentration } (\mu g/L) = \frac{(A_s)(I_s)}{(A_{is})(RF)(V_o)}$$

Equation 3

where:

A_s=Response for the parameter to be measured.
A_{is}=Response for the internal standard.
I_s=Amount of internal standard added to each extract (µg).
V_o=Volume of water extracted (L).

13.2 Report results in µg/L without correction for recovery data. All QC data obtained should be reported with the sample results.

14. Method Performance

14.1 The method detection limit (MDL) is defined as the minimum concentration of a substance that can be measured and reported with 99% confidence that the value is above zero.[1] The MDL concentrations listed in Table 1 were obtained using reagent water.[10] Similar results were achieved using representative wastewaters. The MDL actually achieved in a given analysis will vary depending on instrument sensitivity and matrix effects.

14.2 This method has been tested for linearity of spike recovery from reagent water and has been demonstrated to be applicable over the concentration range from 7×MDL to 3000×MDL.[10]

14.3 This method was tested by 17 laboratories using reagent water, drinking water, surface water, and three industrial wastewaters spiked at six concentrations over the range 1.0 to 70 µg/L.[11] Single operator precision, overall precision, and method accuracy were found to be directly related to the concentration of the parameter and essentially independent of the sample matrix. Linear equations to describe these relationships are presented in Table 3.

References

1. 40 CFR part 136, appendix B.

2. ''Determination of Benzidines in Industrial and Muncipal Wastewaters,'' EPA 600/4–82–022, National Technical Information Service, PB82–196320, Springfield, Virginia 22161, April 1982.

3. ASTM Annual Book of Standards, Part 31, D3694–78. ''Standard Practices for Preparation of Sample Containers and for Preservation of Organic Constituents,'' American Society for Testing and Materials, Philadelphia.

4. ''Carcinogens—Working With Carcinogens,'' Department of Health, Education, and Welfare, Public Health Service, Center for Disease Control, National Institute for Occupational Safety and Health, Publication No. 77–206, August 1977.

5. ''OSHA Safety and Health Standards, General Industry,'' (29 CFR part 1910), Occupational Safety and Health Administration, OSHA 2206 (Revised, January 1976).

6. ''Safety in Academic Chemistry Laboratories,'' American Chemical Society Publication, Committee on Chemical Safety, 3rd Edition, 1979.

7. Provost, L.P., and Elder, R.S. ''Interpretation of Percent Recovery Data,'' *American*

Laboratory, *15*, 58–63 (1983). (The value 2.44 used in the equation in Section 8.3.3 is two times the value 1.22 derived in this report.)

8. ASTM Annual Book of Standards, Part 31, D3370–76. ''Standard Practices for Sampling Water,'' American Society for Testing and Materials, Philadelphia.

9. ''Methods 330.4 (Titrimetric, DPD–FAS) and 330.5 (Spectrophotometric, DPD) for Chlorine Total Residual,'' Methods for Chemical Analysis of Water and Wastes, EPA–600/4–79–020, U.S. Environmental Protection Agency, Environmental Monitoring and Support Laboratory, Cincinnati, Ohio 45268, March 1979.

10. ''EPA Method Study 15, Method 605 (Benzidines),'' EPA 600/4–84–062, National Technical Information Service, PB84–211176, Springfield, Virginia 22161, June 1984.

11. ''EPA Method Validation Study 15, Method 605 (Benzidines),'' Report for EPA Contract 68–03–2624 (In preparation).

TABLE 1—CHROMATOGRAPHIC CONDITIONS AND METHOD DETECTION LIMITS

Parameter	Retention time (min)	Column capacity factor (k′)	Method detection limit (μg/L)
Benzidine	6.1	1.44	0.08
3,3′-Dichlorobenzidine	12.1	3.84	0.13

HPLC Column conditions: Lichrosorb RP–2, 5 micron particle size, in a 25 cm×4.6 mm ID stainless steel column. Mobile Phase: 0.8 mL/min of 50% acetonitrile/50% 0.1M pH 4.7 acetate buffer. The MDL were determined using an electrochemical detector operated at +0.8 V.

TABLE 2—QC ACCEPTANCE CRITERIA—METHOD 605

Parameter	Test conc. (μg/L)	Limit for s (μg/L)	Range for X (μg/L)	Range for P, P_s (percent)
Benzidine	50	18.7	9.1–61.0	D–140
3.3′-Dichlorobenzidine	50	23.6	18.7–50.0	5–128

$\underline{s}$=Standard deviation of four recovery measurements, in μg/L (Section 8.2.4).
$\bar{X}$=Average recovery for four recovery measurements, in μg/L (Section 8.2.4).
P, P_s=Percent recovery measured (Section 8.3.2, Section 8.4.2).
D=Detected; result must be greater than zero.

Note: These criteria are based directly upon the method performance data in Table 3. Where necessary, the limits for recovery have been broadened to assure applicability of the limits to concentrations below those used to develop Table 3.

TABLE 3—METHOD ACCURACY AND PRECISION AS FUNCTIONS OF CONCENTRATION—METHOD 605

Parameter	Accuracy, as recovery, X′(μg/L)	Single analyst precision, s_r' (μg/L)	Overall precision, S′ (μg/L)
Benzidine	0.70C+0.06	$0.28\bar{X}+0.19$	$0.40\bar{X}+0.18$
3,3′-Dichlorobenzidine	0.66C+0.23	$0.39\bar{X}-0.05$	$0.38\bar{X}+0.02$

X′=Expected recovery for one or more measurements of a sample containing a concentration of C, in μg/L.
s_r'=Expected single analyst standard deviation of measurements at an average concentration found of $\bar{X}$, in μg/L.
S′=Expected interlaboratory standard deviation of measurements at an average concentration found of $\bar{X}$, in μg/L.
$\underline{C}$=True value for the concentration, in μg/L.
$\bar{X}$=Average recovery found for measurements of samples containing a concentration of C, in μg/L.

COLUMN: LICHROSORB RP-2
MOBILE PHASE: 50% ACETONITRILE IN ACETATE BUFFER
DETECTOR: ELECTROCHEMICAL AT + 0.8 V

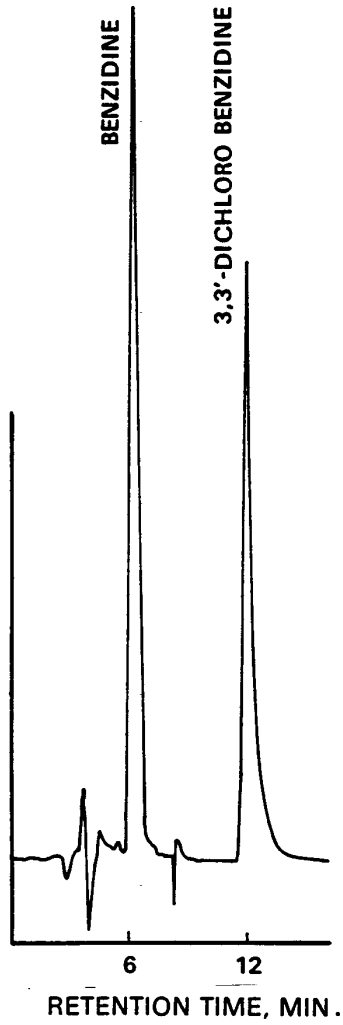


Figure 1. Liquid chromatogram of benzidines.

METHOD 606—PHTHALATE ESTER

1. Scope and Application

1.1 This method covers the determination of certain phthalate esters. The following parameters can be determined by this method:

Parameter	STORET No.	CAS No.
Bis(2-ethylhexyl) phthalate	39100	117–81–7
Butyl benzyl phthalate	34292	85–68–7
Di-n-butyl phthalate	39110	84–74–2
Diethyl phthalate	34336	84–66–2
Dimethyl phthalate	34341	131–11–3
Di-n-octyl phthalate	34596	117–84–0

1.2 This is a gas chromatographic (GC) method applicable to the determination of the compounds listed above in municipal and industrial discharges as provided under 40 CFR 136.1. When this method is used to analyze unfamiliar samples for any or all of the compounds above, compound identifications should be supported by at least one additional qualitative technique. This method describes analytical conditions for a second gas chromatographic column that can be used to confirm measurements made with the primary column. Method 625 provides gas chromatograph/mass spectrometer (GC/MS) conditions appropriate for the qualitative and quantitative confirmation of results for all of the parameters listed above, using the extract produced by this method.

1.3 The method detection limit (MDL, defined in Section 14.1)[1] for each parameter is listed in Table 1. The MDL for a specific wastewater may differ from those listed, depending upon the nature of interferences in the sample matrix.

1.4 The sample extraction and concentration steps in this method are essentially the same as in Methods 608, 609, 611, and 612. Thus, a single sample may be extracted to measure the parameters included in the scope of each of these methods. When cleanup is required, the concentration levels must be high enough to permit selecting aliquots, as necessary, to apply appropriate cleanup procedures. The analyst is allowed the latitude, under Section 12, to select chromatographic conditions appropriate for the simultaneous measurement of combinations of these parameters.

1.5 Any modification of this method, beyond those expressly permitted, shall be considered as a major modification subject to application and approval of alternate test procedures under 40 CFR 136.4 and 136.5.

1.6 This method is restricted to use by or under the supervision of analysts experienced in the use of a gas chromatograph and in the interpretation of gas chromatograms. Each analyst must demonstrate the ability to generate acceptable results with this method using the procedure described in Section 8.2.

2. Summary of Method

2.1 A measured volume of sample, approximately 1–L, is extracted with methylene chloride using a separatory funnel. The methylene chloride extract is dried and exchanged to hexane during concentration to a volume of 10 mL or less. The extract is separated by gas chromatography and the phthalate esters are then measured with an electron capture detector.[2]

2.2 Analysis for phthalates is especially complicated by their ubiquitous occurrence in the environment. The method provides Florisil and alumina column cleanup procedures to aid in the elimination of interferences that may be encountered.

3. Interferences

3.1 Method interferences may be caused by contaminants in solvents, reagents, glassware, and other sample processing hardware that lead to discrete artifacts and/or elevated baselines in gas chromatograms. All of these materials must be routinely demonstrated to be free from interferences under the conditions of the analysis by running laboratory reagent blanks as described in Section 8.1.3.

3.1.1 Glassware must be scrupulously cleaned.[3] Clean all glassware as soon as possible after use by rinsing with the last solvent used in it. Solvent rinsing should be followed by detergent washing with hot water, and rinses with tap water and distilled water. The glassware should then be drained dry, and heated in a muffle furnace at 400 °C for 15 to 30 min. Some thermally stable materials, such as PCBs, may not be eliminated by this treatment. Solvent rinses with acetone and pesticide quality hexane may be substituted for the muffle furnace heating. Thorough rinsing with such solvents usually eliminates PCB interference. Volumetric ware should not be heated in a muffle furnace. After drying and cooling, glassware should be sealed and stored in a clean environment to prevent any accumulation of dust or other contaminants. Store inverted or capped with aluminum foil.

3.1.2 The use of high purity reagents and solvents helps to minimize interference problems. Purification of solvents by distillation in all-glass systems may be required.

3.2 Phthalate esters are contaminants in many products commonly found in the laboratory. It is particularly important to avoid the use of plastics because phthalates are commonly used as plasticizers and are easily extracted from plastic materials. Serious phthalate contamination can result at any time, if consistent quality control is not practiced. Great care must be experienced to prevent such contamination. Exhaustive cleanup of reagents and glassware may be required to eliminate background phthalate contamination.[4,5]

3.3 Matrix interferences may be caused by contaminants that are co-extracted from the sample. The extent of matrix interferences will vary considerably from source to source, depending upon the nature and diversity of the industrial complex or municipality being sampled. The cleanup procedures in Section 11 can be used to overcome many of these interferences, but unique samples may require additional cleanup approaches to achieve the MDL listed in Table 1.

4. Safety

4.1 The toxicity or carcinogenicity of each reagent used in this method has not been precisely defined; however, each chemical compound should be treated as a potential health hazard. From this viewpoint, exposure to these chemicals must be reduced to the lowest possible level by whatever means available. The laboratory is responsible for maintaining a current awareness file of OSHA regulations regarding the safe handling of the chemicals specified in this method. A reference file of material data handling sheets should also be made available to all personnel involved in the chemical analysis. Additional references to laboratory safety are available and have been identified [6–8] for the information of the analyst.

5. Apparatus and Materials

5.1 Sampling equipment, for discrete or composite sampling.

5.1.1 Grab sample bottle—1–L or 1–qt, amber glass, fitted with a screw cap lined with Teflon. Foil may be substituted for Teflon if the sample is not corrosive. If amber bottles are not available, protect samples from light. The bottle and cap liner must be washed, rinsed with acetone or methylene chloride, and dried before use to minimize contamination.

5.1.2 Automatic sampler (optional)—The sampler must incorporate glass sample containers for the collection of a minimum of 250 mL of sample. Sample containers must be kept refrigerated at 4 °C and protected from light during compositing. If the sampler uses a peristaltic pump, a minimum length of compressible silicone rubber tubing may be used. Before use, however, the compressible tubing should be thoroughly rinsed with methanol, followed by repeated rinsings with distilled water to minimize the potential for contamination of the sample. An integrating flow meter is required to collect flow proportional composites.

5.2 Glassware (All specifications are suggested. Catalog numbers are included for illustration only).

5.2.1 Separatory funnel—2–L, with Teflon stopcock.

5.2.2 Drying column—Chromatographic column, approximately 400 mm long × 19 mm ID, with coarse frit filter disc.

5.2.3 Chromatographic column—300 mm long × 10 mm ID, with Teflon stopcock and coarse frit filter disc at bottom (Kontes K–420540–0213 or equivalent).

5.2.4 Concentrator tube, Kuderna-Danish—10–mL, graduated (Kontes K–570050–1025 or equivalent). Calibration must be checked at the volumes employed in the test. Ground glass stopper is used to prevent evaporation of extracts.

5.2.5 Evaporative flask, Kuderna-Danish—500–mL (Kontes K–570001–0500 or equivalent). Attach to concentrator tube with springs.

5.2.6 Snyder column, Kuderna-Danish—Three-ball macro (Kontes K–503000–0121 or equivalent).

5.2.7 Snyder column, Kuderna-Danish—Two-ball micro (Kontes K–569001–0219 or equivalent).

5.2.8 Vials—10 to 15–mL, amber glass, with Teflon-lined screw cap.

5.3 Boiling chips—Approximately 10/40 mesh. Heat to 400 °C for 30 min or Soxhlet extract with methylene chloride.

5.4 Water bath—Heated, with concentric ring cover, capable of temperature control (±2 °C). The bath should be used in a hood.

5.5 Balance—Analytical, capable of accurately weighing 0.0001 g.

5.6 Gas chromatograph—An analytical system complete with gas chromatograph suitable for on-column injection and all required accessories including syringes, analytical columns, gases, detector, and strip-chart recorder. A data system is recommended for measuring peak areas.

5.6.1 Column 1—1.8 m long × 4 mm ID glass, packed with 1.5% SP–2250/1.95% SP–2401 Supelcoport (100/120 mesh) or equivalent. This column was used to develop the method performance statemelts in Section 14. Guidelines for the use of alternate column packings are provided in Section 12.1.

5.6.2 Column 2—1.8 m long × 4 mm ID glass, packed with 3% OV–1 on Supelcoport (100/120 mesh) or equivalent.

5.6.3 Detector—Electron capture detector. This detector has proven effective in the analysis of wastewaters for the parameters listed in the scope (Section 1.1), and was used to develop the method performance statements in Section 14. Guidelines for the use of alternate detectors are provided in Section 12.1.

6. Reagents

6.1 Reagent water—Reagent water is defined as a water in which an interferent is not observed at the MDL of the parameters of interest.

6.2 Acetone, hexane, isooctane, methylene chloride, methanol—Pesticide quality or equivalent.

6.3 Ethyl ether—nanograde, redistilled in glass if necessary.

6.3.1 Ethyl ether must be shown to be free of peroxides before it is used as indicated by

EM Laboratories Quant test strips. (Available from Scientific Products Co., Cat. No. P1126–8, and other suppliers.)

6.3.2 Procedures recommended for removal of peroxides are provided with the test strips. After cleanup, 20 mL of ethyl alcohol preservative must be added to each liter of ether.

6.4 Sodium sulfate—(ACS) Granular, anhydrous. Several levels of purification may be required in order to reduce background phthalate levels to an acceptable level: 1) Heat 4 h at 400 °C in a shallow tray, 2) Heat 16 h at 450 to 500 °C in a shallow tray, 3) Soxhlet extract with methylene chloride for 48 h.

6.5 Florisil—PR grade (60/100 mesh). Purchase activated at 1250 °F and store in the dark in glass containers with ground glass stoppers or foil-lined screw caps. To prepare for use, place 100 g of Florisil into a 500-mL beaker and heat for approximately 16 h at 40 °C. After heating transfer to a 500-mL reagent bottle. Tightly seal and cool to room temperature. When cool add 3 mL of reagent water. Mix thoroughly by shaking or rolling for 10 min and let it stand for at least 2 h. Keep the bottle sealed tightly.

6.6 Alumina—Neutral activity Super I, W200 series (ICN Life Sciences Group, No. 404583). To prepare for use, place 100 g of alumina into a 500-mL beaker and heat for approximately 16 h at 400 °C. After heating transfer to a 500-mL reagent bottle. Tightly seal and cool to room temperature. When cool add 3 mL of reagent water. Mix thoroughly by shaking or rolling for 10 min and let it stand for at least 2 h. Keep the bottle sealed tightly.

6.7 Stock standard solutions (1.00 µg/µL)—Stock standard solutions can be prepared from pure standard materials or purchased as certified solutions.

6.7.1 Prepare stock standard solutions by accurately weighing about 0.0100 g of pure material. Dissolve the material in isooctane and dilute to volume in a 10-mL volumetric flask. Larger volumes can be used at the convenience of the analyst. When compound purity is assayed to be 96% or greater, the weight can be used without correction to calculate the concentration of the stock standard. Commercially prepared stock standards can be used at any concentration if they are certified by the manufacturer or by an independent source.

6.7.2 Transfer the stock standard solutions into Teflon-sealed screw-cap bottles. Store at 4 °C and protect from light. Stock standard solutions should be checked frequently for signs of degradation or evaporation, especially just prior to preparing calibration standards from them.

6.7.3 Stock standard solutions must be replaced after six months, or sooner if comparison with check standards indicates a problem.

6.8 Quality control check sample concentrate—See Section 8.2.1.

7. Calibration

7.1 Establish gas chromatograph operating conditions equivalent to those given in Table 1. The gas chromatographic system can be calibrated using the external standard technique (Section 7.2) or the internal standard technique (Section 7.3).

7.2 External standard calibration procedure:

7.2.1 Prepared calibration standards at a minimum of three concentration levels for each parameter of interest by adding volumes of one or more stock standards to a volumetric flask and diluting to volume with isooctane. One of the external standards should be at a concentration near, but above, the MDL (Table 1) and the other concentrations should correspond to the expected range of concentrations found in real samples or should define the working range of the detector.

7.2.2 Using injections of 2 to 5 µL, analyze each calibration standard according to Section 12 and tabulate peak height or area responses against the mass injected. The results can be used to prepare a calibration curve for each compound. Alternatively, if the ratio of response to amount injected (calibration factor) is a constant over the working range (< 10% relative standard deviation, RSD), linearity through the origin can be assumed and the average ratio or calibration factor can be used in place of a calibration curve.

7.3 Internal standard calibration procedure—To use this approach, the analyst must select one or more internal standards that are similar in analytical behavior to the compounds of interest. The analyst must further demonstrate that the measurement of the internal standard is not affected by method or matrix interferences. Because of these limitations, no internal standard can be suggested that is applicable to all samples.

7.3.1 Prepare calibration standards at a minimum of three concentration levels for each parameter of interest by adding volumes of one or more stock standards to a volumetric flash. To each calibration standard, add a known constant amount of one or more internal standards, and dilute to volume with isooctane. One of the standards should be at a concentraton near, but above, the MDL and the other concentrations should correspond to the expected range of concentrations found in real samples or should define the working range of the detector.

7.3.2 Using injections of 2 to 5 µL, analyze each calibration standard according to Section 12 and tabulate peak height or area responses against concentration for each compound and internal standard. Calculate

response factors (RF) for each compound using Equation 1.

$$RF = \quad (A_s)(C_{is}) \quad (A_{is})(C_s)$$

Equation 1

where:

A_s=Response for the parameter to be measured.

A_{is}=Response for the internal standard.

C_{is}=Concentration of the internal standard (µg/L).

C_s=Concentration of the parameter to be measured (µg/L).

If the RF value over the working range is a constant (< 10% RSD), the RF can be assumed to be invariant and the average RF can be used for calculations. Alternatively, the results can be used to plot a calibration curve of response ratios, A_s/A_{is}, vs. RF.

7.4 The working calibration curve, calibration factor, or RF must be verified on each working day by the measurement of one or more calibration standards. If the response for any parameter varies from the predicted response by more than ±15%, a new calibration curve must be prepared for that compound.

7.5 Before using any cleanup procedure, the analyst must process a series of calibration standards through the procedure to validate elution patterns and the absence of interferences from the reagents.

8. Quality Control

8.1 Each laboratory that uses this method is required to operate a formal quality control program. The minimum requirements of this program consist of an initial demonstration of laboratory capability and an ongoing analysis of spiked samples to evaluate and document data quality. The laboratory must maintain records to document the quality of data that is generated. Ongoing data quality checks are compared with established performance criteria to determine if the results of analyses meet the performance characteristics of the method. When results of sample spikes indicate atypical method performance, a quality control check standard must be analyzed to confirm that the measurements were performed in an in-control mode of operation.

8.1.1 The analyst must make an initial, one-time, demonstration of the ability to generate acceptable accuracy and precision with this method. This ability is established as described in Section 8.2.

8.1.2 In recognition of advances that are occurring in chromatography, the analyst is permitted certain options (detailed in Sections 10.4, 11.1, and 12.1) to improve the separations or lower the cost of measurements. Each time such a modification is made to the method, the analyst is required to repeat the procedure in Section 8.2.

8.1.3 Before processing any samples, the analyst must analyze a reagent water blank to demonstrate that interferences from the analytical system and glassware are under control. Each time a set of samples is extracted or reagents are changed, a reagent water blank must be processed as a safeguard against laboratory contamination.

8.1.4 The laboratory must, on an ongoing basis, spike and analyze a minimum of 10% of all samples to monitor and evaluate laboratory data quality. This procedure is described in Section 8.3.

8.1.5 The laboratory must, on an ongoing basis, demonstrate through the analyses of quality control check standards that the operation of the measurement system is in control. This procedure is described in Section 8.4. The frequency of the check standard analyses is equivalent to 10% of all samples analyzed but may be reduced if spike recoveries from samples (Section 8.3) meet all specified quality control criteria.

8.1.6 The laboratory must maintain performance records to document the quality of data that is generated. This procedure is described in Section 8.5.

8.2 To establish the ability to generate acceptable accuracy and precision, the analyst must perform the following operations.

8.2.1 A quality contrml (QC) check sample concentrate is required containing each parameter of interest at the following concentrations in acetone: butyl benzyl phthalate, 10 µg/mL; bis(2-ethylhexyl) phthalate, 50 µg/mL; di-n-octyl phthalate, 50 µg/mL; any other phthlate, 25 µg/mL. The QC check sample concentrate must be obtained from the U.S. Environmental Protection Agancy, Environmental Monitoring and Support Laboratory in Cincinnati, Ohio, if available. If not available from that source, the QC check sample concentrate must be obtained from another external source. If not available from either source above, the QC check sample concentrate must be prepared by the laboratory using stock standards prepared independently from those used for calibration.

8.2.2 Using a pipet, prepare QC check samples at the test concentrations shown in Table 2 by adding 1.00 mL of QC check sample concentrate to each of four 1–L aliquots of reagent water.

8.2.3 Analyze the well-mixed QC check samples according to the method beginning in Section 10.

8.2.4 Calculate the average recovery ($\bar{X}$) in µg/L, and the standard deviation of the recovery (s) in µg/L, for each parameter using the four results.

8.2.5 For each parameter compare s and $\bar{X}$ with the corresponding acceptance criteria for precision and accuracy, respectively, found in Table 2. If s and $\bar{X}$ for all parameters of interest meet the acceptance criteria, the system performance is acceptable and analysis of actual samples can begin. If

any individual s exceeds the precision limit or any individual X falls outside the range for accuracy, the system performance is unacceptable for that parameter. Locate and correct the source of the problem and repeat the test for all parameters of interest beginning with Section 8.2.2.

8.3 The laboratory must, on an ongoing basis, spike at least 10% of the samples from each sample site being monitored to assess accuracy. For laboratories analyzing one to ten samples per month, at least one spiked sample per month is required.

8.3.1 The concentration of the spike in the sample should be determined as follows:

8.3.1.1 If, as in compliance monitoring, the concentration of a specific parameter in the sample is being checked against a regulatory concentration limit, the spike should be at that limit or 1 to 5 times higher than the background concentration determined in Section 8.3.2, whichever concentration would be larger.

8.3.1.2 If the concentration of a specific parameter in the sample is not being checked against a limit specific to that parameter, the spike should be at the test concentration in Section 8.2.2 or 1 to 5 times higher than the background concentration determined in Section 8.3.2, whichever concentration would be larger.

8.3.1.3 If it is impractical to determine background levels before spiking (e.g., maximum holding times will be exceeded), the spike concentration should be (1) the regulatory concentration limit, if any; or, if none (2) the larger of either 5 times higher than the expected background concentration or the test concentration in Section 8.2.2.

8.3.2 Analyze one sample aliquot to determine the background concentration (B) of each parameter. If necessary, prepare a new QC check sample concentrate (Section 8.2.1) appropriate for the background concentrations in the sample. Spike a second sample aliquot with 1.0 mL of the QC check sample concentrate and analyze it to determine the concentration after spiking (A) of each parameter. Calculate each percent recovery (P) as 100(A-B)%/T, where T is the known true value of the spike.

8.3.3 Compare the percent recovery (P) for each parameter with the corresponding QC acceptance criteria found in Table 2. These acceptance criteria were calculated to include an allowance for error in measurement of both the background and spike concentrations, assuming a spike to background ratio of 5:1. This error will be accounted for to the extent that the analyst's spike to background ratio approaches 5:1.[9] If spiking was performed at a concentration lower than the test concentration in Section 8.2.2, the analyst must use either the QC acceptance criteria in Table 2, or optional QC acceptance criteria calculated for the specific spike concentration. To calculate optional acceptance criteria for the recovery of a parameter: (1) Calculate accuracy (X′) using the equation in Table 3, substituting the spike concentration (T) for C; (2) calculate overall precision (S′) using the equation in Table 3, substituting X′ for $\bar{X}$; (3) calculate the range for recovery at the spike concentration as (100 X′/T)±2.44(100 S′/T)%.[9]

8.3.4 If any individual P falls outside the designated range for recovery, that parameter has failed the acceptance criteria. A check standard containing each parameter that failed the criteria must be analyzed as described in Section 8.4.

8.4 If any parameter fails the acceptance criteria for recovery in Section 8.3, a QC check standard containing each parameter that failed must be prepared and analyzed.

NOTE: The frequency for the required analysis of a QC check standard will depend upon the number of parameters being simultaneously tested, the complexity of the sample matrix, and the performance of the laboratory.

8.4.1 Prepare the QC check standard by adding 1.0 mL of QC check sample concentrate (Section 8.2.1 or 8.3.2) to 1 L of reagent water. The QC check standard needs only to contain the parameters that failed criteria in the test in Section 8.3.

8.4.2 Analyze the QC check standard to determine the concentration measured (A) of each parameter. Calculate each percent recovery (P_s) as 100 (A/T)%, where T is the true value of the standard concentration.

8.4.3 Compare the percent recovery (P_s) for each parameter with the corresponding QC acceptance criteria found in Table 2. Only parameters that failed the test in Section 8.3 need to be compared with these criteria. If the recovery of any such parameter falls outside the designated range, the laboratory performance for that parameter is judged to be out of control, and the problem must be immediately identified and corrected. The analytical result for that parameter in the unspiked sample is suspect and may not be reported for regulatory compliance purposes.

8.5 As part of the QC program for the laboratory, method accuracy for wastewater samples must be assessed and records must be maintained. After the analysis of five spiked wastewater samples as in Section 8.3, calculate the average percent recovery ($\bar{P}$) and the standard deviation of the percent recovery (s_p). Express the accuracy assessment as a percent recovery interval from $\bar{P}-2s_p$ to $\bar{P}+2s_p$. If $\bar{P}$=90% and s_p=10%, for example, the accuracy interval is expressed as 70–110%. Update the accuracy assessment for each parameter on a regular basis (e.g. after each five to ten new accuracy measurements).

8.6 It is recommended that the laboratory adopt additional quality assurance practices for use with this method. The specific practices that are most productive depend upon the needs of the laboratory and

the nature of the samples. Field duplicates may be analyzed to assess the precision of the environmental measurements. When doubt exists over the identification of a peak on the chromatogram, confirmatory techniques such as gas chromatography with a dissimilar column, specific element detector, or mass spectrometer must be used. Whenever possible, the laboratory should analyze standard reference materials and participate in relevant performance evaluation studies.

9. Sample Collection, Preservation, and Handling

9.1 Grab samples must be collected in glass containers. Conventional sampling practices[10] should be followed, except that the bottle must not be prerinsed with sample before collection. Composite samples should be collected in refrigerated glass containers in accordance with the requirements of the program. Automatic sampling equipment must be as free as possible of Tygon tubing and other potential sources of contamination.

9.2 All samples must be iced or refrigerated at 4 °C from the time of collection until extraction.

9.3 All samples must be extracted within 7 days of collection and completely analyzed within 40 days of extraction.[2]

10. Sample Extraction

10.1 Mark the water meniscus on the side of the sample bottle for later determination of sample volume. Pour the entire sample into a 2–L separatory funnel.

10.2 Add 60 mL of methylene chloride to the sample bottle, seal, and shake 30 s to rinse the inner surface. Transfer the solvent to the separatory funnel and extract the sample by shaking the funnel for 2 min. with periodic venting to release excess pressure. Allow the organic layer to separate from the water phase for a minimum of 10 min. If the emulsion interface between layers is more than one-third the volume of the solvent layer, the analyst must employ mechanical techniques to complete the phrase separation. The optimum technique depends upon the sample, but may include stirring, filtration of the emulsion through glass wool, centrifugation, or other physical methods. Collect the methylene chloride extract in a 250-mL Erlenmeyer flask.

10.3 Add a second 60-mL volume of methylene chloride to the sample bottle and repeat the extraction procedure a second time, combining the extracts in the Erlenmeyer flask. Perform a third extraction in the same manner.

10.4 Assemble a Kuderna-Danish (K–D) concentrator by attaching a 10-mL concentrator tube to a 500-mL evaporative flask. Other concentrator devices or techniques may be used in place of the K–D concentrator if the requirements of Section 8.2 are met.

10.5 Pour the combined extract through a solvent-rinsed drying column containing about 10 cm of anhydrous sodium sulfate, and collect the extract in the K–D concentrator. Rinse the Erlenmeyer flask and column with 20 to 30 mL of methylene chloride to complete the quantitative transfer.

10.6 Add one or two clean boiling chips to the evaporative flask and attach a three-ball Snyder column. Prewet the Snyder column by adding about 1 mL of methylene chloride to the top. Place the K–D apparatus on a hot water bath (60 to 65 °C) so that the concentrator tube is partially immersed in the hot water, and the entire lower rounded surface of the flask is bathed with hot vapor. Adjust the vertical position of the apparatus and the water temperature as required to complete the concentration in 15 to 20 min. At the proper rate of distillation the balls of the column will actively chatter but the chambers will not flood with condensed solvent. When the apparent volume of liquid reaches 1 mL, remove the K–D apparatus and allow it to drain and cool for at least 10 min.

10.7 Increase the temperature of the hot water bath to about 80 °C. Momentarily remove the Snyder column, add 50 mL of hexane and a new boiling chip, and reattach the Snyder column. Concentrate the extract as in Section 10.6, except use hexane to prewet the column. The elapsed time of concentration should be 5 to 10 min.

10.8 Remove the Snyder column and rinse the flask and its lower joint into the concentrator tube with 1 to 2 mL of hexane. A 5-mL syringe is recommended for this operation. Adjust the extract volume to 10 mL. Stopper the concentrator tube and store refrigerated if further processing will not be performed immediately. If the extract will be stored longer than two days, it should be transferred to a Teflon-sealed screw-cap vial. If the sample extract requires no further cleanup, proceed with gas chromatographic analysis (Section 12). If the sample requires further cleanup, proceed to Section 11.

10.9 Determine the original sample volume by refilling the sample bottle to the mark and transferring the liquid to a 1000-mL graduated cylinder. Record the sample volume to the nearest 5 mL.

11. Cleanup and Separation

11. Cleanup procedures may not be necessary for a relatively clean sample matrix. If particular circumstances demand the use of a cleanup procedure, the analyst may use either procedure below or any other appropriate procedure. However, the analyst first must demonstrate that the requirements of Section 8.2 can be met using the method as revised to incorporate the cleanup procedure.

11.2 If the entire extract is to be cleaned up by one of the following procedures, it must be concentrated to 2.0 mL. To the concentrator tube in Section 10.8, add a clean boiling chip and attach a two-ball micro-Snyder column. Prewet the column by adding about 0.5 mL of hexane to the top. Place the micro-K–D apparatus on a hot water bath (80 °C) so that the concentrator tube is partially immersed in the hot water. Adjust the vertical position of the apparatus and the water temperature as required to complete the concentration in 5 to 10 min. At the proper rate of distillation the balls of the column will actively chatter but the chambers will not flood. When the apparent volume of liquid reaches about 0.5 mL, remove the K–D apparatus and allow it to drain and cool for at least 10 min. Remove the micro-Snyder column and rinse its lower joint into the concentrator tube with 0.2 mL of hexane. Adjust the final volume to 2.0 mL and proceed with one of the following cleanup procedures.

11.3 Florisil column cleanup for phthalate esters:

11.3.1 Place 10 g of Florisil into a chromatographic column. Tap the column to settle the Florisil and add 1 cm of anhydrous sodium sulfate to the top.

11.3.2 Preelute the column with 40 mL of hexane. The rate for all elutions should be about 2 mL/min. Discard the eluate and just prior to exposure of the sodium sulfate layer to the air, quantitatively transfer the 2-mL sample extract onto the column using an additional 2 mL of hexane to complete the transfer. Just prior to exposure of the sodium sulfate layer to the air, add 40 mL of hexane and continue the elution of the column. Discard this hexane eluate.

11.3.3 Next, elute the column with 100 mL of 20% ethyl ether in hexane (V/V) into a 500-mL K–D flask equipped with a 10-mL concentrator tube. Concentrate the collected fraction as in Section 10.6. No solvent exchange is necessary. Adjust the volume of the cleaned up extract to 10 mL in the concentrator tube and analyze by gas chromatography (Section 12).

11.4 Alumina column cleanup for phthalate esters:

11.4.1 Place 10 g of alumina into a chromatographic column. Tap the column to settle the alumina and add 1 cm of anhydrous sodium sulfate to the top.

11.4.2 Preelute the column with 40 mL of hexane. The rate for all elutions should be about 2 mL/min. Discard the eluate and just prior to exposure of the sodium sulfate layer to the air, quantitatively transfer the 2-mL sample extract onto the column using an additional 2 mL of hexane to complete the transfer. Just prior to exposure of the sodium sulfate layer to the air, add 35 mL of hexane and continue the elution of the column. Discard this hexane eluate.

11.4.3 Next, elute the column with 140 mL of 20% ethyl ether in hexane (V/V) into a 500-mL K–D flask equipped with a 10–mL concentrator type. Concentrate the collected fraction as in Section 10.6. No solvent exchange is necessary. Adjust the volume of the cleaned up extract to 10 mL in the concentrator tube and analyze by gas chromatography (Section 12).

12. Gas Chromatography

12.1 Table 1 summarizes the recommended operating conditions for the gas chromatograph. Included in this table are retention times and MDL that can be achieved under these conditions. Examples of the separations achieved by Column 1 are shown in Figures 1 and 2. Other packed or capillary (open-tubular) columns, chromatographic conditions, or detectors may be used if the requirements of Section 8.2 are met.

12.2 Calibrate the system daily as described in Section 7.

12.3 If the internal standard calibration procedure is being used, the internal staldard must be added to the sample extract and mixed thoroughly immediately before injection into the gas chromatograph.

12.4 Inject 2 to 5 µL of the sample extract or standard into the gas-chromatograph using the solvent-flush technique.[11] Smaller (1.0 µL) volumes may be injected if automatic devices are employed. Record the volume injected to the nearest 0.05 µL, and the resulting peak size in area or peak height units.

12.5 Identify the parameters in the sample by comparing the retention times of the peaks in the sample chromatogram with those of the peaks in standard chromatograms. The width of the retention time window used to make identifications should be based upon measurements of actual retention time variations of standards over the course of a day. Three times the standard deviation of a retention time for a compound can be used to calculate a suggested window size; however, the experience of the analyst should weigh heavily in the interpretation of chromatograms.

12.6 If the response for a peak exceeds the working range of the system, dilute the extract and reanalyze.

12.7 If the measurement of the peak response is prevented by the presence of interferences, further cleanup is required.

13. Calculations

13.1 Determine the concentration of individual compounds in the sample.

13.1.1 If the external standard calibration procedure is used, calculate the amount of material injected from the peak response using the calibration curve or calibration

factor determined in Section 7.2.2. The concentration in the sample can be calculated from Equation 2.

$$\text{Concentration } (\mu g/L) = \frac{(A)(V_t)}{(V_i)(V_s)}$$

Equation 2

where:

A=Amount of material injected (ng).
V_i=Volume of extract injected (µL).
V_t=Volume of total extract (µL).
V_s=Volume of water extracted (mL).

13.1.2 If the internal standard calibration procedure is used, calculate the concentration in the sample using the response factor (RF) determined in Section 7.3.2 and Equation 3.

$$\text{Concentration } (\mu g/L) = \frac{(A_s)(I_s)}{(A_{is})(RF)(V_o)}$$

Equation 3

where:

A_s=Response for the parameter to be measured.
A_{is}=Response for the internal standard.
I_s=Amount of internal standard added to each extract (µg).
V_o=Volume of water extracted (L).

13.2 Report results in µg/L without correction for recovery data. All QC data obtained should be reported with the sample results.

14. Method Performance

14.1 The method detection limit (MDL) is defined as the minimum concentration of a substance that can be measured and reported with 99% confidence that the value is above zero.[1] The MDL concentrations listed in Table 1 were obtained using reagent water.[12] Similar results were achieved using representative wastewaters. The MDL actually achieved in a given analysis will vary depending on instrument sensitivity and matrix effects.

14.2 This method has been tested for linearity of spike recovery from reagent water and has been demonstrated to be applicable over the concentration range from 5 × MDL to 1000 × MDL with the following exceptions: dimethyl and diethyl phthalate recoveries at 1000 × MDL were low (70%); bis-2-ethylhexyl and di-n-octyl phthalate recoveries at 5 × MDL were low (60%).[12]

14.3 This method was tested by 16 laboratories using reagent water, drinking water, surface water, and three industrial wastewaters spiked at six concentrations over the range 0.7 to 106 µg/L.[13] Single operator precision, overall precision, and method accuracy were found to be directly related to the concentration of the parameter and essentially independent of the sample matrix. Linear equations to describe these relationships are presented in Table 3.

References

1. 40 CFR part 136, appendix B.

2. ''Determination of Phthalates in Industrial and Muncipal Wastewaters,'' EPA 600/4–81–063, National Technical Information Service, PB81–232167, Springfield, Virginia 22161, July 1981.

3. ASTM Annual Book of Standards, Part 31, D3694–78. ''Standard Practices for Preparation of Sample Containers and for Preservation of Organic Constituents,'' American Society for Testing and Materials, Philadelphia.

4. Giam, C.S., Chan, H.S., and Nef, G.S. ''Sensitive Method for Determination of Phthalate Ester Plasticizers in Open-Ocean Biota Samples,'' *Analytical Chemistry, 47,* 2225 (1975).

5. Giam, C.S., and Chan, H.S. ''Control of Blanks in the Analysis of Phthalates in Air and Ocean Biota Samples,'' U.S. National Bureau of Standards, Special Publication 442, pp. 701–708, 1976.

6. ''Carcinogens—Working with Carcinogens,'' Department of Health, Education, and Welfare, Public Health Service, Center for Disease Control, National Institute for Occupational Safety and Health, Publication No. 77–206, August 1977.

7. ''OSHA Safety and Health Standards, General Industry,'' (29 CFR part 1910), Occupational Safety and Health Administration, OSHA 2206 (Revised, January 1976).

8. ''Safety in Academic Chemistry Laboratories,'' American Chemical Society Publication, Committee on Chemical Safety, 3rd Edition, 1979.

9. Provost L.P., and Elder, R.S. ''Interpretation of Percent Recovery Data,'' *American Laboratory, 15,* 58–63 (1983). (The value 2.44 used in the equation in Section 8.3.3 is two times the value 1.22 derived in this report.)

10. ASTM Annual Book of Standards, Part 31, D3370–76. ''Standard Practices for Sampling Water,'' American Society for Testing and Materials, Philadelphia.

11. Burke, J.A. ''Gas Chromatography for Pesticide Residue Analysis; Some Practical Aspects,'' *Journal of the Association of Official Analytical Chemists, 48,* 1037 (1965).

12. ''Method Detection Limit and Analytical Curve Studies, EPA Methods 606, 607, and 608,'' Special letter report for EPA Contract 68–03–2606, U.S. Environmental Protection Agency, Environmental Monitoring and Support Laboratory, Cincinnati, Ohio 45268, June 1980.

13. ''EPA Method Study 16 Method 606 (Phthalate Esters),'' EPA 600/4–84–056, National Technical Information Service, PB84–211275, Springfield, Virginia 22161, June 1984.

TABLE 1—CHROMATOGRAPHIC CONDITIONS AND METHOD DETECTION LIMITS

Parameter	Retention time (min)		Method detection limit (µg/L)
	Column 1	Column 2	
Dimethyl phthalate	2.03	0.95	0.29
Diethyl phthalate	2.82	1.27	0.49
Di-n-butyl phthalate	8.65	3.50	0.36
Butyl benzyl phthalate	[a] 6.94	[a] 5.11	0.34
Bis(2-ethylhexyl) phthalate	[a] 8.92	[a] 10.5	2.0
Di-n-octyl phthalate	[a] 16.2	[a] 18.0	3.0

Column 1 conditions: Supelcoport (100/120 mesh) coated with 1.5% SP–2250/1.95% SP–2401 packed in a 1.8 m long × 4 mm ID glass column with 5% methane/95% argon carrier gas at 60 mL/min flow rate. Column temperature held isothermal at 180°C, except where otherwise indicated.

Column 2 conditions: Supelcoport (100/120 mesh) coated with 3% OV–1 packed in a 1.8 m long × 4 mm ID glass column with 5% methane/95% argon carrier gas at 60 mL/min flow rate. Column temperature held isothermal at 200 °C, except where otherwise indicated.

[a] 220 °C column temperature.

TABLE 2—QC ACCEPTANCE CRITERIA—METHOD 606

Parameter	Test conc. (µg/L)	Limit for s (µg/L)	Range for X (µg/L)	Range for P, P_s (percent)
Bis(2-ethylhexyl) phthalate	50	38.4	1.2–55.9	D–158
Butyl benzyl phthalate	10	4.2	5.7–11.0	30–136
Di-n-butyl phthalate	25	8.9	10.3–29.6	23–136
Diethyl phthalate	25	9.0	1.9–33.4	D–149
Dimethyl phathalate	25	9.5	1.3–35.5	D–156
Di-n-octyl phthalate	50	13.4	D–50.0	D–114

$\underline{s}$=Standard deviation of four recovery measurements, in µg/L (Section 8.2.4).
$\bar{X}$=Average recovery for four recovery measurements, in µg/L (Section 8.2.4).
P, P_s=Percent recovery measured (Section 8.3.2, Section 8.4.2).
D=Detected; result must be greater than zero.

Note: These criteria are based directly upon the method performance data in Table 3. Where necessary, the limits for recovery have been broadened to assure applicability of the limits to concentrations below those used to develop Table 3.

TABLE 3—METHOD ACCURACY AND PRECISION AS FUNCTIONS OF CONCENTRATION—METHOD 606

Parameter	Accuracy, as recovery, X′ (µg/L)	Single analyst precision, s_r' (µg/L)	Overall precision, S′ (µg/L)
Bis(2-ethylhexyl) phthalate	0.53C+2.02	$0.80\bar{X}-2.54$	$0.73\bar{X}-0.17$
Butyl benzyl phthalate	0.82C+0.13	$0.26\bar{X}+0.04$	$0.25\bar{X}+0.07$
Di-n-butyl phthalate	0.79C+0.17	$0.23\bar{X}+0.20$	$0.29\bar{X}+0.06$
Diethyl phthalate	0.70C+0.13	$0.27\bar{X}+0.05$	$0.45\bar{X}+0.11$
Dimethyl phthalate	0.73C+0.17	$0.26\bar{X}+0.14$	$0.44\bar{X}+0.31$
Di-n-octyl phthalate	0.35C − 0.71	0.38X+0.71	0.62X+0.34

$\bar{X}'$=Expected recovery for one or more measurements of a sample containing a concentration of C, in µg/L.
s_r'=Expected single analyst standard deviation of measurements at an average concentration found of $\bar{X}$, in µg/L.
S′=Expected interlaboratory standard deviation of measurements at an average concentration found of $\bar{X}$, in µg/L.
C=True value for the concentration, in µg/L.
$\bar{X}$=Average recovery found for measurements of samples containing a concentration of C, in µg/L.

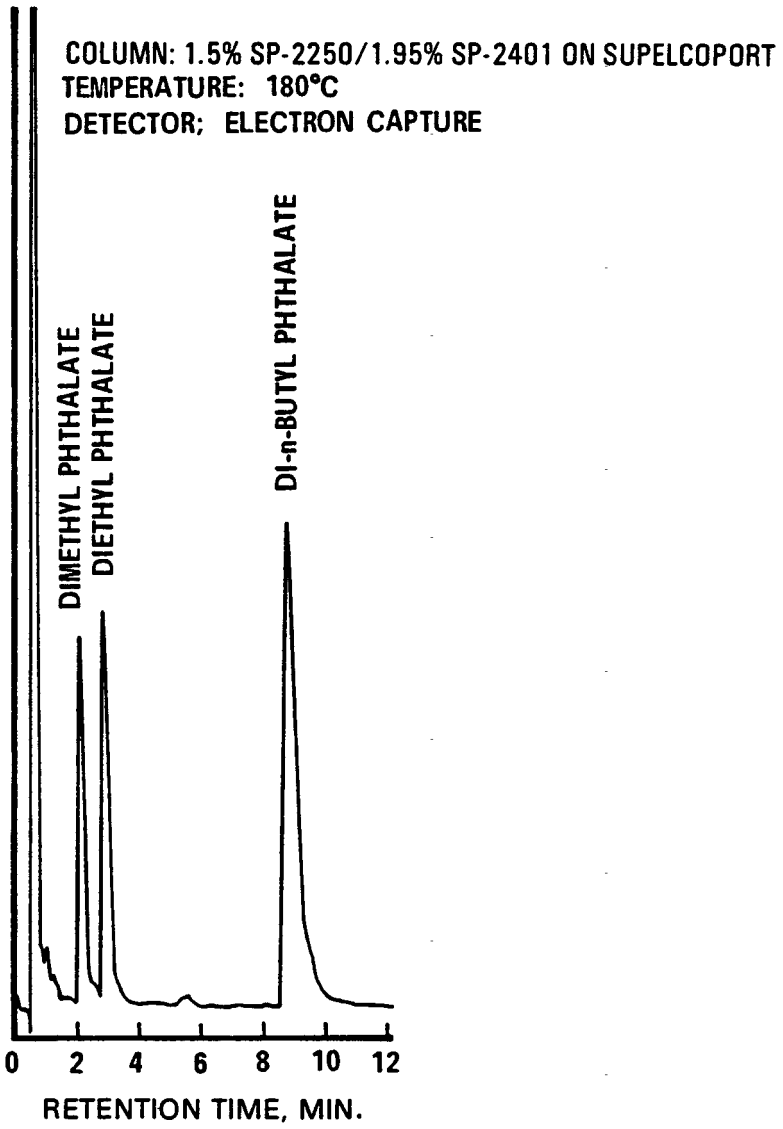


Figure 1. Gas chromatogram of phthalates.

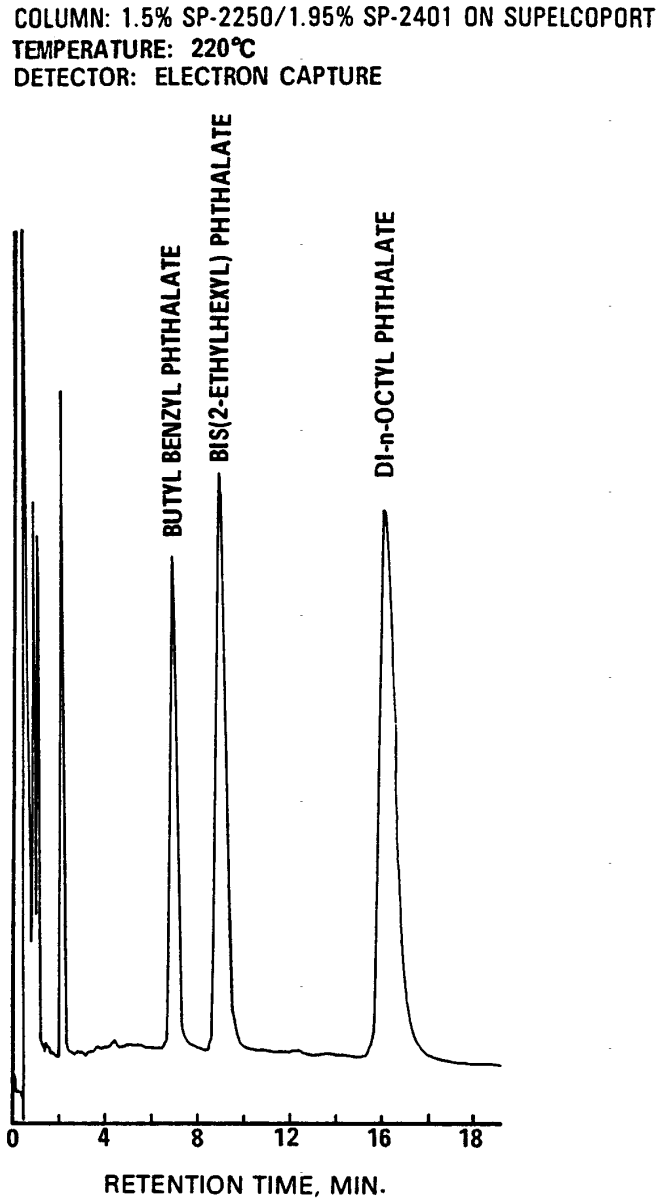


Figure 2. Gas chromatogram of phthalates.

METHOD 607—NITROSAMINES

1. Scope and Application

1.1 This method covers the determination of certain nitrosamines. The following parameters can be determined by this method:

Parameter	Storet No.	CAS No.
N-Nitrosodimethylamine	34438	62–75–9
N-Nitrosodiphenylamine	34433	86–30–6
N-Nitrosodi-n-propylamine	34428	621–64–7

1.2 This is a gas chromatographic (GC) method applicable to the determination of the parameters listed above in municipal and industrial discharges as provided under 40 CFR 136.1. When this method is used to analyze unfamiliar samples for any or all of the compmunds above, compound identifications should be supported by at least one additional qualitative technique. This method describes analytical conditimns for a second gas chromatographic column that can be used to confirm measurements made with the primary column. Method 625 provides gas chromatograph/mass spectrometer (GC/MS) conditions appropriate for the qualitative and quantitative confirmation of results for N-nitrosodi-n-propylamine. In order to confirm the presence of N-nitrosodiphenylamine, the cleanup procedure specified in Section 11.3 or 11.4 must be used. In order to confirm the presence of N-nitrosodimethylamine by GC/MS, Column 1 of this method must be substituted for the column recommended in Method 625. Confirmation of these parameters using GC-high resolution mass spectrometry or a Thermal Energy Analyzer is also recommended. [1,2]

1.3 The method detection limit (MDL, defined in Section 14.1)[3] for each parameter is listed in Table 1. The MDL for a specific wastewater may differ from those listed, depending upon the nature of interferences in the sample matrix.

1.4 Any modification of this method, beyond those expressly permitted, shall be considered as a major modification subject to application and approval of alternate test procedures under 40 CFR 136.4 and 136.5.

1.5 This method is restricted to use by or under the supervision of analysts experienced in the use of a gas chromatograph and in the interpretation of gas chromatograms. Each analyst must demonstrate the ability to generate acceptable results with this method using the procedure described in Section 8.2.

2. Summary of Method

2.1 A measured volume of sample, approximately 1–L, is extracted with methylene chloride using a separatory funnel. The methylene chloride extract is washed with dilute hydrochloric acid to remove free amines, dried, and concentrated to a volume of 10 mL or less. After the extract has been exchanged to methanol, it is separated by gas chromatography and the parameters are then measured with a nitrogen-phosphorus detector.[4]

2.2 The method provides Florisil and alumina column cleanup procedures to separate diphenylamine from the nitrosamines and to aid in the elimination of interferences that may be encountered.

3. Interferences

3.1 Method interferences may be caused by contaminants in solvents, reagents, glassware, and other sample processing hardware that lead to discrete artifacts and/or elevated baselines in gas chromatograms. All of these materials must be routinely demonstrated to be free from interferences under the conditions of the analysis by running laboratory reagent blanks as described in Section 8.1.3.

3.1.1 Glassware must be scrupulously cleaned.[5] Clean all glassware as soon as possible after use by rinsing with the last solvent used in it. Solvent rinsing should be followed by detergent washing with hot water, and rinses with tap water and distilled water. The glassware should then be drained dry, and heated in a muffle furnace at 400 °C for 15 to 30 min. Solvent rinses with acetone and pesticide quality hexane may be substituted for the muffle furnace heating. Volumetric ware should not be heated in a muffle furnace. After drying and cooling, glassware should be sealed and stored in a clean environment to prevent any accumulation of dust or other contaminants. Store inverted or capped with aluminum foil.

3.1.2 The use of high purity reagents and solvents helps to minimize interference problems. Purification of solvents by distillation in all-glass systems may be required.

3.2 Matrix interferences may be caused by contaminants that are co-extracted from the sample. The extent of matrix interferences will vary considerably from source to source, depending upon the nature and diversity of the industrial complex or municipality being sampled. The cleanup procedures in Section 11 can be used to overcome many of these interferences, but unique samples may require additional cleanup approaches to achieve the MDL listed in Table 1.

3.3 N-Nitrosodiphenylamine is reported[6–9] to undergo transnitrosation reactions. Care must be exercised in the heating or concentrating of solutions containing this compound in the presence of reactive amines.

3.4 The sensitive and selective Thermal Energy Analyzer and the reductive Hall detector may be used in place of the nitrogen-phosphorus detector when interferences are encountered. The Thermal Energy Analyzer offers the highest selectivity of the non-MS detectors.

4. Safety

4.1 The toxicity or carcinogenicity of each reagent used in this method has not been precisely defined; however, each chemical compound should be treated as a potential health hazard. From this viewpoint, exposure to these chemicals must be reduced to the lowest possible level by whatever means available. The laboratory is responsible for maintaining a current awareness file of OSHA regulations regarding the safe handling of the chemicals specified in this method. A reference file of material data handling sheets should also be made available to all personnel involved in the chemical analysis. Additional references to laboratory safety are available and have been identified[10–12] for the information of the analyst.

4.2 These nitrosamines are known carcinogens[13–17], therefore, utmost care must be exercised in the handling of these materials. Nitrosamine reference standards and standard solutions should be handled and prepared in a ventilated glove box within a properly ventilated room.

5. Apparatus and Materials

5.1 Sampling equipment, for discrete or composite sampling.

5.1.1 Grab sample bottle—1–L or 1-qt, amber glass, fitted with a screw cap lined with Teflon. Foil may be substituted for Teflon if the sample is not corrosive. If amber bottles are not available, protect samples from light. The bottle and cap liner must be washed, rinsed with acetone or methylene chloride, and dried before use to minimize contamination.

5.1.2 Automatic sampler (optional)—The sampler must incorporate glass sample containers for the collection of a minimum of 250 mL of sample. Sample containers must be kept refrigerated at 4 °C and protected from light during compositing. If the sampler uses a peristaltic pump, a minimum length of compressible silicone rubber tubing may be used. Before use, however, the compressible tubing should be thoroughly rinsed with methanol, followed by repeated rinsings with distilled water to minimize the potential for contamination of the sample. An integrating flowmeter is required to collect flow proportional composites.

5.2 Glassware (All specifications are suggested. Catalog numbers are included for illustration only.):

5.2.1 Separatory funnels—2–L and 250–mL, with Teflon stopcock.

5.2.2 Drying column—Chromatographic column, approximately 400 mm long × 19 mm ID, with coarse frit filter disc.

5.2.3 Concentrator tube, Kuderna-Danish—10-mL, graduated (Kontes K–570050–1025 or equivalent). Calibration must be checked at the volumes employed in the test. Ground glass stopper is used to prevent evaporation of extracts.

5.2.4 Evaporative flask, Kuderna-Danish—500-mL (Kontes K–570001–0500 or equivalent). Attach to concentrator tube with springs.

5.2.5 Snyder column, Kuderna-Danish—Three-ball macro (Kontes K–503000–0121 or equivalent).

5.2.6 Snyder column, Kuderna-Danish—Two-ball micro (Kontes K–569001–0219 or equivalent).

5.2.7 Vials—10 to 15-mL, amber glass, with Teflon-lined screw cap.

5.2.8 Chromatographic column—Approximately 400 mm long × 22 mm ID, with Teflon stopcock and coarse frit filter disc at bottom (Kontes K–420540–0234 or equivalent), for use in Florisil column cleanup procedure.

5.2.9 Chromatographic column—Approximately 300 mm long × 10 mm ID, with Teflon stopcock and coarse frit filter disc at bottom (Kontes K–420540–0213 or equivalent), for use in alumina column cleanup procedure.

5.3 Boiling chips—Approximately 10/40 mesh. Heat to 400 °C for 30 min or Soxhlet extract with methylene chloride.

5.4 Water bath—Heated, with concentric ring cover, capable of temperature control (±2 °C). The bath should be used in a hood.

5.5 Balance—Analytical, capable of accurately weighing 0.0001 g.

5.6 Gas chromatograph—An analytical system complete with gas chromatograph suitable for on-column injection and all required accessories including syringes, analytical columns, gases, detector, and strip-chart recorder. A data system is recommended for measuring peak areas.

5.6.1 Column 1—1.8 m long × 4 mm ID glass, packed with 10% Carbowax 20 M/2% KOH on Chromosorb W–AW (80/100 mesh) or equivalent. This column was used to develop the method performance statements in Section 14. Guidelines for the use of alternate column packings are provided in Section 12.2.

5.6.2 Column 2—1.8 m long x 4 mm ID glass, packed with 10% SP–2250 on Supelcoport (100/120 mesh) or equivalent.

5.6.3 Detector—Nitrogen-phosphorus, reductive Hall, or Thermal Energy Analyzer detector.[1,2] These detectors have proven effective in the analysis of wastewaters for the parameters listed in the scope (Section 1.1). A nitrogen-phosphorus detector was used to develop the method performance statements in Section 14. Guidelines for the use of alternate detectors are provided in Section 12.2.

6. Reagents

6.1 Reagent water—Reagent water is defined as a water in which an interferent is not observed at the MDL of the parameters of interest.

6.2 Sodium hydroxide solution (10 N)—Dissolve 40 g of NaOH (ACS) in reagent water and dilute to 100 ml.

6.3 Sodium thiosulfate—(ACS) Granular.

6.4 Sulfuric acid (1+1)—Slowly, add 50 mL of H_2SO_4 (ACS, sp. gr. 1.84) to 50 mL of reagent water.

6.5 Sodium sulfate—(ACS) Granular, anhydrous. Purify by heating at 400 °C for 4 h in a shallow tray.

6.6 Hydrochloric acid (1+9)—Add one volume of concentrated HCl (ACS) to nine volumes of reagent water.

6.7 Acetone, methanol, methylene chloride, pentane—Pesticide quality or equivalent.

6.8 Ethyl ether—Nanograde, redistilled in glass if necessary.

6.8.1 Ethyl ether must be shown to be free of peroxides before it is used as indicated by EM Laboratories Quant test strips. (Available from Scientific Products Co., Cat No. P1126–8, and other suppliers.)

6.8.2 Procedures recommended for removal of peroxides are provided with the test strips. After cleanup, 20 mL of ethyl alcohol preservative must be added to each liter of ether.

6.9 Florisil—PR grade (60/100 mesh). Purchase activated at 1250 °F and store in the dark in glass containers with ground glass stoppers or foil-lined screw caps. Before use, activate each batch at least 16 h at 130 °C in a foil-covered glass container and allow to cool.

6.10 Alumina—Basic activity Super I, W200 series (ICN Life Sciences Group, No. 404571, or equivalent). To prepare for use, place 100 g of alumina into a 500-mL reagent bottle and add 2 mL of reagent water. Mix the alumina preparation thoroughly by shaking or rolling for 10 min and let it stand for at least 2 h. The preparation should be homogeneous before use. Keep the bottle sealed tightly to ensure proper activity.

6.11 Stock standard solutions (1.00 µg/µL)—Stock standard solutions can be prepared from pure standard materials or purchased as certified solutions.

6.11.1 Prepare stock standard solutions by accurately weighing about 0.0100 g of pure material. Dissolve the material in methanol and dilute to volume in a 10-mL volumetric flask. Larger volumes can be used at the convenience of the analyst. When compound purity is assayed to be 96% or greater, the weight can be used without correction to calculate the concentration of the stock standard. Commercially prepared stock standards can be used at any concentration if they are certified by the manufacturer or by an independent source.

6.11.2 Transfer the stock standard solutions into Teflon-sealed screw-cap bottles. Store at 4 °C and protect from light. Stock standard solutions should be checked frequently for signs of degradation or evaporation, especially just prior to preparing calibration standards from them.

6.11.3 Stock standard solutions must be replaced after six months, or sooner if comparison with check standards indicates a problem.

6.12 Quality control check sample concentrate—See Section 8.2.1.

7. Calibration

7.1 Establish gas chromatographic operating conditions equivalent to those given in Table 1. The gas chromatographic system can be calibrated using the external standard technique (Section 7.2) or the internal standard technique (Section 7.3).

7.2 External standard calibration procedure:

7.2.1 Prepare calibration standards at a minimum of three concentration levels for each parameter of interest by adding volumes of one or more stock standards to a volumetric flask and diluting to volume with methanol. One of the external standards should be at a concentraton near, but above, the MDL (Table 1) and the other concentrations should correspond to the expected range of concentrations found in real samples or should define the working range of the detector.

7.2.2 Using injections of 2 to 5 µL, analyze each calibration standard according to Section 12 and tabulate peak height or area responses against the mass injected. The results can be used to prepare a calibration curve for each compound. Alternatively, if the ratio of response to amount injected (calibration factor) is a constant over the working range (< 10% relative standard deviation, RSD), linearity through the origin can be assumed and the average ratio or calibration factor can be used in place of a calibration curve.

7.3 Internal standard calibration procedure—To use this approach, the analyst must select one or more internal standards that are similar in analytical behavior to the compounds of interest. The analyst must further demonstrate that the measurement of the internal standard is not affected by method or matrix interferences. Because of these limitations, no internal standard can be suggested that is applicable to all samples.

7.3.1 Prepare calibration standards at a minimum of three concentration levels for each parameter of interest by adding volumes of one or more stock standards to a volumetric flask. To each calibration standard, add a known constant amount of one or more internal standards, and dilute to volume with methanol. One of the standards should be at a concentration near, but above, the MDL and the other concentrations should correspond to the expected range of concentrations found in real samples or should define the working range of the detector.

7.3.2 Using injections of 2 to 5 µL, analyze each calibration standard according to Section 12 and tabulate peak height or area responses against concentration for each compound and internal standard. Calculate response factors (RF) for each compound using Equation 1.

$$RF = \frac{(A_s)(C_{is})}{(A_{is})(C_s)}$$

Equation 1

where:

A_s=Response for the parameter to be measured.

A_{is}=Response for the internal standard.

C_{is}=Concentration of the internal standard (µg/L).

C_s=Concentration of the parameter to be measured (µg/L).

If the RF value over the working range is a constant (<10% RSD), the RF can be assumed to be invariant and the average RF can be used for calculations. Alternatively, the results can be used to plot a calibration curve of response ratios, A_s/A_{is}, vs. RF.

7.4 The working calibration curve, calibration factor, or RF must be verified on each working day by the measurement of one or more calibration standards. If the response for any parameter varies from the predicted response by more than ±15%, a new calibration curve must be prepared for that compound.

7.5 Before using any cleanup procedure, the analyst must process a series of calibration standards through the procedure to validate elution patterns and the absence of interferences from the reagents.

8. Quality Control

8.1 Each laboratory that uses this method is required to operate a formal quality control program. The minimum requirements of this program consist of an initial demonstration of laboratory capability and an ongoing analysis of spiked samples to evaluate and document data quality. The laboratory must maintain records to document the quality of data that is generated. Ongoing data quality checks are compared with established performance criteria to determine if the results of analyses meet the performance characteristics of the method. When results of sample spikes indicate atypical method performance, a quality control check standard must be analyzed to confirm that the measurements were performed in an in-control mode of operation.

8.1.1 The analyst must make an initial, one-time, demonstration of the ability to generate acceptable accuracy and precision with this method. This ability is established as described in Section 8.2.

8.1.2 In recognition of advances that are occurring in chromatography, the analyst is permitted certain options (detailed in Sections 10.4, 11.1, and 12.2) to improve the separations or lower the cost of measurements. Each time such a modification is made to the method, the analyst is required to repeat the procedure in Section 8.2.

8.1.3 Before processing any samples, the analyst must analyze a reagent water blank to demonstrate that interferences from the analytical system and glassware are under control. Each time a set of samples is extracted or reagents are changed, a reagent water blank must be processed as a safeguard against laboratory contamination.

8.1.4 The laboratory must, on an ongoing basis, spike and analyze a minimum of 10% of all samples to monitor and evaluate laboratory data quality. This procedure is described in Section 8.3.

8.1.5 The laboratory must, on an ongoing basis, demonstrate through the analyses of quality control check standards that the operation of the measurement system is in control. This procedure is described in Section 8.4. The frequency of the check standard analyses is equivalent to 10% of all samples analyzed but may be reduced if spike recoveries from samples (Section 8.3) meet all specified quality control criteria.

8.1.6 The laboratory must maintain performance records to document the quality of data that is generated. This procedure is described in Section 8.5.

8.2 To establish the ability to generate acceptable accuracy and precision, the analyst must perform the following operations.

8.2.1 A quality control (QC) check sample concentrate is required containing each parameter of interest at a concentration of 20 µg/mL in methanol. The QC check sample concentrate must be obtained from the U.S. Environmental Protection Agency, Environmental Monitoring and Support Laboratory in Cincinnati, Ohio, if available. If not available from that source, the QC check sample concentrate must be obtained from another external source. If not available from either source above, the QC check sample concentrate must be prepared by the laboratory using stock standards prepared independently from those used for calibration.

8.2.2 Using a pipet, prepare QC check samples at a concentration of 20 µg/L by adding 1.00 mL of QC check sample concentrate to each of four 1–L aliquots of reagent water.

8.2.3 Analyze the well-mixed QC check samples according to the method beginning in Section 10.

8.2.4 Calculate the average recovery ($\bar{X}$) in µg/L, and the standard deviation of the recovery (s) in µg/L, for each parameter using the four results.

8.2.5 For each parameter compare s and $\bar{X}$ with the corresponding acceptance criteria for precision and accuracy, respectively, found in Table 2. If s and $\bar{X}$ for all parameters of interest meet the acceptance criteria, the system performance is acceptable

and analysis of actual samples can begin. If any individual s exceeds the precision limit or any individual X falls outside the range for accuracy, the system performance is unacceptable for that parameter. Locate and correct the source of the problem and repeat the test for all parameters of interest beginning with Section 8.2.2.

8.3 The laboratory must, on an ongoing basis, spike at least 10% of the samples from each sample site being monitored to assess accuracy. For laboratories analyzing one to ten samples per month, at least one spiked sample per month is required.

8.3.1 The concentration of the spike in the sample should be determined as follows:

8.3.1.1 If, as in compliance monitoring, the concentration of a specific parameter in the sample is being checked against a regulatory concentration limit, the spike should be at that limit or 1 to 5 times higher than the background concentration determined in Section 8.3.2, whichever concentration would be larger.

8.3.1.2 If the concentration of a specific parameter in the sample is not being checked against a limit specific to that parameter, the spike should be at 20 µg/L or 1 to 5 times higher than the background concentration determined in Section 8.3.2, whichever concentration would be larger.

8.3.1.3 If it is impractical to determine background levels before spiking (e.g., maximum holding times will be exceeded), the spike concentration should be (1) the regulatory concentration limit, if any; or, if none (2) the larger of either 5 times higher than the expected background concentration or 20 µg/L.

8.3.2 Analyze one sample aliquot to determine the background concentration (B) of each parameter. If necessary, prepare a new QC check sample concentrate (Section 8.2.1) appropriate for the background concentrations in the sample. Spike a second sample aliquot with 1.0 mL of the QC check sample concentrate and analyze it to determine the concentration after spiking (A) of each parameter. Calculate each percent recovery (P) as 100(A–B)%/T, where T is the known true value of the spike.

8.3.3 Compare the percent recovery (P) for each parameter with the corresponding QC acceptance criteria found in Table 2. These acceptance criteria were caluclated to include an allowance for error in measurement of both the background and spike concentrations, assuming a spike to background ratio of 5:1. This error will be accounted for to the extent that the analyst's spike to background ratio approaches 5:1.[18] If spiking was performed at a concentration lower than 20 µg/L, the analyst must use either the QC acceptance criteria in Table 2, or optional QC acceptance criteria caluclated for the specific spike concentration. To calculate optional acceptance crtieria for the recovery of a parameter: (1) Calculate accuracy (X′) using the equation in Table 3, substituting the spike concentration (T) for C; (2) calculate overall precision (S′) using the equation in Table 3, substituting X′ for $\bar{X}$; (3) calculate the range for recovery at the spike concentration as (100 X′/T) ± 2.44(100 S′/T)%.[18]

8.3.4 If any individual P falls outside the designated range for recovery, that parameter has failed the acceptance criteria. A check standard containing each parameter that failed the criteria must be analyzed as described in Section 8.4.

8.4 If any parameter fails the acceptance criteria for recovery in Section 8.3, a QC check standard containing each parameter that failed must be prepared and analyzed.

NOTE: The frequency for the required analysis of a QC check standard will depend upon the number of parameters being simultaneously tested, the complexity of the sample matrix, and the performance of the laboratory.

8.4.1 Prepare the QC check standard by adding 1.0 mL of QC check sample concentrate (Section 8.2.1 or 8.3.2) to 1 L of reagent water. The QC check standard needs only to contain the parameters that failed criteria in the test in Section 8.3.

8.4.2 Analyze the QC check standard to determine the concentration measured (A) of each parameter. Calculate each percent recovery (P_s) as 100 (A/T)%, where T is the true value of the standard concentration.

8.4.3 Compare the percent recovery (P_s) for each parameter with the corresponding QC acceptance criteria found in Table 2. Only parameters that failed the test in Section 8.3 need to be compared with these criteria. If the recovery of any such parameter falls outside the designated range, the laboratory performance for that parameter is judged to be out of control, and the problem must be immediately identified and corrected. The analytical result for that parameter in the unspiked sample is suspect and may not be reported for regulatory compliance purposes.

8.5 As part of the QC program for the laboratory, method accuracy for wastewater samples must be assessed and records must be maintained. After the analysis of five spiked wastewater samples as in Section 8.3, calculate the average percent recovery ($\bar{P}$) and the standard deviation of the percent recovery (s_p). Express the accuracy assessment as a percent recovery interval from $\bar{P}-2s_p$ to $\bar{P}+2s_p$. If $\bar{P}$=90% and s_p=10%, for example, the accuracy interval is expressed as 70–110%. Update the accuracy assessment for each parameter on a regular basis (e.g. after each five to ten new accuracy measurements).

8.6 It is recommended that the laboratory adopt additional quality assurance practices for use with this method. The specific practices that are most productive depend upon the needs of the laboratory and the nature of

the samples. Field duplicates may be analyzed to assess the precision of the environmental measurements. When doubt exists over the identification of a peak on the chromatogram, confirmatory techniques such as gas chromatography with a dissimilar column, specific element detector, or mass spectrometer must be used. Whenever possible, the laboratory should analyze standard reference materials and participate in relevant performance evaluation studies.

9. Sample Collection, Preservation, and Handling

9.1 Grab samples must be collected in glass containers. Conventional sampling practices[19] should be followed, except that the bottle must not be prerinsed with sample before collection. Composite samples should be collected in refrigerated glass containers in accordance with the requirements of the program. Automatic sampling equipment must be as free as possible of Tygon tubing and other potential sources of contamination.

9.2 All samples must be iced or refrigerated at 4 °C from the time of collection until extraction. Fill the sample bottles and, if residual chlorine is present, add 80 mg of sodium thiosulfate per liter of sample and mix well. EPA Methods 330.4 and 330.5 may be used for measurement of residual chlorine.[20] Field test kits are available for this purpose. If N-nitrosodiphenylamine is to be determined, adjust the sample pH to 7 to 10 with sodium hydroxide solution or sulfuric acid.

9.3 All samples must be extracted within 7 days of collection and completely analyzed within 40 days of extraction.[4]

9.4 Nitrosamines are known to be light sensitive.[7] Samples should be stored in amber or foil-wrapped bottles in order to minimize photolytic decomposition.

10. Sample Extraction

10.1 Mark the water meniscus on the side of the sample bottle for later determination of sample volume. Pour the entire sample into a 2–L separatory funnel. Check the pH of the sample with wide-range pH paper and adjust to within the range of 5 to 9 with sodium hydroxide solution or sulfuric acid.

10.2 Add 60 mL of methylene chloride to the sample bottle, seal, and shake 30 s to rinse the inner surface. Transfer the solvent to the separatory funnel and extract the sample by shaking the funnel for 2 min with periodic venting to release excess pressure. Allow the organic layer to separate from the water phase for a minimum of 10 min. If the emulsion interface between layers is more than one-third the volume of the solvent layer, the analyst must employ mechanical techniques to complete the phase separation. The optimum technique depends upon the sample, but may include stirring, filtration of the emulsion through glass wool, centrifugation, or other physical methods. Collect the methylene chloride extract in a 250-mL Erlenmeyer flask.

10.3 Add a second 60-mL volume of methylene chloride to the sample bottle and repeat the extraction procedure a second time, combining the extracts in the Erlenmeyer flask. Perform a third extraction in the same manner.

10.4 Assemble a Kuderna-Danish (K–D) concentrator by attaching a 10-mL concentrator tube to a 500-mL evaporative flask. Other concentration devices or techniques may be used in place of the K–D concentrator if the requirements of Section 8.2 are met.

10.5 Add 10 mL of hydrochloric acid to the combined extracts and shake for 2 min. Allow the layers to separate. Pour the combined extract through a solvent-rinsed drying column containing about 10 cm of anhydrous sodium sulfate, and collect the extract in the K–D concentrator. Rinse the Erlenmeyer flask and column with 20 to 30 mL of methylene chloride to complete the quantitative transfer.

10.6 Add one or two clean boiling chips to the evaporative flask and attach a three-ball Snyder column. Prewet the Snyder column by adding about 1 mL of methylene chloride to the top. Place the K–D apparatus on a hot water bath (60 to 65°C) so that the concentrator tube is partially immersed in the hot water, and the entire lower rounded surface of the flask is bathed with hot vapor. Adjust the vertical position of the apparatus and the water temperature as required to complete the concentration in 15 to 20 min. At the proper rate of distillation the balls of the column will actively chatter but the chambers will not flood with condensed solvent. When the apparent volume of liquid reaches 1 mL, remove the K–D apparatus and allow it to drain and cool for at least 10 min.

10.7 Remove the Snyder column and rinse the flask and its lower joint into the concentrator tube with 1 to 2 mL of methylene chloride. A 5-mL syringe is recommended for this operation. Stopper the concentrator tube and store refrigerated if further processing will not be performed immediately. If the extract will be stored longer than two days, it should be transferred to a Teflon-sealed screw-cap vial. If N-nitrosodiphenylamine is to be measured by gas chromatography, the analyst must first use a clean-up column to eliminate diphenylamine interference (Section 11). If N-nitrosodiphenylamine is of no interest, the analyst may proceed directly with gas chromatographic analysis (Section 12).

10.8 Determine the original sample volume by refilling the sample bottle to the mark and transferring the liquid to a 1000-

mL graduated cylinder. Record the sample volume to the nearest 5 mL.

11. Cleanup and Separation

11.1 Cleanup procedures may not be necessary for a relatively clean sample matrix. If particular circumstances demand the use of a cleanup procedure, the analyst may use either procedure below or any other appropriate procedure. However, the analyst first must demonstrate that the requirements of Section 8.2 can be met using the method as revised to incorporate the cleanup procedure. Diphenylamine, if present in the original sample extract, must be separated from the nitrosamines if N-nitrosodiphenylamine is to be determined by this method.

11.2 If the entire extract is to be cleaned up by one of the following procedures, it must be concentrated to 2.0 mL. To the concentrator tube in Section 10.7, add a clean boiling chip and attach a two-ball micro-Snyder column. Prewet the column by adding about 0.5 mL of methylene chloride to the top. Place the micr-K–D apparatus on a hot water bath (60 to 65°C) so that the concentrator tube is partially immersed in the hot water. Adjust the vertical position of the apparatus and the water temperature as required to complete the concentration in 5 to 10 min. At the proper rate of distillation the balls of the column will actively chatter but the chambers will not flood. When the apparent volume of liquid reaches about 0.5 mL, remove the K–D apparatus and allow it to drain and cool for at least 10 min. Remove the micro-Snyder column and rinse its lower joint into the concentrator tube with 0.2 mL of methylene chloride. Adjust the final volume to 2.0 mL and proceed with one of the following cleanup procedures.

11.3 Florisil column cleanup for nitrosamines:

11.3.1 Place 22 g of activated Florisil into a 22-mm ID chromatographic column. Tap the column to settle the Florisil and add about 5 mm of anhydrous sodium sulfate to the top.

11.3.2 Preelute the column with 40 mL of ethyl ether/pentane (15+85)(V/V). Discard the eluate and just prior to exposure of the sodium sulfate layer to the air, quantitatively transfer the 2-mL sample extract onto the column using an additional 2 mL of pentane to complete the transfer.

11.3.3 Elute the column with 90 mL of ethyl ether/pentane (15+85)(V/V) and discard the eluate. This fraction will contain the diphenylamine, if it is present in the extract.

11.3.4 Next, elute the column with 100 mL of acetone/ethyl ether (5+95)(V/V) into a 500-mL K–D flask equipped with a 10-mL concentrator tube. This fraction will contain all of the nitrosamines listed in the scope of the method.

11.3.5 Add 15 mL of methanol to the collected fraction and concentrate as in Section 10.6, except use pentane to prewet the column and set the water bath at 70 to 75°C. When the apparatus is cool, remove the Snyder column and rinse the flask and its lower joint into the concentrator tube with 1 to 2 mL of pentane. Analyze by gas chromatography (Section 12).

11.4 Alumina column cleanup for nitrosamines:

11.4.1 Place 12 g of the alumina preparation (Section 6.10) into a 10-mm ID chromatographic column. Tap the column to settle the alumina and add 1 to 2 cm of anhydrous sodium sulfate to the top.

11.4.2 Preelute the column with 10 mL of ethyl ether/pentane (3+7)(V/V). Discard the eluate (about 2 mL) and just prior to exposure of the sodium sulfate layer to the air, quantitatively transfer the 2 mL sample extract onto the column using an additional 2 mL of pentane to complete the transfer.

11.4.3 Just prior to exposure of the sodium sulfate layer to the air, add 70 mL of ethyl ether/pentane (3+7)(V/V). Discard the first 10 mL of eluate. Collect the remainder of the eluate in a 500–mL K–D flask equipped with a 10 mL concentrator tube. This fraction contains N-nitrosodiphenylamine and probably a small amount of N-nitrosodi-n-propylamine.

11.4.4 Next, elute the column with 60 mL of ethyl ether/pentane (1+1)(V/V), collecting the eluate in a second K–D flask equipped with a 10–mL concentrator tube. Add 15 mL of methanol to the K–D flask. This fraction will contain N-nitrosodimethylamine, most of the N-nitrosodi-n-propylamine and any diphenylamine that is present.

11.4.5 Concentrate both fractions as in Section 10.6, except use pentane to prewet the column. When the apparatus is cool, remove the Snyder column and rinse the flask and its lower joint into the concentrator tube with 1 to 2 mL of pentane. Analyze the fractions by gas chromatography (Section 12).

12. Gas Chromatography

12.1 N-nitrosodiphenylamine completely reacts to form diphenylamine at the normal operating temperatures of a GC injection port (200 to 250°C). Thus, N-nitrosodiphenylamine is chromatographed and detected as diphenylamine. Accurate determination depends on removal of diphenylamine that may be present in the original extract prior to GC analysis (See Section 11).

12.2 Table 1 summarizes the recommended operating conditions for the gas chromatograph. Included in this table are retention times and MDL that can be achieved under these conditions. Examples of the separations achieved by Column 1 are shown in Figures 1 and 2. Other packed or capillary (open-tubular) columns, chromatographic

conditions, or detectors may be used if the requirements of Section 8.2 are met.

12.3 Calibrate the system daily as described in Section 7.

12.4 If the extract has not been subjected to one of the cleanup procedures in Section 11, it is necessary to exchange the solvent from methylene chloride to methanol before the thermionic detector can be used. To a 1 to 10-mL volume of methylene chloride extract in a concentrator tube, add 2 mL of methanol and a clean boiling chip. Attach a two-ball micro-Snyder column to the concentrator tube. Prewet the column by adding about 0.5 mL of methylene chloride to the top. Place the micro-K–D apparatus on a boiling (100°C) water bath so that the concentrator tube is partially immersed in the hot water. Adjust the vertical position of the apparatus and the water temperature as required to complete the concentration in 5 to 10 min. At the proper rate of distillation the balls of the column will actively chatter but the chambers will not flood. When the apparent volume of liquid reaches about 0.5 mL, remove the K–D apparatus and allow it to drain and cool for at least 10 min. Remove the micro-Snyder column and rinse its lower joint into the concentrator tube with 0.2 mL of methanol. Adjust the final volume to 2.0 mL.

12.5 If the internal standard calibration procedure is being used, the internal standard must be added to the sample extract and mixed thoroughly immediately before injection into the gas chromatograph.

12.6 Inject 2 to 5 µL of the sample extract or standard into the gas chromatograph using the solvent-flush technique.[21] Smaller (1.0 µL) volumes may be injected if automatic devices are employed. Record the volume injected to the nearest 0.05 µL, and the resulting peak size in area or peak height units.

12.7 Identify the parameters in the sample by comparing the retention times of the peaks in the sample chromatogram with those of the peaks in standard chromatograms. The width of the retention time window used to make identifications should be based upon measurements of actual retention time variations of standards over the course of a day. Three times the standard deviation of a retention time for a compound can be used to calculate a suggested window size; however, the experience of the analyst should weigh heavily in the interpretation of chromatograms.

12.8 If the response for a peak exceeds the working range of the system, dilute the extract and reanalyze.

12.9 If the measurement of the peak response is prevented by the presence of interferences, further cleanup is required.

13. Calculations

13.1 Determine the concentration of individual compounds in the sample.

13.1.1 If the external standard calibration procedure is used, calculate the amount of material injected from the peak response using the calibration curve or calibration factor determined in Section 7.2.2. The concentration in the sample can be calculated from Equation 2.

$$\text{Concentration } (\mu g/L) = \frac{(A)(V_t)}{(V_i)(V_s)}$$

Equation 2

where:

A=Amount of material injected (ng).
V_i=Volume of extract injected (µL).
V_t=Volume of total extract (µL).
V_s=Volume of water extracted (mL).

13.1.2 If the internal standard calibration procedure is used, calculate the concentration in the sample using the response factor (RF) determined in Section 7.3.2 and Equation 3.

$$RF = \frac{(A_s)(C_{is})}{(A_{is})(C_s)}$$

Equation 3

where:

A_s=Response for the parameter to be measured.
A_{is}=Response for the internal standard.
I_s=Amount of internal standard added to each extract (µg).
V_o=Volume of water extracted (L).

13.2 Report results in µg/L without correction for recovery data. All QC data obtained should be reported with the sample results.

14. Method Performance

14.1 The method detection limit (MDL) is defined as the minimum concentration of a substance that can be measured and reported with 99% confidence that the value is above zero.[3] The MDL concentrations listed in Table 1 were obtained using reagent water.[22] Similar results were achieved using representative wastewaters. The MDL actually achieved in a given analysis will vary depending on instrument sensitivity and matrix effects.

14.2 This method has been tested for linearity of spike recovery from reagent water and has been demonstrated to be applicable over the concentration range from 4 x MDL to 1000 x MDL.[22]

14.3 This method was tested by 17 laboratories using reagent water, drinking water, surface water, and three industrial wastewaters spiked at six concentrations

over the range 0.8 to 55 μg/L.[23] Single operator precision, overall precision, and method accuracy were found to be directly related to the concentration of the parameter and essentially independent of the sample matrix. Linear equations to describe these relationships are presented in Table 3.

References

1. Fine, D.H., Lieb, D., and Rufeh, R. ''Principle of Operation of the Thermal Energy Analyzer for the Trace Analysis of Volatile and Non-volatile N-nitroso Compounds,'' *Journal of Chromatography, 107,* 351 (1975).

2. Fine, D.H., Hoffman, F., Rounbehler, D.P., and Belcher, N.M. ''Analysis of N-nitroso Compounds by Combined High Performance Liquid Chromatography and Thermal Energy Analysis,'' Walker, E.A., Bogovski, P. and Griciute, L., Editors, N-nitroso Compounds—Analysis and Formation, Lyon, International Agency for Research on Cancer (IARC Scientific Publications No. 14), pp. 43–50 (1976).

3. 40 CFR part 136, appendix B.

4. ''Determination of Nitrosamines in Industrial and Municipal Wastewaters,'' EPA 600/4–82–016, National Technical Information Service, PB82–199621, Springfield, Virginia 22161, April 1982.

5. ASTM Annual Book of Standards, Part 31, D3694–78. ''Standard Practices for Preparation of Sample Containers and for Preservation of Organic Constituents,'' American Society for Testing and Materials, Philadelphia.

6. Buglass, A.J., Challis, B.C., and Osborn, M.R. ''Transnitrosation and Decomposition of Nitrosamines,'' Bogovski, P. and Walker, E.A., Editors, N-nitroso Compounds in the Environment, Lyon, International Agency for Research on Cancer (IARC Scientific Publication No. 9), pp. 94–100 (1974).

7. Burgess, E.M., and Lavanish, J.M. ''Photochemical Decomposition of N-nitrosamines,'' *Tetrahedon Letters,* 1221 (1964)

8. Druckrey, H., Preussmann, R., Ivankovic, S., and Schmahl, D. ''Organotrope Carcinogene Wirkungen bei 65 Verschiedenen N-NitrosoVerbindungen an BD-Ratten,'' *Z. Krebsforsch., 69,* 103 (1967).

9. Fiddler, W. ''The Occurrence and Determination of N-nitroso Compounds,'' *Toxicol. Appl. Pharmacol., 31,* 352 (1975).

10. ''Carcinogens—Working With Carcinogens,'' Department of Health, Education, and Welfare, Public Health Service, Center for Disease Control, National Institute for Occupational Safety and Health, Publication No. 77–206, August 1977.

11. ''OSHA Safety and Health Standards, General Industry,'' (29 CFR Part 1910), Occupational Safety and Health Administration, OSHA 2206 (Revised, January 1976).

12. ''Safety in Academic Chemistry Laboratories,'' American Chemical Society Publication, Committee on Chemical Safety, 3rd Edition, 1979.

13. Lijinsky, W. ''How Nitrosamines Cause Cancer,'' *New Scientist, 73,* 216 (1977).

14. Mirvish, S.S. ''N-Nitroso compounds: Their Chemical and in vivo Formation and Possible Importance as Environmental Carcinogens,'' *J. Toxicol. Environ. Health, 3,* 1267 (1977).

15. ''Reconnaissance of Environmental Levels of Nitrosamines in the Central United States,'' EPA–330/1–77–001, National Enforcement Investigations Center, U.S. Environmental Protection Agency (1977).

16. ''Atmospheric Nitrosamine Assessment Report,'' Office of Air Quality Planning and Standards, U.S. Environmental Protection Agency, Research Triangle Park, North Carolina (1976).

17. ''Scientific and Technical Assessment Report on Nitrosamines,'' EPA–660/6–7–001, Office of Research and Development, U.S. Environmental Protection Agency (1976).

18. Provost, L.P., and Elder, R.S. ''Interpretation of Percent Recovery Data,'' *American Laboratory, 15,* 58–63 (1983). (The value 2.44 used in the equation in Section 8.3.3 is two times the value of 1.22 derived in this report.)

19. ASTM Annual Book of Standards, Part 31, D3370–76. ''Standard Practices for Sampling Water,'' American Society for Testing and Materials, Philadelphia.

20. ''Methods 330.4 (Titrimetric, DPD–FAS) and 330.5 (Spectrophotometric, DPD) for Chlorine, Total Residual,'' Methods for Chemical Analysis of Water and Wastes, EPA–600/4–79–020, U.S. Environmental Protection Agency, Environmental Monitoring and Support Laboratory, Cincinnati, Ohio 45268, March 1979.

21. Burke, J. A. ''Gas Chromatography for Pesticide Residue Analysis; Some Practical Aspects,'' *Journal of the Association of Official Analytical Chemists, 48,* 1037 (1965).

22. ''Method Detection Limit and Analytical Curve Studies EPA Methods 606, 607, and 608,'' Special letter report for EPA Contract 68–03–2606, U.S. Environmental Protection Agency, Environmental Monitoring and Support Laboratory, Cincinnati, Ohio 45268, June 1980.

23. ''EPA Method Study 17 Method 607—Nitrosamines,'' EPA 600/4–84–051, National Technical Information Service, PB84–207646, Springfield, Virginia 22161, June 1984.

TABLE 1—CHROMATOGRAPHIC CONDITIONS AND METHOD DETECTION LIMITS

Parameter	Retention time (min)		Method detection limit (μg/L)
	Column 1	Column 2	
N-Nitrosodimethylamine	4.1	0.88	0.15
N-Nitrosodi-n-propylamine	12.1	4.2	.46

TABLE 1—CHROMATOGRAPHIC CONDITIONS AND METHOD DETECTION LIMITS—Continued

Parameter	Retention time (min)		Method detection limit (µg/L)
	Column 1	Column 2	
N-Nitrosodiphenylamine [a]	[b] 12.8	[c] 6.4	.81

Column 1 conditions: Chromosorb W–AW (80/100 mesh) coated with 10% Carbowax 20 M/2% KOH packed in a 1.8 m long x 4mm ID glass column with helium carrier gas at 40 mL/min flow rate. Column temperature held isothermal at 110 °C, except where otherwise indicated.

Column 2 conditions: Supelcoport (100/120 mesh) coated with 10% SP–2250 packed in a 1.8 m long x 4 mm ID glass column with helium carrier gas at 40 mL/min flow rate. Column temperature held isothermal at 120 °C, except where otherwise indicated.

[a] Measured as diphenylamine.
[b] 220 °C column temperature.
[c] 210 °C column temperature.

TABLE 2—QC ACCEPTANCE CRITERIA—METHOD 607

Parameter	Test conc. (µg/L)	Limit for s (µg/L)	Range for $\bar{X}$ (µg/L)	Range for P, P_s (percent)
N–Nitrosodimethylamine	20	3.4	4.6–20.0	13–109
N–Nitrosodiphenyl	20	6.1	2.1–24.5	D–139
N–Nitrosodi-n-propylamine	20	5.7	11.5–26.8	45–146

s=Standard deviation for four recovery measurements, in µg/L (Section 8.2.4).
$\bar{X}$=Average recovery for four recovery measurements, in µg/L (Section 8.2.4).
P, P_s=Percent recovery measured (Section 8.3.2, Section 8.4.2).
D=Detected; result must be greater than zero.

NOTE: These criteria are based directly upon the method performance data in Table 3. Where necessary, the limits for recovery have been broadened to assure applicability of the limits to concentrations below those used to develop Table 3.

TABLE 3—METHOD ACCURACY AND PRECISION AS FUNCTIONS OF CONCENTRATION—METHOD 607

Parameter	Accuracy, as recovery, X′ (µg/L)	Single analyst precision, s_r' (µg/L)	Overall precision, S′ (µg/L)
N–Nitrosodimethylamine	0.37C+0.06	$0.25\bar{X}-0.04$	$0.25\bar{X}+0.11$
N–Nitrosodiphenylamine	0.64C+0.52	$0.36\bar{X}-1.53$	$0.46\bar{X}-0.47$
N–Nitrosodi-n-propylamine	0.96C − 0.07	$0.15\bar{X}+0.13$	$0.21\bar{X}+0.15$

X′=Expected recovery for one or more measurements of a sample containing a concentration of C, in µg/L.
s_r'=Expected single analyst standard deviation of measurements at an average concentration found of $\bar{X}$, in µg/L.
S′=Expected interlaboratory standard deviation of measurements at an average concentration found of X, in µg/L.
C=True value for the concentration, in µg/L.
X=Average recovery found for measurements of samples containing a concentration of C, in µg/L.

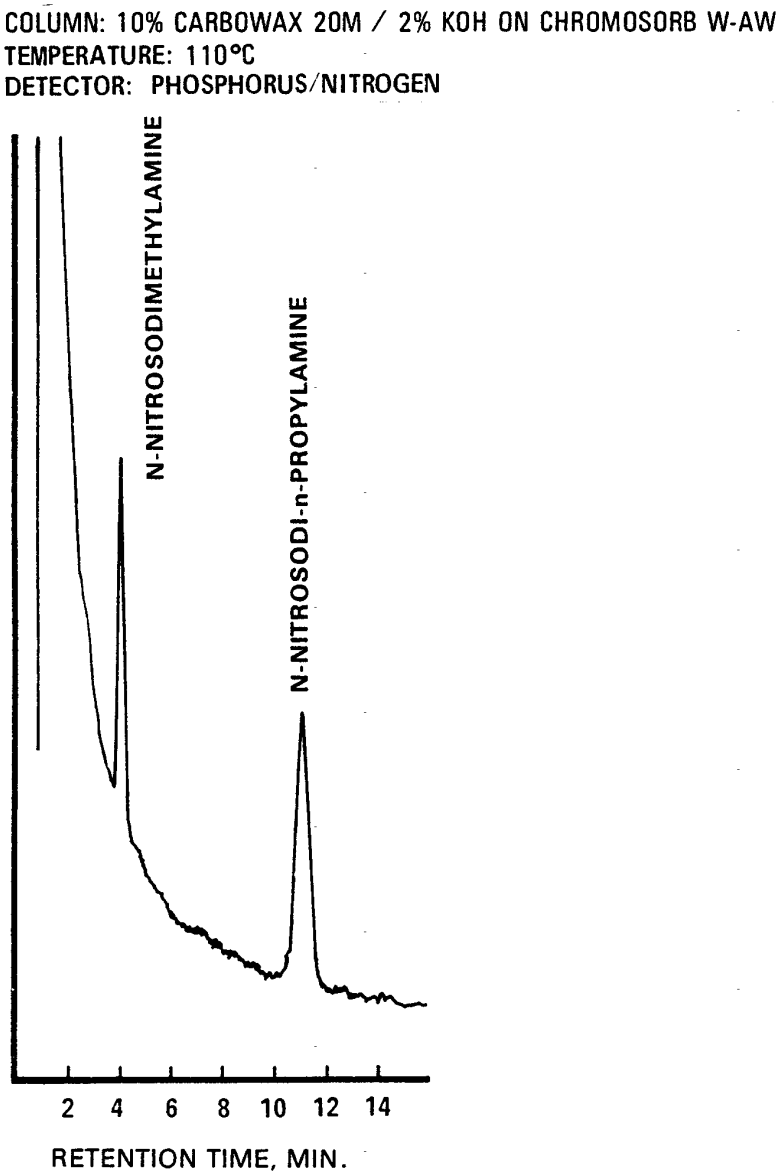


Figure 1. Gas chromatogram of nitrosamines.

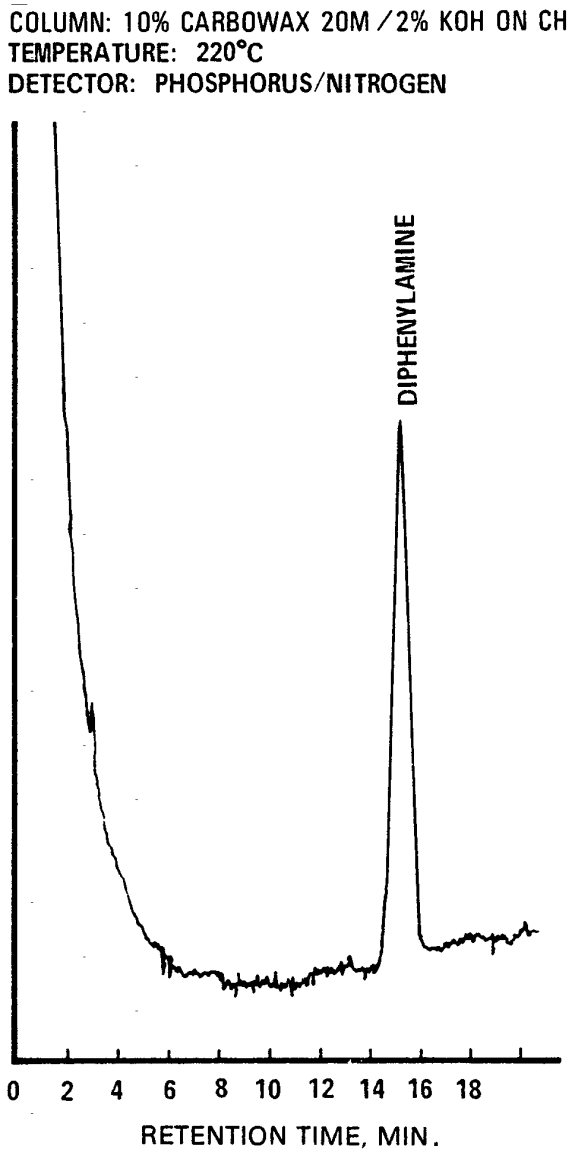


Figure 2. Gas chromatogram of N-nitrosodiphenylamine as diphenylamine.

METHOD 608—ORGANOCHLORINE PESTICIDES AND PCBS

1. Scope and Application

1.1 This method covers the determination of certain organochlorine pesticides and PCBs. The following parameters can be determined by this method:

Parameter	STORET No.	CAS No.
Aldrin	39330	309–00–2
α–BHC	39337	319–84–6
β–BHC	39338	319–85–7
δ–BHC	34259	319–86–8
γ–BHC	39340	58–89–9
Chlordane	39350	57–74–9
4,4′–DDD	39310	72–54–8
4,4′–DDE	39320	72–55–9
4,4′–DDT	39300	50–29–3
Dieldrin	39380	60–57–1
Endosulfan I	34361	959–98–8
Endosulfan II	34356	33212–65–9
Endosulfan sulfate	34351	1031–07–8
Eldrin	39390	72–20–8
Endrin aldehyde	34366	7421–93–4
Heptachlor	39410	76–44–8
Heptachlor epoxide	39420	1024–57–3
Toxaphene	39400	8001–35–2
PCB–1016	34671	12674–11–2
PCB–1221	39488	1104–28–2
PCB–1232	39492	11141–16–5
PCB–1242	39496	53469–21–9
PCB–1248	39500	12672–29–6
PCB–1254	39504	11097–69–1
PCB–1260	39508	11096–82–5

1.2 This is a gas chromatographic (GC) method applicable to the determination of the compounds listed above in municipal and industrial discharges as provided under 40 CFR 136.1. When this method is used to analyze unfamiliar samples for any or all of the compounds above, compound identifications should be supported by at least one additional qualitative technique. This method describes analytical conditions for a second gas chromatographic column that can be used to confirm measurements made with the primary column. Method 625 provides gas chromatograph/mass spectrometer (GC/MS) conditions appropriate for the qualitative and quantitative confirmation of results for all of the parameters listed above, using the extract produced by this method.

1.3 The method detection limit (MDL, defined in Section 14.1)[1] for each parameter is listed in Table 1. The MDL for a specific wastewater may differ from those listed, depending upon the nature of interferences in the sample matrix.

1.4 The sample extraction and concentration steps in this method are essentially the same as in Methods 606, 609, 611, and 612. Thus, a single sample may be extracted to measure the parameters included in the scope of each of these methods. When cleanup is required, the concentration levels must be high enough to permit selecting aliquots, as necessary, to apply appropriate cleanup procedures. The analyst is allowed the latitude, under Section 12, to select chromatographic conditions appropriate for the simultaneous measurement of combinations of these parameters.

1.5 Any modification of this method, beyond those expressly permitted, shall be considered as a major modification subject to application and approval of alternate test procedures under 40 CFR 136.4 and 136.5.

1.6 This method is restricted to use by or under the supervision of analysts experienced in the use of a gas chromatograph and in the interpretation of gas chromatograms. Each analyst must demonstrate the ability to generate acceptable results with this method using the procedure described in Section 8.2.

2. Summary of Method

2.1 A measured volume of sample, approximately 1–L, is extracted with methylene chloride using a separatory funnel. The methylene chloride extract is dried and exchanged to hexane during concentration to a volume of 10 mL or less. The extract is separated by gas chromatography and the parameters are then measured with an electron capture detector.[2]

2.2 The method provides a Florisil column cleanup procedure and an elemental sulfur removal procedure to aid in the elimination of interferences that may be encountered.

3. Interferences

3.1 Method interferences may be caused by contaminants in solvents, reagents, glassware, and other sample processing hardware that lead to discrete artifacts and/or elevated baselines in gas chromatograms. All of these materials must be routinely demonstrated to be free from interferences under the conditions of the analysis by running laboratory reagent blanks as described in Section 8.1.3.

3.1.1 Glassware must be scrupulously cleaned.[3] Clean all glassware as soon as possible after use by rinsing with the last solvent used in it. Solvent rinsing should be followed by detergent washing with hot water, and rinses with tap water and distilled water. The glassware should then be drained dry, and heated in a muffle furnace at 400 °C for 15 to 30 min. Some thermally stable materials, such as PCBs, may not be eliminated by this treatment. Solvent rinses with acetone and pesticide quality hexane may be substituted for the muffle furnace heating. Thorough rinsing with such solvents usually eliminates PCB interference. Volumetric ware should not be heated in a muffle furnace. After drying and cooling, glassware should be sealed and stored in a clean environment to prevent any accumulation of dust or other contaminants. Store inverted or capped with aluminum foil.

3.1.2 The use of high purity reagents and solvents helps to minimize interference problems. Purification of solvents by distillation in all-glass systems may be required.

3.2 Interferences by phthalate esters can pose a major problem in pesticide analysis when using the electron capture detector. These compounds generally appear in the chromatogram as large late eluting peaks, especially in the 15 and 50% fractions from Florisil. Common flexible plastics contain varying amounts of phthalates. These phthalates are easily extracted or leached from such materials during laboratory operations. Cross contamination of clean glassware routinely occurs when plastics are handled during extraction steps, especially when solvent-wetted surfaces are handled. Interferences from phthalates can best be minimized by avoiding the use of plastics in the laboratory. Exhaustive cleanup of reagents and glassware may be required to eliminate background phthalate contamination.[4,5] The interferences from phthalate esters can be avoided by using a microcoulometric or electrolytic conductivity detector.

3.3 Matrix interferences may be caused by contaminants that are co-extracted from the sample. The extent of matrix interferences will vary considerably from source to source, depending upon the nature and diversity of the industrial complex or municipality being sampled. The cleanup procedures in Section 11 can be used to overcome many of these interferences, but unique samples may require additional cleanup approaches to achieve the MDL listed in Table 1.

4. Safety

4.1 The toxicity or carcinogenicity of each reagent used in this method has not been precisely defined; however, each chemical compound should be treated as a potential health hazard. From this viewpoint, exposure to these chemicals must be reduced to the lowest possible level by whatever means available. The laboratory is responsible for maintaining a current awareness file of OSHA regulations regarding the safe handling of the chemicals specified in this method. A reference file of material data handling sheets should also be made available to all personnel involved in the chemical analysis. Additional references to laboratory safety are available and have been identified[6–8] for the information of the analyst.

4.2 The following parameters covered by this method have been tentatively classified as known or suspected, human or mammalian carcinogens: 4,4′-DDT, 4,4′–DDD, the BHCs, and the PCBs. Primary standards of these toxic compounds should be prepared in a hood. A NIOSH/MESA approved toxic gas respirator should be worn when the analyst handles high concentrations of these toxic compounds.

5. Apparatus and Materials

5.1 Sampling equipment, for discrete or composite sampling.

5.1.1 Grab sample bottle—1–L or 1-qt, amber glass, fitted with a screw cap lined with Teflon. Foil may be substituted for Teflon if the sample is not corrosive. If amber bottles are not available, protect samples from light. The bottle and cap liner must be washed, rinsed with acetone or methylene chloride, and dried before use to minimize contamination.

5.1.2 Automatic sampler (optional)—The sampler must incorporate glass sample containers for the collection of a minimum of 250 mL of sample. Sample containers must be kept refrigerated at 4°C and protected from light during composting. If the sampler uses a peristaltic pump, a minimum length of compressible silicone rubber tubing may be used. Before use, however, the compressible tubing should be thoroughly rinsed with methanol, followed by repeated rinsings with distilled water to minimize the potential for contamination of the sample. An integrating flow meter is required to collect flow proportional composites.

5.2. Glassware (All specifications are suggested. Catalog numbers are included for illustration only.):

5.2.1 Separatory funnel—2–L, with Teflon stopcock.

5.2.2 Drying column—Chromatographic column, approximately 400 mm long × 19 mm ID, with coarse frit filter disc.

5.2.3 Chromatographic column—400 mm long × 22 mm ID, with Teflon stopcock and coarse frit filter disc (Kontes K–42054 or equivalent).

5.2.4 Concentrator tube, Kuderna-Danish—10–mL, graduated (Kontes K–570050–1025 or equivalent). Calibration must be checked at the volumes employed in the test. Ground glass stopper is used to prevent evaporation of extracts.

5.2.5 Evaporative flask, Kuderna-Danish—500–mL (Kontes K–570001–0500 or equivalent). Attach to concentrator tube with springs.

5.2.6 Snyder column, Kuderna/Danish—Three-ball macro (Kontes K–503000–0121 or equivalent).

5.2.7 Vials—10 to 15–mL, amber glass, with Teflon-lined screw cap.

5.3 Boiling chips—Approximately 10/40 mesh. Heat to 400°C for 30 min or Soxhlet extract with methylene chloride.

5.4 Water bath—Heated, with concentric ring cover, capable of temperature control (±2°C). The bath should be used in a hood.

5.5 Balance—Analytical, capable of accurately weighing 0.0001 g.

5.6 Gas chromatograph—An analytical system complete with gas chromatograph suitable for on-column injection and all required accessories including syringes, analytical columns, gases, detector, and strip-

chart recorder. A data system is recommended for measuring peak areas.

5.6.1 Column 1—1.8 m long × 4 mm ID glass, packed with 1.5% SP–2250/1.95% SP–2401 on Supelcoport (100/120 mesh) or equivalent. This column was used to develop the method performance statements in Section 14. Guidelines for the use of alternate column packings are provided in Section 12.1.

5.6.2 Column 2—1.8 m long × 4 mm ID glass, packed with 3% OV–1 on Supelcoport (100/120 mesh) or equivalent.

5.6.3 Detector—Electron capture detector. This detector has proven effective in the analysis of wastewaters for the parameters listed in the scope (Section 1.1), and was used to develop the method performance statements in Section 14. Guidelines for the use of alternate detectors are provided in Section 12.1.

6. Reagents

6.1 Reagent water—Reagent water is defined as a water in which an interferent is not observed at the MDL of the parameters of interest.

6.2 Sodium hydroxide solution (10 N)—Dissolve 40 g of NaOH (ACS) in reagent water and dilute to 100 mL.

6.3 Sodium thiosulfate—(ACS) Granular.

6.4 Sulfuric acid (1+1)—Slowly, add 50 mL to H_2SO_4 (ACS, sp. gr. 1.84) to 50 mL of reagent water.

6.5 Acetone, hexane, isooctane, methylene chloride—Pesticide quality or equivalent.

6.6 Ethyl ether—Nanograde, redistilled in glass if necessary.

6.6.1 Ethyl ether must be shown to be free of peroxides before it is used as indicated by EM Laboratories Quant test strips. (Available from Scientific Products Co., Cat. No. P1126–8, and other suppliers.)

6.6.2 Procedures recommended for removal of peroxides are provided with the test strips. After cleanup, 20 mL of ethyl alcohol preservative must be added to each liter of ether.

6.7 Sodium sulfate—(ACS) Granular, anhydrous. Purify by heating at 400°C for 4 h in a shallow tray.

6.8 Florisil—PR grade (60/100 mesh). Purchase activated at 1250°F and store in the dark in glass containers with ground glass stoppers or foil-lined screw caps. Before use, activate each batch at least 16 h at 130°C in a foil-covered glass container and allow to cool.

6.9 Mercury—Triple distilled.

6.10 Copper powder—Activated.

6.11 Stock standard solutions (1.00 µg/µL)—Stock standard solutions can be prepared from pure standard materials or purchased as certified solutions.

6.11.1 Prepare stock standard solutions by accurately weighing about 0.0100 g of pure material. Dissolve the material in isooctane and dilute to volume in a 10-mL volumetric flask. Larger volumes can be used at the convenience of the analyst. When compound purity is assayed to be 96% or greater, the weight can be used without correction to calculate the concentration of the stock standard. Commercially prepared stock standards can be used at any concentration if they are certified by the manufacturer or by an independent source.

6.11.2 Transfer the stock standard solutions into Teflon-sealed screw-cap bottles. Store at 4°C and protect from light. Stock standard solutions should be checked frequently for signs of degradation or evaporation, especially just prior to preparing calibration standards from them.

6.11.3 Stock standard solutions must be replaced after six months, or sooner if comparison with check standards indicates a problem.

6.12 Quality control check sample concentrate—See Section 8.2.1.

7. Calibration

7.1 Establish gas chromatographic operating conditions equivalent to those given in Table 1. The gas chromatographic system can be calibrated using the external standard technique (Section 7.2) or the internal standard technique (Section 7.3).

7.2 External standard calibration procedure:

7.2.1 Prepare calibration standards at a minimum of three concentration levels for each parameter of interest by adding volumes of one or more stock standards to a volumetric flask and diluting to volume with isooctane. One of the external standards should be at a concentration near, but above, the MDL (Table 1) and the other concentrations should correspond to the expected range of concentrations found in real samples or should define the working range of the detector.

7.2.2 Using injections of 2 to 5 µL, analyze each calibration standard according to Section 12 and tabulate peak height or area responses against the mass injected. The results can be used to prepare a calibration curve for each compound. Alternatively, if the ratio of response to amount injected (calibration factor) is a constant over the working range (<10% relative standard deviation, RSD), linearity through the origin can be assumed and the average ratio or calibration factor can be used in place of a calibration curve.

7.3 Internal standard calibration procedure—To use this approach, the analyst must select one or more internal standards that are similar in analytical behavior to the compounds of interest. The analyst must further demonstrate that the measurement of the internal standard is not affected by method or matrix interferences. Because of these limitations, no internal standard can

be suggested that is applicable to all samples.

7.3.1 Prepare calibration standards at a minimum of three concentration levels for each parameter of interest by adding volumes of one or more stock standards to a volumetric flask. To each calibration standard, add a known constant amount of one or more internal standards, and dilute to volume with isooctane. One of the standards should be at a concentration near, but above, the MDL and the other concentrations should correspond to the expected range of concentrations found in real samples or should define the working range of the detector.

7.3.2 Using injections of 2 to 5 µL, analyze each calibration standard according to Section 12 and tabulate peak height or area responses against concentration for each compound and internal standard. Calculate response factors (RF) for each compound using Equation 1.

$$RF = \frac{(A_s)(C_{is})}{(A_{is})(C_s)}$$

Equation 1

where:

A_s=Response for the parameter to be measured.

A_{is}=Response for the internal standard.

C_{is}=Concentration of the internal standard (µg/L).

C_s=Concentraton of the parameter to be measured (µg/L).

If the RF value over the working range is a constant (< 10% RSD), the RF can be assumed to be invariant and the average RF can be used for calculations. Alternatively, the results can be used to plot a calibration curve of response ratios, A_s/A_{is}, vs. RF.

7.4 The working calibration curve, calibration factor, or RF must be verified on each working day by the measurement of one or more calibration standards. If the response for any parameter varies from the predicted response by more than ±15%, the test must be repeated using a fresh calibration standard. Alternatively, a new calibration curve must be prepared for that compound.

7.5 The cleanup procedure in Section 11 utilizes Florisil column chromatography. Florisil from different batches or sources may vary in adsorptive capacity. To standardize the amount of Florisil which is used, the use of lauric acid value[9] is suggested. The referenced procedure determines the adsorption from hexane solution of lauric acid (mg) per g of Florisil. The amount of Florisil to be used for each column is calculated by dividing 110 by this ratio and multiplying by 20 g.

7.6 Before using any cleanup procedure, the analyst must process a series of calibration standards through the procedure to validate elution patterns and the absence of interferences from the reagents.

8. Quality Control

8.1 Each laboratory that uses this method is required to operate a formal quality control program. The minimum requirements of this program consist of an initial demonstration of laboratory capability and an ongoing analysis of spiked samples to evaluate and document data quality. The laboratory must maintain records to document the quality of data that is generated. Ongoing data quality checks are compared with established performance criteria to determine if the results of analyses meet the performance characteristics of the method. When results of sample spikes indicate atypical method performance, a quality control check standard must be analyzed to confirm that the measurements were performed in an in-control mode of operation.

8.1.1 The analyst must make an initial, one-time, demonstration of the ability to generate acceptable accuracy and precision with this method. This ability is established as described in Section 8.2.

8.1.2 In recognition of advances that are occurring in chromatography, the analyst is permitted certain options (detailed in Sections 10.4, 11.1, and 12.1) to improve the separations or lower the cost of measurements. Each time such a modification is made to the method, the analyst is required to repeat the procedure in Section 8.2.

8.1.3 Before processing any samples, the analyst must analyze a reagent water blank to demonstrate that interferences from the analytical system and glassware are under control. Each time a set of samples is extracted or reagents are changed, a reagent water blank must be processed as a safeguard against laboratory contamination.

8.1.4 The laboratory must, on an ongoing basis, spike and analyze a minimum of 10% of all samples to monitor and evaluate laboratory data quality. This procedure is described in Section 8.3.

8.1.5 The laboratory must, on an ongoing basis, demonstrate through the analyses of quality control check standards that the operation of the measurement system is in control. This procedure is described in Section 8.4. The frequency of the check standard analyses is equivalent to 10% of all samples analyzed but may be reduced if spike recoveries from samples (Section 8.3) meet all specified quality control criteria.

8.1.6 The laboratory must maintain performance records to document the quality of data that is generated. This procedure is described in Section 8.5.

8.2 To establish the ability to generate acceptable accuracy and precision, the analyst must perform the following operations.

8.2.1 A quality control (QC) check sample concentrate is required containing each single-component parameter of interest at the following concentrations in acetone: 4,4′–DDD, 10 µg/mL; 4,4′–DDT, 10 µg/mL; endosulfan II, 10 µg/mL; endosulfan sulfate, 10 µg/mL; endrin, 10 µg/mL; any other single-component pesticide, 2 µg/mL. If this method is only to be used to analyze for PCBs, chlordane, or toxaphene, the QC check sample concentrate should contain the most representative multicomponent parameter at a concentration of 50 µg/mL in acetone. The QC check sample concentrate must be obtained from the U.S. Environmental Protection Agency, Environmental Monitoring and Support Laboratory in Cincinnati, Ohio, if available. If not available from that source, the QC check sample concentrate must be obtained from another external source. If not available from either source above, the QC check sample concentrate must be prepared by the laboratory using stock standards prepared independently from those used for calibration.

8.2.2 Using a pipet, prepare QC check samples at the test concentrations shown in Table 3 by adding 1.00 mL of QC check sample concentrate to each of four 1–L aliquots of reagent water.

8.2.3 Analyze the well-mixed QC check samples according to the method beginning in Section 10.

8.2.4 Calculate the average recovery ($\bar{X}$) in µg/mL; and the standard deviation of the recovery (s) in µg/mL, for each parameter using the four results.

8.2.5 For each parameter compare s and $\bar{X}$ with the corresponding acceptance criteria for precision and accuracy, respectively, found in Table 3. If s and $\bar{X}$ for all parameters of interest meet the acceptance criteria, the system performance is acceptable and analysis of actual samples can begin. If any individual s exceeds the precision limit or any individual $\bar{X}$ falls outside the range for accuracy, the system performance is unacceptable for that parameter.

NOTE: The large number of parameters in Table 3 present a substantial probability that one or more will fail at least one of the acceptance criteria when all parameters are analyzed.

8.2.6 When one or more of the parameters tested fail at least one of the acceptance criteria, the analyst must proceed according to Section 8.2.6.1 or 8.2.6.2.

8.2.6.1 Locate and correct the source of the problem and repeat the test for all parameters of interest beginning with Section 8.2.2.

8.2.6.2 Beginning with Section 8.2.2, repeat the test only for those parameters that failed to meet criteria. Repeated failure, however, will confirm a general problem with the measurement system. If this occurs, locate and correct the source of the problem and repeat the test for all compmunds of interest beginning with Section 8.2.2.

8.3 The laboratory must, on an ongoing basis, spike at least 10% of the samples from each sample site being monitored to assess accuracy. For laboratories analyzing one to ten samples per month, at least one spiked sample per month is required.

8.3.1 The concentration of the spike in the sample should be determined as follows:

8.3.1.1 If, as in compliance monitoring, the concentration of a specific parameter in the sample is being checked against a regulatory concentration limit, the spike should be at that limit or 1 to 5 times higher than the background concentration determined in Section 8.3.2, whichever concentration would be larger.

8.3.1.2 If the concentration of a specific parameter in the sample is not being checked against a limit specific to that parameter, the spike should be at the test concentration in Section 8.2.2 or 1 to 5 times higher than the background concentration determined in Section 8.3.2, whichever concentration would be larger.

8.3.1.3 If it is impractical to determine background levels before spiking (e.g., maximum holding times will be exceeded), the spike concentration should be (1) the regulatory concentration limit, if any; or, if none (2) the larger of either 5 times higher than the expected background concentration or the test concentration in Section 8.2.2.

8.3.2 Analyze one sample aliquot to determine the background concentration (B) of each parameter. If necessary, prepare a new QC check sample concentrate (Section 8.2.1) appropriate for the background concentrations in the sample. Spike a second sample aliquot with 1.0 mL of the QC check sample concentrate and analyze it to determine the concentration after spiking (A) of each parameter. Calculate each percent recovery (P) as 100(A–B)%/T, where T is the known true value of the spike.

8.3.3 Compare the percent recovery (P) for each parameter with the corresponding QC acceptance criteria found in Table 3. These acceptance criteria were calculated to include an allowance for error in measurement of both the background and spike concentrations, assuming a spike to background ratio of 5:1. This error will be accounted for to the extent that the analyst's spike to background ratio approaches 5:1.[10] If spiking was performed at a concentration lower than the test concentration in Section 8.2.2, the analyst must use either the QC acceptance criteria in Table 3, or optional QC acceptance criteria calculated for the specific spike concentration. To calculate optional acceptance criteria for the recovery of a parameter: (1)

Calculate accuracy (X′) using the equation in Table 4, substituting the spike concentration (T) for C; (2) calculate overall precision (S′) using the equation in Table 4, substituting X′ for X; (3) calculate the range for recovery at the spike concentration as (100 X′/T)±2.44(100 S′/T)%.[10]

8.3.4 If any individual P falls outside the designated range for recovery, that parameter has failed the acceptance criteria. A check standard containing each parameter that failed the criteria must be analyzed as described in Section 8.4.

8.4 If any parameter fails the acceptance criteria for recovery in Section 8.3, a QC check standard containing each parameter that failed must be prepared and analyzed.

NOTE: The frequency for the required analysis of a QC check standard will depend upon the number of parameters being simultaneously tested, the complexity of the sample matrix, and the performance of the laboratory. If the entire list of parameters in Table 3 must be measured in the sample in Section 8.3, the probability that the analysis of a QC check standard will be required is high. In this case the QC check standard should be routinely analyzed with the spike sample.

8.4.1 Prepare the QC check standard by adding 1.0 mL of QC check sample concentrate (Section 8.2.1 or 8.3.2) to 1 L of reagent water. The QC check standard needs only to contain the parameters that failed criteria in the test in Section 8.3.

8.4.2 Analyze the QC check standards to determine the concentration measured (A) of each parameter. Calculate each percent recovery (P_s) as 100 (A/T)%, where T is the true value of the standard concentration.

8.4.3 Compare the percent recovery (P_s) for each parameter with the corresponding QC acceptance criteria found in Table 3. Only parameters that failed the test in Section 8.3 need to be compared with these criteria. If the recovery of any such parameter falls outside the designated range, the laboratory performance for that parameter is judged to be out of control, and the problem must be immediately identified and corrected. The analytical result for that parameter in the unspiked sample is suspect and may not be reported for regulatory compliance purposes.

8.5 As part of the QC program for the laboratory, method accuracy for wastewater samples must be assessed and records must be maintained. After the analysis of five spiked wastewater samples as in Section 8.3, calculate the average percent recovery ($\bar{P}$) and the standard deviation of the percent recovery (s_p). Express the accuracy assessment as a percent recovery interval from $\bar{P}-2\ s_p$ to $\bar{P}+2\ s_p$. If $\bar{P}$=90% and s_p=10%, for example, the accuracy interval is expressed as 70–110%. Update the accuracy assessment for each parameter on a regular basis (e.g. after each five to ten new accuracy measurements).

8.6 It is recommended that the laboratory adopt additional quality assurance practices for use with this method. The specific practices that are most productive depend upon the needs of the laboratory and the nature of the samples. Field duplicates may be analyzed to assess the precision of the environmental measurements. When doubt exists over the identification of a peak on the chromatogram, confirmatory techniques such as gas chromatography with a dissimilar column, specific element detector, or mass spectrometer must be used. Whenever possible, the laboratory should analyze standard reference materials and participate in relevant performance evaluation studies.

9. Sample Collection, Preservation, and Handling

9.1 Grab samples must be collected in glass containers. Conventional sampling practices[11] should be followed, except that the bottle must not be prerinsed with sample before collection. Composite samples should be collected in refrigerated glass containers in accordance with the requirements of the program. Automatic sampling equipment must be as free as possible of Tygon tubing and other potential sources of contamination.

9.2 All samples must be iced or refrigerated at 4 °C from the time of collection until extraction. If the samples will not be extracted within 72 h of collection, the sample should be adjusted to a pH range of 5.0 to 9.0 with sodium hydroxide solution or sulfuric acid. Record the volume of acid or base used. If aldrin is to be determined, add sodium thiosulfate when residual chlorine is present. EPA Methods 330.4 and 330.5 may be used for measurement of residual chlorine.[12] Field test kits are available for this purpose.

9.3 All samples must be extracted within 7 days of collection and completely analyzed within 40 days of extraction.[2]

10. Sample Extraction

10.1 Mark the water meniscus on the side of the sample bottle for later determination of sample volume. Pour the entire sample into a 2–L separatory funnel.

10.2 Add 60 mL of methylene chloride to the sample bottle, seal, and shake 30 s to rinse the inner surface. Transfer the solvent to the separatory funnel and extract the sample by shaking the funnel for 2 min. with periodic venting to release excess pressure. Allow the organic layer to separate from the water phase for a minimum of 10 min. If the emulsion interface between layers is more than one-third the volume of the solvent layer, the analyst must employ mechanical techniques to complete the phase separation.

The optium technique depends upon the sample, but may include stirring, filtration of the emulsion through glass wool, centrifugation, or other physical methods. Collect the methylene chloride extract in a 250–mL Erlenmeyer flask.

10.3 Add a second 60-mL volume of methylene chloride to the sample bottle and repeat the extraction procedure a second time, combining the extracts in the Erlenmeyer flask. Perform a third extraction in the same manner.

10.4 Assemble a Kuderna-Danish (K–D) concentrator by attaching a 10-mL concentrator tube to a 500-mL evaporative flask. Other concentration devices or techniques may be used in place of the K–D concentrator if the requirements of Section 8.2 are met.

10.5 Pour the combined extract through a solvent-rinsed drying column containing about 10 cm of anhydrous sodium sulfate, and collect the extract in the K–D concentrator. Rinse the Erlenmeyer flask and column with 20 to 30 mL of methylene chloride to complete the quantitative transfer.

10.6 Add one or two clean boiling chips to the evaporative flask and attach a three-ball Snyder column. Prewet the Snyder column by adding about 1 mL of methylene chloride to the top. Place the K–D apparatus on a hot water bath (60 to 65 °C) so that the concentrator tube is partially immersed in the hot water, and the entire lower rounded surface of the flask is bathed with hot vapor. Adjust the vertical position of the apparatus and the water temperature as required to complete the concentration in 15 to 20 min. At the proper rate of distillation the balls of the column will actively chatter but the chambers will not flood with condensed solvent. When the apparent volume of liquid reaches 1 mL, remove the K–D apparatus and allow it to drain and cool for at least 10 min.

10.7 Increase the temperature of the hot water bath to about 80 °C. Momeltarily remove the Snyder column, add 50 mL of hexane and a new boiling chip, and reattach the Snyder column. Concentrate the extract as in Section 10.6, except use hexane to prewet the column. The elapsed time of concentration should be 5 to 10 min.

10.8 Remove the Snyder column and rinse the flask and its lower joint into the concentrator tube with 1 to 2 mL of hexane. A 5-mL syringe is recommended for this operation. Stopper the concentrator tube and store refrigerated if further processing will not be performed immediately. If the extract will be stored longer than two days, it should be transferred to a Teflon-sealed screw-cap vial. If the sample extract requires no further cleanup, proceed with gas chromatographic analysis (Section 12). If the sample requires further cleanup, proceed to Section 11.

10.9 Determine the original sample volume by refilling the sample bottle to the mark and transferring the liquid to a 1000-mL graduated cylinder. Record the sample volume to the nearest 5 mL.

11. Cleanup and Separation

11.1 Cleanup procedures may not be necessary for a relatively clean sample matrix. If particular circumstances demand the use of a cleanup procedure, the analyst may use either procedure below or any other appropriate procedure. However, the analyst first must demonstrate that the requirements of Section 8.2 can be met using the method as revised to incorporate the cleanup procedure. The Florisil column allows for a select fractionation of the compounds and will eliminate polar interferences. Elemental sulfur, which interferes with the electron capture gas chromatography of certain pesticides, can be removed by the technique described in Section 11.3.

11.2 Florisil column cleanup:

11.2.1 Place a weight of Florisil (nominally 20 g) predetermined by calibration (Section 7.5), into a chromatographic column. Tap the column to settle the Florisil and add 1 to 2 cm of anhydrous sodium sulfate to the top.

11.2.2 Add 60 mL of hexane to wet and rinse the sodium sulfate and Florisil. Just prior to exposure of the sodium sulfate layer to the air, stop the elution of the hexane by closing the stopcock on the chromatographic column. Discard the eluate.

11.2.3 Adjust the sample extract volume to 10 mL with hexane and transfer it from the K–D concentrator tube onto the column. Rinse the tube twice with 1 to 2 mL of hexane, adding each rinse to the column.

11.2.4 Place a 500-mL K–D flask and clean concentrator tube under the chromatographic column. Drain the column into the flask until the sodium sulfate layer is nearly exposed. Elute the column with 200 mL of 6% ethyl ether in hexane (V/V) (Fraction 1) at a rate of about 5 mL/min. Remove the K–D flask and set it aside for later concentration. Elute the column again, using 200 mL of 15% ethyl ether in hexane (V/V) (Fraction 2), into a second K–D flask. Perform the third elution using 200 mL of 50% ethyl ether in hexane (V/V) (Fraction 3). The elution patterns for the pesticides and PCBs are shown in Table 2.

11.2.5 Concentrate the fractions as in Section 10.6, except use hexane to prewet the column and set the water bath at about 85 °C. When the apparatus is cool, remove the Snyder column and rinse the flask and its lower joint into the concentrator tube with hexane. Adjust the volume of each fraction to 10 mL with hexane and analyze by gas chromatography (Section 12).

11.3 Elemental sulfur will usually elute entirely in Fraction 1 of the Florisil column

cleanup. To remove sulfur interference from this fraction or the original extract, pipet 1.00 mL of the concentrated extract into a clean concentrator tube or Teflon-sealed vial. Add one to three drops of mercury and seal.[13] Agitate the contents of the vial for 15 to 30 s. Prolonged shaking (2 h) may be required. If so, this may be accomplished with a reciprocal shaker. Alternatively, activated copper powder may be used for sulfur removal.[14] Analyze by gas chromatography.

12. Gas Chromatography

12.1 Table 1 summarizes the recommended operating conditions for the gas chromatograph. Included in this table are retention times and MDL that can be achieved under these conditions. Examples of the separations achieved by Column 1 are shown in Figures 1 to 10. Other packed or capillary (open-tubular) columns, chromatographic conditions, or detectors may be used if the requirements of Section 8.2 are met.

12.2 Calibrate the system daily as described in Section 7.

12.3 If the internal standard calibration procedure is being used, the internal standard must be added to the sample extract and mixed thoroughly immediately before injection into the gas chromatograph.

12.4 Inject 2 to 5 µL of the sample extract or standard into the gas chromatograph using the solvent-flush technique.[15] Smaller (1.0 uL) volumes may be injected if automatic devices are employed. Record the volume injected to the nearest 0.05 µL, the total extract volume, and the resulting peak size in area or peak height units.

12.5 Identify the parameters in the sample by comparing the retention times of the peaks in the sample chromatogram with those of the peaks in standard chromatograms. The width of the retention time window used to make identifications should be based upon measurements of actual retention time variations of standards over the course of a day. Three times the standard deviation of a retention time for a compound can be used to calculate a suggested window size; however, the experience of the analyst should weigh heavily in the interpretation of chromatograms.

12.6 If the response for a peak exceeds the working range of the system, dilute the extract and reanalyze.

12.7 If the measurement of the peak response is prevented by the presence of interferences, further cleanup is required.

13. Calculations

13.1 Determine the concentration of individual compounds in the sample.

13.1.1 If the external standard calibration procedure is used, calculate the amount of material injected from the peak response using the calibration curve or calibration factor determined in Section 7.2.2. The concentration in the sample can be calculated from Equation 2.

$$\text{Concentration } (\mu g/L) = \frac{(A)(V_t)}{(V_i)(V_s)}$$

Equation 2

where:

A=Amount of material injected (ng).
V_i=Volume of extract injected (µL).
V_t=Volume of total extract (µL).
V_s=Volume of water extracted (mL).

13.1.2 If the internal standard calibration procedure is used, calculate the concentration in the sample using the response factor (RF) determined in Section 7.3.2 and Equation 3.

$$\text{Concentration } (\mu g/L) = \frac{(A_s)(I_s)}{(A_{is})(RF)(V_o)}$$

Equation 3

where:

A_s=Response for the parameter to be measured.
A_{is}=Response for the internal standard.
I_s=Amount of internal standard added to each extract (µg).
V_o=Volume of water extracted (L).

13.2 When it is apparent that two or more PCB (Aroclor) mixtures are present, the Webb and McCall procedure[16] may be used to identify and quantify the Aroclors.

13.3 For multicomponent mixtures (chlordane, toxaphene, and PCBs) match retention times of peaks in the standards with peaks in the sample. Quantitate every identifiable peak unless interference with individual peaks persist after cleanup. Add peak height or peak area of each identified peak in the chromatogram. Calculate as total response in the sample versus total response in the standard.

13.4 Report results in µg/L without correction for recovery data. All QC data obtained should be reported with the sample results.

14. Method Performance

14.1 The method detection limit (MDL) is defined as the minimum concentration of a substance that can be measured and reported with 99% confidence that the value is above zero.[1] The MDL concentrations listed in Table 1 were obtained using reagent water.[17] Similar results were achieved using representative wastewaters. The MDL actually achieved in a given analysis will vary depending on instrument sensitivity and matrix effects.

14.2 This method has been tested for linearity of spike recovery from reagent water and has been demonstrated to be applicable

over the concentration range from 4×MDL to 1000×MDL with the following exceptions: Chlordane recovery at 4×MDL was low (60%); Toxaphene recovery was demonstrated linear over the range of 10×MDL to 1000×MDL.[17]

14.3 This method was tested by 20 laboratories using reagent water, drinking water, surface water, and three industrial wastewaters spiked at six concentrations.[18] Concentrations used in the study ranged from 0.5 to 30 µg/L for single-component pesticides and from 8.5 to 400 µg/L for multicomponent parameters. Single operator precision, overall precision, and method accuracy were found to be directly related to the concentration of the parameter and essentially independent of the sample matrix. Linear equations to describe these relationships are presented in Table 4.

REFERENCES

1. 40 CFR part 136, appendix B.

2. ''Determination of Pesticides and PCBs in Industrial and Municipal Wastewaters,'' EPA 600/4–82–023, National Technical Information Service, PB82–214222, Springfield, Virginia 22161, April 1982.

3. ASTM Annual Book of Standards, Part 31, D3694–78. ''Standard Practices for Preparation of Sample Containers and for Preservation of Organic Constituents,'' American Society for Testing and Materials, Philadelphia.

4. Giam, C.S., Chan, H.S., and Nef, G.S., ''Sensitive Method for Determination of Phthalate Ester Plasticizers in Open-Ocean Biota Samples,'' *Analytical Chemistry, 47,* 2225 (1975).

5. Giam, C.S., Chan, H.S. ''Control of Blanks in the Analysis of Phthalates in Air and Ocean Biota Samples,'' U.S. National Bureau of Standards, Special Publication 442, pp. 701–708, 1976.

6. ''Carcinogens—Working With Carcinogens,'' Department of Health, Education, and Welfare, Public Health Service, Center for Disease Control, National Institute for Occupational Safety and Health, Publication No. 77–206, August 1977.

7. ''OSHA Safety and Health Standards, General Industry,'' (29 CFR part 1910), Occupational Safety and Health Administration, OSHA 2206 (Revised, January 1976).

8. ''Safety in Academic Chemistry Laboratories,'' American Chemical Society Publication, Committee on Chemical Safety, 3rd Edition, 1979.

9. Mills, P.A. ''Variation of Florisil Activity: Simple Method for Measuring Absorbent Capacity and Its Use in Standardizing Florisil Columns,'' *Journal of the Association of Official Analytical Chemists, 51,* 29, (1968).

10. Provost, L.P., and Elder, R.S. ''Interpretation of Percent Recovery Data,'' *American Laboratory, 15,* 58–63 (1983). (The value 2.44 used in the equation in Section 8.3.3 is two times the value 1.22 derived in this report.)

11. ASTM Annual Book of Standards, Part 31, D3370–76. ''Standard Practices for Sampling Water,'' American Society for Testing and Materials, Philadelphia.

12. ''Methods 330.4 (Titrimetric, DPD–FAS) and 330.5 (Spectrophotometric, DPD) for Chlorine, Total Residual,'' Methods for Chemical Analysis of Water and Wastes, EPA–600/4–79–020, U.S. Environmental Protection Agency, Environmental Monitoring and Support Laboratory, Cincinnati, Ohio 45268, March 1979.

13. Goerlitz, D.F., and Law, L.M. *Bulletin for Environmental Contamination and Toxicology, 6,* 9 (1971).

14. ''Manual of Analytical Methods for the Analysis of Pesticides in Human and Environmental Samples,'' EPA–600/8–80–038, U.S. Environmental Protection Agency, Health Effects Research Laboratory, Research Triangle Park, North Carolina.

15. Burke, J.A. ''Gas Chromatography for Pesticide Residue Analysis; Some Practical Aspects,'' *Journal of the Association of Official Analytical Chemists, 48,* 1037 (1965).

16. Webb, R.G., and McCall, A.C. ''Quantitative PCB Standards for Election Capture Gas Chromatography,'' *Journal of Chromatographic Science, 11,* 366 (1973).

17. ''Method Detection Limit and Analytical Curve Studies, EPA Methods 606, 607, and 608,'' Special letter report for EPA Contract 68–03–2606, U.S. Environmental Protection Agency, Environmental Monitoring and Support Laboratory, Cincinnati, Ohio 45268, June 1980.

18. ''EPA Method Study 18 Method 608—Organochlorine Pesticides and PCBs,'' EPA 600/4–84–061, National Technical Information Service, PB84–211358, Springfield, Virginia 22161, June 1984.

TABLE 1—CHROMATOGRAPHIC CONDITIONS AND METHOD DETECTION LIMITS

Parameter	Retention time (min)		Method detection limit (µg/L)
	Col. 1	Col. 2	
α-BHC	1.35	1.82	0.003
γ-BHC	1.70	2.13	0.004
β-BHC	1.90	1.97	0.006
Heptachlor	2.00	3.35	0.003
δ-BHC	2.15	2.20	0.009
Aldrin	2.40	4.10	0.004
Heptachlor epoxide	3.50	5.00	0.083
Endosulfan I	4.50	6.20	0.014
4,4′-DDE	5.13	7.15	0.004
Dieldrin	5.45	7.23	0.002
Endrin	6.55	8.10	0.006
4,4′-DDD	7.83	9.08	0.011
Endosulfan II	8.00	8.28	0.004
4,4′-DDT	9.40	11.75	0.012
Endrin aldehyde	11.82	9.30	0.023
Endosulfan sulfate	14.22	10.70	0.066
Chlordane	mr	mr	0.014
Toxaphene	mr	mr	0.24

TABLE 1—CHROMATOGRAPHIC CONDITIONS AND METHOD DETECTION LIMITS—Continued

Parameter	Retention time (min)		Method detection limit (µg/L)
	Col. 1	Col. 2	
PCB–1016	mr	mr	nd
PCB–1221	mr	mr	nd
PCB–1232	mt	mr	nd
PCB–1242	mr	mr	0.065
PCB–1248	mr	mr	nd
PCB–1254	mr	mr	nd
PCB–1260	mr	mr	nd

AColumn 1 conditions: Supelcoport (100/120 mesh) coated with 1.5% SP–2250/1.95% SP–2401 packed in a 1.8 m long × 4 mm ID glass column with 5% methane/95% argon carrier gas at 60 mL/min flow rate. Column temperature held isothermal at 200 °C, except for PCB–1016 through PCB–1248, should be measured at 160 °C.

AColumn 2 conditions: Supelcoport (100/120 mesh) coated with 3% OV–1 packed in a 1.8 m long × 4 mm ID glass column with 5% methane/95% argon carrier gas at 60 mL/min flow rate. Column temperature held isothermal at 200 °C for the pesticides; at 140 °C for PCB–1221 and 1232; and at 170 °C for PCB–1016 and 1242 to 1268.

Amr=Multiple peak response. See Figures 2 thru 10.

And=Not determined.

TABLE 2—DISTRIBUTION OF CHLORINATED PESTICIDES AND PCBS INTO FLORISIL COLUMN FRACTIONS [2]

Parameter	Percent recovery by fraction [a]		
	1	2	3
Aldrin	100		
α-BHC	100		
β-BHC	97		
δ-BHC	98		
γ-BHC	100		
Chlordane	100		
4,4′–DDD	99		
4,4′–DDE	98		
4,4′–DDT	100		
Dieldrin	0	100	
Endosulfan I	37	64	
Endosulfan II	0	7	91
Endosulfan sulfate	0	0	106
Endrin	4	96	
Endrin aldehyde	0	68	26
Heptachlor	100		
Heptachlor epoxide	100		
Toxaphene	96		
PCB–1016	97		
PCB–1221	97		
PCB–1232	95	4	
PCB–1242	97		
PCB–1248	103		

TABLE 2—DISTRIBUTION OF CHLORINATED PESTICIDES AND PCBS INTO FLORISIL COLUMN FRACTIONS [2]—Continued

Parameter	Percent recovery by fraction [a]		
	1	2	3
PCB–1254	90		
PCB–1260	95		

[a] Eluant composition:
Fraction 1–6% ethyl ether in hexane.
Fraction 2–15% ethyl ether in hexane.
Fraction 3–50% ethyl ether in hexane.

TABLE 3—QC ACCEPTANCE CRITERIA—METHOD 608

Parameter	Test conc. (µg/L)	Limit for s (µg/L)	Range for X (µg/L)	Range for P, P_s(%)
Aldrin	2.0	0.42	1.08–2.24	42–122
α-BHC	2.0	0.48	0.98–2.44	37–134
β-BHC	2.0	0.64	0.78–2.60	17–147
δ-BHC	2.0	0.72	1.01–2.37	19–140
γ-BHC	2.0	0.46	0.86–2.32	32–127
Chlordane	50	10.0	27.6–54.3	45–119
4,4′–DDD	10	2.8	4.8–12.6	31–141
4,4′–DDE	2.0	0.55	1.08–2.60	30–145
4,4′–DDT	10	3.6	4.6–13.7	25–160
Dieldrin	2.0	0.76	1.15–2.49	36–146
Endosulfan I	2.0	0.49	1.14–2.82	45–153
Endosulfan II	10	6.1	2.2–17.1	D–202
Endosulfan Sulfate	10	2.7	3.8–13.2	26–144
Endrin	10	3.7	5.1–12.6	30–147
Heptachlor	2.0	0.40	0.86–2.00	34–111
Heptachlor epoxide	2.0	0.41	1.13–2.63	37–142
Toxaphene	50.0	12.7	27.8–55.6	41–126
PCB-1016	50	10.0	30.5–51.5	50–114
PCB-1221	50	24.4	22.1–75.2	15–178
PCB-1232	50	17.9	14.0–98.5	10–215
PCB-1242	50	12.2	24.8–69.6	39–150
PCB-1248	50	15.9	29.0–70.2	38–158
PCB-1254	50	13.8	22.2–57.9	29–131
PCB-1260	50	10.4	18.7–54.9	8–127

s=Standard deviation of four recovery measurements, in µg/L (Section 8.2.4).

$\bar{X}$=Average recovery for four recovery measurements, in µg/L (Section 8.2.4).

P, P_s=Percent recovery measured (Section 8.3.2, Section 8.4.2).

D=Detected; result must be greater than zero.

NOTE: These criteria are based directly upon the method performance data in Table 4. Where necessary, the limits for recovery have been broadened to assure applicability of the limits to concentrations below those used to develop Table 4.

TABLE 4—METHOD ACCURACY AND PRECISION AS FUNCTIONS OF CONCENTRATION—METHOD 608

Parameter	Accuracy, as recovery, X′ (µg/L)	Single analyst precision, s_r′ (µg/L)	Overall precision, S′ (µg/L)
Aldrin	0.81C+0.04	$0.16\bar{X}-0.04$	$0.20\bar{X}-0.01$
α-BHC	0.84C+0.03	$0.13\bar{X}+0.04$	$0.23\bar{X}-0.00$
β-BHC	0.81C+0.07	$0.22\bar{X}-0.02$	$0.33\bar{X}-0.05$
δ-BHC	0.81C+0.07	$0.18\bar{X}+0.09$	$0.25\bar{X}+0.03$
γ-BHC	0.82C−0.05	$0.12\bar{X}+0.06$	$0.22\bar{X}+0.04$
Chlordane	0.82C−0.04	$0.13\bar{X}+0.13$	$0.18\bar{X}+0.18$
4,4′-DDD	0.84C+0.30	$0.20\bar{X}-0.18$	$0.27\bar{X}-0.14$
4,4′-DDE	0.85C+0.14	$0.13\bar{X}+0.06$	$0.28\bar{X}-0.09$
4,4′-DDT	0.93C−0.13	$0.17\bar{X}+0.39$	$0.31\bar{X}-0.21$
Dieldrin	0.90C+0.02	$0.12\bar{X}+0.19$	$0.16\bar{X}+0.16$

TABLE 4—METHOD ACCURACY AND PRECISION AS FUNCTIONS OF CONCENTRATION—METHOD 608—Continued

Parameter	Accuracy, as recovery, X′ (μg/L)	Single analyst precision, s_r′ (μg/L)	Overall precision, S′ (μg/L)
Endosulfan I	0.97C+0.04	0.10$\bar{X}$+0.07	0.18$\bar{X}$+0.08
Endosulfan II	0.93C+0.34	0.41$\bar{X}$—0.65	0.47$\bar{X}$ − 0.20
Endosulfan Sulfate	0.89C − 0.37	0.13$\bar{X}$+0.33	0.24$\bar{X}$+0.35
Endrin	0.89C − 0.04	0.20$\bar{X}$+0.25	0.24$\bar{X}$+0.25
Heptachlor	0.69C+0.04	0.06$\bar{X}$+0.13	0.16$\bar{X}$+0.08
Heptachlor epoxide	0.89C+0.10	0.18$\bar{X}$ − 0.11	0.25$\bar{X}$ − 0.08
Toxaphene	0.80C+1.74	0.09$\bar{X}$+3.20	0.20$\bar{X}$+0.22
PCB–1016	0.81C+0.50	0.13$\bar{X}$+0.15	0.15$\bar{X}$+0.45
PCB–1221	0.96C+0.65	0.29$\bar{X}$ − 0.76	0.35$\bar{X}$ − 0.62
PCB–1232	0.91C+10.79	0.21$\bar{X}$ − 1.93	0.31$\bar{X}$+3.50
PCB–1242	0.93C+0.70	0.11$\bar{X}$+1.40	0.21$\bar{X}$+1.52
PCB–1248	0.97C+1.06	0.17$\bar{X}$+0.41	0.25$\bar{X}$ − 0.37
PCB–1254	0.76C+2.07	0.15$\bar{X}$+1.66	0.17$\bar{X}$+3.62
PCB–1260	0.66C+3.76	0.22$\bar{X}$ − 2.37	0.39$\bar{X}$ − 4.86

X′=Expected recovery for one or more measurements of a sample containing a concentration of C, in μg/L.
s_r′=Expected single analyst standard deviation of measurements at an average concentration found of $\bar{X}$, in μg/L.
S′=Expected interlaboratory standard deviation of measurements at an average concentration found of $\bar{X}$, in μg/L.
C=True value for the concentration, in μg/L.
$\bar{X}$=Average recovery found for measurements of samples containing a concentration of C, in μg/L.

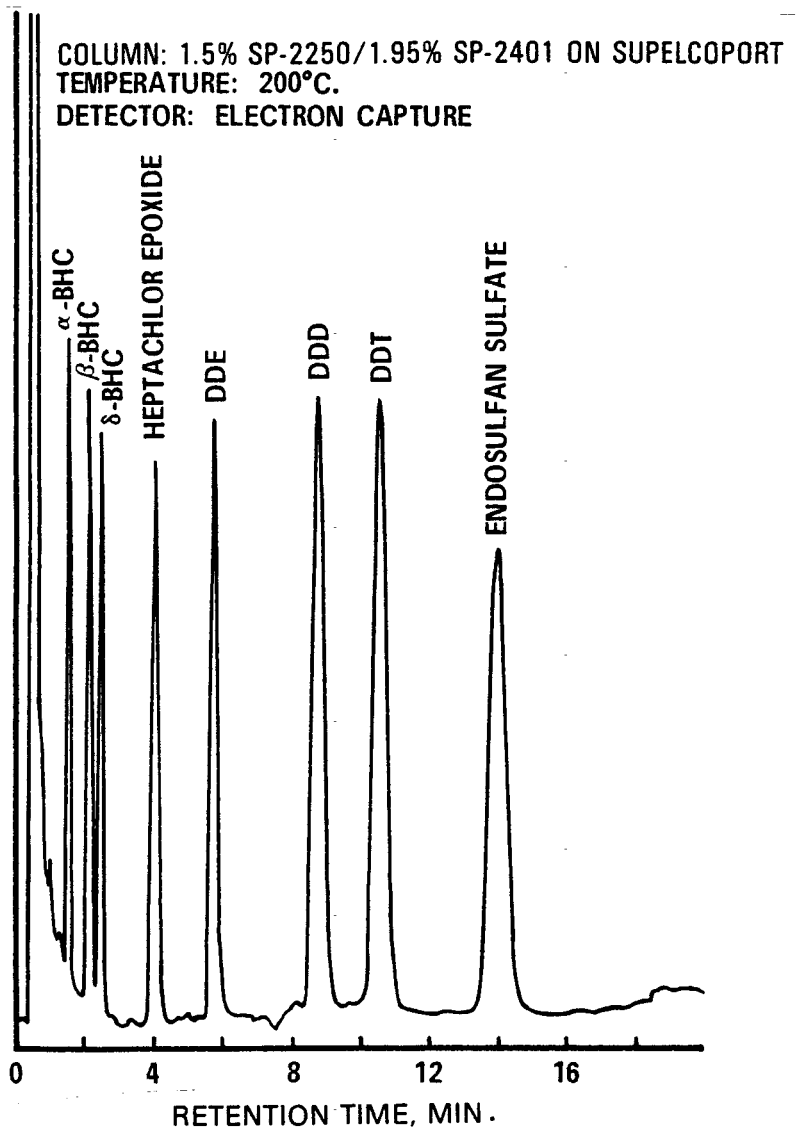


Figure 1. Gas chromatogram of pesticides.

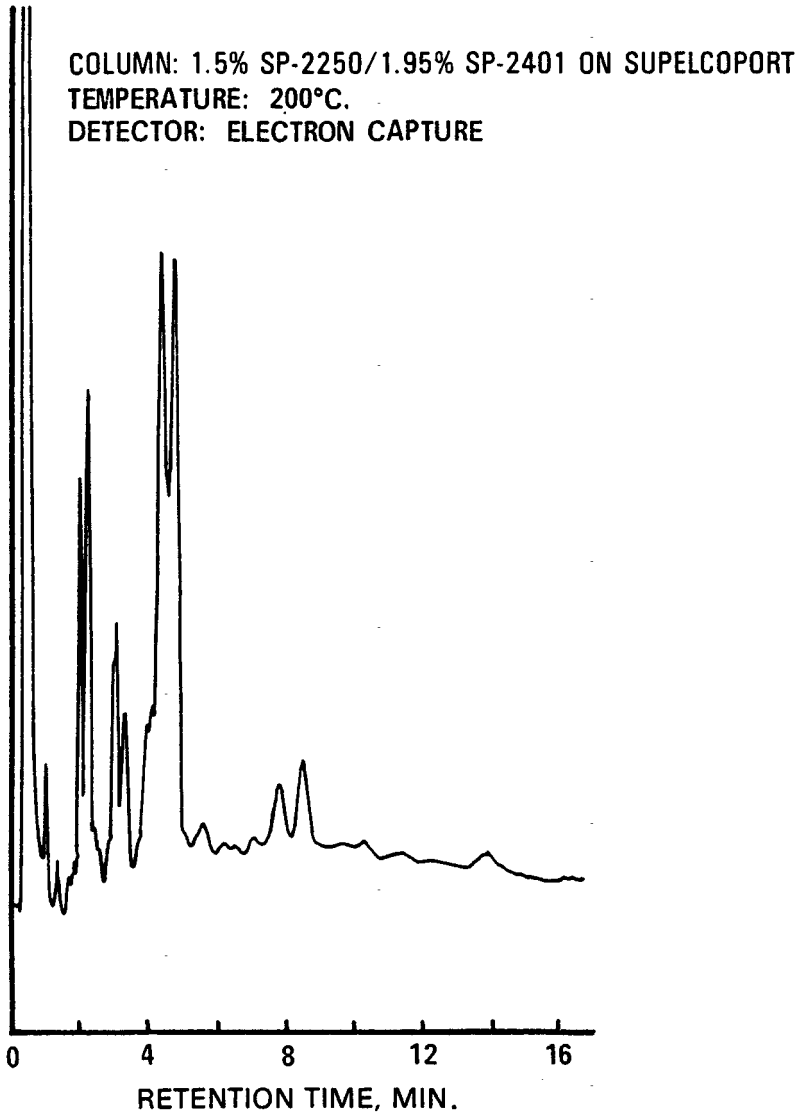


Figure 2. Gas chromatogram of chlordane.

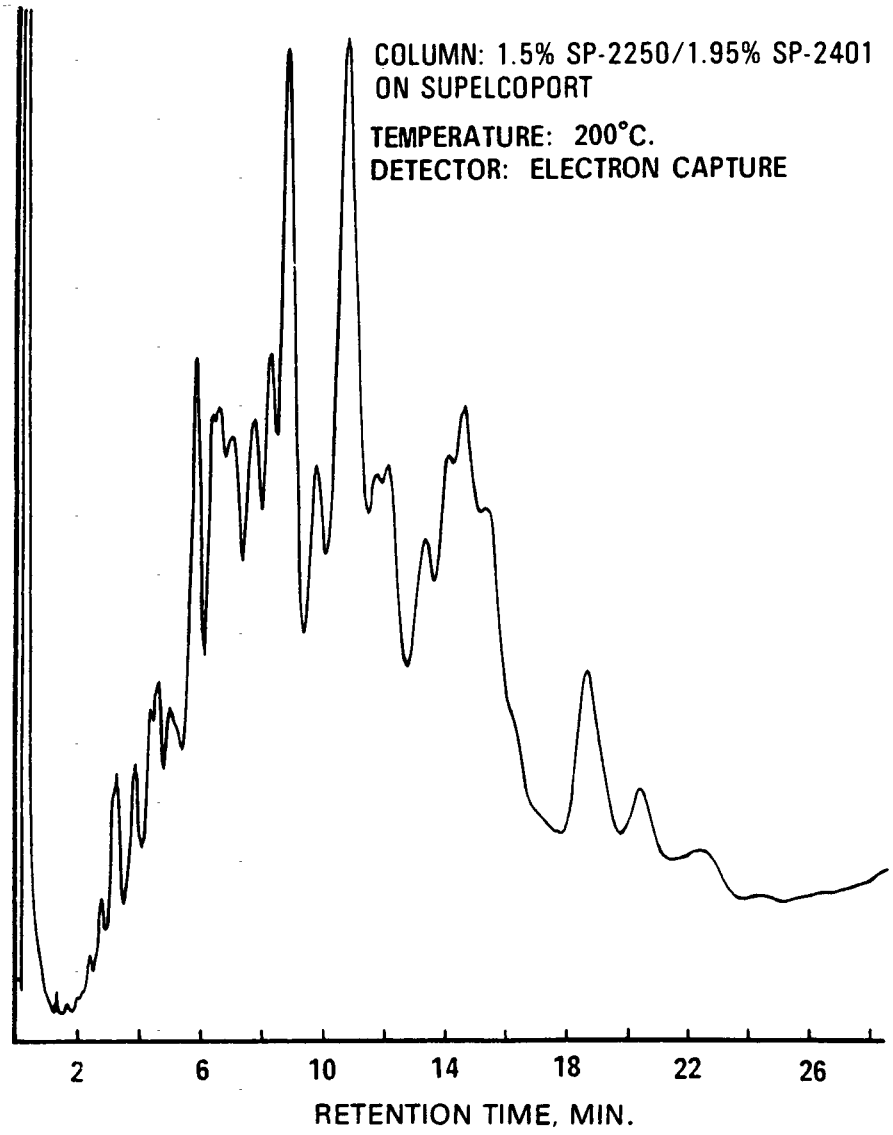


Figure 3. Gas chromatogram of toxaphene.

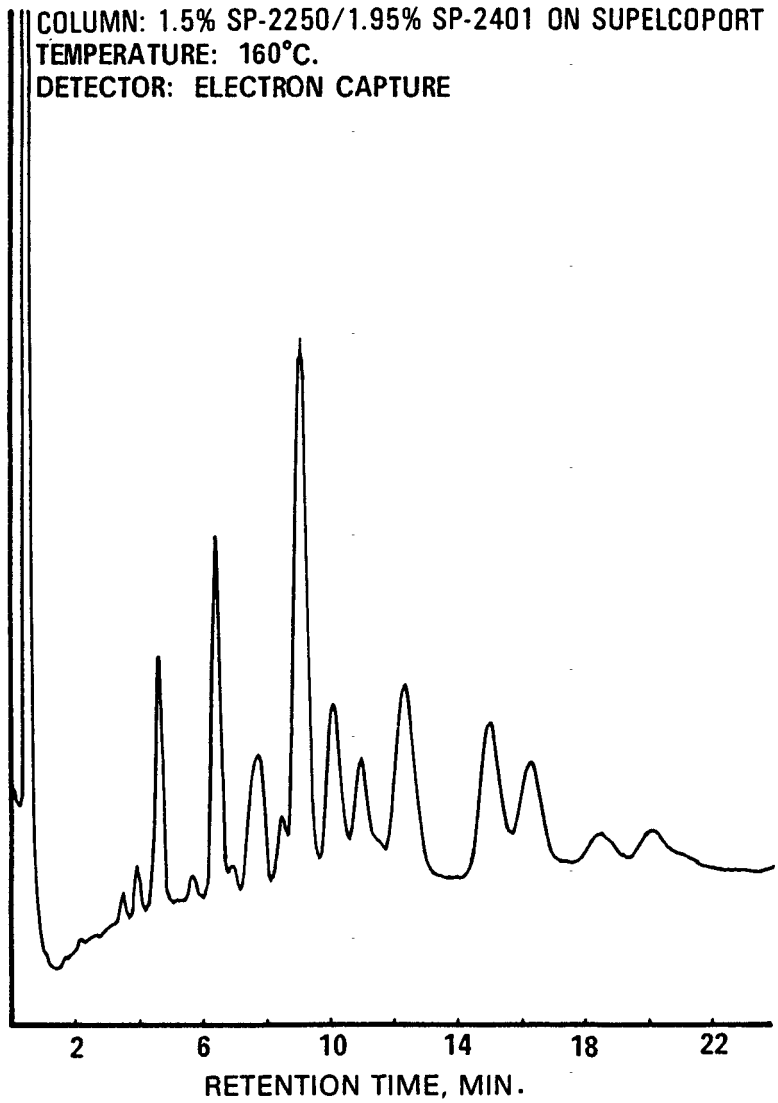


Figure 4. Gas chromatogram of PCB-1016.

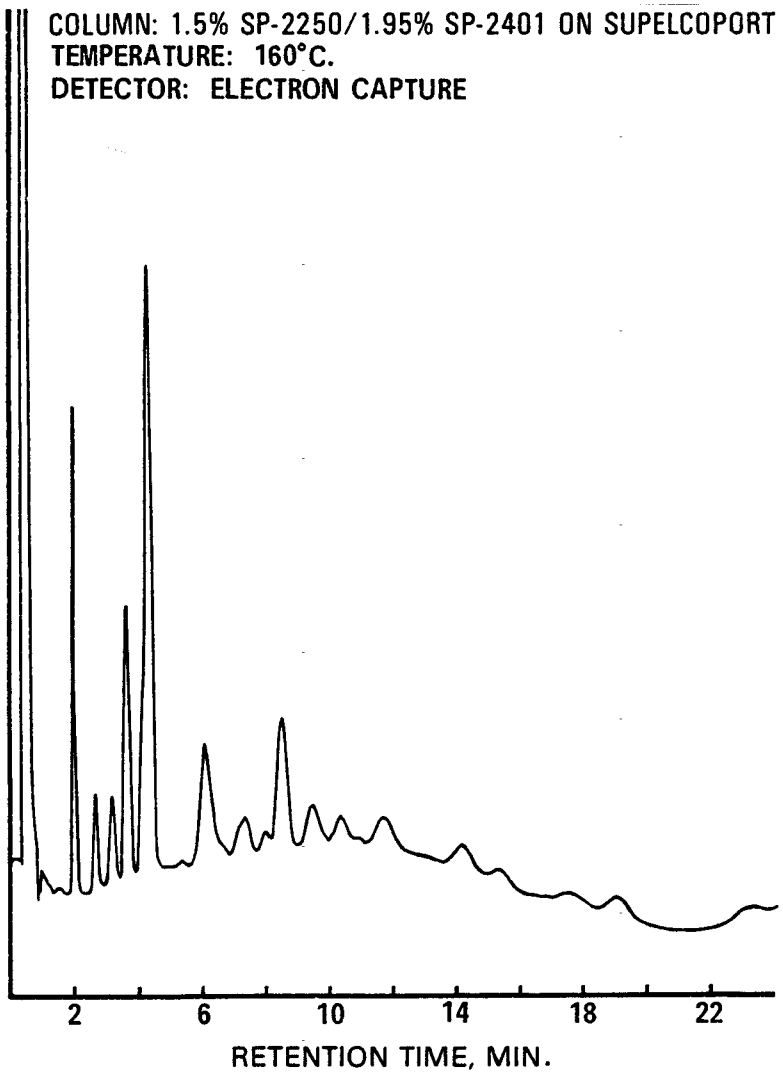


Figure 5. Gas chromatogram of PCB-1221.

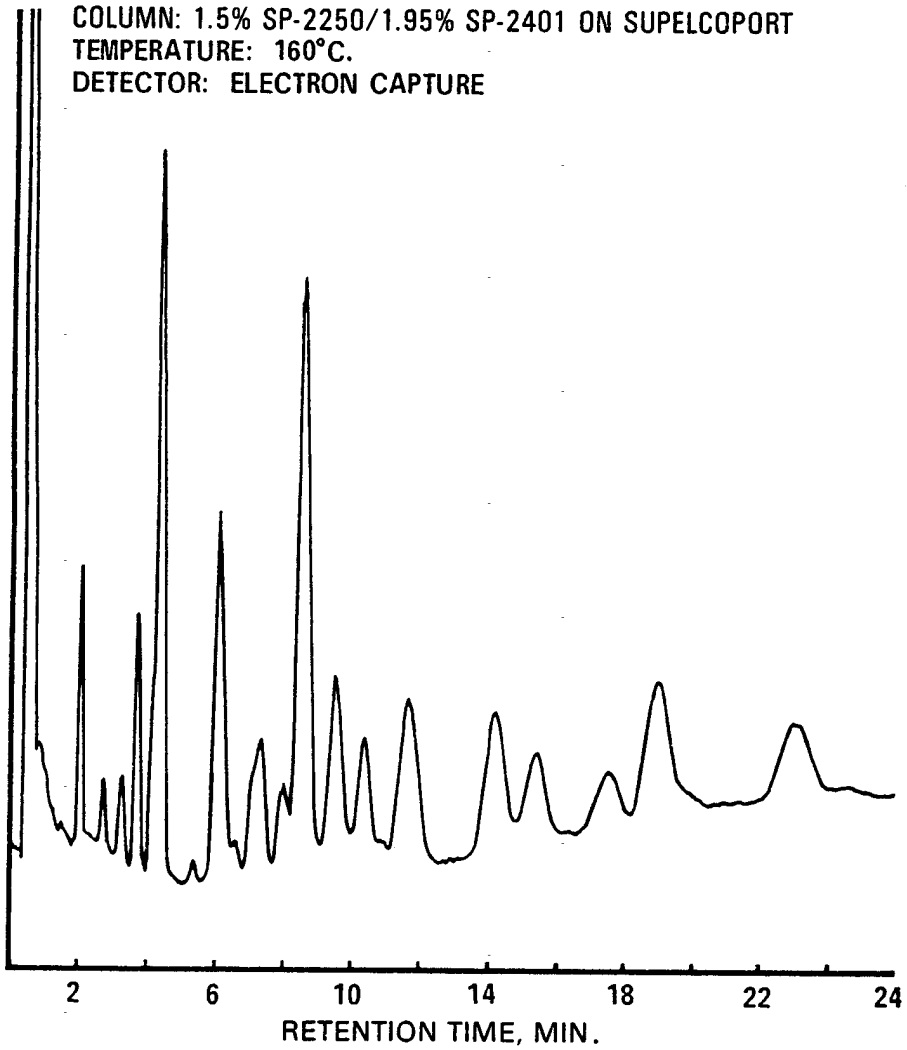


Figure 6. Gas chromatogram of PCB-1232.

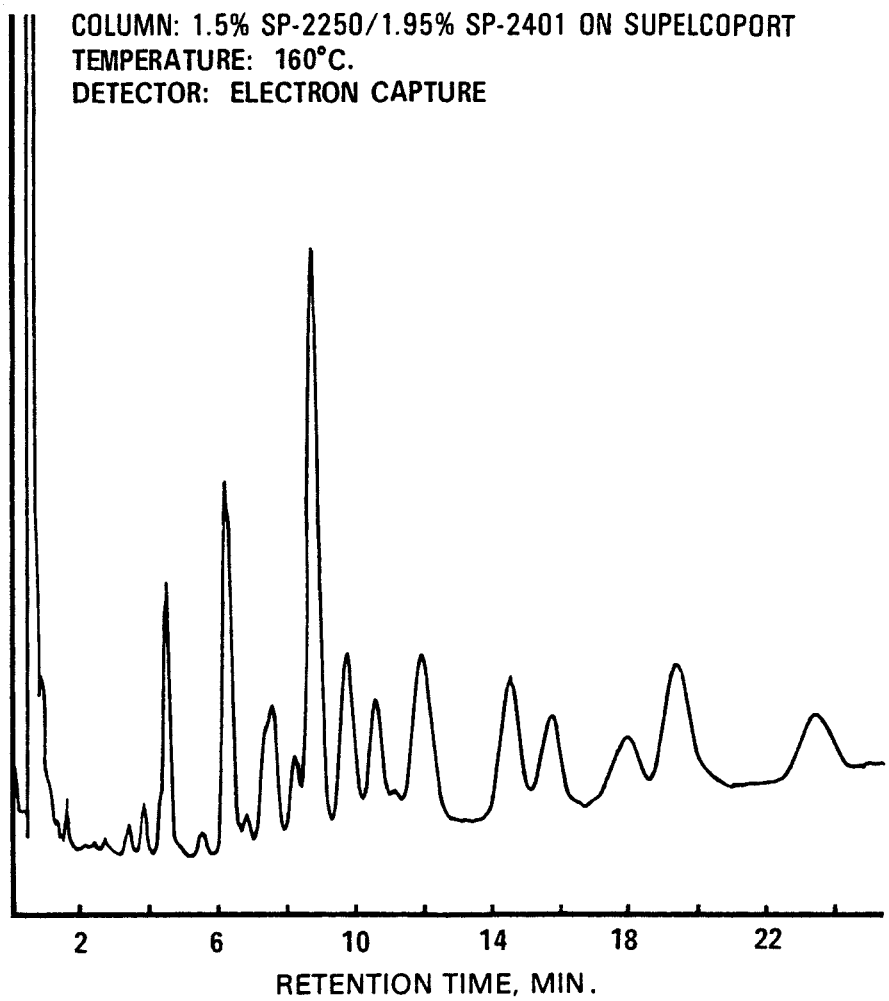


Figure 7. Gas chromatogram of PCB-1242.

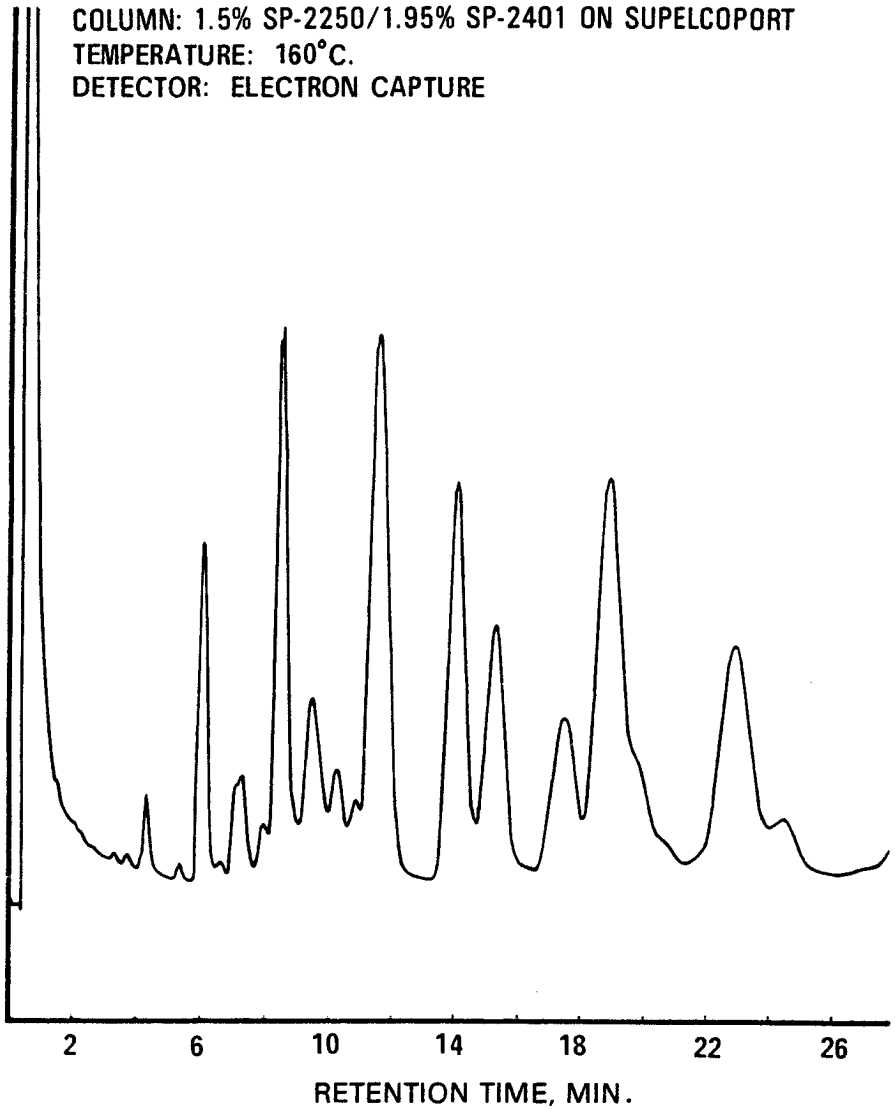


Figure 8. Gas chromatogram of PCB-1248.

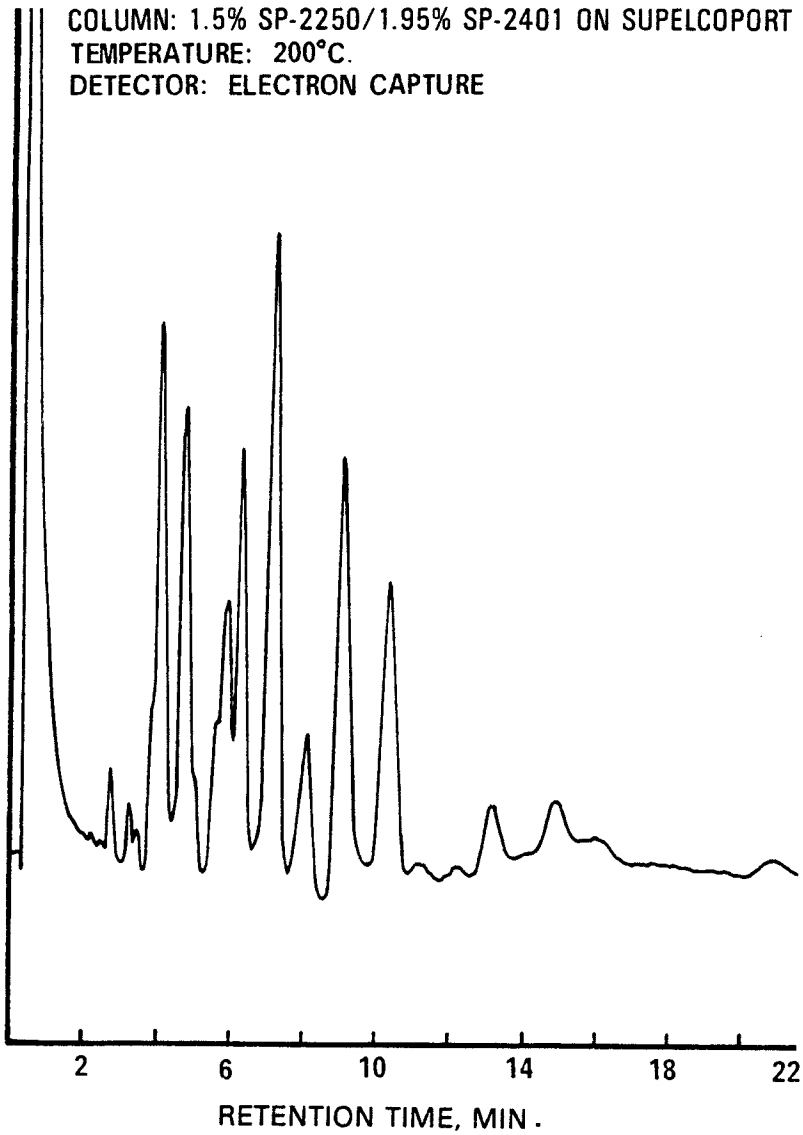


Figure 9. Gas chromatogram of PCB-1254.

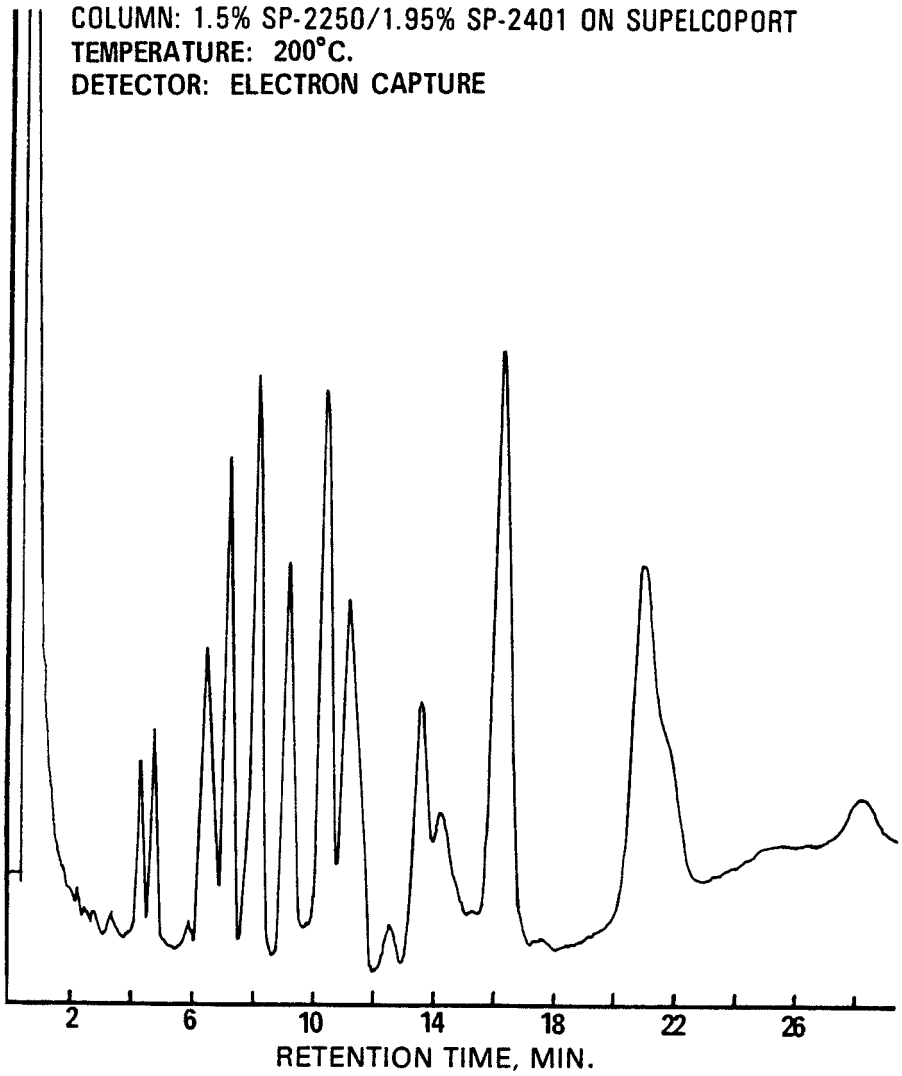


Figure 10. Gas chromatogram of PCB-1260.

METHOD 609—NITROAROMATICS AND ISOPHORONE

1. Scope and Application

1.1 This method covers the determination of certain nitroaromatics and isophorone. The following parameters may be determined by this method:

Parameter	STORET No.	CAS No.
2,4-Dinitrotoluene	34611	121–14–2
2,6-Dinitrotoluene	34626	606–20–2
Isophorone	34408	78–59–1
Nitrobenzene	34447	98–95–3

1.2 This is a gas chromatographic (GC) method applicable to the determination of the compounds listed above in municipal and industrial discharges as provided under 40 CFR 136.1. When this method is used to analyze unfamiliar samples for any or all of the compounds above, compound identifications should be supported by at least one additional qualitative technique. This method describes analytical conditions for a second gas chromatographic column that can be used to confirm measurements made with the primary column. Method 625 provides gas chromatograph/mass spectrometer (GC/MS) conditions appropriate for the qualitative and quantitative confirmation of results for all of the parameters listed above, using the extract produced by this method.

1.3 The method detection limit (MDL, defined in Section 14.1)[1] for each parameter is listed in Table 1. The MDL for a specific wastewater may differ from those listed, depending upon the nature of interferences in the sample matrix.

1.4 The sample extraction and concentration steps in this method are essentially the same as in Methods 606, 608, 611, and 612. Thus, a single sample may be extracted to measure the parameters included in the scope of each of these methods. When cleanup is required, the concentration levels must be high enough to permit selecting aliquots, as necessary, to apply appropriate cleanup procedures. The analyst is allowed the latitude, under Section 12, to select chromatographic conditions appropriate for the simultaneous measurement of combinations of these parameters.

1.5 Any modification of this method, beyond those expressly permitted, shall be considered as a major modification subject to application and approval of alternate test procedures under 40 CFR 136.4 and 136.5.

1.6 This method is restricted to use by or under the supervision of analysts experienced in the use of a gas chromatograph and in the interpretation of gas chromatograms. Each analyst must demonstrate the ability to generate acceptable results with this method using the procedure described in Section 8.2.

2. Summary of Method

2.1 A measured volume of sample, approximately 1–L, is extracted with methylene chloride using a separatory funnel. The methylene chloride extract is dried and exchanged to hexane during concentration to a volume of 10 mL or less. Isophorone and nitrobenzene are measured by flame ionization detector gas chromatography (FIDGC). The dinitrotoluenes are measured by electron capture detector gas chromatography (ECDGC).[2]

2.2 The method provides a Florisil column cleanup procedure to aid in the elimination of interferences that may be encountered.

3. Interferences

3.1 Method interferences may be caused by contaminants in solvents, reagents, glassware, and other sample processing hardware that lead to discrete artifacts and/or elevated baseliles in gas chromatograms. All of these materials must be routinely demonstrated to be free from interferences under the conditions of the analysis by running laboratory reagent blanks as described in Section 8.1.3.

3.1.1 Glassware must be scrupulously cleaned.[3] Clean all glassware as soon as possible after use by rinsing with the last solvent used in it. Solvent rinsing should be followed by detergent washing with hot water, and rinses with tap water and distilled water. The glassware should then be drained dry, and heated in a muffle furnace at 400 °C for 15 to 30 min. Some thermally stable materials, such as PCBs, may not be eliminated by this treatment. Solvent rinses with acetone and pesticide quality hexane may be substituted for the muffle furnace heating. Thorough rinsing with such solvents usually eliminates PCB interference. Volumetric ware should not be heated in a muffle furnace. After drying and cooling, glassware should be sealed and stored in a clean environment to prevent any accumulation of dust or other contaminants. Store inverted or capped with aluminum foil.

3.1.2 The use of high purity reagents and solvents helps to minimize interference problems. Purification of solvents by distillation in all-glass systems may be required.

3.2 Matrix interferences may be caused by contaminants that are co-extracted from the sample. The extent of matrix interferences will vary considerably from source to source, depending upon the nature and diversity of the industrial complex or municipality being sampled. The cleanup procedure in Section 11 can be used to overcome many of these interferences, but unique samples may require additional cleanup approaches to achieve the MDL listed in Table 1.

4. Safety

4.1 The toxicity or carcinogenicity of each reagent used in this method has not been precisely defined; however, each chemical compound should be treated as a potential health hazard. From this viewpoint, exposure to these chemicals must be reduced to the lowest possible level by whatever means available. The laboratory is responsible for maintaining a current awareness file of OSHA regulations regarding the safe handling of the chemicals specified in this method. A reference file of material data handling sheets should also be made available to all personnel involved in the chemical analysis. Additional references to laboratory safety are available and have been identified[4-6] for the information of the analyst.

5. Apparatus and Materials

5.1 Sampling equipment, for discrete or composite sampling.

5.1.1 Grab sample bottle—1–L or 1-qt, amber glass, fitted with a screw cap lined with Teflon. Foil may be substituted for Teflon if the sample is not corrosive. If amber bottles are not available, protect samples from light. The bottle and cap liner must be washed, rinsed with acetone or methylene chloride, and dried before use to minimize contamination.

5.1.2 Automatic sampler (optional)—The sampler must incorporate glass sample containers for the collection of a minimum of 250 mL of sample. Sample containers must be kept refrigerated at 4°C and protected from light during compositing. If the sampler uses a peristaltic pump, a minimum length of compressible silicone rubber tubing may be used. Before use, however, the compressible tubing should be thoroughly rinsed with methanol, followed by repeated rinsings with distilled water to minimize the potential for contamination of the sample. An integrating flow meter is required to collect flow proportional composites.

5.2 Glassware (All specifications are suggested. Catalog numbers are included for illustration only.):

5.2.1 Separatory funnel—2–L, with Teflon stopcock.

5.2.2 Drying column—Chromatographic column, approximately 400 mm long x 19 mm ID, with coarse frit filter disc.

5.2.3 Chromatographic column—100 mm long x 10 mm ID, with Teflon stopcock.

5.2.4 Concentrator tube, Kuderna-Danish—10-mL, graduated (Kontes K–570050–1025 or equivalent). Calibration must be checked at the volumes employed in the test. Ground glass stopper is used to prevent evaporation of extracts.

5.2.5 Evaporative flask, Kuderna-Danish—500-mL (Kontes K–570001–0500 or equivalent). Attach to concentrator tube with springs.

5.2.6 Snyder column, Kuderna-Danish—Three-ball macro (Kontes K–503000–0121 or equivalent).

5.2.7 Snyder column, Kuderna-Danish—Two-ball micro (Kontes K–569001–0219 or equivalent).

5.2.8 Vials—10 to 15-mL, amber glass, with Teflon-lined screw cap.

5.3 Boiling chips—Approximately 10/40 mesh. Heat to 400°C for 30 min or Soxhlet extract with methylene chloride.

5.4 Water bath—Heated, with concentric ring cover, capable of temperature control (± 2°C). The bath should be used in a hood.

5.5 Balance—Analytical, capable of accurately weighing 0.0001 g.

5.6 Gas chromatograph—An analytical system complete with gas chromatograph suitable for on-column injection and all required accessories including syringes, analytical columns, gases, detector, and strip-chart recorder. A data system is recommended for measuring peak areas.

5.6.1 Column 1—1.2 m long x 2 or 4 mm ID glass, packed with 1.95% QF–1/1.5% OV–17 on Gas-Chrom Q (80/100 mesh) or equivalent. This column was used to develop the method performance statements given in Section 14. Guidelines for the use of alternate column packings are provided in Section 12.1.

5.6.2 Column 2—3.0 m long x 2 or 4 mm ID glass, packed with 3% OV–101 on Gas-Chrom Q (80/100 mesh) or equivalent.

5.6.3 Detectors—Flame ionization and electron capture detectors. The flame ionization detector (FID) is used when determining isophorone and nitrobenzene. The electron capture detector (ECD) is used when determining the dinitrotoluenes. Both detectors have proven effective in the analysis of wastewaters and were used in develop the method performance statements in Section 14. Guidelines for the use to alternate detectors are provided in Section 12.1.

6. Reagents

6.1 Reagent water—Reagent water is defined as a water in which an interferent is not observed at the MDL of the parameters of interest.

6.2 Sodium hydroxide solution (10 N)—Dissolve 40 g of NaOH (ACS) in reagent water and dilute to 100 mL.

6.3 Sulfuric acid (1+1)—Slowly, add 50 mL of H_2SO_4 (ACS, sp. gr. 1.84) to 50 mL of reagent water.

6.4 Acetone, hexane, methanol, methylene chloride—Pesticide quality or equivalent.

6.5 Sodium sulfate—(ACS) Granular, anhydrous. Purify by heating at 400 °C for 4 h in a shallow tray.

6.6 Florisil—PR grade (60/100 mesh). Purchase activated at 1250 °F and store in dark in glass containers with ground glass stoppers or foil-lined screw caps. Before use, activate each batch at least 16 h at 200 °C in a

foil-covered glass container and allow to cool.

6.7 Stock standard solutions (1.00 µg/µL)—Stock standard solutions can be prepared from pure standard materials or purchased as certified solutions.

6.7.1 Prepare stock standard solutions by accurately weighing about 0.0100 g of pure material. Dissolve the material in hexane and dilute to volume in a 10-mL volumetric flask. Larger volumes can be used at the convenience of the analyst. When compound purity is assayed to be 96% or greater, the weight can be used without correction to calculate the concentration of the stock standard. Commercially prepared stock standards can be used at any concentration if they are certified by the manufacturer or by an independent source.

6.7.2 Transfer the stock standard solutions into Teflon-sealed screw-cap bottles. Store at 4 °C and protect from light. Stock standard solutions should be checked frequently for signs of degradation or evaporation, especially just prior to preparing calibration standards from them.

6.7.3 Stock standard solutions must be replaced after six months, or sooner if comparison with check standards indicates a problem.

6.8 Quality control check sample concentrate—See Section 8.2.1.

7. Calibration

7.1 Establish gas chromatographic operating conditions equivalent to those given in Table 1. The gas chromatographic system can be calibrated using the external standard technique (Section 7.2) or the internal standard technique (Section 7.3).

7.2 External standard calibration procedure:

7.2.1 Prepare calibration standards at a minimum of three concentration levels for each parameter of interest by adding volumes of one or more stock standards to a volumetric flask and diluting to volume with hexane. One of the external standards should be at a concentration near, but above, the MDL (Table 1) and the other concentrations should correspond to the expected range of concentrations found in real samples or should define the working range of the detector.

7.2.2 Using injections of 2 to 5 µL, analyze each calibration standard according to Section 12 and tabulate peak height or area responses against the mass injected. The results can be used to prepare a calibration curve for each compound. Alternatively, if the ratio of response to amount injected (calibration factor) is a constant over the working range (< 10% relative standard deviation, RSD) linearity through the origin can be assumed and the average ratio or calibration factor can be used in place of a calibration curve.

7.3 Internal standard calibration procedure—To use this approach, the analyst must select one or more internal standards that are similar in analytical behavior to the compounds of interest. The analyst must further demonstrate that the measurement of the internal standard is not affected by method or matrix interferences. Because of these limitations, no internal standard can be suggested that is applicable to all samples.

7.3.1 Prepare calibration standards at a minimum of three concentration levels for each parameter of interest by adding volumes of one or more stock standards to a volumetric flash. To each calibration standard, add a known constant amount of one or more internal standards, and dilute to volume with hexane. One of the standards should be at a concentration near, but above, the MDL and the other concentrations should correspond to the expected range of concentrations found in real samples or should define the working range of the detector.

7.3.2 Using injections of 2 to 5 µL, analyze each calibration standard according to Section 12 and tabulate peak height or area responses against concentration for each compound and internal standard. Calculate response factors (RF) for each compound using Equation 1.

Equation 1.

$$RF = \frac{(A_s)(C_{is})}{(A_{is})(C_s)}$$

where:

A_s=Response for the parameter to be measured.

A_{is}=Response for the internal standard.

C_{is}=Concentration of the internal standard (µg/L).

C_s=Concentration of the parameter to be measured (µg/L).

If the RF value over the working range is a constant (< 10% RSD), the RF can be assumed to be invariant and the average RF can be used for calculations. Alternatively, the results can be used to plot a calibration curve of response ratios, A_s/A_{is}, vs. RF.

7.4 The working calibration curve, calibration factor, or RF must be verified on each working day by the measurement of one or more calibration standards. If the response for any parameter varies from the predicted response by more than ± 15%, a new calibration curve must be prepared for that compound.

7.5 Before using any cleanup procedure, the analyst must process a series of calibration standards through the procedure to validate elution patterns and the absence of interferences from the reagents.

8. Quality Control

8.1 Each laboratory that uses this method is required to operate a formal quality control program. The minimum requirements of this program consist of an initial demonstration of laboratory capability and an ongoing analysis of spiked samples to evaluate and document data quality. The laboratory must maintain records to document the quality of data that is generated. Ongoing data quality checks are compared with established performance criteria to determine if the results of analyses meet the performance characteristics of the method. When results of sample spikes indicate atypical method performance, a quality control check standard must be analyzed to confirm that the measurements were performed in an in-control mode of operation.

8.1.1 The analyst must make an initial, one-time, demonstration of the ability to generate acceptable accuracy and precision with this method. This ability is established as described in Section 8.2.

8.1.2 In recognition of advances that are occurring in chromatography, the analyst is permitted certain options (detailed in Sections 10.4, 11.1, and 12.1) to improve the separations or lower the cost of measurements. Each time such a modification is made to the method, the analyst is required to repeat the procedure in Section 8.2.

8.1.3 Before processing any samples, the analyst must analyze a reagent water blank to demonstrate that interferences from the analytical system and glassware are under control. Each time a set of samples is extracted or reagents are changed, a reagent water blank must be processed as a safeguard against laboratory contamination.

8.1.4 The laboratory must, on an ongoing basis, spike and analyze a minimum of 10% of all samples to monitor and evaluate laboratory data quality. This procedure is described in Section 8.3.

8.1,5 The laboratory must, on an ongoing basis, demonstrate through the analyses of quality control check standards that the operation of the measurement system is in control. This procedure is described in Section 8.4. The frequency of the check standard analyses is equivalent to 10% of all samples analyzed but may be reduced if spike recoveries from samples (Section 8.3) meet all specified quality control criteria.

8.1.6 The laboratory must maintain performance records to document the quality of data that is generated. This procedure is described in Section 8.5.

8.2 To establish the ability to generate acceptable accuracy and precision, the analyst must perform the following operations.

8.2.1 A quality control (QC) check sample concentrate is required containing each parameter of interest in acetone at a concentration of 20 µg/mL for each dinitrotoluene and 100 µg/mL for isophorone and nitrobenzene. The QC check sample concentrate must be obtained from the U.S. Environmental Protection Agency, Environmental Monitoring and Support Laboratory in Cincinnati, Ohio, if available. If not available from that source, the QC check sample concentrate must be obtained from another external source. If not available from either source above, the QC check sample concentrate must be prepared by the laboratory using stock standards prepared independently from those used for calibration.

8.2.2 Using a pipet, prepare QC check samples at the test concentrations shown in Table 2 by adding 1.00 mL of QC check sample concentrate to each of four 1–L aliquots of reagent water.

8.2.3 Analyze the well-mixed QC check samples according to the method beginning in Section 10.

8.2.4 Calculate the average recovery ($\bar{X}$) in µg/L, and the standard deviation of the recovery (s) in µg/L, for each parameter using the four results.

8.2.5 For each parameter compare s and $\bar{X}$ with the corresponding acceptance criteria for precision and accuracy, respectively, found in Table 2. If s and $\bar{X}$ for all parameters of interest meet the acceptance criteria, the system performance is acceptable and analysis of actual samples can begin. If any individual s exceeds the precision limit or any individual $\bar{X}$ falls outside the range for accuracy, the system performance is unacceptable for that parameter. Locate and correct the source of the problem and repeat the test for all parameters of interest beginning with Section 8.2.2.

8.3 The laboratory must, on an ongoing basis, spike at least 10% of the samples from each sample site being monitored to assess accuracy. For laboratories analyzing one to ten samples per month, at least one spiked sample per month is required.

8.3.1 The concentration of the spike in the sample should be determined as follows:

8.3.1.1 If, as in compliance monitoring, the concentration of a specific parameter in the sample is being checked against a regulatory concentration limit, the spike should be at that limit or 1 to 5 times higher than the background concentration determined in Section 8.3.2, whichever concentration would be larger.

8.3.1.2 If the concentration of a specific parameter in the sample is not being checked against a limit specific to that parameter, the spike should be at the test concentration in Section 8.2.2 or 1 to 5 times higher than the background concentration determined in Section 8.3.2, whichever concentration would be larger.

8.3.1.3 If it is impractical to determile background levels before spiking (e.g., maximum holding times will be exceeded), the

spike concentration should be (1) the regulatory concentration limit, if any; or, if none (2) the larger of either 5 times higher than the expected background concentration or the test concentration in Section 8.2.2.

8.3.2 Analyze one sample aliquot to determine the background concentration (B) of each parameter. If necessary, prepare a new QC check sample concentrate (Section 8.2.1) appropriate for the background concentrations in the sample. Spike a second sample aliquot with 1.0 mL of the QC check sample concentrate and analyze it to determine the concentration after spiking (A) of each parameter. Calculate each percent recovery (P) as 100 (A–B)%/T, where T is the known true value of the spike.

8.3.3 Compare the percent recovery (P) for each parameter with the corresponding QC acceptance criteria found in Table 2. These acceptance criteria were calculated to include an allowance for error in measurement of both the background and spike concentrations, assuming a spike to background ratio of 5:1. This error will be accounted for to the extent that the analyst's spike to background ratio approaches 5:1.[7] If spiking was performed at a concentration lower than the test concentration in Section 8.2.2, the analyst must use either the QC acceptance criteria in Table 2, or optional QC acceptance criteria calculated for the specific spike concentration. To calculate optional acceptance criteria for the recovery of a parameter: (1) Calculate accuracy (X′) using the equation in Table 3, substituting the spike concentration (T) for C; (2) calculate overall precision (S′) using the equation in Table 3, substituting X′ for X8; (3) calculate the range for recovery at the spike concentration as (100 X′/T) ± 2.44 (100 S′/T)%.[7]

8.3.4 If any individual P falls outside the designated range for recovery, that parameter has failed the acceptance criteria. A check standard containing each parameter that failed the criteria must be analyzed as described in Section 8.4.

8.4. If any parameter fails the acceptance criteria for recovery in Section 8.3, a QC check standard containing each parameter that failed must be prepared and analyzed.

NOTE: The frequency for the required analysis of a QC check standard will depend upon the number of parameters being simultaneously tested, the complexity of the sample matrix, and the performance of the laboratory.

8.4.1 Prepare the QC check standard by adding 1.0 mL of QC check sample concentrate (Section 8.2.1 or 8.3.2) to 1 L of reagent water. The QC check standard needs only to contain the parameters that failed criteria in the test in Section 8.3.

8.4.2 Analyze the QC check standard to determine the concentration measured (A) of each parameter. Calculate each percent recovery (P_s) as 100 (A/T)%, where T is the true value of the standard concentration.

8.4.3 Compare the percent recovery (P_s) for each parameter with the corresponding QC acceptance criteria found in Table 2. Only parameters that failed the test in Section 8.3 need to be compared with these criteria. If the recovery of any such parameter falls outside the designated range, the laboratory performance for that parameter is judged to be out of control, and the problem must be immediately identified and corrected. The analytical result for that parameter in the unspiked sample is suspect and may not be reported for regulatory compliance purposes.

8.5 As part of QC program for the laboratory, method accuracy for wastewater samples must be assessed and records must be maintained. After the analysis of five spiked wastewater samples as in Section 8.3, calculate the average percent recovery ($\bar{P}$) and the standard deviation of the percent recovery (s_p). Express the accuracy assessment as a percent recovery interval from $\bar{P}-2s_p$ to $\bar{P}+2s_p$. If $\bar{P}$=90% and s_p = 10%, for example, the accuracy interval is expressed as 70–110%. Update the accuracy assessment for each parameter on a regular basis (e.g. after each five to ten new accuracy measurements).

8.6 It is recommended that the laboratory adopt additional quality assurance practices for use with this method. The specific practices that are most productive depend upon the needs of the laboratory and the nature of the samples. Field duplicates may be analyzed to assess the precision of the environmental measurements. When doubt exists over the identification of a peak on the chromatogram, confirmatory techniques such as gas chromatography with a dissimilar column, specific element detector, or mass spectrometer must be used. Whenever possible, the laboratory should analyze standard reference materials and participate in relevant performance evaluation studies.

9. Sample Collection, Preservation, and Handling

9.1 Grab samples must be collected in glass containers. Conventional sampling practices[8] should be followed, except that the bottle must not be prerinsed with sample before collection. Composite samples should be collected in refrigerated glass containers in accordance with the requirements of the program. Automatic sampling equipment must be as free as possible of Tygon tubing and other potential sources of contamination.

9.2 All samples must be iced or refrigerated at 4 °C from the time of collection until extraction.

9.3 All samples must be extracted within 7 days of collection and completely analyzed within 40 days of extraction.[2]

10. Sample Extraction

10.1 Mark the water meniscus on the side of the sample bottle for later determination of sample volume. Pour the entire sample into a 2–L separatory funnel. Check the pH of the sample with wide-range pH paper and adjust to within the range of 5 to 9 with sodium hydroxide solution or sulfuric acid.

10.2 Add 60 mL of methylene chloride to the sample bottle, seal, and shake 30 s to rinse the inner surface. Transfer the solvent to the separatory funnel and extract the sample by shaking the funnel for 2 min. with periodic venting to release excess pressure. Allow the organic layer to separate from the water phase for a minimum of 10 min. If the emulsion interface between layers is more than one-third the volume of the solvent layer, the analyst must employ mechanical techniques to complete the phase separation. The optimum technique depends upon the sample, but may include stirring, filtration of the emulsion through glass wool, centrifugation, or other physical methods. Collect the methylene chloride extract in a 250-mL Erlenmeyer flask.

10.3 Add a second 60-mL volume of methylene chloride to the sample bottle and repeat the extraction procedure a second time, combining the extracts in the Erlenmeyer flask. Perform a third extraction in the same manner.

10.4 Assemble a Kuderna-Danish (K–D) concentrator by attaching a 10-mL concentrator tube to a 500-mL evaporative flask. Other concentration devices or techniques may be used in place of the K–D concentrator if the requirements of Section 8.2 are met.

10.5 Pour the combined extract through a solvent-rinsed drying column containing about 10 cm of anhydrous sodium sulfate, and collect the extract in the K–D concentrator. Rinse the Erlenmeyer flask and column with 20 to 30 mL of methylene chloride to complete the quantitative transfer.

10.6 Sections 10.7 and 10.8 describe a procedure for exchanging the methylene chloride solvent to hexane while concentrating the extract volume to 1.0 mL. When it is not necessary to achieve the MDL in Table 2, the solvent exchange may be made by the addition of 50 mL of hexane and concentration to 10 mL as described in Method 606, Sections 10.7 and 10.8.

10.7 Add one or two clean boiling chips to the evaporative flask and attach a three-ball Snyder column. Prewet the Snyder column by adding about 1 mL of methylene chloride to the top. Place the K–D apparatus on a hot water bath (60 to 65°C) so that the concentrator tube is partially immersed in the hot water, and the entire lower rounded surface of the flask is bathed with hot vapor. Adjust the vertical position of the apparatus and the water temperature as required to complete the concentration in 15 to 20 min. At the proper rate of distillation the balls of the column will actively chatter but the chambers will not flood with condensed solvent. When the apparent volume of liquid reaches 1 mL, remove the K–D apparatus and allow it to drain and cool for at least 10 min.

10.8 Remove the Snyder column and rinse the flask and its lower joint into the concentrator tube with 1 to 2 mL of methylene chloride. A 5-mL syringe is recommended for this operation. Add 1 to 2 mL of hexane and a clean boiling chip to the concentrator tube and attach a two-ball micro-Snyder column. Prewet the column by adding about 0.5 mL of hexane to the top. Place the micro-K–D apparatus on a hot water bath (60 to 65°C) so that the concentrator tube is partially immersed in the hot water. Adjust the vertical position of the apparatus and the water temperature as required to complete the concentration in 5 to 10 min. At the proper rate of distillation the balls of the column will actively chatter but the chambers will not flood. When the apparent volume of liquid reaches 0.5 mL, remove the K–D apparatus and allow it to drain and cool for at least 10 min.

10.9 Remove the micro-Snyder column and rinse its lower joint into the concentrator tube with a minimum amount of hexane. Adjust the extract volume to 1.0 mL. Stopper the concentrator tube and store refrigerated if further processing will not be performed immediately. If the extract will be stored longer than two days, it should be transferred to a Teflon-sealed screw-cap vial. If the sample extract requires no further cleanup, proceed with gas chromatographic analysis (Section 12). If the sample requires further cleanup, proceed to Section 11.

10.10 Determine the original sample volume by refilling the sample bottle to the mark and transferring the liquid to a 1000-mL graduated cylinder. Record the sample volume to the nearest 5 mL.

11. Cleanup and Separation

11.1 Cleanup procedures may not be necessary for a relatively clean sample matrix. If particular circumstances demand the use of a cleanup procedure, the analyst may use the procedure below or any other appropriate procedure. However, the analyst first must demonstrate that the requirements of Section 8.2 can be met using the method as revised to incorporate the cleanup procedure.

11.2 Florisil column cleanup:

11.2.1 Prepare a slurry of 10 g of activated Florisil in methylene chloride/hexane (1+9)(V/V) and place the Florisil into a chromatographic column. Tap the column to settle the Florisil and add 1 cm of anhydrous sodium sulfate to the top. Adjust the elution rate to about 2 mL/min.

11.2.2 Just prior to exposure of the sodium sulfate layer to the air, quantitatively transfer the sample extract onto the column using an additional 2 mL of hexane to complete the transfer. Just prior to exposure of the sodium sulfate layer to the air, add 30 mL of methylene chloride/hexane (1 + 9)(V/V) and continue the elution of the column. Discard the eluate.

11.2.3 Next, elute the column with 30 mL of acetone/methylene chloride (1 + 9)(V/V) into a 500-mL K–D flask equipped with a 10-mL concentrator tube. Concentrate the collected fraction as in Sections 10.6, 10.7, 10.8, and 10.9 including the solvent exchange to 1 mL of hexane. This fraction should contain the nitroaromatics and isophorone. Analyze by gas chromatography (Section 12).

12. Gas Chromatography

12.1 Isophorone and nitrobenzene are analyzed by injection of a portion of the extract into an FIDGC. The dinitrotoluenes are analyzed by a separate injection into an ECDGC. Table 1 summarizes the recommended operating conditions for the gas chromatograph. Included in this table are retention times and MDL that can be achieved under these conditions. Examples of the separations achieved by Column 1 are shown in Figures 1 and 2. Other packed or capillary (open-tubular) columns, chromatographic conditions, or detectors may be used if the requirements of Section 8.2 are met.

12.2 Calibrate the system daily as described in Section 7.

12.3 If the internal standard calibration procedure is being used, the internal standard must be added to the same extract and mixed thoroughly immediately before injection into the gas chromatograph.

12.4 Inject 2 to 5 µL of the sample extract or standard into the gas chromatograph using the solvent-flush technique.[9] Smaller (1.0 µL) volumes may be injected if automatic devices are employed. Record the volume injected to the nearest 0.05 µL, the total extract volume, and the resulting peak size in area or peak height units.

12.5 Identify the parameters in the sample by comparing the retention times of the peaks in the sample chromatogram with those of the peaks in standard chromatograms. The width of the retention time window used to make identifications should be based upon measurements of actual retention time variations of standards over the course of a day. Three times the standard deviation of a retention time for a compound can be used to calculate a suggested window size; however, the experience of the analyst should weigh heavily in the interpretation of chromatograms.

12.6 If the response for a peak exceeds the working range of the system, dilute the extract and reanalyze.

12.7 If the measurement of the peak response is prevented by the presence of interferences, further cleanup is required.

13. Calculations

13.1 Determine the concentration of individual compounds in the sample.

13.1.1 If the external standard calibration procedure is used, calculate the amount of material injected from the peak response using the calibration curve or calibration factor determined in Section 7.2.2. The concentration in the sample can be calculated from Equation 2.

$$\text{Concentration } (\mu g/L) = \frac{(A)(V_t)}{(V_i)(V_s)}$$

Equation 2

where:

A=Amount of material injected (ng).
V_i=Volume of extract injected (µL).
V_t=Volume of total extract (µL).
V_s=Volume of water extracted (mL).

13.1.2 If the internal standard calibration procedure is used, calculate the concentration in the sample using the response factor (RF) determined in Section 7.3.2 and Equation 3.

$$\text{Concentration } (\mu g/L) = \frac{(A_s)(I_s)}{(A_{is})(RF)(V_o)}$$

Equation 3

where:

A_s=Response for the parameter to be measured.
A_{is}=Response for the internal standard.
I_s=Amount of internal standard added to each extract (µg).
V_o=Volume of water extracted (L).

13.2 Report results in µg/L without correction for recovery data. All QC data obtained should be reported with the sample results.

14. Method Performance

14.1 The method detection limit (MDL) is defined as the minimum concentration of a substance that can be measured and reported with 99% confidence that the value is above zero.[1] The MDL concentrations listed in Table 1 were obtained using reagent water.[10] Similar results were achieved using representative wastewaters. The MDL actually achieved in a given analysis will vary depending on instrument sensitivity and matrix effects.

14.2 This method has been tested for linearity of spike recovery from reagent water and has been demonstrated to be applicable over the concentration range from 7×MDL to 1000×MDL.[10]

14.3 This method was tested by 18 laboratories using reagent water, drinking water, surface water, and three industrial wastewaters spiked at six concentrations over the range 1.0 to 515 µg/L.[11] Single operator precision, overall precision, and method accuracy were found to be directly related to the concentration of the parameter and essentially independent of the sample matrix. Linear equations to describe these relationships are presented in Table 3.

REFERENCES

1. 40 CFR part 136, appendix B.

2. ''Determination of Nitroaromatic Compounds and Isophorone in Industrial and Municipal Wastewaters,'' EPA 600/ 4–82–024, National Technical Information Service, PB82–208398, Springfield, Virginia 22161, May 1982.

3. ASTM Annual Book of Standards, Part 31, D3694–78. ''Standard Practices for Preparation of Sample Containers and for Preservation of Organic Constituents,'' American Society for Testing and Materials, Philadelphia.

4. ''Carcinogens—Working With Carcinogens,'' Department of Health, Education, and Welfare, Public Health Service, Center for Disease Control, National Institute for Occupational Safety and Health, Publication No. 77–206, August 1977.

5. ''OSHA Safety and Health Standards, General Industry,'' (29 CFR part 1910), Occupational Safety and Health Administration, OSHA 2206 (Revised, January 1976).

6. ''Safety in Academic Chemistry Laboratories,'' American Chemical Society Publication, Committee on Chemical Safety, 3rd Edition, 1979.

7. Provost, L.P., and Elder, R.S. ''Interpretation of Percent Recovery Data,'' *American Laboratory, 15,* 58–63 (1983). (The value 2.44 used in the equation in Section 8.3.3 is two times the value 1.22 derived in this report.)

8. ASTM Annual Book of Standards, Part 31, D3370–76. ''Standard Practices for Sampling Water,'' American Society for Testing and Materials, Philadelphia.

9. Burke, J.A. ''Gas Chromatography for Pesticide Residue Analysis; Some Practical Aspects,'' *Journal of the Association of Official Analytical Chemists, 48,* 1037 (1965).

10. ''Determination of Method Detection Limit and Analytical Curve for EPA Method 609—Nitroaromatics and Isophorone,'' Special letter report for EPA Contract 68–03–2624, U.S. Environmental Protection Agency, Environmental Monitoring and Support Laboratory, Cincinnati, Ohio 45268, June 1980.

11. ''EPA Method Study 19, Method 609 (Nitroaromatics and Isophorone),'' EPA 600/ 4–84–018, National Technical Information Service, PB84–176908, Springfield, Virginia 22161, March 1984.

TABLE 1—CHROMATOGRAPHIC CONDITIONS AND METHOD DETECTION LIMITS

Parameter	Retention time (min)		Method detection limit (µg/L)	
	Col. 1	Col. 2	ECDGC	FIDGC
Nitrobenzene	3.31	4.31	13.7	3.6
2,6-Dinitrotoluene	3.52	4.75	0.01	–
Isophorone	4.49	5.72	15.7	5.7
2,4-Dinitrotoluene	5.35	6.54	0.02	–

AAColumn 1 conditions: Gas-Chrom Q (80/100 mesh) coated with 1.95% QF–1/1.5% OV–17 packed in a 1.2 m long × 2 mm or 4 mm ID glass column. A 2 mm ID column and nitrogen carrier gas at 44 mL/min flow rate were used when determining isophorone and nitrobenzene by FIDGC. The column temperature was held isothermal at 85 °C. A 4 mm ID column and 10% methane/90% argon carrier gas at 44 mL/min flow rate were used when determining the dinitrotoluenes by ECDGC. The column temperature was held isothermal at 145 °C.

AAColumn 2 conditions: Gas-Chrom Q (80/100 mesh) coated with 3% OV–101 packed in a 3.0 m long × 2 mm or 4 mm ID glass column. A 2 mm ID column and nitrogen carrier gas at 44 mL/min flow rate were used when determining isophorone and nitrobenzene by FIDGC. The column temperature was held isothermal at 100 °C. A 4 mm ID column and 10% methane/90% argon carrier gas at 44 mL/min flow rate were used when determining the dinitrotoluenes by ECDGC. The column temperature was held isothermal at 150 °C.

TABLE 2—QC ACCEPTANCE CRITERIA—METHOD 609

Parameter	Test Conc. (µg/L)	Limit for s (µg/L)	Range for $\bar{X}$ (µg/L)	Range for P, P_s (%)
2,4-Dinitrotoluene	20	5.1	3.6–22.8	6–125
2,6-Dinitrotoluene	20	4.8	3.8–23.0	8–126
Isophorone	100	32.3	8.0–100.0	D–117
Nitrobenzene	100	33.3	25.7–100.0	6–118

s=Standard deviation of four recovery measurements, in µg/L (Section 8.2.4).
$\bar{X}$=Average recovery for four recovery measurements, in µg/L (Section 8.2.4).
P, P_s=Percent recovery measured (Section 8.3.2, Section 8.4.2).
D=Detected; result must be greater than zero.

NOTE: These criteria are based directly upon the method performance data in Table 3. Where necessary, the limits for recovery have been broadened to assure applicability of the limits to concentrations below those used to develop Table 3.

TABLE 3—METHOD ACCURACY AND PRECISION AS FUNCTIONS OF CONCENTRATION—METHOD 609

Parameter	Accuracy, as recovery, X′ (μg/L)	Single analyst precision, s_r' (μg/L)	Overall precision, S′ (μg/L)
2,4-Dinitrotoluene	0.65C+0.22	$0.20\bar{X}+0.08$	$0.37\bar{X}-0.07$
2,6-Dinitrotoluene	0.66C+0.20	$0.19\bar{X}+0.06$	$0.36\bar{X}-0.00$
Isophorone	0.49C+2.93	$0.28\bar{X}+2.77$	$0.46\bar{X}+0.31$
Nitrobenzene	0.60C+2.00	$0.25\bar{X}+2.53$	$0.37\bar{X}-0.78$

X′=Expected recovery for one or more measurements of a sample containing a concentration of C, in μg/L.
s_r'=Expected single analyst standard deviation of measurements at an average concentration found of $\bar{X}$, in μg/L.
S′=Expected interlaboratory standard deviation of measurements at an average concentration found of X, in μg/L.
C=True value for the concentration, in μg/L.
$\bar{X}$=Average recovery found for measurements of samples containing a concentration of C, in μg/L.

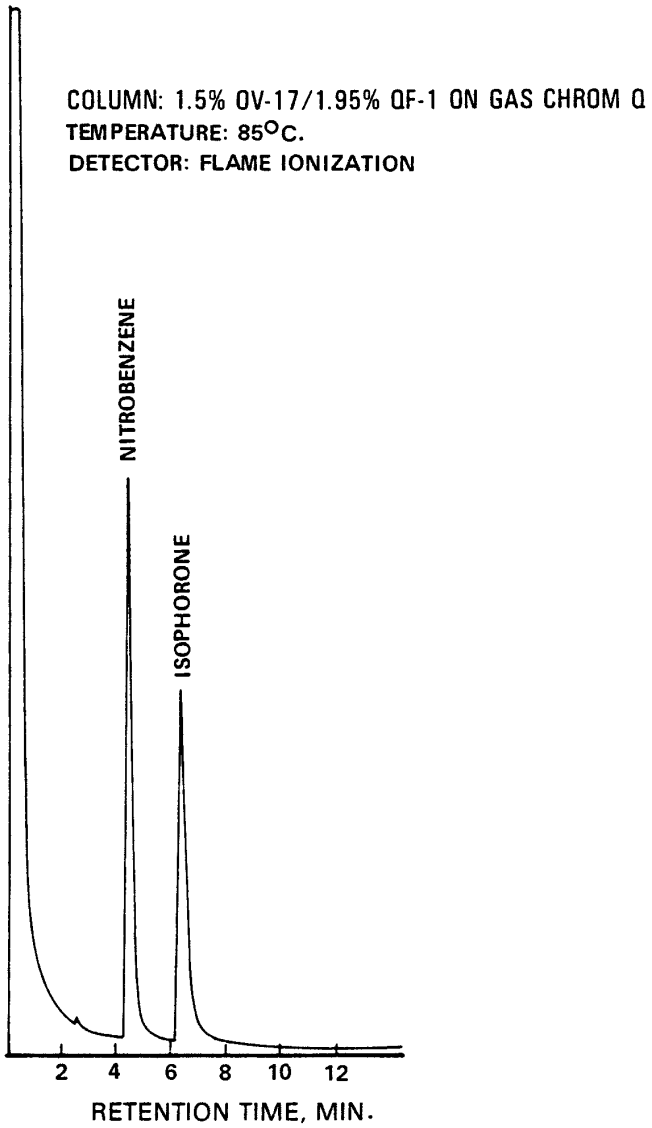


Figure 1. Gas chromatogram of nitrobenzene and isophorone.

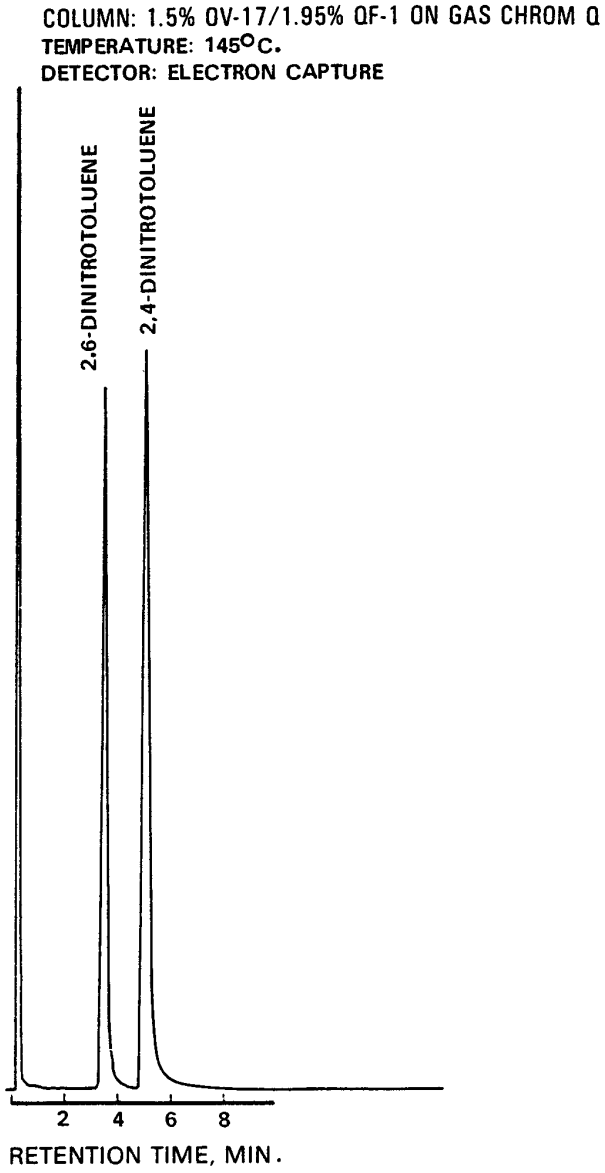


Figure 2. Gas chromatogram of dinitrotoluenes.

METHOD 610—POLYNUCLEAR AROMATIC HYDROCARBONS

1. Scope and Application

1.1 This method covers the determination of certain polynuclear aromatic hydrocarbons (PAH). The following parameters can be determined by this method:

Parameter	STORET No.	CAS No.
Acenaphthene	34205	83–32–9
Acenaphthylene	34200	208–96–8
Anthracene	34220	120–12–7
Benzo(a)anthracene	34526	56–55–3
Benzo(a)pyrene	34247	50–32–8
Benzo(b)fluoranthene	34230	205–99–2
Benzo(ghi)perylene	34521	191–24–2
Benzo(k)fluoranthene	34242	207–08–9
Chrysene	34320	218–01–9
Dibenzo(a,h)anthracene	34556	53–70–3
Fluoranthene	34376	206–44–0
Fluorene	34381	86–73–7
Indeno(1,2,3-cd)pyrene	34403	193–39–5
Naphthalene	34696	91–20–3
Phenanthrene	34461	85–01–8
Pyrene	34469	129–00–0

1.2 This is a chromatographic method applicable to the determination of the compounds listed above in municipal and industrial discharges as provided under 40 CFR 136.1. When this method is used to analyze unfamiliar samples for any or all of the compounds above, compound identifications should be supported by at least one additional qualitative technique. Method 625 provides gas chromatograph/mass spectrometer (GC/MS) conditions appropriate for the qualitative and quantitative confirmation of results for many of the parameters listed above, using the extract produced by this method.

1.3 This method provides for both high performance liquid chromatographic (HPLC) and gas chromatographic (GC) approaches for the determination of PAHs. The gas chromatographic procedure does not adequately resolve the following four pairs of compounds: Anthracene and phenanthrene; chrysene and benzo(a)anthracene; benzo(b)fluoranthene and benzo(k)fluoranthene; and dibenzo(a,h) anthracene and indeno (1,2,3-cd)pyrene. Unless the purpose for the analysis can be served by reporting the sum of an unresolved pair, the liquid chromatographic approach must be used for these compounds. The liquid chromatographic method does resolve all 16 of the PAHs listed.

1.4 The method detection limit (MDL, defined in Section 15.1)[1] for each parameter is listed in Table 1. The MDL for a specific wastewater may differ from those listed, depending upon the nature of interferences in the sample matrix.

1.5 The sample extraction and concentration steps in this method are essentially the same as in Methods 606, 608, 609, 611, and 612. Thus, a single sample may be extracted to measure the parameters included in the scope of each of these methods. When cleanup is required, the concentration levels must be high enough to permit selecting aliquots, as necessary, to apply appropriate cleanup procedures. Selection of the aliquots must be made prior to the solvent exchange steps of this method. The analyst is allowed the latitude, under Sections 12 and 13, to select chromatographic conditions appropriate for the simultaneous measurement of combinations of these parameters.

1.6 Any modification of this method, beyond those expressly permitted, shall be considered as a major modification subject to application and approval of alternate test procedures under 40 CFR 136.4 and 136.5.

1.7 This method is restricted to use by or under the supervision of analysts experienced in the use of HPLC and GC systems and in the interpretation of liquid and gas chromatograms. Each analyst must demonstrate the ability to generate acceptable results with this method using the procedure described in Section 8.2.

2. Summary of Method

2.1 A measured volume of sample, approximately 1–L, is extracted with methylene chloride using a separatory funnel. The methylene chloride extract is dried and concentrated to a volume of 10 mL or less. The extract is then separated by HPLC or GC. Ultraviolet (UV) and fluorescence detectors are used with HPLC to identify and measure the PAHs. A flame ionization detector is used with GC.[2]

2.2 The method provides a silica gel column cleanup procedure to aid in the elimination of interferences that may be encountered.

3. Interferences

3.1 Method interferences may be caused by contaminants in solvents, reagents, glassware, and other sample processing hardward that lead to discrete artifacts and/or elevated baselines in the chromatograms. All of these materials must be routinely demonstrated to be free from interferences under the conditions of the analysis by running laboratory reagent blanks as described in Section 8.1.3.

3.1.1 Glassware must be scrupulously cleaned.[3] Clean all glassware as soon as possible after use by rinsing with the last solvent used in it. Solvent rinsing should be followed by detergent washing with hot water, and rinses with tap water and distilled water. The glassware should then be drained dry, and heated in a muffle furnace at 400 °C for 15 to 30 min. Some thermally stable materials, such as PCBs, may not be eliminated by this treatment. Solvent rinses with acetone and pesticide quality hexane may be

substituted for the muffle furnace heating. Thorough rinsing with such solvents usually eliminates PCB interference. Volumetric ware should not be heated in a muffle furnace. After drying and cooling, glassware should be sealed and stored in a clean environment to prevent any accumulation of dust or other contaminants. Store inverted or capped with aluminum foil.

3.1.2 The use of high purity reagents and solvents helps to minimize interference problems. Purification of solvents by distillation in all-glass systems may be required.

3.2 Matrix interferences may be caused by contaminants that are co-extracted from the sample. The extent of matrix interferences will vary considerably from source to source, depending upon the nature and diversity of the industrial complex or municipality being sampled. The cleanup procedure in Section 11 can be used to overcome many of these interferences, but unique samples may require additional cleanup approaches to achieve the MDL listed in Table 1.

3.3 The extent of interferences that may be encountered using liquid chromatographic techniques has not been fully assessed. Although the HPLC conditions described allow for a unique resolution of the specific PAH compounds covered by this method, other PAH compounds may interfere.

4. Safety

4.1 The toxicity or carcinogenicity of each reagent used in this method have not been precisely defined; however, each chemical compound should be treated as a potential health hazard. From this viewpoint, exposure to these chemicals must be reduced to the lowest possible level by whatever means available. The laboratory is responsible for maintaining a current awareness file of OSHA regulations regarding the safe handling of the chemicals specified in this method. A reference file of material data handling sheets should also be made available to all personnel involved in the chemical analysis. Additional references to laboratory safety are available and have been identified[4-6] for the information of the analyst.

4.2 The following parameters covered by this method have been tentatively classified as known or suspected, human or mammalian carcinogens: benzo(a)anthracene, benzo(a)pyrene, and dibenzo(a,h)-anthracene. Primary standards of these toxic compounds should be prepared in a hood. A NIOSH/MESA approved toxic gas respirator should be worn when the analyst handles high concentrations of these toxic compounds.

5. Apparatus and Materials

5.1 Sampling equipment, for discrete or composite sampling.

5.1.1 Grab sample bottle—1–L or 1-qt, amber glass, fitted with a screw cap lined with Teflon. Foil may be substituted for Teflon if the sample is not corrosive. If amber bottles are not available, protect samples from light. The bottle and cap liner must be washed, rinsed with acetone or methylene chloride, and dried before use to minimize contamination.

5.1.2 Automatic sampler (optional)—The sampler must incorporate glass sample containers for the collection of a minimum of 250 mL of sample. Sample containers must be kept refrigerated at 4°C and protected from light during compositing. If the sampler uses a peristaltic pump, a minimum length of compressible silicone rubber tubing may be used. Before use, however, the compressible tubing should be thoroughly rinsed with methanol, followed by repeated rinsings with distilled water to minimize the potential for contamination of the sample. An integrating flow meter is required to collect flow proportional composites.

5.2 Glassware (All specifications are suggested. Catalog numbers are included for illustration only.):

5.2.1 Separatory funnel—2–L, with Teflon stopcock.

5.2.2 Drying column—Chromatographic column, approximately 400 mm long × 19 mm ID, with coarse frit filter disc.

5.2.3 Concentrator tube, Kuderna-Danish—10-mL, graduated (Kontes K–570050–1025 or equivalent). Calibration must be checked at the volumes employed in the test. Ground glass stopper is used to prevent evaporation of extracts.

5.2.4 Evaporative flask, Kuderna-Danish—500-mL (Kontes K–570001–0500 or equivalent). Attach to concentrator tube with springs.

5.2.5 Snyder column, Kuderna-Danish—Three-ball macro (Kontes K–503000–0121 or equivalent).

5.2.6 Snyder column, Kuderna-Danish—Two-ball micro (Kontes K–569001–0219 or equivalent).

5.2.7 Vials—10 to 15-mL, amber glass, with Teflon-lined screw cap.

5.2.8 Chromatographic column—250 mm long x 10 mm ID, with coarse frit filter disc at bottom and Teflon stopcock.

5.3 Boiling chips—Approximately 10/40 mesh. Heat to 400 °C for 30 min or Soxhlet extract with methylene chloride.

5.4 Water bath—Heated, with concentric ring cover, capable of temperature control (±2 °C). The bath should be used in a hood.

5.5 Balance—Analytical, capable of accurately weighing 0.0001 g.

5.6 High performance liquid chromatograph (HPLC)—An analytical system complete with column supplies, high pressure syringes, detectors, and compatible strip-chart recorder. A data system is recommended for measuring peak areas and retention times.

5.6.1 Gradient pumping system—Constant flow.

5.6.2 Reverse phase column—HC–ODS Sil-X, 5 micron particle diameter, in a 25 cm x 2.6 mm ID stainless steel column (Perkin Elmer No. 089–0716 or equivalent). This column was used to develop the method performance statements in Section 15. Guidelines for the use of alternate column packings are provided in Section 12.2.

5.6.3 Detectors—Fluorescence and/or UV detectors. The fluorescence detector is used for excitation at 280 nm and emission greater than 389 nm cutoff (Corning 3–75 or equivalent). Fluorometers should have dispersive optics for excitation and can utilize either filter or dispersive optics at the emission detector. The UV detector is used at 254 nm and should be coupled to the fluorescence detector. These detectors were used to develop the method performance statements in Section 15. Guidelines for the use of alternate detectors are provided in Section 12.2.

5.7 Gas chromatograph—An analytical system complete with temperature programmable gas chromatograph suitable for on-column or splitless injection and all required accessories including syringes, analytical columns, gases, detector, and strip-chart recorder. A data system is recommended for measuring peak areas.

5.7.1 Column—1.8 m long x 2 mm ID glass, packed with 3% OV–17 on Chromosorb W–AW–DCMS (100/120 mesh) or equivalent. This column was used to develop the retention time data in Table 2. Guidelines for the use of alternate column packings are provided in Section 13.3.

5.7.2 Detector—Flame ionization detector. This detector has proven effective in the analysis of wastewaters for the parameters listed in the scope (Section 1.1), excluding the four pairs of unresolved compounds listed in Section 1.3. Guidelines for the use of alternate detectors are provided in Section 13.3.

6. Reagents

6.1 Reagent water—Reagent water is defined as a water in which an interferent is not observed at the MDL of the parameters of interest.

6.2 Sodium thiosulfate—(ACS) Granular.

6.3 Cyclohexane, methanol, acetone, methylene chloride, pentane—Pesticide quality or equivalent.

6.4 Acetonitrile—HPLC quality, distilled in glass.

6.5 Sodium sulfate—(ACS) Granular, anhydrous. Purify by heating at 400 °C for 4 h in a shallow tray.

6.6 Silica gel—100/200 mesh, desiccant, Davison, grade-923 or equivalent. Before use, activate for at least 16 h at 130 °C in a shallow glass tray, loosely covered with foil.

6.7 Stock standard solutions (1.00 µg/µL)—Stock standard solutions can be prepared from pure standard materials or purchased as certified solutions.

6.7.1 Prepare stock standard solutions by accurately weighing about 0.0100 g of pure material. Dissolve the material in acetonitrile and dilute to volume in a 10-mL volumetric flask. Larger volumes can be used at the convenience of the analyst. When compound purity is assayed to be 96% or greater, the weight can be used without correction to calculate the concentration of the stock standard. Commercially prepared stock standards can be used at any concentration if they are certified by the manufacturer or by an independent source.

6.7.2 Transfer the stock standard solutions into Teflon-sealed screw-cap bottles. Store at 4 °C and protect from light. Stock standard solutions should be checked frequently for signs of degradation or evaporation, especially just prior to preparing calibration standards from them.

6.7.3 Stock standard solutions must be replaced after six months, or sooner if comparison with check standards indicates a problem.

6.8 Quality control check sample concentrate—See Section 8.2.1.

7. Calibration

7.1 Establish liquid or gas chromatographic operating conditions equivalent to those given in Table 1 or 2. The chromatographic system can be calibrated using the external standard technique (Section 7.2) or the internal standard technique (Section 7.3).

7.2 External standard calibration procedure:

7.2.1 Prepare calibration standards at a minimum of three concentration levels for each parameter of interest by adding volumes of one or more stock standards to a volumetric flask and diluting to volume with acetonitrile. One of the external standards should be at a concentration near, but above, the MDL (Table 1) and the other concentrations should correspond to the expected range of concentrations found in real samples or should define the working range of the detector.

7.2.2 Using injections of 5 to 25 µL for HPLC and 2 to 5 µL for GC, analyze each calibration standard according to Section 12 or 13, as appropriate. Tabulate peak height or area responses against the mass injected. The results can be used to prepare a calibration curve for each compound. Alternatively, if the ratio of response to amount injected (calibration factor) is a constant over the working range (<10% relative standard deviation, RSD), linearity through the origin can be assumed and the average ratio or calibration factor can be used in place of a calibration curve.

7.3 Internal standard calibration procedure—To use this approach, the analyst must select one or more internal standards that are similar in analytical behavior to the

compounds of interest. The analyst must further demonstrate that the measurement of the internal standard is not affected by method or matrix interferences. Because of these limitations, no internal standard can be suggested that is applicable to all samples.

7.3.1 Prepare calibration standards at a minimum of three concentration levels for each parameter of interest by adding volumes of one or more stock standards to a volumetric flask. To each calibration standard, add a known constant amount of one or more internal standards, and dilute to volume with acetonitrile. One of the standards should be at a concentration near, but above, the MDL and the other concentrations should correspond to the expected range of concentrations found in real samples or should define the working range of the detector.

7.3.2 Using injections of 5 to 25 µL for HPLC and 2 to 5 µL for GC, analyze each calibration standard according to Section 12 or 13, as appropriate. Tabulate peak height or area responses against concentration for each compound and internal standard. Calculate response factors (RF) for each compound using Equation 1.

$$RF = \frac{(A_s)(C_{is})}{(A_{is})(C_s)}$$

Equation 1

where:

A_s=Response for the parameter to be measured.

A_{is}=Response for the internal standard.

C_{is}=Concentration of the internal standard (µg/L).

C_s=Concentration of the parameter to be measured (µg/L).

If the RF value over the working range is a constant (<10% RSD), the RF can be assumed to be invariant and the average RF can be used for calculations. Alternatively, the results can be used to plot a calibration curve of response ratios, A_s/A_{is}, vs. RF.

7.4 The working calibration curve, calibration factor, or RF must be verified on each working day by the measurement of one or more calibration standards. If the response for any parameter varies from the predicted response by more than ±15%, the test must be repeated using a fresh calibration standard. Alternatively, a new calibration curve must be prepared for that compound.

7.5 Before using any cleanup procedure, the analyst must process a series of calibration standards through the procedure to validate elution patterns and the absence of interferences from the reagents.

8. Quality Control

8.1 Each laboratory that uses this method is required to operate a formal quality control program. The minimum requirements of this program consist of an initial demonstration of laboratory capability and an ongoing analysis of spiked samples to evaluate and document data quality. The laboratory must maintain records to document the quality of data that is generated. Ongoing data quality checks are compared with established performance criteria to determine if the results of analyses meet the performance characteristics of the method. When results of sample spikes indicate atypical method performance, a quality control check standard must be analyzed to confirm that the measurements were performed in an in-control mode of operation.

8.1.1 The analyst must make an initial, one-time, demonstration of the ability to generate acceptable accuracy and precision with this method. This ability is established as described in Section 8.2.

8.1.2 In recognition of advances that are occurring in chromatography, the analyst is permitted certain options (detailed in Sections 10.4, 11.1, 12.2, and 13.3) to improve the separations or lower the cost of measurements. Each time such a modification is made to the method, the analyst is required to repeat the procedure in Section 8.2.

8.1.3 Before processing any samples the analyst must analyze a reagent water blank to demonstrate that interferences from the analytical system and glassware are under control. Each time a set of samples is extracted or reagents are changed a reagent water blank must be processed as a safeguard against laboratory contamination.

8.1.4 The laboratory must, on an ongoing basis, spike and analyze a minimum of 10% of all samples to monitor and evaluate laboratory data quality. This procedure is described in Section 8.3.

8.1.5 The laboratory must, on an ongoing basis, demonstrate through the analyses of quality control check standards that the operation of the measurement system is in control. This procedure is described in Section 8.4. The frequency of the check standard analyses is equivalent to 10% of all samples analyzed but may be reduced if spike recoveries from samples (Section 8.3) meet all specified quality control criteria.

8.1.6 The laboratory must maintain performance records to document the quality of data that is generated. This procedure is described in Section 8.5.

8.2 To establish the ability to generate acceptable accuracy and precision, the analyst must perform the following operations.

8.2.1 A quality control (QC) check sample concentrate is required containing each parameter of interest at the following concentrations in acetonitrile: 100 µg/mL of any

of the six early-eluting PAHs (naphthalene, acenaphthylene, acenaphthene, fluorene, phenanthrene, and anthracene); 5 µg/mL of benzo(k)fluoranthene; and 10 µg/mL of any of the other PAHs. The QC check sample concentrate must be obtained from the U.S. Environmental Protection Agency, Environmental Monitoring and Support Laboratory in Cincinnati, Ohio, if available. If not available from that source, the QC check sample concentrate must be obtained from another external source. If not available from either source above, the QC check sample concentrate must be prepared by the laboratory using stock standards prepared independently from those used for calibration.

8.2.2 Using a pipet, prepare QC check samples at the test concentrations shown in Table 3 by adding 1.00 mL of QC check sample concentrate to each of four 1–L aliquots of reagent water.

8.2.3 Analyze the well-mixed QC check samples according to the method beginning in Section 10.

8.2.4 Calculate the average recovery ($\bar{X}$) in µg/L, and the standard deviation of the recovery (s) in µg/L, for each parameter using the four results.

8.2.5 For each parameter compare s and $\bar{X}$ with the corresponding acceptance criteria for precision and accuracy, respectively, found in Table 3. If s and $\bar{X}$ for all parameters of interest meet the acceptance criteria, the system performance is acceptable and analysis of actual samples can begin. If any individual s exceeds the precision limit or any individual $\bar{X}$ falls outside the range for accuracy, the system performance is unacceptable for that parameter.

NOTE: The large number of parameters in Table 3 present a substantial probability that one or more will fail at least one of the acceptance criteria when all parameters are analyzed.

8.2.6 When one or more of the parameters tested fail at least one of the acceptance criteria, the analyst must proceed according to Section 8.2.6.1 or 8.2.6.2.

8.2.6.1 Locate and correct the source of the problem and repeat the test for all parameters of interest beginning with Section 8.2.2.

8.2.6.2 Beginning with Section 8.2.2, repeat the test only for those parameters that failed to meet criteria. Repeated failure, however, will confirm a general problem with the measurement system. If this occurs, locate and correct the source of the problem and repeat the test for all compounds of interest beginning with Section 8.2.2.

8.3 The laboratory must, on an ongoing basis, spike at least 10% of the samples from each sample site being monitored to assess accuracy. For laboratories analyzing one to ten samples per month, at least one spiked sample per month is required.

8.3.1 The concentration of the spike in the sample should be determined as follows:

8.3.1.1 If, as in compliance monitoring, the concentration of a specific parameter in the sample is being checked against a regulatory concentration limit, the spike should be at that limit or 1 to 5 times higher than the background concentration determined in Section 8.3.2, whichever concentration would be larger.

8.3.1.2 If the concentration of a specific parameter in the sample is not being checked against a limit specific to that parameter, the spike should be at the test concentration in Section 8.2.2 or 1 to 5 times higher than the background concentration determined in Section 8.3.2, whichever concentration would be larger.

8.3.1.3 If it is impractical to determine background levels before spiking (e.g., maximum holding times will be exceeded), the spike concentration should be (1) the regulatory concentration limit, if any; or, if none, (2) the larger of either 5 times higher than the expected background concentration or the test concentration in Section 8.2.2.

8.3.2 Analyze one sample aliquot to determine the background concentration (B) of each parameter. If necessary, prepare a new QC check sample concentrate (Section 8.2.1) appropriate for the background concentrations in the sample. Spike a second sample aliquot with 1.0 mL of the QC check sample concentrate and analyze it to determine the concentration after spiking (A) of each parameter. Calculate each percent recovery (P) as 100 (A–B)%/T, where T is the known true value of the spike.

8.3.3 Compare the percent recovery (P) for each parameter with the corresponding QC acceptance criteria found in Table 3. These acceptance criteria were calculated to include an allowance for error in measurement of both the background and spike concentrations, assuming a spike to background ratio of 5:1. This error will be accounted for to the extent that the analyst's spike to background ratio approaches 5:1.[7] If spiking was performed at a concentration lower than the test concentration in Section 8.2.2, the analyst must use either the QC acceptance criteria in Table 3, or optional QC acceptance criteria calculated for the specific spike concentration. To calculate optional acceptance criteria for the recovery of a parameter: (1) Calculate accuracy (X′) using the equation in Table 4, substituting the spike concentration (T) for C; (2) calculate overall precision (S′) using the equation in Table 4, substituting X′ for X; (3) calculate the range for recovery at the spike concentration as (100 X′/T)±2.44(100 S′/T)%.[7]

8.3.4 If any individual P falls outside the designated range for recovery, that parameter has failed the acceptance criteria. A check standard containing each parameter

that failed the critiera must be analyzed as described in Section 8.4.

8.4 If any parameter fails the acceptance criteria for recovery in Section 8.3, a QC check standard containing each parameter that failed must be prepared and analyzed.

NOTE: The frequency for the required analysis of a QC check standard will depend upon the number of parameters being simultaneously tested, the complexity of the sample matrix, and the performance of the laboratory. If the entire list of parameters in Table 3 must be measured in the sample in Section 8.3, the probability that the analysis of a QC check standard will be required is high. In this case the QC check standard should be routinely analyzed with the spike sample.

8.4.1 Prepare the QC check standard by adding 1.0 mL of QC check sample concentrate (Section 8.2.1 or 8.3.2) to 1 L of reagent water. The QC check standard needs only to contain the parameters that failed criteria in the test in Section 8.3.

8.4.2 Analyze the QC check standard to determine the concentration measured (A) of each parameter. Calculate each percent recovery (P_s) as 100 (A/T)%, where T is the true value of the standard concentration.

8.4.3 Compare the percent recovery (P_s) for each parameter with the corresponding QC acceptance criteria found in Table 3. Only parameters that failed the test in Section 8.3 need to be compared with these criteria. If the recovery of any such parameter falls outside the designated range, the laboratory performance for that parameter is judged to be out of control, and the problem must be immediately identified and corrected. The analytical result for that parameter in the unspiked sample is suspect and may not be reported for regulatory compliance purposes.

8.5 As part of the QC program for the laboratory, method accuracy for wastewater samples must be assessed and records must be maintained. After the analysis of five spiked wastewater samples as in Section 8.3, calculate the average percent recovery (P) and the standard deviation of the percent recovery (s_p). Express the accuracy assessment as a percent recovery interval from $P–2s_p$ to $P+2s_p$. If P=90% and s_p=10%, for example, the accuracy interval is expressed as 70–110%. Update the accuracy assessment for each parameter on a regular basis (e.g. after each five to ten new accuracy measurements).

8.6 It is recommended that the laboratory adopt additional quality assurance practices for use with this method. The specific practices that are most productive depend upon the needs of the laboratory and the nature of the samples. Field duplicates may be analyzed to assess the precision of the environmental measurements. When doubt exists over the identification of a peak on the chromatogram, confirmatory techniques such as gas chromatography with a dissimilar column, specific element detector, or mass spectrometer must be used. Whenever possible, the laboratory should analyze standard reference materials and participate in relevant performance evaluation studies.

9. Sample Collection, Preservation, and Handling

9.1 Grab samples must be collected in glass containers. Conventional sampling practices[8] should be followed, except that the bottle must not be prerinsed with sample before collection. Composite samples should be collected in refrigerated glass containers in accordance with the requirements of the program. Automatic sampling equipment must be as free as possible of Tygon tubing and other potential sources of contamination.

9.2 All samples must be iced or refrigerated at 4°C from the time of collection until extraction. PAHs are known to be light sensitive; therefore, samples, extracts, and standards should be stored in amber or foil-wrapped bottles in order to minimize photolytic decomposition. Fill the sample bottles and, if residual chlorine is present, add 80 mg of sodium thiosulfate per liter of sample and mix well. EPA Methods 330.4 and 330.5 may be used for measurement of residual chlorine.[9] Field test kits are available for this purpose.

9.3 All samples must be extracted within 7 days of collection and completely analyzed within 40 days of extraction.[2]

10. Sample Extraction

10.1 Mark the water meniscus on the side of the sample bottle for later determination of sample volume. Pour the entire sample into a 2–L separatory funnel.

10.2 Add 60 mL of methylene chloride to the sample bottle, seal, and shake 30 s to rinse the inner surface. Transfer the solvent to the separatory funnel and extract the sample by shaking the funnel for 2 min. with periodic venting to release excess pressure. Allow the organic layer to separate from the water phase for a minimum of 10 min. If the emulsion interface between layers is more than one-third the volume of the solvent layer, the analyst must employ mechanical techniques to complete the phase separation. The optimum technique depends upon the sample, but may include stirring, filtration of the emulsion through glass wool, centrifugation, or other physical methods. Collect the methylene chloride extract in a 250–mL Erlenmeyer flask.

10.3 Add a second 60-mL volume of methylene chloride to the sample bottle and repeat the extraction procedure a second time, combining the extracts in the Erlenmeyer flask. Perform a third extraction in the same manner.

10.4 Assemble a Kuderna-Danish (K–D) concentrator by attaching a 10-mL concentrator tube to a 500-mL evaporative flask. Other concentration devices or techniques may be used in place of the K–D concentrator if the requirements of Section 8.2 are met.

10.5 Pour the combined extract through a solvent-rinsed drying column containing about 10 cm of anhydrous sodium sulfate, and collect the extract in the K–D concentrator. Rinse the Erlenmeyer flask and column with 20 to 30 mL of methylene chloride to complete the quantitative transfer.

10.6 Add one or two clean boiling chips to the evaporative flask and attach a three-ball Snyder column. Prewet the Snyder column by adding about 1 mL of methylene chloride to the top. Place the K–D apparatus on a hot water bath (60 to 65 °C) so that the concentrator tube is partially immersed in the hot water, and the entire lower rounded surface of the flask is bathed with hot vapor. Adjust the vertical position of the apparatus and the water temperature as required to complete the concentration in 15 to 20 min. At the proper rate of distillation the balls of the column will actively chatter but the chambers will not flood with condensed solvent. When the apparent volume of liquid reaches 1 mL, remove the K–D apparatus and allow it to drain and cool for at least 10 min.

10.7 Remove the Snyder column and rinse the flask and its lower joint into the concentrator tube with 1 to 2 mL of methylene chloride. A 5-mL syringe is recommended for this operation. Stopper the concentrator tube and store refrigerated if further processing will not be performed immediately. If the extract will be stored longer than two days, it should be transferred to a Teflon-sealed screw-cap vial and protected from light. If the sample extract requires no further cleanup, proceed with gas or liquid chromatographic analysis (Section 12 or 13). If the sample requires further cleanup, proceed to Section 11.

10.8 Determine the original sample volume by refilling the sample bottle to the mark and transferring the liquid to a 1000-mL graduated cylinder. Record the sample volume to the nearest 5 mL.

11. Cleanup and Separation

11.1 Cleanup procedures may not be necessary for a relatively clean sample matrix. If particular circumstances demand the use of a cleanup procedure, the analyst may use the procedure below or any other appropriate procedure. However, the analyst first must demonstrate that the requirements of Section 8.2 can be met using the methods as revised to incorporate the cleanup procedure.

11.2 Before the silica gel cleanup technique can be utilized, the extract solvent must be exchanged to cyclohexane. Add 1 to 10 mL of the sample extract (in methylene chloride) and a boiling chip to a clean K–D concentrator tube. Add 4 mL of cyclohexane and attach a two-ball micro-Snyder column. Prewet the column by adding 0.5 mL of methylene chloride to the top. Place the micro-K–D apparatus on a boiling (100 °C) water bath so that the concentrator tube is partially immersed in the hot water. Adjust the vertical position of the apparatus and the water temperature as required to complete concentration in 5 to 10 min. At the proper rate of distillation the balls of the column will actively chatter but the chambers will not flood. When the apparent volume of the liquid reaches 0.5 mL, remove the K–D apparatus and allow it to drain and cool for at least 10 min. Remove the micro-Snyder column and rinse its lower joint into the concentrator tube with a minimum amount of cyclohexane. Adjust the extract volume to about 2 mL.

11.3 Silica gel column cleanup for PAHs:

11.3.1 Prepare a slurry of 10 g of activiated silica gel in methylene chloride and place this into a 10-mm ID chromatographic column. Tap the column to settle the silica gel and elute the methylene chloride. Add 1 to 2 cm of anhydrous sodium sulfate to the top of the silica gel.

11.3.2 Preelute the column with 40 mL of pentane. The rate for all elutions should be about 2 mL/min. Discard the eluate and just prior to exposure of the sodium sulfate layer to the air, transfer the 2-mL cyclohexane sample extract onto the column using an additional 2 mL cyclohexane to complete the transfer. Just prior to exposure of the sodium sulfate layer to the air, add 25 mL of pentane and continue the elution of the column. Discard this pentane eluate.

11.3.3 Next, elute the column with 25 mL of methylene chloride/pentane (4+6)(V/V) into a 500-mL K–D flask equipped with a 10-mL concentrator tube. Concentrate the collected fraction to less than 10 mL as in Section 10.6. When the apparatus is cool, remove the Snyder column and rinse the flask and its lower joint with pentane. Proceed with HPLC or GC analysis.

12. High Performance Liquid Chromatography

12.1 To the extract in the concentrator tube, add 4 mL of acetonitrile and a new boiling chip, then attach a two-ball micro-Snyder column. Concentrate the solvent as in Section 10.6, except set the water bath at 95 to 100 °C. When the apparatus is cool, remove the micro-Snyder column and rinse its lower joint into the concentrator tube with about 0.2 mL of acetonitrile. Adjust the extract volume to 1.0 mL.

12.2 Table 1 summarizes the recommended operating conditions for the HPLC. Included in this table are retention times, capacity factors, and MDL that can be achieved under

these conditions. The UV detector is recommended for the determination of naphthalene, acenaphthylene, acenapthene, and fluorene and the fluorescence detector is recommended for the remaining PAHs. Examples of the separations achieved by this HPLC column are shown in Figures 1 and 2. Other HPLC columns, chromatographic conditions, or detectors may be used if the requirements of Section 8.2 are met.

12.3 Calibrate the system daily as described in Section 7.

12.4 If the internal standard calibration procedure is being used, the internal standard must be added to the sample extract and mixed thoroughly immediately before injection into the instrument.

12.5 Inject 5 to 25 µL of the sample extract or standard into the HPLC using a high pressure syringe or a constant volume sample injection loop. Record the volume injected to the nearest 0.1 µL, and the resulting peak size in area or peak height units. Re-equilibrate the HPLC column at the initial gradient conditions for at least 10 min between injections.

12.6 Identify the parameters in the sample by comparing the retention time of the peaks in the sample chromatogram with those of the peaks in standard chromatograms. The width of the retention time window used to make identifications should be based upon measurements of actual retention time variations of standards over the course of a day. Three times the standard deviation of a retention time for a compound can be used to calculate a suggested window size; however, the experience of the analyst should weigh heavily in the interpretation of chromatograms.

12.7 If the response for a peak exceeds the working range of the system, dilute the extract with acetonitrile and reanalyze.

12.8 If the measurement of the peak response is prevented by the presence of interferences, further cleanup is required.

13. Gas Chromatography

13.1 The packed column GC procedure will not resolve certain isomeric pairs as indicated in Section 1.3 and Table 2. The liquid chromatographic procedure (Section 12) must be used for these parameters.

13.2 To achieve maximum sensitivity with this method, the extract must be concentrated to 1.0 mL. Add a clean boiling chip to the methylene chloride extract in the concentrator tube. Attach a two-ball micro-Snyder column. Prewet the micro-Snyder column by adding about 0.5 mL of methylene chloride to the top. Place the micro-K–D apparatus on a hot water bath (60 to 65 °C) so that the concentrator tube is partially immersed in the hot water. Adjust the vertical position of the apparatus and the water temperature as required to complete the concentration in 5 to 10 min. At the proper rate of distillation the balls will actively chatter but the chambers will not flood. When the apparent volume of liquid reaches 0.5 mL, remove the K–D apparatus and allow it to drain and cool for at least 10 min. Remove the micro-Snyder column and rinse its lower joint into the concentrator tube with a minimum amount of methylene chloride. Adjust the final volume to 1.0 mL and stopper the concentrator tube.

13.3 Table 2 summarizes the recommended operating conditions for the gas chromatograph. Included in this table are retention times that were obtained under these conditions. An example of the separations achieved by this column is shown in Figure 3. Other packed or capillary (open-tubular) columns, chromatographic conditions, or detectors may be used if the requirements of Section 8.2 are met.

13.4 Calibrate the gas chromatographic system daily as described in Section 7.

13.5 If the internal standard calibration procedure is being used, the internal standard must be added to the sample extract and mixed thoroughly immediately before injection into the gas chromatograph.

13.6 Inject 2 to 5 µL of the sample extract or standard into the gas chromatograph using the solvent-flush technique.[10] Smaller (1.0 µL) volumes may be injected if automatic devices are employed. Record the volume injected to the nearest 0.05 µL, and the resulting peak size in area or peak height units.

13.7 Identify the parameters in the sample by comparing the retention times of the peaks in the sample chromatogram with those of the peaks in standard chromatograms. The width of the retention time window used to make identifications should be based upon measurements of actual retention time variations of standards over the course of a day. Three times the standard deviation of a retention time for a compound can be used to calculate a suggested window size; however, the experience of the analyst should weigh heavily in the interpretation of chromatograms.

13.8 If the response for a peak exceeds the working range of the system, dilute the extract and reanalyze.

13.9 If the measurement of the peak response is prevented by the presence of interferences, further cleanup is required.

14. Calculations

14.1 Determine the concentration of individual compounds in the sample.

14.1.1 If the external standard calibration procedure is used, calculate the amount of material injected from the peak response using the calibration curve or calibration factor determined in Section 7.2.2. The concentration in the sample can be calculated from Equation 2.

$$\text{Concentration } (\mu g/L) = \frac{(A)(V_t)}{(V_i)(V_s)}$$

Equation 2

where:

A=Amount of material injected (ng).

V_i=Volume of extract injected (µL).

V_t=Volume of total extract (µL).

V_s=Volume of water extracted (mL).

13.1.2 If the internal standard calibration procedure is used, calculate the concentration in the sample using the response factor (RF) determined in Section 7.3.2 and Equation 3.

$$\text{Concentration } (\mu g/L) = \frac{(A_s)(I_s)}{(A_{is})(RF)(V_o)}$$

Equation 3

where:

A_s=Response for the parameter to be measured.

A_{is}=Response for the internal standard.

I_s=Amount of internal standard added to each extract (µg).

V_o=Volume of water extracted (L).

14.2 Report results in µg/L without correction for recovery data. All QC data obtained should be reported with the sample results.

15. Method Performance

15.1 The method detection limit (MDL) is defined as the minimum concentration of a substance that can be measured and reported with 99% confidence that the value is above zero.[1] The MDL concentrations listed in Table 1 were obtained using reagent water.[11] Similar results were achieved using representative wastewaters. MDL for the GC approach were not determined. The MDL actually achieved in a given analysis will vary depending on instrument sensitivity and matrix effects.

15.2 This method has been tested for linearity of spike recovery from reagent water and has been demonstrated to be applicable over the concentration range from 8 × MDL to 800 × MDL[11] with the following exception: benzo(ghi)perylene recovery at 80 × and 800 × MDL were low (35% and 45%, respectively).

15.3 This method was tested by 16 laboratories using reagent water, drinking water, surface water, and three industrial wastewaters spiked at six concentrations over the range 0.1 to 425 µg/L.[12] Single operator precision, overall precision, and method accuracy were found to be directly related to the concentration of the parameter and essentially independent of the sample matrix. Linear equations to describe these relationships are presented in Table 4.

References

1. 40 CFR part 136, appendix B.

2. ''Determination of Polynuclear Aromatic Hydrocarbons in Industrial and Municipal Wastewaters,'' EPA 600/4–82–025, National Technical Information Service, PB82–258799, Springfield, Virginia 22161, June 1982.

3. ASTM Annual Book of Standards, Part 31, D3694–78. ''Standard Practices for Preparation of Sample Containers and for Preservation of Organic Constituents,'' American Society for Testing and Materials, Philadelphia.

4. ''Carcinogens—Working With Carcinogens,'' Department of Health, Education, and Welfare, Public Health Service, Center for Disease Control, National Institute for Occupational Safety and Health, Publication No. 77–206, August 1977.

5. ''OSHA Safety and Health Standards, General Industry,'' (29 CFR part 1910), Occupational Safety and Health Administration, OSHA 2206 (Revised, January 1976).

6. ''Safety in Academic Chemistry Laboratories,'' American Chemical Society Publication, Committee on Chemical Safety, 3rd Edition, 1979.

7. Provost, L.P., and Elder, R.S. ''Interpretation of Percent Recovery Data,'' *American Laboratory, 15*, 58–63 (1983). (The value 2.44 used in the equation in Section 8.3.3 is two times the value 1.22 derived in this report.)

8. ASTM Annual Book of Standards, Part 31, D3370–76. ''Standard Practices for Sampling Water,'' American Society for Testing and Materials, Philadelphia.

9. ''Methods 330.4 (Titrimetric, DPD–FAS) and 330.5 (Spectrophotometric, DPD) for Chlorine, Total Residual,'' Methods for Chemical Analysis of Water and Wastes, EPA–600/4–79–020, U.S. Environmental Protection Agency, Environmental Monitoring and Support Laboratory, Cincinnati, Ohio 45268, March 1979.

10. Burke, J.A. ''Gas Chromatography for Pesticide Residue Analysis; Some Practical Aspects,'' *Journal of the Association of Official Analytical Chemists, 48,* 1037 (1965).

11. Cole, T., Riggin, R., and Glaser, J. ''Evaluation of Method Detection Limits and Analytical Curve for EPA Method 610—PNAs,'' International Symposium on Polynuclear Aromatic Hydrocarbons, 5th, Battelle's Columbus Laboratories, Columbus, Ohio (1980).

12. ''EPA Method Study 20, Method 610 (PNA's),'' EPA 600/4–84–063, National Technical Information Service, PB84–211614, Springfield, Virginia 22161, June 1984.

TABLE 1—HIGH PERFORMANCE LIQUID CHROMATOGRAPHY CONDITIONS AND METHOD DETECTION LIMITS

Parameter	Retention time (min)	Column capacity factor (k′)	Method detection limit (μg/L) [a]
Naphthalene	16.6	12.2	1.8
Acenaphthylene	18.5	13.7	2.3
Acenaphthene	20.5	15.2	1.8
Fluorene	21.2	15.8	0.21
Phenanthrene	22.1	16.6	0.64
Anthracene	23.4	17.6	0.66
Fluoranthene	24.5	18.5	0.21
Pyrene	25.4	19.1	0.27
Benzo(a)anthracene	28.5	21.6	0.013
Chrysene	29.3	22.2	0.15
Benzo(b)fluoranthene	31.6	24.0	0.018
Benzo(k)fluoranthene	32.9	25.1	0.017
Benzo(a)pyrene	33.9	25.9	0.023
Dibenzo(a,h)anthracene	35.7	27.4	0.030
Benzo(ghi)perylene	36.3	27.8	0.076
Indeno(1,2,3-cd)pyrene	37.4	28.7	0.043

AAAHPLC column conditions: Reverse phase HC–ODS Sil–X, 5 micron particle size, in a 25 cm × 2.6 mm ID stainless steel column. Isocratic elution for 5 min. using acetonitrile/water (4+6), then linear gradient elution to 100% acetonitrile over 25 min. at 0.5 mL/min flow rate. If columns having other internal diameters are used, the flow rate should be adjusted to maintain a linear velocity of 2 mm/sec.

[a] The MDL for naphthalene, acenaphthylene, acenaphthene, and fluorene were determined using a UV detector. All others were determined using a fluorescence detector.

TABLE 2—GAS CHROMATOGRAPHIC CONDITIONS AND RETENTION TIMES

Parameter	Retention time (min)
Naphthalene	4.5
Acenaphthylene	10.4
Acenaphthene	10.8
Fluorene	12.6
Phenanthrene	15.9
Anthracene	15.9
Fluoranthene	19.8
Pyrene	20.6
Benzo(a)anthracene	24.7
Chrysene	24.7
Benzo(b)fluoranthene	28.0
Benzo(k)fluoranthene	28.0
Benzo(a)pyrene	29.4
Dibenzo(a,h)anthracene	36.2
Indeno(1,2,3-cd)pyrene	36.2
Benzo(ghi)perylene	38.6

GC Column conditions: Chromosorb W–AW–DCMS (100/120 mesh) coated with 3% OV–17 packed in a 1.8 x 2 mm ID glass column with nitrogen carrier gas at 40 mL/min. flow rate. Column temperature was held at 100 °C for 4 min., then programmed at 8 °C/min. to a final hold at 280 °C.

TABLE 3—QC ACCEPTANCE CRITERIA—METHOD 610

Parameter	Test conc. (μg/L)	Limit for s (μg/L)	Range for $\bar{X}$ (μg/L)	Range for P, P_s (%)
Acenaphthene	100	40.3	D–105.7	D–124
Acenaphthylene	100	45.1	22.1–112.1	D–139
Anthracene	100	28.7	11.2–112.3	D–126
Benzo(a)anthracene	10	4.0	3.1–11.6	12–135
Benzo(a)pyrene	10	4.0	0.2–11.0	D–128
Benzo(b)fluor-anthene	10	3.1	1.8–13.8	6–150
Benzo(ghi)perylene	10	2.3	D–10.7	D–116
Benzo(k)fluo-ranthene	5	2.5	D–7.0	D–159
Chrysene	10	4.2	D–17.5	D–199
Dibenzo(a,h)an-thracene	10	2.0	0.3–10.0	D–110
Fluoranthene	10	3.0	2.7–11.1	14–123
Fluorene	100	43.0	D–119	D–142
Indeno(1,2,3–cd)pyrene	10	3.0	1.2–10.0	D–116
Naphthalene	100	40.7	21.5–100.0	D–122
Phenanthrene	100	37.7	8.4–133.7	D–155
Pyrene	10	3.4	1.4–12.1	D–140

s=Standard deviation of four recovery measurements, in μg/L (Section 8.2.4).
$\bar{X}$=Average recovery for four recovery measurements, in μg/L (Section 8.2.4).
P, P_s=Percent recovery measured (Section 8.3.2, Section 8.4.2).
D=Detected; result must be greater than zero.

NOTE: These criteria are based directly upon the method performance data in Table 4. Where necessary, the limits for recovery have been broadened to assure applicability of the limits to concentrations below those used to develop Table 4.

TABLE 4—METHOD ACCURACY AND PRECISION AS FUNCTIONS OF CONCENTRATION—METHOD 610

Parameter	Accuracy, as recovery, X′ (μg/L)	Single analyst precision, s_r' (μg/L)	Overall precision, S′ (μg/L)
Acenaphthene	$0.52C+0.54$	$0.39\bar{X}+0.76$	$0.53\bar{X}+1.32$
Acenaphthylene	$0.69C-1.89$	$0.36\bar{X}+0.29$	$0.42\bar{X}+0.52$
Anthracene	$0.63C-1.26$	$0.23\bar{X}+1.16$	$0.41\bar{X}+0.45$
Benzo(a)anthracene	$0.73C+0.05$	$0.28\bar{X}+0.04$	$0.34\bar{X}+0.02$
Benzo(a)pyrene	$0.56C+0.01$	$0.38\bar{X}-0.01$	$0.53\bar{X}-0.01$
Benzo(b)fluoranthene	$0.78C+0.01$	$0.21\bar{X}+0.01$	$0.38\bar{X}-0.00$
Benzo(ghi)perylene	$0.44C+0.30$	$0.25\bar{X}+0.04$	$0.58\bar{X}+0.10$
Benzo(k)fluoranthene	$0.59C+0.00$	$0.44\bar{X}-0.00$	$0.69\bar{X}+0.01$
Chrysene	$0.77C-0.18$	$0.32\bar{X}-0.18$	$0.66\bar{X}-0.22$
Dibenzo(a,h)anthracene	$0.41C+0.11$	$0.24\bar{X}+0.02$	$0.45\bar{X}+0.03$

TABLE 4—METHOD ACCURACY AND PRECISION AS FUNCTIONS OF CONCENTRATION—METHOD 610—Continued

Parameter	Accuracy, as recovery, X′ (μg/L)	Single analyst precision, s_r' (μg/L)	Overall precision, S′ (μg/L)
Fluoranthene	0.68C + 0.07	0.22$\bar{X}$ + 0.06	0.32$\bar{X}$ + 0.03
Fluorene	0.56C − 0.52	0.44$\bar{X}$ − 1.12	0.63$\bar{X}$ − 0.65
Indeno(1,2,3–cd)pyrene	0.54C + 0.06	0.29$\bar{X}$ + 0.02	0.42$\bar{X}$ + 0.01
Naphthalene	0.57C − 0.70	0.39$\bar{X}$ − 0.18	0.41$\bar{X}$ + 0.74
Phenanthrene	0.72C − 0.95	0.29$\bar{X}$ + 0.05	0.47$\bar{X}$ − 0.25
Pyrene	0.69C − 0.12	0.25$\bar{X}$ + 0.14	0.42$\bar{X}$ − 0.00

X′=Expected recovery for one or more measurements of a sample containing a concentration of C, in μg/L.
s_r'=Expected single analyst standard deviation of measurements at an average concentration found of $\bar{X}$, in μg/L.
S′=Expected interlaboratory standard deviation of measurements at an average concentration found of X, in μg/L.
C=True value for the concentration, in μg/L.
X=Average recovery found for measurements of samples containing a concentration of C, in μg/L.

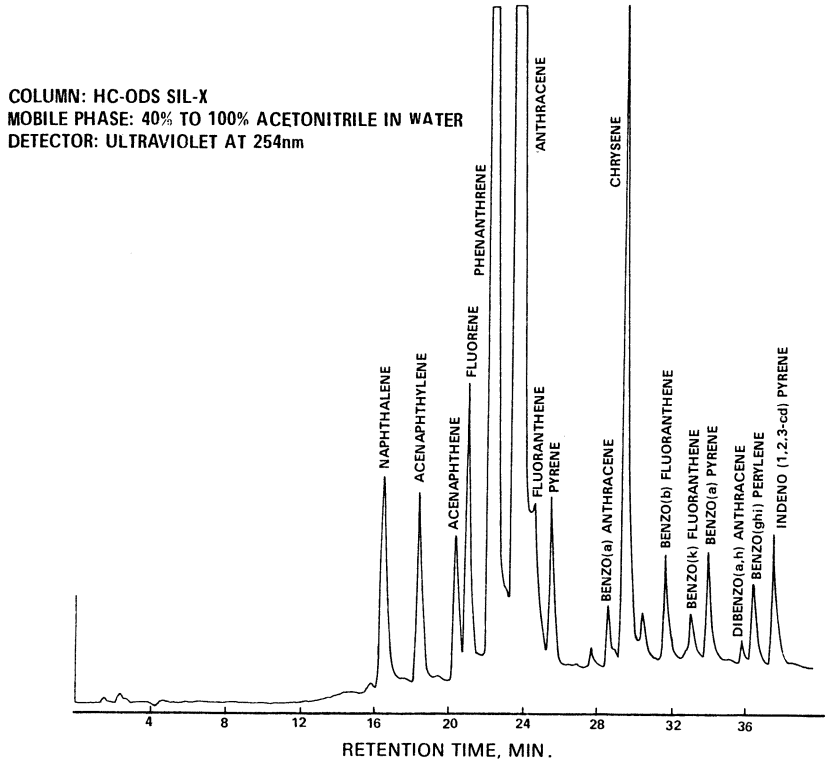


Figure 1. Liquid chromatogram of polynuclear aromatic hydrocarbons.

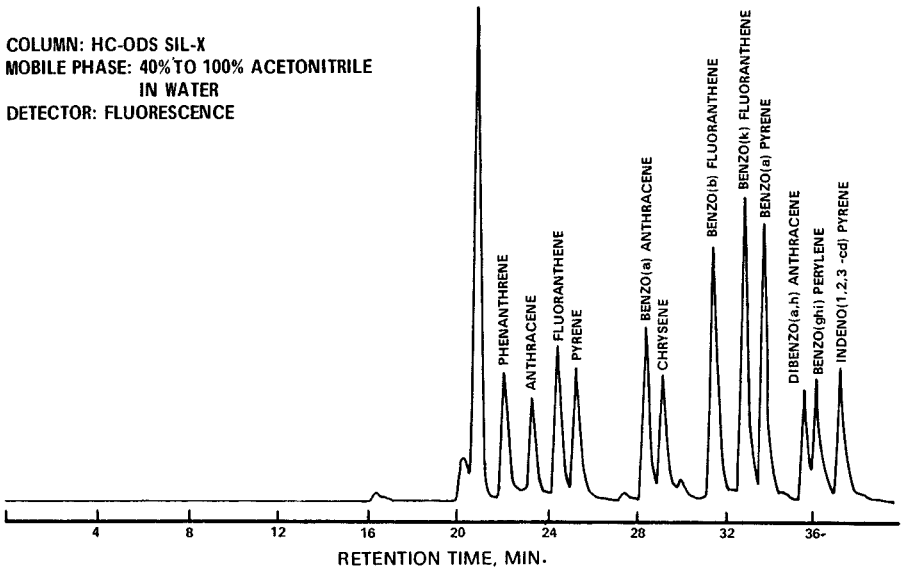


Figure 2. Liquid chromatogram of polynuclear aromatic hydrocarbons.

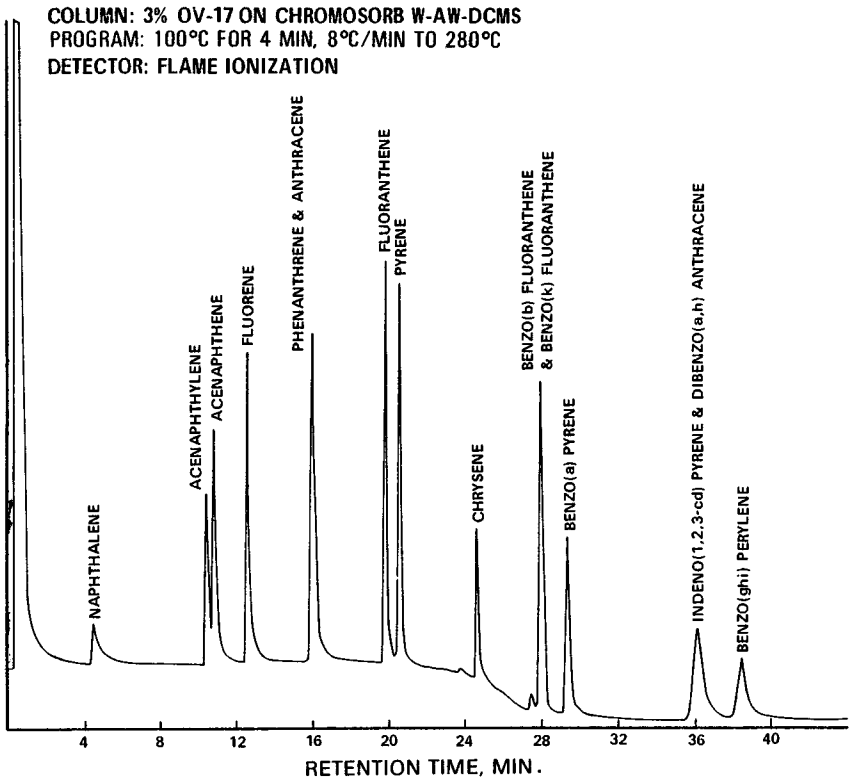


Figure 3. Gas chromatogram of polynuclear aromatic hydrocarbons.

METHOD 611—HALOETHERS

1. Scope and Application

1.1 This method covers the determination of certain haloethers. The following parameters can be determined by this method:

Parameter	STORET No.	CAS No.
Bis(2-chloroethyl) ether	34273	111–44–4
Bis(2-chloroethoxy) methane	34278	111–91–1
Bis(2-chloroisopropyl) ether	34283	108–60–1
4-Bromophenyl phenyl ether	34636	101–55–3
4-Chlorophenyl phenyl either	34641	7005–72–3

1.2 This is a gas chromatographic (GC) method applicable to the determination of the compounds listed above in municipal and industrial discharges as provided under 40 CFR 136.1. When this method is used to analyze unfamiliar samples for any or all of the compounds above, compound identifications should be supported by at least one additional qualitative technique. This method describes analytical conditions for a second gas chromatographic column that can be used to confirm measurements made with the primary column. Method 625 provides gas chromatograph/mass spectrometer (GC/MS) conditions appropriate for the qualitative and quantitative confirmation of results for all of the parameters listed above, using the extract produced by this method.

1.3 The method detection limit (MDL, defined in Section 14.1)[1] for each parameter is listed in Table 1. The MDL for a specific wastewater may differ from those listed, depending upon the nature of interferences in the sample matrix.

1.4 The sample extraction and concentration steps in this method are essentially the same as in Methods 606, 608, 609, and 612. Thus, a single sample may be extracted to measure the parameters included in the scope of each of these methods. When cleanup is required, the concentration levels must be high enough to permit selecting aliquots, as necessary, to apply appropriate cleanup procedures. The analyst is allowed the latitude, under Section 12, to select chromatographic conditions appropriate for the simultaneous measurement of combinations of these parameters.

1.5 Any modification of this method, beyond those expressly permitted, shall be considered as a major modification subject to application and approval of alternate test procedures under 40 CFR 136.4 and 136.5.

1.6 This method is restricted to use by or under the supervision of analysts experienced in the use of a gas chromatograph and in the interpretation of gas chromatograms. Each analyst must demonstrate the ability to generate acceptable results with this method using the procedure described in Section 8.2.

2. Summary of Method

2.1 A measured volume of sample, approximately 1–L, is extracted with methylene chloride using a separatory funnel. The methylene chloride extract is dried and exchanged to hexane during concentration to a volume of 10 mL or less. The extract is separated by gas chromatography and the parameters are then measured with a halide specific detector.[2]

2.2 The method provides a Florisil column cleanup procedure to aid in the elimination of interferences that may be encountered.

3. Interferences

3.1 Method interferences may be caused by contaminants in solvents, reagents, glassware, and other sample processing hardware that lead to discrete artifacts and/or elevated baselines in gas chromatograms. All of these materials must be routinely demonstrated to be free from interferences under the conditions of the analysis by running laboratory reagent blanks as described in Section 8.1.3.

3.1.1 Glassware must be scrupulously cleaned.[3] Clean all glassware as soon as possible after use by rinsing with the last solvent used in it. Solvent rinsing should be followed be detergent washing with hot water, and rinses with tap water and distilled water. The glassware should then be drained dry, and heated in a muffle furnace at 400 °C for 15 to 30 min. Some thermally stable materials, such a PCBs, may not be eliminated by this treatment. Solvent rinses with acetone and pesticide quality hexane may be substituted for the muffle furnace heating. Thorough rinsing with such solvents usually eliminates PCB interference. Volumetric ware should not be heated in a muffle furnace. After drying and cooling, glassware should be sealed and stored in a clean environment to prevent any accumulation of dust or other contaminants. Store inverted or capped with aluminum foil.

3.1.2 The use of high purity reagents and solvents helps to minimize interference problems. Purification of solvents by distillation in all-glass systems may be required.

3.2 Matrix interferences may be caused by contaminants that are co-extracted from the sample. The extent of matrix interferences will vary considerably from source to source, depending upon the nature and diversity of the industrial complex or municipality being sampled. The cleanup procedure in Section 11 can be used to overcome many of these interferences, but unique samples may require additional cleanup approaches to achieve the MDL listed in Table 1.

3.3 Dichlorobenzenes are known to coelute with haloethers under some gas chromatographic conditions. If these materials are present together in a sample, it may be necessary to analyze the extract

with two different column packings to completely resolve all of the compounds.

4. Safety

4.1 The toxicity or carcinogenicity of each reagent used in this method has not been precisely defined; however, each chemical compound should be treated as a potential health hazard. From this viewpoint, exposure to these chemicals must be reduced to the lowest possible level by whatever means available. The laboratory is responsible for maintaining a current awareness file of OSHA regulations regarding the safe handling of the chemicals specified in this method. A reference file of material data handling sheets should also be made available to all personnel involved in the chemical analysis. Additional references to laboratory safety are available and have been identified[4-6] for the information of the analyst.

5. Apparatus and Materials

5.1 Sampling equipment, for discrete or composite sampling.

5.1.1 Grab sample bottle—1-L or 1-qt, amber glass, fitted with a screw cap lined with Teflon. Foil may be substituted for Teflon if the sample is not corrosive. If amber bottles are not available, protect samples from light. The bottle and cap liner must be washed, rinsed with acetone or methylene chloride, and dried before use to minimize contamination.

5.1.2 Automatic sampler (optional)—The sampler must incorporate glass sample containers for the collection of a minimum of 250 mL of sample. Sample containers must be kept refrigerated at 4 °C and protected from light during compositing. If the sampler uses a peristaltic pump, a minimum length of compressible silicone rubber tubing may be used. Before use, however, the compressible tubing should be thoroughly rinsed with methanol, followed by repeated rinsings with distilled water to minimize the potential for contamination of the sample. An integrating flow meter is required to collect flow proportional composites.

5.2 Glassware (All specifications are suggested. Catalog numbers are included for illustration only.):

5.2.1 Separatory funnel—2-L, with Teflon stopcock.

5.2.2 Drying column—Chromatographic column, approximately 400 mm long x 19 mm ID, with coarse frit filter disc.

5.2.3 Chromatographic column—400 mm long x 19 mm ID, with Teflon stopcock and coarse frit filter disc at bottom (Kontes K–420540–0224 or equivalent).

5.2.4 Concentrator tube, Kuderna-Danish—10-mL, graduated (Kontes K–570050–1025 or equivalent). Calibration must be checked at the volumes employed in the test. Ground glass stopper is used to prevent evaporation of extracts.

5.2.5 Evaporative flask, Kuderna-Danish—500-mL (Kontes K–570001–0500 or equivalent). Attach to concentrator tube with springs.

5.2.6 Snyder column, Kuderna-Danish—Three-ball macro (Kontes K–503000–0121 or equivalent).

5.2.7 Vials—10 to 15-mL, amber glass, with Teflon-lined screw cap.

5.3 Boiling chips—Approximately 10/40 mesh. Heat to 400 °C for 30 min or Soxhlet extract with methylene chloride.

5.4 Water bath—Heated, with concentric ring cover, capable of temperature control (±2°C). The bath should be used in a hood.

5.5 Balance—Analytical, capable of accurately weighing 0.0001 g.

5.6 Gas chromatograph—An analytical system complete with temperature programmable gas chromatograph suitable for on-column injection and all required accessories including syringes, analytical columns, gases, detector, and strip-chart recorder. A data system is recommended for measuring peak areas.

5.6.1 Column 1—1.8 m long x 2 mm ID glass, packed with 3% SP–1000 on Supelcoport (100/120 mesh) or equivalent. This column was used to develop the method performance statements in Section 14. Guidelines for the use of alternate column packings are provided in Section 12.1.

5.6.2 Column 2—1.8 m long x 2 mm ID glass, packed with 2,6-diphenylene oxide polymer (60/80 mesh), Tenax, or equivalent.

5.6.3 Detector—Halide specific detector: electrolytic conductivity or microcoulometric. These detectors have proven effective in the analysis of wastewaters for the parameters listed in the scope (Section 1.1). The Hall conductivity detector was used to develop the method performance statements in Section 14. Guidelines for the use of alternate detectors are provided in Section 12.1. Although less selective, an electron capture detector is an acceptable alternative.

6. Reagents

6.1 Reagent water—Reagent water is defined as a water in which an interferent is not observed at the MDL of the parameters of interest.

6.2 Sodium thiosulfate—(ACS) Granular.

6.3 Acetone, hexane, methanol, methylene chloride, petroleum ether (boiling range 30–60 °C)—Pesticide quality or equivalent.

6.4 Sodium sulfate—(ACS) Granular, anhydrous. Purify by heating at 400 °C for 4 h in a shallow tray.

6.5 Florisil—PR Grade (60/100 mesh). Purchase activated at 1250 °F and store in the dark in glass containers with ground glass stoppers or foil-lined screw caps. Before use, activate each batch at least 16 h at 130 °C in a foil-covered glass container and allow to cool.

6.6 Ethyl ether—Nanograde, redistilled in glass if necessary.

6.6.1 Ethyl ether must be shown to be free of peroxides before it is used as indicated by EM Laboratories Quant test strips. (Available from Scientific Products Co., Cat. No. P1126–8, and other suppliers.)

6.6.2 Procedures recommended for removal of peroxides are provided with the test strips. After cleanup, 20 mL of ethyl alcohol preservative must be added to each liter of ether.

6.7 Stock standard solutions (1.00 µg/µL)—Stock standard solutions can be prepared from pure standard materials or purchased as certified solutions.

6.7.1 Prepare stock standard solutions by accurately weighing about 0.0100 g of pure material. Dissolve the material in acetone and dilute to volume in a 10-mL volumetric flask. Larger volumes can be used at the convenience of the analyst. When compound purity is assayed to be 96% or greater, the weight can be used without correction to calculate the concentration of the stock standard. Commercially prepared stock standards can be used at any concentration if they are certified by the manufacturer or by an independent source.

6.7.2 Transfer the stock standard solutions into Teflon-sealed screw-cap bottles. Store at 4 °C and protect from light. Stock standard solutions should be checked frequently for signs of degradation or evaporation, especially just prior to preparing calibration standards from them.

6.7.3 Stock standard solutions must be replaced after six months, or sooner if comparison with check standards indicates a problem.

6.8 Quality control check sample concentrate—See Section 8.2.1.

7. Calibration

7.1 Establish gas chromatographic operating conditions equivalent to those given in Table 1. The gas chromatographic system can be calibrated using the external standard technique (Section 7.2) or the internal standard technique (Section 7.3).

7.2 External standard calibration procedure:

7.2.1 Prepare calibration standards at a minimum of three concentration levels for each parameter of interest by adding volumes of one or more stock standards to a volumetric flask and diluting to volume with hexane. One of the external standards should be at a concentration near, but above, the MDL (Table 1) and the other concentrations should correspond to the expected range of concentrations found in real samples or should define the working range of the detector.

7.2.2 Using injections of 2 to 5 µL, analyze each calibration standard according to Section 12 and tabulate peak height or area responses against the mass injected. The results can be used to prepare a calibration curve for each compound. Alternatively, if the ratio of response to amount injected (calibration factor) is a constant over the working range (<10% relative standard deviation, RSD), linearity through the origin can be assumed and the average ratio or calibration factor can be used in place of a calibration curve.

7.3 Internal standard calibration procedure—To use this approach, the analyst must select one or more internal standards that are similar in analytical behavior to the compounds of interest. The analyst must further demonstrate that the measurement of the internal standard is not affected by method or matrix interferences. Because of these limitations, no internal standard can be suggested that is applicable to all samples.

7.3.1 Prepare calibration standards at a minimum of three concentration levels for each parameter of interest by adding volumes of one or more stock standards to a volumetric flask. To each calibration standard, add a known constant amount of one or more internal standards, and dilute to volume with hexane. One of the standards should be at a concentration near, but above, the MDL and the other concentrations should correspond to the expected range of concentrations found in real samples or should define the working range of the detector.

7.3.2 Using injections of 2 to 5 µL, analyze each calibration standard according to Section 12 and tabulate peak height or area responses against concentration for each compound and internal standard. Calculate response factors (RF) for each compound using Equation 1.

$$RF = \frac{(A_s)(C_{is})}{(A_{is})(C_s)}$$

Equation 1

where:

A_s=Response for the parameter to be measured.

A_{is}=Response for the internal standard.

C_{is}=Concentration of the internal standard (µg/L).

C_s=Concentration of the parameter to be measured (µg/L).

If the RF value over the working range is a constant (< 10% RSD), the RF can be assumed to be invariant and the average RF can be used for calculations. Alternatively, the results can be used to plot a calibration curve of response ratios, A_s/A_{is}, vs. RF.

7.4 The working calibration curve, calibration factor, or RF must be verified on each working day by the measurement of one

or more calibration standards. If the response for any parameter varies from the predicted response by more than ±15%, a new calibration curve must be prepared for that compound.

7.5 The cleanup procedure in Section 11 utilizes Florisil column chromatography. Florisil from different batches or sources may vary in adsorptive capacity. To standardize the amount of Florisil which is used, the use of lauric acid value[7] is suggested. The referenced procedure determines the adsorption from hexane solution of lauric acid (mg) per g of Florisil. The amount of Florisil to be used for each column is calculated by dividing 110 by this ratio and multiplying by 20 g.

7.6 Before using any cleanup procedure, the analyst must process a series of calibration standards through the procedure to validate elution patterns and the absence of interferences from the reagents.

8. Quality Control

8.1 Each laboratory that uses this method is required to operate a formal quality control program. The minimum requirements of this program consist of an initial demonstration of laboratory capability and an ongoing analysis of spiked samples to evaluate and document data quality. The laboratory must maintain records to document the quality of data that is generated. Ongoing data quality checks are compared with established performance criteria to determine if the results of analyses meet the performance characteristics of the method. When results of sample spikes indicate atypical method performance, a quality control check standard must be analyzed to confirm that the measurements were performed in an in-control mode of operation.

8.1.1 The analyst must make an initial, one-time, demonstration of the ability to generate acceptable accuracy and precision with this method. This ability is established as described in Section 8.2.

8.1.2 In recognition of advances that are occurring in chromatography, the analyst is permitted certain options (detailed in Sections 10.4, 11.1, and 12.1) to improve the separations or lower the cost of measurements. Each time such a modification is made to the method, the analyst is required to repeat the procedure in Section 8.2.

8.1.3 Before processing any samples, the analyst must analyze a reagent water blank to demonstrate that interferences from the analytical system and glassware are under control. Each time a set of samples is extracted or reagents are changed, a reagent water blank must be processed as a safeguard against laboratory contamination.

8.1.4 The laboratory must, on an ongoing basis, spike and analyze a minimum of 10% of all samples to monitor and evaluate laboratory data quality. This procedure is described in Section 8.3.

8.1.5 The laboratory must, on an ongoing basis, demonstrate through the analyses of quality control check standards that the operation of the measurement system is in control. This procedure is described in Section 8.4. The frequency of the check standard analyses is equivalent to 10% of all samples analyzed but may be reduced if spike recoveries from samples (Section 8.3) meet all specified quality control criteria.

8.1.6 The laboratory must maintain performance records to document the quality of data that is generated. This procedure is described in Section 8.5.

8.2 To establish the ability to generate acceptable accuracy and precision, the analyst must perform the following operations.

8.2.1 A quality control (QC) check sample concentrate is required containing each parameter of interest at a concentration of 100 µg/mL in acetone. The QC check sample concentrate must be obtained from the U.S. Environmental Protection Agency, Environmental Monitoring and Support Laboratory in Cincinnati, Ohio, if available. If not available from that source, the QC check sample concentrate must be obtained from another external source. If not available from either source above, the QC check sample concentrate must be prepared by the laboratory using stock standards prepared independently from those used for calibration.

8.2.2 Using a pipet, prepare QC check samples at a concentration of 100 µg/L by adding 1.00 mL of QC check sample concentrate to each of four 1–L aliquots of reagent water.

8.2.3 Analyze the well-mixed QC check samples according to the method beginning in Section 10.

8.2.4 Calculate the average recovery ($\bar{X}$) in µg/L, and the standard deviation of the recovery (s) in µg/L, for each parameter using the four results.

8.2.5 For each parameter compare s and $\bar{X}$ with the corresponding acceptance criteria for precision and accuracy, respectively, found in Table 2. If s and $\bar{X}$ for all parameters of interest meet the acceptance criteria, the system performance is acceptable and analysis of actual samples can begin. If any individual s exceeds the precision limit or any individual $\bar{X}$ falls outside the range for accuracy, the system performance is unacceptable for that parameter. Locate and correct the source of the problem and repeat the test for all parameters of interest beginning with Section 8.2.2.

8.3 The laboratory must, on an ongoing basis, spike at least 10% of the samples from each sample site being monitored to assess accuracy. For laboratories analyzing one to ten samples per month, at least one spiked sample per month is required.

8.3.1. The concentration of the spike in the sample should be determined as follows:

8.3.1.1 If, as in compliance monitoring, the concentration of a specific parameter in the sample is being checked against a regulatory concentration limit, the spike should be at that limit or 1 to 5 times higher than the background concentration determined in Section 8.3.2, whichever concentration would be larger.

8.3.1.2 If the concentration of a specific parameter in the sample is not being checked against a limit specific to that parameter, the spike should be at 100 µg/L or 1 to 5 times higher than the background concentration determined in Section 8.3.2, whichever concentration would be larger.

8.3.1.3 If it is impractical to determine background levels before spiking (e.g., maximum holding times will be exceeded), the spike concentration should be (1) the regulatory concentration limit, if any; or, if none (2) the larger of either 5 times higher than the expected background concentration or 100 µg/L.

8.3.2 Analyze one sample aliquot to determine the background concentration (B) of each parameter. If necessary, prepare a new QC check sample concentrate (Section 8.2.1) appropriate for the background concentrations in the sample. Spike a second sample aliquot with 1.0 mL of the QC check sample concentrate and analyze it to determine the concentration after spiking (A) of each parameter. Calculate each percent recovery (P) as 100(A–B)%/T, where T is the known true value of the spike.

8.3.3 Compare the percent recovery (P) for each parameter with the corresponding QC acceptance criteria found in Table 2. These acceptance criteria were calculated to include an allowance for error in measurement of both the background and spike concentrations, assuming a spike to background ratio of 5:1. This error will be accounted for to the extent that the analyst's spike to background ratio approaches 5:1.[8] If spiking was performed at a concentration lower than 100 µg/L, the analyst must use either the QC acceptance criteria in Table 2, or optional QC acceptance criteria calculated for the specific spike concentration. To calculate optional acceptance criteria for the recovery of a parameter: (1) Calculate accuracy (X′) using the equation in Table 3, substituting the spike concentration (T) for C; (2) calculate overall precision (S′) using the equation in Table 3, substituting X′ for $\bar{X}$; (3) calculate the range for recovery at the spike concentration as (100 X′/T)±2.44(100 S′/T)%.[8]

8.3.4 If any individual P falls outside the designated range for recovery, that parameter has failed the acceptance criteria. A check standard containing each parameter that failed the criteria must be analyzed as described in Section 8.4.

8.4 If any parameter fails the acceptance criteria for recovery in Section 8.3, a QC check standard containing each parameter that failed must be prepared and analyzed.

NOTE: The frequency for the required analysis of a QC check standard will depend upon the number of parameters being simultaneously tested, the complexity of the sample matrix, and the performance of the laboratory.

8.4.1 Prepare the QC check standard by adding 1.0 m/L of QC check sample concentrate (Section 8.2.1 or 8.3.2) to 1 L of reagent water. The QC check standard needs only to contain the parameters that failed criteria in the test in Section 8.3.

8.4.2 Analyze the QC check standard to determine the concentration measured (A) of each parameter. Calculate each percent recovery (P_s) as 100 (A/T)%, where T is the true value of the standard concentration.

8.4.3 Compare the percent recovery (P_s) for each parameter with the corresponding QC acceptance criteria found in Table 2. Only parameters that failed the test in Section 8.3 need to be compared with these criteria. If the recovery of any such parameter falls outside the designated range, the laboratory performance for that parameter is judged to be out of control, and the problem must be immediately identified and corrected. The analytical result for that parameter in the unspiked sample is suspect and may not be reported for regulatory compliance purposes.

8.5 As part of the QC program for the laboratory, method accuracy for wastewater samples must be assessed and records must be maintained. After the analysis of five spiked wastewater samples as in Section 8.3, calculate the average percent recovery ($\bar{P}$) and the standard deviation of the percent recovery (s_p). Express the accuracy assessment as a percent recovery interval from $\bar{P}-2s_p$ to $\bar{P}+2s_p$. If $\bar{P}$=90% and s_p=10%, for example, the accuracy interval is expressed as 70–110%. Update the accuracy assessment for each parameter on a regular basis (e.g. after each five to ten new accuracy measurements).

8.6 It is recommended that the laboratory adopt additional quality assurance practices for use with this method. The specific practices that are most productive depend upon the needs of the laboratory and the nature of the samples. Field duplicates may be analyzed to assess the precision of the environmental measurements. When doubt exists over the identification of a peak on the chromatogram, confirmatory techniques such as gas chromatography with a dissimilar column, specific element detector, or mass spectrometer must be used. Whenever possible, the laboratory should analyze standard reference materials and participate in relevant performance evaluation studies.

9. Sample Collection, Preservation, and Handling

9.1 Grab samples must be collected in glass containers. Conventional sampling practices[9] should be followed, except that the bottle must not be prerinsed with sample before collection. Composite samples should be collected in refrigerated glass containers in accordance with the requirements of the program. Automatic sampling equipment must be as free as possible of Tygon tubing and other potential sources of contamination.

9.2 All samples must be iced or refrigerated at 4°C from the time of collection until extraction. Fill the sample bottles and, if residual chlorine is present, add 80 mg of sodium thiosulfate per liter of sample and mix well. EPA Methods 330.4 and 330.5 may be used for measurement of residual chlorine.[10] Field test kits are available for this purpose.

9.3 All samples must be extracted within 7 days of collection and completely analyzed within 40 days of extraction.[2]

10. Sample Extraction

10.1 Mark the water meniscus on the side of the sample bottle for later determination of sample volume. Pour the entire sample into a 2-L separatory funnel.

10.2 Add 60 mL methylene chloride to the sample bottle, seal, and shake 30 s to rinse the inner surface. Transfer the solvent to the separatory funnel and extract the sample by shaking the funnel for 2 min with periodic venting to release excess pressure. Allow the organic layer to separate from the water phase for a minimum of 10 min. If the emulsion interface between layers is more than one-third the volume of the solvent layer, the analyst must employ mechanical techniques to complete the phase separation. The optimum technique depends upon the sample, but may include stirring, filtration of the emulsion through glass wool, centrifugation, or other physical methods. Collect the methylene chloride extract in a 250-mL Erlenmeyer flask.

10.3 Add a second 60-mL volume of methylene chloride to the sample bottle and repeat the extraction procedure a second time, combining the extracts in the Erlenmeyer flask. Perform a third extraction in the same manner.

10.4 Assemble a Kuderna-Danish (K–D) concentrator by attaching a 10-mL concentrator tube to a 500-mL evaporative flask. Other concentration devices or techniques may be used in place of the K–D concentrator if the requirements of Section 8.2 are met.

10.5 Pour the combined extract through a solvent-rinsed drying column containing about 10 cm of anhydrous sodium sulfate, and collect the extract in the K–D concentrator. Rinse the Erlenmeyer flask and column with 20 to 30 mL of methylene chloride to complete the quantitative transfer.

10.6 Add one or two clean boiling chips to the evaporative flask and attach a three-ball Snyder column. Prewet the Snyder column by adding about 1 mL of methylene chloride to the top. Place the K–D apparatus on a hot water bath (60 to 65 °C) so that the concentrator tube is partially immersed in the hot water, and the entire lower rounded surface of the flask is bathed with hot vapor. Adjust the vertical position of the apparatus and the water temperature as required to complete the concentration in 15 to 20 min. At the proper rate of distillation the balls of the column will actively chatter but the chambers will not flood with condensed solvent. When the apparent volume of liquid reaches 1 mL, remove the K–D apparatus and allow it to drain and cool for at least 10 min.

NOTE: Some of the haloethers are very volatile and significant losses will occur in concentration steps if care is not exercised. It is important to maintain a constant gentle evaporation rate and not to allow the liquid volume to fall below 1 to 2 mL before removing the K–D apparatus from the hot water bath.

10.7 Momentarily remove the Snyder column, add 50 mL of hexane and a new boiling chip, and reattach the Snyder column. Raise the temperature of the water bath to 85 to 90 °C. Concentrate the extract as in Section 10.6, except use hexane to prewet the column. The elapsed time of concentration should be 5 to 10 min.

10.8 Remove the Snyder column and rinse the flask and its lower joint into the concentrator tube with 1 to 2 mL of hexane. A 5-mL syringe is recommended for this operation. Stopper the concentrator tube and store refrigerated if further processing will not be performed immediately. If the extract will be stored longer than two days, it should be transferred to a Teflon-sealed screw-cap vial. If the sample extract requires no further cleanup, proceed with gas chromatographic analysis (Section 12). If the sample requires further cleanup, proceed to Section 11.

10.9 Determine the original sample volume by refilling the sample bottle to the mark and transferring the liquid to a 1000-mL graduated cylinder. Record the sample volume to the nearest 5 mL.

11. Cleanup and Separation

11.1 Cleanup procedures may not be necessary for a relatively clean sample matrix. If particular circumstances demand the use of a cleanup procedure, the analyst may use the procedure below or any other appropriate procedure. However, the analyst first must demonstrate that the requirements of Section 8.2 can be met using the method as revised to incorporate the cleanup procedure.

11.2 Florisil column cleanup for haloethers:

11.2.1 Adjust the sample extract volume to 10 mL.

11.2.2 Place a weight of Florisil (nominally 20 g) predetermined by calibration (Section 7.5), into a chromatographic column. Tap the column to settle the Florisil and add 1 to 2 cm of anhydrous sodium sulfate to the top.

11.2.3 Preelute the column with 50 to 60 mL of petroleum ether. Discard the eluate and just prior to exposure of the sodium sulfate layer to the air, quantitatively transfer the sample extract onto the column by decantation and subsequent petroleum ether washings. Discard the eluate. Just prior to exposure of the sodium sulfate layer to the air, begin eluting the column with 300 mL of ethyl ether/petroleum ether (6+94) (V/V). Adjust the elution rate to approximately 5 mL/min and collect the eluate in a 500-mL K–D flask equipped with a 10-mL concentrator tube. This fraction should contain all of the haloethers.

11.2.4 Concentrate the fraction as in Section 10.6, except use hexane to prewet the column. When the apparatus is cool, remove the Snyder column and rinse the flask and its lower joint into the concentrator tube with hexane. Adjust the volume of the cleaned up extract to 10 mL with hexane and analyze by gas chromatography (Section 12).

12. Gas Chromatography

12.1 Table 1 summarizes the recommended operating conditions for the gas chromatograph. Included in this table are retention times and MDL that can be achieved under these conditions. Examples of the separations achieved by Columns 1 and 2 are shown in Figures 1 and 2, respectively. Other packed or capillary (open-tubular) columns, chromatographic conditions, or detectors may be used if the requirements of Section 8.2 are met.

12.2 Calibrate the system daily as described in Section 7.

12.3 If the internal standard calibration procedure is being used, the internal standard must be added to the sample extract and mixed thoroughly immediately before injection into the gas chromatrograph.

12.4 Inject 2 to 5 µL of the sample extract or standard into the gas chromatograph using the solvent-flush technique.[11] Smaller (1.0 µL) volumes may be injected if automatic devices are employed. Record the volume injected to the nearest 0.05 µL, the total extract volume, and the resulting peak size in area or peak height units.

12.5 Identify the parameters in the sample by comparing the retention times of the peaks in the sample chromatogram with those of the peaks in standard chromatograms. The width of the retention time window used to make identifications should be based upon measurements of actual retention time variations of standards over the course of a day. Three times the standard deviation of a retention time for a compound can be used to calculate a suggested window size; however, the experience of the analyst should weight heavily in the interpretation of chromatograms.

12.6 If the response for a peak exceeds the working range of the system, dilute the extract and reanalyze.

12.7 If the measurement of the peak response is prevented by the presence of interferences, further cleanup is required.

13. Calculations

13.1 Determine the concentration of individual compounds in the sample.

13.1.1 If the external standard calibration procedure is used, calculate the amount of material injected from the peak response using the calibration curve or calibration factor determined in Section 7.2.2. The concentration in the sample can be calculated from Equation 2.

$$\text{Concentration } (\mu g/L) = \frac{(A)(V_t)}{(V_i)(V_s)}$$

Equation 2

where:

A=Amount of material injected (ng).

V_i=Volume of extract injected (µL).

V_t=Volume of total extract (µL).

V_s=Volume of water extracted (mL).

13.1.2 If the internal standard calibration procedure is used, calculate the concentration in the sample using the response factor (RF) determined in Section 7.3.2 and Equation 3.

$$\text{Concentration } (\mu g/L) = \frac{(A_s)(I_s)}{(A_{is})(RF)(V_o)}$$

Equation 3

where:

A_s=Response for the parameter to be measured.

A_{is}=Response for the internal standard.

I_s=Amount of internal standard added to each extract (µg).

V_o=Volume of water extracted (L).

13.2 Report results in µg/L without correction for recovery data. All QC data obtained should be reported with the sample results.

14. Method Performance

14.1 The method detection limit (MDL) is defined as the minimum concentration of a substance that can be measured and reported with 99% confidence that the value is above zero.[1] The MDL concentrations listed in Table 1 were obtained using reagent water.[12]

Similar results were achieved using representative wastewaters. The MDL actually achieved in a given analysis will vary depending on instrument sensitivity and matrix effects.

14.2 This method has been tested for linearity of spike recovery from reagent water and has been demonstrated to be applicable over the concentration range from 4 × MDL to 1000 × MDL.[12]

14.3 This method was tested by 20 laboratories using reagent water, drinking water, surface water, and three industrial wastewaters spiked at six concentrations over the range 1.0 to 626 µ/L.[12] Single operator precision, overall precision, and method accuracy were found to be directly related to the concentration of the parameter and essentially independent of the sample matrix. Linear equations to describe these relationships are presented in Table 3.

REFERENCES

1. 40 CFR part 136, appendix B.

2. ''Determination of Haloethers in Industrial and Municipal Wastewaters,'' EPA 600/4–81–062, National Technical Information Service, PB81–232290, Springfield, Virginia 22161, July 1981.

3. ASTM Annual Book of Standards, Part 31, D3694–78. ''Standard Practices for Preparation of Sample Containers and for Preservation of Organic Constitutents,'' American Society for Testing and Materials, Philadelphia.

4. ''Carcinogens—Working Carcinogens, '' Department of Health, Education, and Welfare, Public Health Services, Center for Disease Control, National Institute for Occupational Safety and Health, Publication No. 77–206, August 1977.

5. ''OSHA Safety and Health Standards, General Industry,'' (29 CFR part 1910), Occupational Safety and Health Administration, OSHA 2206 (Revised, January 1976).

6. ''Safety in Academic Chemistry Laboratories,'' American Chemical Society Publication, Committee on Chemical Safety, 3rd Edition, 1979.

7. Mills., P.A. ''Variation of Florisil Activity: Simple Method for Measuring Absorbent Capacity and Its Use in Standardizing Florisil Columns,'' *Journal of the Association of Official Analytical Chemists, 51,* 29 (1968).

8. Provost, L.P., and Elder, R.S. ''Interpretation of Percent Recovery Data,'' *American Laboratory, 15,* 58–63 (1983). (The value 2.44 used in the equation in Section 8.3.3 is two times the value 1.22 derived in this report.)

9. ASTM Annual Book of Standards, Part 31, D3370–76. ''Standard Practices for Sampling Water,'' American Society for Testing and Materials, Philadelphia.

10. ''Methods 330.4 (Titrimetric, DPD–FAS) and 330.5 (Spectrophotometric, DPD) for Chlorine, Total Residual,'' Methods for Chemical Analysis of Water and Wastes, EPA–600/4–79–020, U.S. Environmental Protection Agency, Environmental Monitoring and Support Laboratory, Cincinnati, Ohio 45268, March 1979.

11. Burke, J.A. ''Gas Chromatography for Pesticide Residue Analysis; Some Practical Aspects,'' *Journal of the Association of Official Analytical Chemists, 48,* 1037 (1965).

12. ''EPA Method Study 21, Method 611, Haloethers,'' EPA 600/4–84–052, National Technical Information Service, PB84–205939, Springfield, Virginia 22161, June 1984.

TABLE 1—CHROMATOGRAPHIC CONDITIONS AND METHODS DETECTION LIMITS

Parameters	Retention time (min)		Method detection limit (µ/L)
	Column 1	Column 2	
Bis(2-chloroisopropyl) ether	8.4	9.7	0.8
Bis(2-chloroethyl) ether	9.3	9.1	0.3
Bis(2-chloroethoxy) methane	13.1	10.0	0.5
4-Chlorophenyl ether	19.4	15.0	3.9
4-Bromophenyl phenyl ether	21.2	16.2	2.3

AColumn 1 conditions: Supelcoport (100/120 mesh) coated with 3% SP–1000 packed in a 1.8 m long x 2 mm ID glass column with helium carrier gas at 40 mL/min. flow rate. Column temperature held at 60 °C for 2 min. after injection then programmed at 8 ° C/min. to 230 °C and held for 4 min. Under these conditions the retention time for Aldrin is 22.6 min.

AColumn 2 conditions: Tenax–GC (60/80 mesh) packed in a 1.8 m long x 2mm ID glass column with helium carrier gas at 40 mL/min. flow rate. Column temperature held at 150 °C for 4 min. after injection then programmed at 16 °C/min. to 310 °C. Under these conditions the retention time for Aldrin is 18.4 min.

TABLE 2—QC ACCEPTANCE CRITERIA—METHOD 611

Parameter	Test conc. (µg/L)	Limit for s (µg/L)	Range for $\bar{X}$ (µg/L)	Range for P, P_s percent
Bis (2-chloroethyl)ether	100	26.3	26.3–136.8	11–152
Bis (2-chloroethoxy)methane	100	25.7	27.3–115.0	12–128
Bis (2-chloroisopropyl)ether	100	32.7	26.4–147.0	9–165
4-Bromophenyl phenyl ether	100	39.3	7.6 –167.5	D–189
4-Chlorophenyl phenyl ether	100	30.7	15.4–152.5	D–170

$\bar{s}$=Standard deviation of four recovery measurements, in µg/L (Section 8.2.4).
$\bar{X}$=Average recovery for four recovery measurements, in µg/L (Section 8.2.4).
P, P_s=Percent recovery measured (Section 8.3.2, Section 8.4.2).
D=Detected; result must be greater than zero.

NOTE: These criteria are based directly upon the method performance data in Table 3. Where necessary, the limits for recovery have been broadened to assure applicability of the limits to concentrations below those used to develop Table 3.

TABLE 3—METHOD ACCURACY AND PRECISION AS FUNCTIONS OF CONCENTRATION—METHOD 611

Parameter	Accuracy, as recovery, X′ (μg/L)	Single analyst precision, s_r' (μg/L)	Overall precision, S′ (μg/L)
Bis(2-chloroethyl) ether	0.81C+0.54	0.19$\bar{X}$+0.28	0.35$\bar{X}$+0,36
Bis(2-chloroethoxy) methane	0.71C+0.13	0.20$\bar{X}$+0.15	0.33$\bar{X}$+0.11
Bis(2-chloroisopropyl) ether	0.85C+1.67	0.20$\bar{X}$+1.05	0.36$\bar{X}$+0.79
4–Bromophenyl phenyl ether	0.85C+2.55	0.25$\bar{X}$+0.21	0.47$\bar{X}$+0.37
4–Chlorophenyl phenyl ether	0.82C+1.97	0.18$\bar{X}$+2.13	0.41$\bar{X}$+0.55

X′ = Expected recovery for one or more measuremelts of a sample containing a concentration of C, in μg/L.
s_r' = Expected single analyst standard deviation of measurements at an average concentration found of $\bar{X}$, in μg/L.
S′ = Expected interlaboratory standard deviation of measurements at an average concentration found of $\bar{X}$, in μg/L.
C =True value for the concentration, in μg/L.
$\bar{X}$ = Average recovery found for measurements of samples containing a concentration of C, in μg/L.

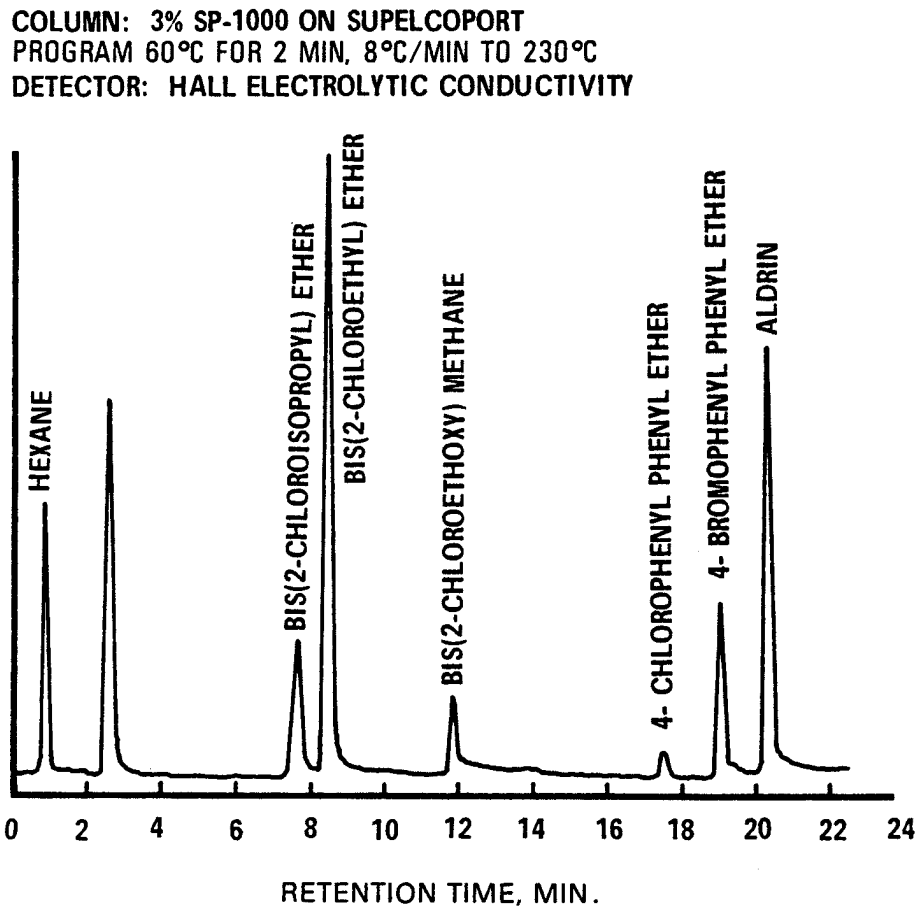


Figure 1. Gas chromatogram of haloethers.

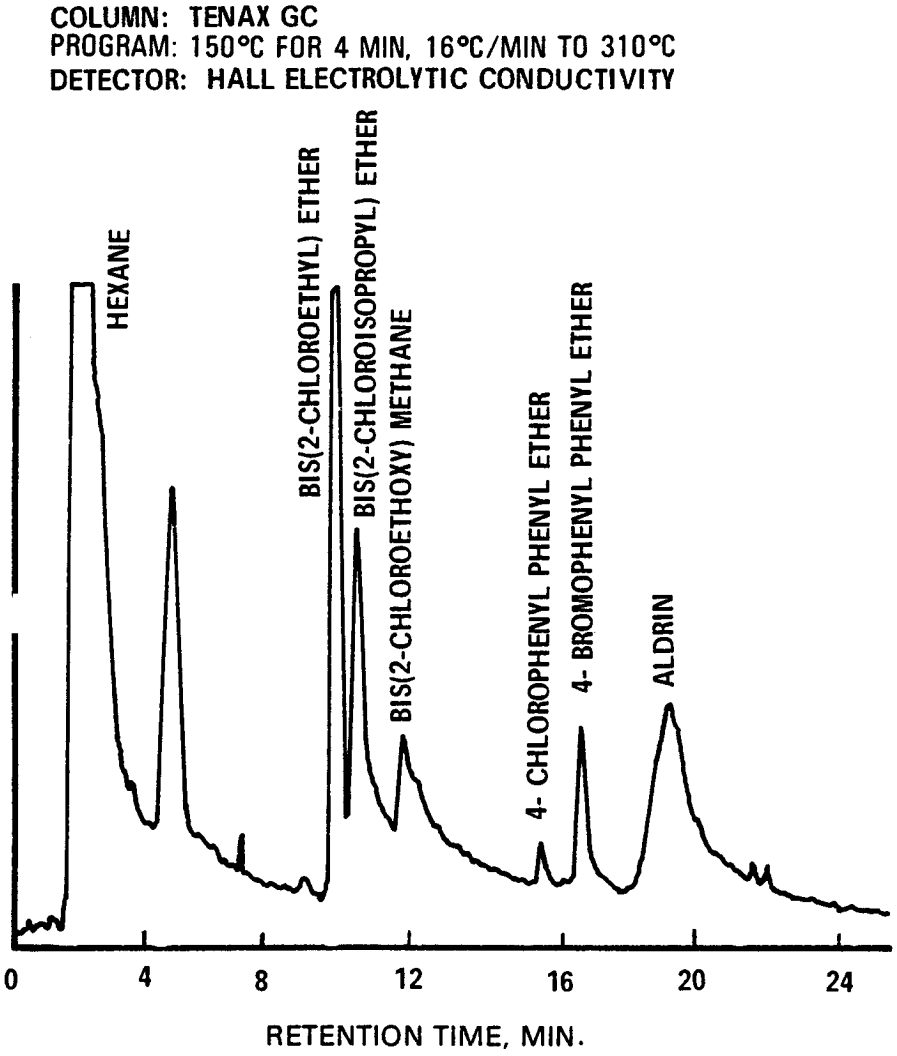


Figure 2. Gas chromatogram of haloethers.

METHOD 612—CHLORINATED HYDROCARBONS

1. Scope and Application

1.1 This method covers the determination of certain chlorinated hydrocarbons. The following parameters can be determined by this method:

Parameter	STORET No.	CAS No.
2-Chloronaphthalene	34581	91–58–7
1,2-Dichlorobenzene	34536	95–50–1
1,3-Dichlorobenzene	34566	541–73–1
1,4-Dichlorobenzene	34571	106–46–7
Hexachlorobenzene	39700	118–74–1
Hexachlorobutadiene	34391	87–68–3
Hexachlorocyclopentadiene	34386	77–47–4
Hexachloroethane	34396	67–72–1
1,2,4-Trichlorobenzene	34551	120–82–1

1.2 This is a gas chromatographic (GC) method applicable to the determination of the compounds listed above in municipal and industrial discharges as provided under 40 CFR 136.1. When this method is used to analyze unfamiliar samples for any or all of the compounds above, compound identifications should be supported by at least one additional qualitative technique. This method describes a second gas chromatographic column that can be used to confirm measurements made with the primary column. Method 625 provides gas chromatograph/mass spectrometer (GC/MS) conditions appropriate for the qualitative and quantitative confirmation of results for all of the parameters listed above, using the extract produced by this method.

1.3 The method detection limit (MDL, defined in Section 14.1)[1] for each parameter is listed in Table 1. The MDL for a specific wastewater may differ from those listed, depending upon the nature of interferences in the sample matrix.

1.4 The sample extraction and concentration steps in this method are essentially the same as in Methods 606, 608, 609, and 611. Thus, a single sample may be extracted to measure the parameters included in the scope of each of these methods. When cleanup is required, the concentration levels must be high enough to permit selecting aliquots, as necessary, to apply appropriate cleanup procedures. The analyst is allowed the latitude, under Section 12, to select chromatographic conditions appropriate for the simultaneous measurement of combinations of these parameters.

1.5 Any modification of this method, beyond those expressly permitted, shall be considered as a major modification subject to application and approval of alternate test procedures under 40 CFR 136.4 and 136.5.

1.6 This method is restricted to use by or under the supervision of analysts experienced in the use of a gas chromatograph and in the interpretation of gas chromatograms. Each analyst must demonstrate the ability to generate acceptable results with this method using the procedure described in Section 8.2.

2. Summary of Method

2.1 A measured volume of sample, approximately 1–L, is extracted with methylene chloride using a separatory funnel. The methylene chloride extract is dried and exchanged to hexane during concentration to a volume of 10 mL or less. The extract is separated by gas chromatography and the parameters are then measured with an electron capture detector.[2]

2.2 The method provides a Florisil column cleanup procedure to aid in the elimination of interferences that may be encountered.

3. Interferences

3.1 Method interferences may be caused by contaminants in solvents, reagents, glassware, and other sample processing hardware that lead to discrete artifacts and/or elevated baselines in gas chromatograms. All of these materials must be routinely demonstrated to be free from interferences under the conditions of the analysis by running laboratory reagent blanks as described in Section 8.1.3.

3.1.1 Glassware must be scrupulously cleaned.[3] Clean all glassware as soon as possible after use by rinsing with the last solvent used in it. Solvent rinsing should be followed by detergent washing with hot water, and rinses with tap water and distilled water. The glassware should then be drained dry, and heated in a muffle furnace at 400 °C for 15 to 30 min. Some thermally stable materials, such as PCBs, may not be eliminated by this treatment. Solvent rinses with acetone and pesticide quality hexane may be substituted for the muffle furnace heating. Thorough rinsing with such solvents usually eliminates PCB interference. Volumetric ware should not be heated in a muffle furnace. After drying and cooling, glassware should be sealed and stored in a clean environment to prevent any accumulation of dust or other contaminants. Store inverted or capped with aluminum foil.

3.1.2 The use of high purity reagents and solvents helps to minimize interference problems. Purification of solvents by distillation in all-glass systems may be required.

3.2 Matrix interferences may be caused by contaminants that are co-extracted from the sample. The extent of matrix interferences will vary considerably from source to source, depending upon the nature and diversity of the industrial complex or municipality being sampled. The cleanup procedure in Section 11 can be used to overcome many of these interferences, but unique samples may require additional cleanup approaches to achieve the MDL listed in Table 1.

4. Safety

4.1 The toxicity or carcinogenicity of each reagent used in this method has not been precisely defined; however, each chemical compound should be treated as a potential health hazard. From this viewpoint, exposure to these chemicals must be reduced to the lowest possible level by whatever means available. The laboratory is responsible for maintaining a current awareness file of OSHA regulations regarding the safe handling of the chemicals specified in this method. A reference file of material data handling sheets should also be made available to all personnel involved in the chemical analysis. Additional references to laboratory safety are available and have been identified[4–6] for the information of the analyst.

5. Apparatus and Materials

5.1 Sampling equipment, for discrete or composite sampling.

5.1.1 Grab sample bottle—1cL or 1-qt, amber glass, fitted with a screw cap lined with Teflon. Foil may be substituted for Teflon if the sample is not corrosive. If amber bottles are not available, protect samples from light. The bottle and cap liner must be washed, rinsed with acetone or methylene chloride, and dried before use to minimize contamination.

5.1.2 Automatic sampler (optional)—The sampler must incorporate glass sample containers for the collection of a minimum of 250 mL of sample. Sample containers must be kept refrigerated at 4 °C and protected from light during compositing. If the sampler uses a peristaltic pump, a minimum length of compressible silicone rubber tubing may be used. Before use, however, the compressible tubing should be thoroughly rinsed with methanol, followed by repeated rinsings with distilled water to minimize the potential for contamination of the sample. An integrating flow meter is required to collect flow proportional composites.

5.2 Glassware (All specifications are suggested. Catalog numbers are included for illustration only.):

5.2.1 Separatory funnel—2–L, with Teflon stopcock.

5.2.2 Drying column—Chromatographic column, approximately 400 mm long x 19 mm ID, with coarse frit filter disc.

5.2.3 Chromatographic column—300 long x 10 mm ID, with Teflon stopcock and coarse frit filter disc at bottom.

5.2.4 Concentrator tube, Kuderna-Danish—10-mL, graduated (Kontes K–570050–1025 or equivalent). Calibration must be checked at the volumes employed in the test. Ground glass stopper is used to prevent evaporation of extracts.

5.2.5 Evaporative flask, Kuderna-Danish—500-mL (Kontes K–570001–0500 or equivalent). Attach to concentrator tube with springs.

5.2.6 Snyder column, Kuderna-Danish—Three-ball macro (Kontes K–503000–0121 or equivalent).

5.2.7 Vials—10 to 15-mL, amber glass, with Teflon-lined screw cap.

5.3 Boiling chips—Approximately 10/40 mesh. Heat to 400 °C for 30 min or Soxhlet extract with methylene chloride.

5.4 Water bath—Heated, with concentric ring cover, capable of temperature control (± 2 °C). The bath should be used in a hood.

5.5 Balance—Analytical, capable of accurately weighing 0.0001 g.

5.6 Gas chromatograph—An analytical system complete with gas chromatograph suitable for on-column injection and all required accessories including syringes, analytical columns, gases, detector, and strip-chart recorder. A data system is recommended for measuring peak areas.

5.6.1 Column 1—1.8 m long x 2 mm ID glass, packed with 1% SP–1000 on Supelcoport (100/120 mesh) or equivalent. Guidelines for the use of alternate column packings are provide in Section 12.1.

5.6.2 Column 2—1.8 m long × 2 mm ID glass, packed with 1.5% OV–1/2.4% OV–225 on Supelcoport (80/100 mesh) or equivalent. This column was used to develop the method performance statements in Section 14.

5.6.3 Detector— Electron capture detector. This detector has proven effective in the analysis of wastewaters for the parameters listed in the scope (Section 1.1), and was used to develop the method performance statements in Section 14. Guidelines for the use of alternate detectors are provided in Section 12.1.

6. Reagents

6.1 Reagent water— Reagent water is defined as a water in which an interferent is not observed at the MDL of the parameters of interest.

6.2 Acetone, hexane, isooctane, methanol, methylene chloride, petroleum ether (boiling range 30 to 60 °C)—Pesticide quality or equivalent.

6.3 Sodium sulfate—(ACS) Granular, anhydrous. Purify heating at 400 °C for 4 h in a shallow tray.

6.4 Florisil—PR grade (60/100 mesh). Purchase activated at 1250 °F and store in the dark in glass containers with ground glass stoppers or foil-lined screw caps. Before use, activate each batch at least 16 h at 130 ° C in a foil-covered glass container and allow to cool.

6.5 Stock standard solution (1.00 µg/µL)—Stock standard solutions can be prepared from pure standard materials or purchased as certified solutions.

6.5.1 Prepare stock standard solutions by accurately weighing about 0.0100 g of pure material. Dissolve the material in isooctane and dilute to volume in a 120-mL volumetric

flask. Larger volumes can be used at the convenience of the analyst. When compound purity is assayed to be 96% or greater, the weight can be used without correction to calculate the concentration of the stock standard. Commercially prepared stock standards can be used at any concentration if they are certified by the manufacturer or by an independent source.

6.5.2 Transfer the stock standard solutions into Teflon-sealed screw-cap bottles. Store at 4 °C and protect from light. Stock standard solutions should be checked frequently for signs of degradation or evaporation, especially just prior to preparing calibration standards from them.

6.5.3 Stock standard solutions must be replaced after six months, or sooner if comparision with check standards indicates a problem.

6.6 Quality control check sample concentrate—See Section 8.2.1.

7. Calibration

7.1 Establish gas chromatographic operating conditions equivalent to those given in Table 1. The gas chromatographic system can be calibrated using the external standard technique (Section 7.2) or the internal standard technique (Section 7.3).

7.2 External standard calibration procedure:

7.2.1 Prepare calibration standards at a minimum of three concentration levels for each parameter of interest by adding volumes of one or more stock standards to a volumetric flask and diluting to volume with isooctane. One of the external standards should be at a concentration near, but above, the MDL (Table 1) and the other concentrations should correspond to the expected range of concentrations found in real samples or should define the working range of the detector.

7.2.2 Using injections of 2 to 5 µL, analyze each calibration standard according to Section 12 and tabulate peak height or area responses against the mass injected. The results can be used to prepare a calibration curve for each compound. Alternatively, if the ratio of response to amount injected (calibration factor) is a constant over the working range (<10% relative standard deviation, RSD), linearity through the origin can be assumed and the average ratio or calibration factor can be used in place of a calibration curve.

7.3 Internal standard calibration procedure—To use this approach, the analyst must select one or more internal standards that are similar in analytical behavior to the compounds of interest. The analyst must further demonstrate that the measurement of the internal standard is not affected by method or matrix interferences. Because of these limitations, no internal standard can be suggested that is applicable to all samples.

7.3.1 Prepare calibration standards at a minimum of three concentration levels for each parameter of interest by adding volumes of one or more stock standards to a volumetric flask. To each calibration standard, add a known constant amount of one or more internal standards, and dilute to volume with isooctane. One of the standards should be at a concentration near, but above, the MDL and the other concentrations should correspond to the expected range of concentrations found in real samples or should define the working range of the detector.

7.3.2 Using injections of 2 to 5 µL, analyze each calibration standard according to Section 12 and tabulate peak height or area responses against concentration for each compound and internal standard. Calculate response factors (RF) for each compound using Equation 1.

$$RF = \frac{(A_s)(C_{is})}{(A_{is})(C_s)}$$

Equation 1

where:

A_s=Response for the parameter to be measured.

A_{is}=Response for the internal standard.

C_{is}=Concentration of the internal standard (µg/L).

C_s=Concentration of the parameter to be measured (µg/L).

If the RF value over the working range is a constant (<10% RSD), the RF can be assumed to be invariant and the average RF can be used for calculations. Alternatively, the results can be used to plot a calibration curve of response ratios, A_s/A_{is}, vs. RF.

7.4 The working calibration curve, calibration factor, or RF must be verified on each working day by the measurement of one or more calibration standards. If the response for any parameter varies from the predicted response by more than ±15%, a new calibration curve must be prepared for that compound.

7.5 Before using any cleanup procedure, the analyst must process a series of calibration standards through the procedure to validate elution patterns and the absence of interferences from the reagents.

8. Quality Control

8.1 Each laboratory that uses this method is required to operate a formal quality control program. The minimum requirements of this program consist of an initial demonstration of laboratory capability and an ongoing analysis of spiked samples to evaluate and document data quality. The laboratory must maintain records to document the quality of

data that is generated. Ongoing data quality checks are compared with established performance criteria to determine if the results of analyses meet the performance characteristics of the method. When the results of sample spikes indicate atypical method performance, a quality control check standard must be analyzed to confirm that the measurements were performed in an in-control mode of operation.

8.1.1 The analyst must make an initial, one-time, demonstration of the ability to generate acceptable accuracy and precision with this method. This ability is established as described in Section 8.2.

8.1.2 In recognition of advances that are occurring in chromatography, the analyst is permitted certain options (detailed in Sections 10.4, 11.1, and 12.1) to improve the separations or lower the cost of measurements. Each time such modification is made to the method, the analyst is required to repeat the procedure in Section 8.2.

8.1.3 Before processing any samples, the analyst must analyze a reagent water blank to demonstrate that interferences from the analytical system and glassware are under control. Each time a set of samples is extracted or reagents are changed, a reagent water blank must be processed as a safeguard against laboratory contamination.

8.1.4 The laboratory must, on an ongoing basis, spike and analyze a minimum of 10% of all samples to monitor and evaluate laboratory data quality. This procedure is described in Section 8.3.

8.1.5 The laboratory must, on an ongoing basis, demonstrate through the analyses of quality control check standards that the operation of the measurement system is in control. This procedure is described in Section 8.4. The frequency of the check standard analyses is equivalent to 10% of all samples analyzed but may be reduced if spike recoveries from samples (Section 8.3) meet all specified quality control criteria.

8.1.6 The laboratory must maintain performance records to document the quality of data that is generated. This procedure is described in Section 8.5.

8.2 To establish the ability to generate acceptable accuracy and precision, the analyst must perform the following operations.

8.2.1 A quality control (QC) check sample concentrate is required containing each parameter of interest at the following concentrations in acetone: Hexachloro-substituted parameters, 10 µg/mL; any other chlorinated hydrocarbon, 100 µg/mL. The QC check sample concentrate must be obtained from the U.S. Environmental Protection Agency, Environmental Monitoring and Support Laboratory in Cincinnati, Ohio, if available. If not available from that source, the QC check sample concentrate must be obtained from another external source. If not available from either source above, the QC check sample concentrate must be prepared by the laboratory using stock standards prepared independently from those used for calibration.

8.2.2 Using a pipet, prepare QC check samples at the test concentrations shown in Table 2 by adding 1.00 mL of QC check sample concentrate to each of four 1–L aliquots of reagent water.

8.2.3 Analyze the well-mixed QC check samples according to the method beginning in Section 10.

8.2.4 Calculate the average recovery ($\bar{X}$) in µg/L, and the standard deviation of the recovery (s) in µg/L, for each parameter using the four results.

8.2.5 For each parameter compare s and $\bar{X}$ with the corresponding acceptance criteria for precision and accuracy, respectively, found in Table 2. If s and $\bar{X}$ for all parameters of interest meet the acceptance criteria, the system performance is acceptable and analysis of actual samples can begin. If any individual s exceeds the precision limit or any individual $\bar{X}$ falls outside the range for accuracy, the system performance is unacceptable for that parameter.

NOTE: The large number of parameters in Table 2 presents a substantial probability that one or more will fail at least one of the acceptance criteria when all parameters are analyzed.

8.2.6 When one or more of the parameters tested fail at least one of the acceptance criteria, the analyst must proceed according to Section 8.2.6.1 or 8.2.6.2.

8.2.6.1 Locate and correct the source of the problem and repeat the test for all parameters of interest beginning with Section 8.2.2.

8.2.6.2 Beginning with Section 8.2.2, repeat the test only for those parameters that failed to meet criteria. Repeated failure, however, will confirm a general problem with the measurement system. If this occurs, locate and correct the source of the problem and repeat the test for all compounds of interest beginning with Section 8.2.2.

8.3 The laboratory must, on an ongoing basis, spike at least 10% of the samples from each sample site being monitored to assess accuracy. For laboratories analyzing one to ten samples per month, at least one spike sample per month is required.

8.3.1 The concentration of the spike in the sample should be determined as follows:

8.3.1.1 If, as in compliance monitoring, the concentration of a specific parameter in the sample is being checked against a regulatory concentration limit, the spike should be at that limit or 1 to 5 times higher than the background concentration determined in Section 8.3.2, whichever concentration would be larger.

8.3.1.2 If the concentration of a specific parameter in the sample is not being

checked against a limit specific to that parameter, the spike should be at the test concentration in Section 8.2.2 or 1 to 5 times higher than the background concentration determined in Section 8.3.2, whichever concentration would be larger.

8.3.1.3 If it is impractical to determine background levels before spiking (e.g., maximum holding times will be exceeded), the spike concentration should be (1) the regulatory concentration limit, if any; or, if none by (2) the larger of either 5 times higher than the expected background concentration or the test concentration in Section 8.2.2.

8.3.2 Analyze one sample aliquot to determine the background concentration (B) of each parameter. In necessary, prepare a new QC check sample concentrate (Section 8.2.1) appropriate for the background concentrations in the sample. Spike a second sample aliquot with 1.0 mL of the QC check sample concentrate and analyze it to determine the concentration after spiking (A) of each parameter. Calculate each percent recovery (P) as 100 (A–B)%/T, where T is the known true value of the spike.

8.3.3 Compare the percent recovery (P) for each parameter with the corresponding QC acceptance criteria found in Table 2. These acceptance criteria were calculated to include an allowance for error in measurement of both the background and spike concentrations, assuming a spike to background ratio of 5:1. This error will be accounted for to the extent that the analyst's spike to background ratio approaches 5:1.[7] If spiking was performed at a concentration lower than the test concentration in Section 8.2.2, the analyst must use either the QC acceptance criteria in Table 2, or optional QC acceptance criteria calculated for the specific spike concentration. To calculate optional acceptance criteria for the recovery of a parameter: (1) Calculate accuracy (X′) using the equation in Table 3, substituting the spike concentration (T) for C; (2) calculate overall precision (S′) using the equation in Table 3, substituting X′ for X; (3) calculate the range for recovery at the spike concentration as (100 X′/T) ± 2.44 (100 S′/T)%.[7]

8.3.4 If any individual P falls outside the designated range for recovery, that parameter has failed the acceptance criteria. A check standard containing each parameter that failed the criteria must be analyzed as described in Section 8.4.

8.4. If any parameter fails the acceptance criteria for recovery in Section 8.3, a QC check standard containing each parameter that failed must be prepared and analyzed.

NOTE: The frequency for the required analysis of a QC check standard will depend upon the number of parameters being simultaneously tested, the complexity of the sample matrix, and the performance of the laboratory.

8.4.1 Prepare the QC check standard by adding 1.0 mL of QC check sample concentrate (Sections 8.2.1 or 8.3.2) to 1 L of reagent water. The QC check standard needs only to contain the parameters that failed criteria in the test in Section 8.3.

8.4.2 Analyze the QC check standard to determine the concentration measured (A) of each parameter. Calculate each percent recovery (P_s) as 100 (A/T)%, where T is the true value of the standard concentration.

8.4.3 Compare the percent recovery (P_s) for each parameter with the corresponding QC acceptance criteria found in Table 2. Only parameters that failed the test in Section 8.3 need to be compared with these criteria. If the recovery of any such parameter falls outside the designated range, the laboratory performance for that parameter is judged to be out of control, and the problem must be immediately identified and corrected. The analytical result for that parameter in the unspiked sample is suspect and may not be reported for regulatory compliance purposes.

8.5 As part of the QC program for the laboratory, method accuracy for wastewater samples must be assessed and records must be maintained. After the analysis of five spiked wastewater samples as in Section 8.3, calculate the average percent recovery (P) and the standard deviation of the percent recovery (s_p). Express the accuracy assessment as a percent recovery interval from $P-2s_p$ to $P+2s_p$. If P=90% and s_p=10%, for example, the accuracy interval is expressed as 70–110%. Update the accuracy assessment for each parameter on a regular basis (e.g. after each five to ten new accuracy measurements).

8.6 It is recommended that the laboratory adopt additional quality assurance practices for use with this method. The specific practices that are most productive depend upon the needs of the laboratory and the nature of the samples. Field duplicates may be analyzed to assess the precision of the environmental measurements. When doubt exists over the identification of a peak on the chromatogram, confirmatory techniques such as gas chromatography with a dissimilar column, specific element detector, or mass spectrometer must be used. Whenever possible, the laboratory should analyze standard reference materials and participate in relevent performance evaluation studies.

9. Sample Collection, Preservation, and Handling

9.1 Grab samples must be collected in glass containers. Conventional sampling practices[8] should be followed, except that the bottle must not be prerinsed with sample before collection. Composite samples should be collected in refrigerated glass containers in accordance with the requirements of the program. Automatic sampling equipment must be as free as possible of Tygon tubing

and other potential sources of contamination.

9.2 All samples must be iced or refrigerated at 4°C from the time of collection until extraction.

9.3 All samples must be extracted within 7 days of collection and completely analyzed within 40 days of extraction.[2]

10. Sample Extraction

10.1 Mark the water meniscus on the side of the sample bottle for later determination of sample volume. Pour the entire sample into a 2–L separatory funnel.

10.2 Add 60 mL of methylele chloride to the sample bottle, seal, and shake 30 s to rinse the inner surface. Transfer the solvent to the separatory funnel and extract the sample by shaking the funnel for 2 min with periodic venting to release excess pressure. Allow the organic layer to separate from the water phase for a minimum of 10 min. If the emulsion interface between layers is more than one-third the volume of the solvent layer, the analyst must employ mechanical techniques to complete the phase separation. The optimum technique depends upon the sample, but may include stirring, filtration of the emulsion through glass wool, centrifugation, or other physical methods. Collect the methylene chloride extract in a 250-mL Erlenmeyer flask.

10.3 Add a second 60-mL volume of methylene chloride to the sample bottle and repeat the extraction procedure a second time, combining the extracts in the Erlenmeyer flask. Perform a third extraction in the same manner.

10.4 Assemble a Kuderna-Danish (K–D) concentrator by attaching a 10-mL concentrator tube to a 500-mL evaporative flask. Other concentration devices or techniques may be used in place of the K–D concentrator if the requirements of Section 8.2 are met.

10.5 Pour the combined extract through a solvent-rinsed drying column containing about 10 cm of anhydrous sodium sulfate, and collect the extract in the K–D concentrator. Rinse the Erlenmeyer flask and column with 20 to 30 mL of methylene chloride to complete the quantitative transfer.

10.6 Add one or two clean boiling chips to the evaporative flask and attach a three-ball Snyder column. Prewet the Snyder column by adding about 1 mL of methylene chloride to the top. Place the K–D apparatus on a hot water bath (60 to 65 °C) so that the concentrator tube is partially immersed in the hot water, and the entire lower rounded surface of the flask is bathed with hot vapor. Adjust the vertical position of the apparatus and the water temperature as required to complete the concentration in 15 to 20 min. At the proper rate of distillation the balls of the column will actively chatter but the chambers will not flood with condensed solvent. When the apparent volume of liquid reaches 1 to 2 mL, remove the K–D apparatus and allow it to drain and cool for at least 10 min.

NOTE: The dichloribenzenes have a sufficiently high volatility that significant losses may occur in concentration steps if care is not exercised. It is important to maintain a constant gentle evaporation rate and not to allow the liquid volume to fall below 1 to 2 mL before removing the K–D apparatus from the hot water bath.

10.7 Momentarily remove the Snyder column, add 50 mL of hexane and a new boiling chip, and reattach the Snyder column. Raise the tempeature of the water bath to 85 to 90 °C. Concentrate the extract as in Section 10.6, except use hexane to prewet the column. The elapsed time of concentration should be 5 to 10 min.

10.8 Romove the Snyder column and rinse the flask and its lower joint into the concentrator tube with 1 to 2 mL of hexane. A 5-mL syringe is recommended for this operation. Stopper the concentrator tube and store refrigerated if further processing will not be performed immediately. If the extract will be stored longer than two days, it should be transferred to a Teflon-sealed screw-cap vial. If the sample extract requires no further cleanup, proceed with gas chromatographic analysis (Section 12). If the sample requires further cleanup, proceed to Section 11.

10.9 Determine the original sample volume by refilling the sample bottle to the mark and transferring the liquid to a 1000-mL graduated cylinder. Record the sample volume to the nearest 5 mL.

11. Cleanup and Separation

11.1 Cleanup procedures may not be necessary for a relatively clean sample matrix. If particular circumstances demand the use of a cleanup procedure, the analyst may use the procedure below or any other appropriate procedure. However, the analyst first must demonstrate that the requirements of Section 8.2 can be met using the method as revised to incorporate the cleanup procedure.

11.2 Florisil column cleanup for chlorinated hydrocarbons:

11.2.1 Adjust the sample extract to 10 mL with hexane.

11.2.2 Place 12 g of Florisil into a chromatographic column. Tap the column to settle the Florisil and add 1 to 2 cm of anhydrous sodium sulfate to the top.

11.2.3 Preelute the column with 100 mL of petroleum ether. Discard the eluate and just prior to exposure of the sodium sulfate layer to the air, quantitatively transfer the sample extract onto the column by decantation and subsequent petroleum ether washings. Discard the eluate. Just prior to exposure of the sodium sulfate layer to the air, begin

eluting the column with 200 mL of petroleum ether and collect the eluate in a 500-mL K–D flask equipped with a 10-mL concentrator tube. This fraction should contain all of the chlorinated hydrocarbons.

11.2.4 Concentrate the fraction as in Section 10.6, except use hexane to prewet the column. When the apparatus is cool, remove the Snyder column and rinse the flask and its lower joint into the concentrator tube with hexane. Analyze by gas chromatography (Section 12).

12. Gas Chromatography

12.1 Table 1 summarizes the recommended operating conditions for the gas chromatograph. Included in this table are retention times and MDL that can be achieved under these conditions. Examples of the separations achieved by Columl 2 are shown in Figures 1 and 2. Other packed or capillary (open-tubular) columns, chromatographic conditions, or detectors may be used if the requirements of Section 8.2 are met.

12.2 Calibrate the system daily as described in Section 7.

12.3 If the internal standard calibration procedure is being used, the internal standard must be added to the sample extract and mixed throughly immediately before injection into the gas chromatograph.

12.4 Inject 2 to 5 µL of the sample extract or standard into the gas chromatograph using the solvent-flush techlique.[9] Smaller (1.0 µL) volumes may be injected if automatic devices are employed. Record the volume injected to the nearest 0.05 µL, the total extract volume, and the resulting peak size in area or peak height units.

12.5 Identify the parameters in the sample by comparing the retention times of the peaks in the sample chromatogram with those of the peaks in standard chromatograms. The width of the retention time window used to make identifications should be based upon measurements of actual retention time variations of standards over the course of a day. Three times the standard deviation of a retention time for a compound can be used to calculate a suggested window size; however, the experience of the analyst should weigh heavily in the interpretation of chromatograms.

12.6 If the response for a peak exceeds the working range of the system, dilute the extract and reanalyze.

12.7 If the measurement of the peak response is prevented by the presence of interferences, further cleanup is required.

13. Calculations

13.1 Determine the concentration of individual compounds in the sample.

13.1.1 If the external standard calibration procedure is used, calculate the amount of material injected from the peak response using the calibration curve or calibration factor determined in Section 7.2.2. The concentration in the sample can be calculated from Equation 2.

$$\text{Concentration } (\mu g/L) = \frac{(A)(V_t)}{(V_i)(V_s)}$$

Equation 2

where:

A=Amount of material injected (ng).
V_i=Volume of extract injected (µL).
V_t=Volume of total extract (µL).
V_s=Volume of water extracted (mL).

13.1.2 If the internal standard calibration procedure is used, calculate the concentration in the sample using the response factor (RF) determined in Section 7.3.2 and Equation 3.

$$\text{Concentration } (\mu g/L) = \frac{(A_s)(I_s)}{(A_{is})(RF)(V_o)}$$

Equation 3

where:

A_s=Response for the parameter to be measured.
A_{is}=Response for the internal standard.
I_s=Amount of internal standard added to each extract (µg).
V_o=Volume of water extracted (L).

13.2 Report results in µg/L without correction for recovery data. All QC data obtained should be reported with the sample results.

14. Method Performance

14.1 The method detection limit (MDL) is defined as the minimum concentration of a substance that can be measured and reported with 99% confidence that the value is above zero.[1] The MDL concentrations listed in Table 1 were obtained using reagent water.[10] Similar results were achieved using representative wastewaters. The MDL actually achieved in a given analysis will vary depending on instrument sensitivity and matrix effects.

14.2 This method has been tested for linearity of spike recovery from reagent water and has been demonstrated to be applicable over the concentration range from 4×MDL to 1000×MDL.[10]

14.3 This method was tested by 20 laboratories using reagent water, drinking water, surface water, and three industrial wastewaters spiked at six concentrations over the range 1.0 to 356 µg/L.[11] Single operator precision, overall precision, and method accuracy were found to be directly related to the concentration of the parameter and essentially independent of the sample matrix. Linear equations to describe these relationships are presented in Table 3.

REFERENCES

1. 40 CFR part 136, appendix B.

2. ''Determination of Chlorinated Hydrocarbons In Industrial and Municipal Wastewaters, ''EPA 6090/4–84–ABC, National Technical Information Service, PBXYZ, Springfield, Virginia, 22161 November 1984.

3. ASTM Annual Book of Standards, Part 31, D3694–78. ''Standard Practices for Preparation of Sample Containers and for Preservation of Organic Constituents,'' American Society for Testing and Materials, Philadelphia.

4. ''Carcinogens—Working With Carcinogens,'' Department of Health, Education, and Welfare, Public Health Service, Center for Disease Control, National Institute for Occupational Safety and Health, Publication No. 77–206, August 1977.

5. ''OSHA Safety and Health Standards, General Industry,'' (29 CFR part 1910), Occupational Safety and Health Administration, OSHA 2206 (Revised, January 1976).

6. ''Safety in Academic Chemistry Laboratories,'' American Chemical Society Publication, Committee on Chemical Safety, 3rd Edition, 1979.

7. Provost, L.P., and Elder, R.S. ''Interpretation of Percent Recovery Data,''*American Laboratory*, *15*, 58–63 (1983). (The value 2.44 used in the equation in Section 8.3.3 is two times the value 1.22 derived in this report.)

8. ASTM Annual Book of Standards, Part 31, D3370–76. ''Standard Practices for Sampling Water,'' American Society for Testing and Materials, Philadelphia.

9. Burke, J.A. ''Gas Chromatography for Pesticide Residue Analysis; Some Practical Aspects,'' *Journal of the Association of Official Analytical Chemists, 48,* 1037 (1965).

10. ''Development of Detection Limits, EPA Method 612, Chlorinated Hydrocarbons,'' Special letter report for EPA Contract 68–03–2625, U.S. Environmental Protection Agency, Environmental Monitoring and Support Laboratory, Cincinnati, Ohio 45268.

11. ''EPA Method Study Method 612—Chlorinated Hydrocarbons,'' EPA 600/4–84–039, National Technical Information Service, PB84–187772, Springfield, Virginia 22161, May 1984.

12. ''Method Performance for Hexachlorocyclopentadiene by Method 612,'' Memorandum from R. Slater, U.S. Environmental Protection Agency, Environmental Monitoring and Support Laboratory, Cincinnati, Ohio 45268, December 7, 1983.

TABLE 1—CHROMATOGRAPHIC CONDITIONS AND METHOD DETECTION LIMITS

Parameter	Retention time (min)		Method detection limit (µg/L)
	Column 1	Column 2	
1,3-Dichlorobenzene	4.5	6.8	1.19
Hexachloroethane	4.9	8.3	0.03
1,4-Dichlorobenzene	5.2	7.6	1.34
1,2-Dichlorobenzene	6.6	9.3	1.14
Hexachlorobutadiene	7.7	20.0	0.34
1,2,4-Trichlorobenzene	15.5	22.3	0.05
Hexachlorocyclopentadiene	nd	[c] 16.5	0.40
2-Chloronaphthalene	[a] 2.7	[b] 3.6	0.94
Hexachlorobenzene	[a] 5.6	[b] 10.1	0.05

Column 1 conditions: Supelcoport (100/120 mesh) coated with 1% SP–1000 packed in a 1.8 m x 2 mm ID glass column with 5% methane/95% argon carrier gas at 25 mL/min. flow rate. Column temperature held isothermal at 65 °C, except where otherwise indicated.

Column 2 conditions: Supelcoport (80/100 mesh) coated with 1.5% OV–1/2.4% OV–225 packed in a 1.8 m x 2 mm ID glass column with 5% methane/95% argon carrier gas at 25 mL/min. flow rate. Column temperature held isothermal at 75 °C, except where otherwise indicated.

nd=Not determined.
[a] 150 °C column temperature.
[b] 165 °C column temperature.
[c] 100 °C column temperature.

TABLE 2—QC ACCEPTANCE CRITERIA—METHOD 612

Parameter	Test conc. (µg/L)	Limit for s (µg/L)	Range for $\bar{X}$ (µg/L)	Range for P, P_s (percent)
2-Chloronaphthalene	100	37.3	29.5–126.9	9–148
1,2-Dichlorobenzene	100	28.3	23.5–145.1	9–160
1,3-Dichlorobenzene	100	26.4	7.2–138.6	D–150
1,4-Dichlorobenzene	100	20.8	22.7–126.9	13–137
Hexachlorobenzene	10	2.4	2.6–14.8	15–159
Hexachlorobutadiene	10	2.2	D–12.7	D–139
Hexachlorocyclopentadiene	10	2.5	D–10.4	D–111
Hexachloroethane	10	3.3	2.4–12.3	8–139

TABLE 2—QC ACCEPTANCE CRITERIA—METHOD 612—Continued

Parameter	Test conc. (μg/L)	Limit for s (μg/L)	Range for $\bar{X}$ (μg/L)	Range for P, P_s (percent)
1,2,4-Trichlorobenzene	100	31.6	20.2–133.7	5–149

s=Standard deviation of four recovery measurements, in μg/L (Section 8.2.4).
X=Average recovery for four recovery measurements, in μg/L (Section 8.2.4).
P, P_s=Percent recovery measured (Section 8.3.2, Section 8.4.2).
D=Detected; result must be greater than zero.

NOTE: These criteria are based directly upon the method performance data in Table 3. Where necessary, the limits for recovery have been broadened to assure applicability of the limits to concentrations below those used to develop Table 3.

TABLE 3—METHOD ACCURACY AND PRECISION AS FUNCTIONS OF CONCENTRATION—METHOD 612

Parameter	Acccuracy, as recovery, X′ (μg/L)	Single analyst precision, s_r' (μg/L)	Overall precision, S′ (μg/L)
2-Chloronaphthalene	0.75C+3.21	$0.28\bar{X}-1.17$	$0.38\bar{X}-1.39$
1,2-Dichlorobenzene	0.85C−0.70	$0.22\bar{X}-2.95$	$0.41\bar{X}-3.92$
1,3-Dichlorobenzene	0.72C+0.87	$0.21\bar{X}-1.03$	$0.49\bar{X}-3.98$
1,4-Dichlorobenzene	0.72C+2.80	$0.16\bar{X}-0.48$	$0.35\bar{X}-0.57$
Hexachlorobenzene	0.87C−0.02	$0.14\bar{X}+0.07$	$0.36\bar{X}-0.19$
Hexachlorobutadiene	0.61C+0.03	$0.18\bar{X}+0.08$	$0.53\bar{X}-0.12$
Hexachlorocyclopentadiene[a]	0.47C	$0.24\bar{X}$	$0.50\bar{X}$
Hexachloroethane	0.74C−0.02	$0.23\bar{X}+0.07$	$0.36\bar{X}-0.00$
1,2,4-Trichlorobenzene	0.76C+0.98	$0.23\bar{X}-0.44$	$0.40\bar{X}-1.37$

X′=Expected recovery for one or more measurements of a sample containing a concentration of C, in μg/L.
s_r'=Expected single analyst standard deviation of measurements at an average concentration found of $\bar{X}$, in μg/L.
S′=Expected interlaboratory standard deviation of measurements at an average concentration found of $\bar{X}$, in μg/L.
C=True value for the concentration, in μg/L.
$\bar{X}$=Average recovery found for measurements of samples containing a concentration of C, in μg/L.

[a] Estimates based upon the performance in a single laboratory.[12]

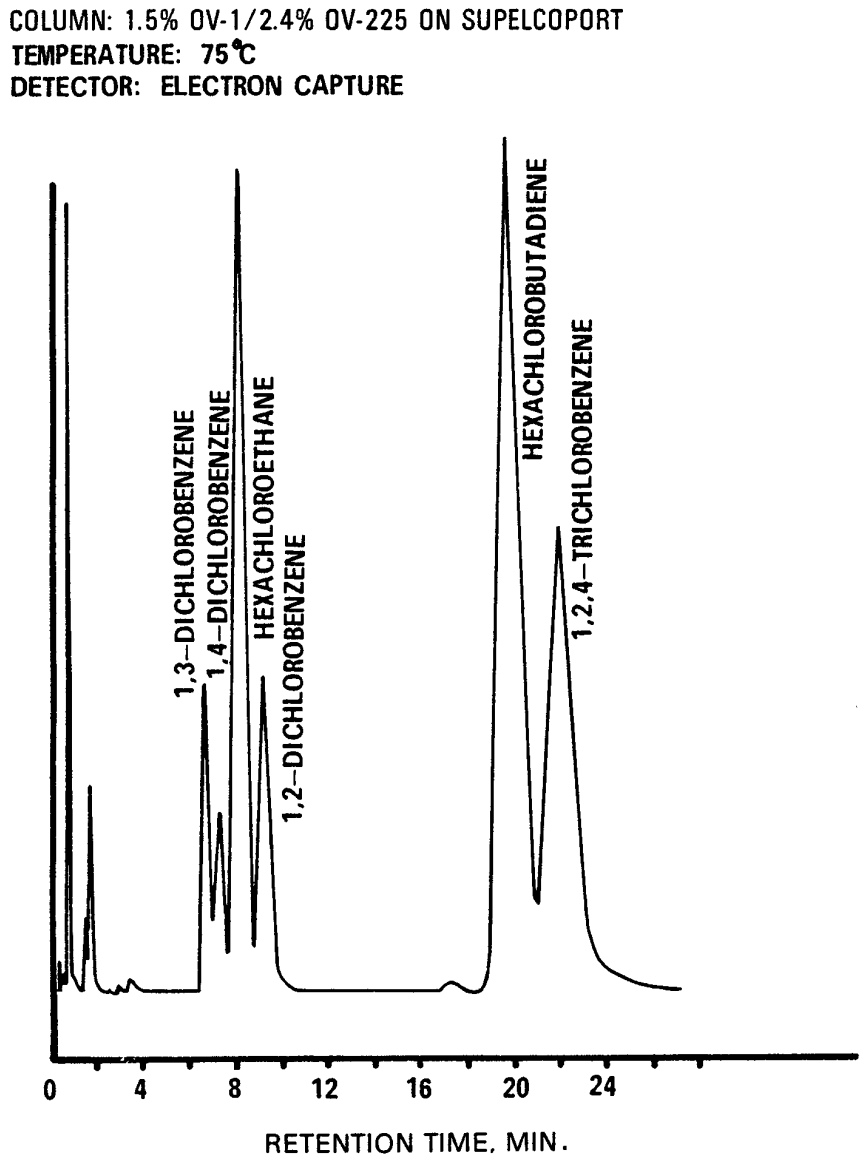


Figure 1. Gas chromatogram of chlorinated hydrocarbons.

COLUMN: 1.5% OV-1/2.4% OV-225 ON SUPELCOPORT
TEMPERATURE: 165°C
DETECTOR: ELECTRON CAPTURE

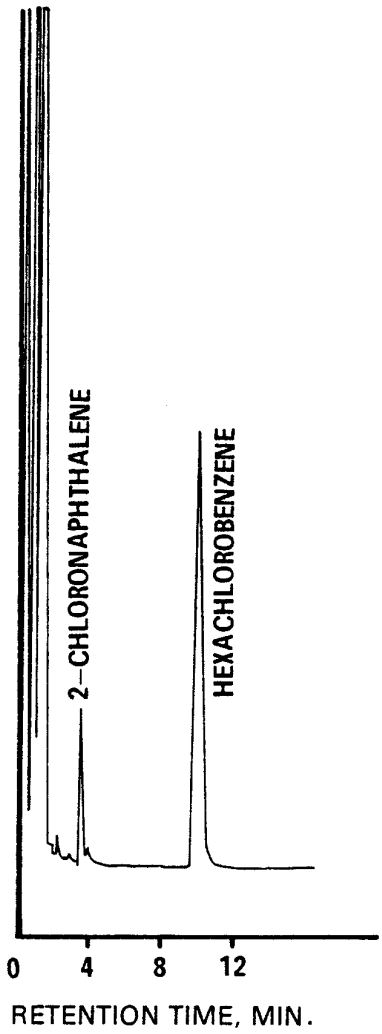


Figure 2. Gas chromatogram of chlorinated hydrocarbons.

METHOD 613—2,3,7,8-TETRACHLORODIBENZO-P-DIOXIN

1. Scope and Application

1.1 This method covers the determination of 2,3,7,8-tetrachlorodibenzo-p-dioxin (2,3,7,8-TCDD). The following parameter may be determined by this method:

Parameter	STORET No.	GAS No.
2,3,7,8-TCDD	34675	1746–01–6

1.2 This is a gas chromatographic/mass spectrometer (GC/MS) method applicable to the determination of 2,3,7,8–TCDD in municipal and industrial discharges as provided under 40 CFR 136.1. Method 625 may be used to screen samples for 2,3,7,8–TCDD. When the screening test is positive, the final qualitative confirmation and quantification must be made using Method 613.

1.3 The method detection limit (MDL, defined in Section 14.1)[1] for 2,3,7,8–TCDD is listed in Table 1. The MDL for a specific wastewater may be different from that listed, depending upon the nature of interferences in the sample matrix.

1.4 Because of the extreme toxicity of this compound, the analyst must prevent exposure to himself, of to others, by materials knows or believed to contain 2,3,7,8–TCDD. Section 4 of this method contains guidelines and protocols that serve as minimum safe-handling standards in a limited-access laboratory.

1.5 Any modification of this method, beyond those expressly permitted, shall be considered as a major modification subject to application and approval of alternate test procedures under 40 CFR 136.4 and 136.5.

1.6 This method is restricted to use by or under the supervision of analysts experienced in the use of a gas chromatograph/mass spectrometer and in the interpretation of mass spectra. Each analyst must demonstrate the ability to generate acceptable results with this method using the procedure described in Section 8.2.

2. Summary of Method

2.1 A measured volume of sample, approximately 1–L, is spiked with an internal standard of labeled 2,3,7,8–TCDD and extracted with methylene chloride using a separatory funnel. The methylene chloride extract is exchanged to hexane during concentration to a volume of 1.0 mL or less. The extract is then analyzed by capillary column GC/MS to separate and measure 2,3,7,8–TCDD.[2,3]

2.2 The method provides selected column chromatographic cleanup proceudres to aid in the elimination of interferences that may be encountered.

3. Interferences

3.1 Method interferences may be caused by contaminants in solvents, reagents, glassware, and other sample processing hardware that lead to discrete artifacts and/or elevated backgrounds at the masses (m/z) monitored. All of these materials must be routinely demonstrated to be free from interferences under the conditions of the analysis by running laboratory reagent blanks as described in Section 8.1.3.

3.1.1 Glassware must be scrupulously cleaned.[4] Clean all glassware as soon as possible after use by rinsing with the last solvent used in it. Solvent rinsing should be followed by detergent washing with hot water, and rinses with tap water and distilled water. The glassware should then be drained dry, and heated in a muffle furnace at 400 °C for 15 to 30 min. Some thermally stable materials, such as PCBs, may not be eliminated by the treatment. Solvent rinses with acetone and pesticide quality hexane may be substituted for the muffle furnace heating. Thorough rinsing with such solvents usually eliminates PCB interference. Volumetric ware should not be heated in a muffle furnace. After drying and cooling, glassware should be sealed and stored in a clean environment to prevent any accumulation of dust or other contaminants. Store inverted or capped with aluminum foil.

3.1.2 The use of high purity reagents and solvents helps to mininmize interference problems. Purification of solvents by distillation in all-glass systems may be required.

3.2 Matrix interferences may be caused by contaminants that are coextracted from the sample. The extent of matrix interferences will vary considerably from source to source, depending upon the nature and diversity of the industrial complex or municipality being sampled. 2,3,7,8–TCDD is often associated with other interfering chlorinated compounds which are at concentrations several magnitudes higher than that of 2,3,7,8–TCDD. The cleanup producers in Section 11 can be used to overcome many of these interferences, but unique samples may require additional cleanup approaches[1,5–7] to eliminate false positives and achieve the MDL listed in Table 1.

3.3 The primary column, SP–2330 or equivalent, resolves 2,3,7,8–TCDD from the other 21 TCDD insomers. Positive results using any other gas chromatographic column must be confirmed using the primary column.

4. Safety

4.1 The toxicity or carcinogenicity of each reagent used in this method has not been precisely defined; however, each chemical compound should be treated as a potential health hazard. From this viewpoint, exposure to these chemicals must be reduced to

the lowest possible level by whatever means available. The laboratory is responsible for maintaining a current awareness file of OSHA regulations regarding the safe handling of the chemicals specified in this method. A reference file of material data handling sheets should also be made available to all personnel involved in the chemical analysis. Additional references to laboratory safety are available and have been identified[8–10] for the information of the analyst. Benzene and 2,3,7,8–TCDD have been identified as suspected human or mammalian carcinogens.

4.2 Each laboratory must develop a strict safety program for handling 2,3,7,8–TCDD. The following laboratory practices are recommended:

4.2.1 Contamination of the laboratory will be minimized by conducting all manipulations in a hood.

4.2.2 The effluents of sample splitters for the gas chromatograph and roughing pumps on the GC/MS should pass through either a column of activated charcoal or be bubbled through a trap containing oil or high-boiling alcohols.

4.2.3 Liquid waste should be dissolved in methanol or ethanol and irradiated with ultraviolet light with a wavelength greater than 290 nm for several days. (Use F 40 BL lamps or equivalent). Analyze liquid wastes and dispose of the solutions when 2,3,7,8–TCDD can no longer be detected.

4.3 Dow Chemical U.S.A. has issued the following precautimns (revised November 1978) for safe handling of 2,3,7,8–TCDD in the laboratory:

4.3.1 The following statements on safe handling are as complete as possible on the basis of available toxicological information. The precautions for safe handling and use are necessarily general in nature since detailed, specific recommendations can be made only for the particular exposure and circumstances of each individual use. Inquiries about specific operations or uses may be addressed to the Dow Chemical Company. Assistance in evaluating the health hazards of particular plant conditions may be obtained from certain consulting laboratories and from State Departments of Health or of Labor, many of which have an industrial health service. 2,3,7,8–TCDD is extremely toxic to laboratory animals. However, it has been handled for years without injury in analytical and biological laboratories. Techniques used in handling radioactive and infectious materials are applicable to 2,3,7,8,–TCDD.

4.3.1.1 Protective equipment—Throwaway plastic gloves, apron or lab coat, safety glasses, and a lab hood adequate for radioactive work.

4.3.1.2 Training—Workers must be trained in the proper method of removing contaminated gloves and clothing without contacting the exterior surfaces.

4.3.1.3 Personal hygiene—Thorough washing of hands and forearms after each manipulation and before breaks (coffee, lunch, and shift).

4.3.1.4 Confinement—Isolated work area, posted with signs, segregated glassware and tools, plastic-backed absorbent paper on benchtops.

4.3.1.5 Waste—Good technique includes minimizing contaminated waste. Plastic bag liners should be used in waste cans. Janitors must be trained in the safe handling of waste.

4.3.1.6 Disposal of wastes—2,3,7,8–TCDD decomposes above 800 °C. Low-level waste such as absorbent paper, tissues, animal remains, and plastic gloves may be burned in a good incinerator. Gross quantities (milligrams) should be packaged securely and disposed through commercial or governmental channels which are capable of handling high-level radioactive wastes or extremely toxic wastes. Liquids should be allowed to evaporate in a good hood and in a disposable container. Residues may then be handled as above.

4.3.1.7 Decontamination—For personal decontamination, use any mild soap with plenty of scrubbing action. For decontamination of glassware, tools, and surfaces, Chlorothene NU Solvent (Trademark of the Dow Chemical Company) is the least toxic solvent shown to be effective. Satisfactory cleaning may be accomplished by rinsing with Chlorothene, then washing with any detergent and water. Dishwater may be disposed to the sewer. It is prudent to minimize solvent wastes because they may require special disposal through commercial sources which are expensive.

4.3.1.8 Laundry—Clothing known to be contaminated should be disposed with the precautions described under Section 4.3.1.6. Lab coats or other clothing worn in 2,3,7,8–TCDD work areas may be laundered.

Clothing should be collected in plastic bags. Persons who convey the bags and launder the clothing should be advised of the hazard and trained in proper handling. The clothing may be put into a washer without contact if the launderer knows the problem. The washer should be run through a cycle before being used again for other clothing.

4.3.1.9 Wipe tests—A useful method of determining cleanliness of work surfaces and tools is to wipe the surface with a piece of filter paper. Extraction and analysis by gas chromatography can achieve a limit of sensitivity of 0.1 µg per wipe. Less than 1 µg of 2,3,7,8–TCDD per sample indicates acceptable cleanliness; anything higher warrants further cleaning. More than 10 µg on a wipe sample constitutes an acute hazard and requires prompt cleaning before further use of the equipment or work space. A high (>10 µg)

2,3,7,8–TCDD level indicates that unacceptable work practices have been employed in the past.

4.3.1.10 Inhalation—Any procedure that may produce airborne contamination must be done with good ventilation. Gross losses to a ventilation system must not be allowed. Handling of the dilute solutions normally used in analytical and animal work presents no inhalation hazards except in the case of an accident.

4.3.1.11 Accidents—Remove contaminated clothing immediately, taking precautions not to contaminate skin or other articles. Wash exposed skin vigorously and repeatedly until medical attention is obtained.

5. Apparatus and Materials

5.1 Sampling equipment, for discrete or composite sampling.

5.1.1 Grab sample bottle—1–L or 1-qt, amber glass, fitted with a screw cap lined with Teflon. Foil may be substituted for Teflon if the sample is not corrosive. If amber bottles are not available, protect samples from light. The bottle and cap liner must be washed, rinsed with acetone or methylene chloride, and dried before use to minimize contamination.

5.1.2 Automatic sampler (optional)—The sampler must incorporate glass sample containers for the collection of a minimum of 250 mL of sample. Sample containers must be kept refrigerated at 4 °C and protected from light during compositing. If the sampler uses a peristaltic pump, a minimum length of compressible silicone rubber tubing may be used. Before use, however, the compressible tubing should be thoroughly rinsed with methanol, followed by repeated rinsings with distilled water to minimize the potential for contamination of the sample. An integrating flow meter is required to collect flow proportional composites.

5.1.3 Clearly label all samples as ''POISON'' and ship according to U.S. Department of Transportation regulations.

5.2 Glassware (All specifications are suggested. Catalog numbers are included for illustration only.):

5.2.1 Separatory funnels—2–L and 125-mL, with Teflon stopcock.

5.2.2 Concentrator tube, Kuderna-Danish—10-mL, graduated (Kontes K–570050–1025 or equivalent). Calibration must be checked at the volumes employed in the test. Ground glass stopper is used to prevent evaporation of extracts.

5.2.3 Evaporative flask, Kuderna-Danish—500–mL (Kontes K–570001–0500 or equivalent). Attach to concentrator tube with springs.

5.2.4 Snyder column, Kuderna-Danish—Three-ball macro (Kontes K–503000–0121 or equivalent).

5.2.5 Snyder column, Kuderna-Danish—Two-ball micro (Kontes K–569001–0219 or equivalent).

5.2.6 Vials—10 to 15–mL, amber glass, with Teflon-lined screw cap.

5.2.7 Chromatographic column—300 mm long × 10 mm ID, with Teflon stopcock and coarse frit filter disc at bottom.

5.2.8 Chromatographic column—400 mm long × 11 mm ID, with Teflon stopcock and coarse frit filter disc at bottom.

5.3 Boiling chips—Approximately 10/40 mesh. Heat to 400 °C for 30 min or Soxhlet extract with methylene chloride.

5.4 Water bath—Heated, with concentric ring cover, capable of temperature control (±2 °C). The bath should be used in a hood.

5.5 GC/MS system:

5.5.1 Gas chromatograph—An analytical system complete with a temperature programmable gas chromatograph and all required accessories including syringes, analytical columns, and gases. The injection port must be designed for capillary columns. Either split, splitless, or on-column injection techniques may be employed, as long as the requirements of Section 7.1.1 are achieved.

5.5.2 Column—60 m long × 0.25 mm ID glass or fused silica, coated with SP–2330 (or equivalent) with a film thickness of 0.2 µm. Any equivalent column must resolve 2, 3, 7, 8–TCDD from the other 21 TCDD isomers.[16]

5.5.3 Mass spectrometer—Either a low resolution mass spectrometer (LRMS) or a high resolution mass spectrometer (HRMS) may be used. The mass spectrometer must be equipped with a 70 V (nominal) ion source and be capable of aquiring m/z abundance data in real time selected ion monitoring (SIM) for groups of four or more masses.

5.5.4 GC/MS interface—Any GC to MS interface can be used that achieves the requirements of Section 7.1.1. GC to MS interfaces constructed of all glass or glass-lined materials are recommended. Glass surfaces can be deactivated by silanizing with dichlorodimethylsilane. To achieve maximum sensitivity, the exit end of the capillary column should be placed in the ion source. A short piece of fused silica capillary can be used as the interface to overcome problems associated with straightening the exit end of glass capillary columns.

5.5.5 The SIM data acquired during the chromatographic program is defined as the Selected Ion Current Profile (SICP). The SICP can be acquired under computer control or as a real time analog output. If computer control is used, there must be software available to plot the SICP and report peak height or area data for any m/z in the SICP between specified time or scan number limits.

5.6 Balance—Analytical, capable of accurately weighing 0.0001 g.

6. Reagents

6.1 Reagent water—Reagent water is defined as a water in which an interferent is not observed at the MDL of 2, 3, 7, 8–TCDD.

6.2 Sodium hydroxide solution (10 N)—Dissolve 40 g of NaOH (ACS) in reagent water and dilute to 100 mL. Wash the solution with methylene chloride and hexane before use.

6.3 Sodium thiosulfate—(ACS) Granular.

6.4 Sulfuric acid—Concentrated (ACS, sp. gr. 1.84).

6.5 Acetone, methylene chloride, hexane, benzene, ortho-xylene, tetradecane—Pesticide quality or equivalent.

6.6 Sodium sulfate—(ACS) Granular, anhydrous. Purify by heating at 400 °C for 4 h in a shallow tray.

6.7 Alumina—Neutral, 80/200 mesh (Fisher Scientific Co., No. A–540 or equivalent). Before use, activate for 24 h at 130 °C in a foil-covered glass container.

6.8 Silica gel—High purity grade, 100/120 mesh (Fisher Scientific Co., No. S–679 or equivalent).

6.9 Stock standard solutions (1.00 µg/µL)—Stock standard solutimns can be prepared from pure standard materials or purchased as certified solutions. Acetone should be used as the solvent for spiking solutions; ortho-xylene is recommended for calibration standards for split injectors; and tetradecane is recommended for splitless or on-colum injectors. Analyze stock internal standards to verify the absence of native 2,3,7,8–TCDD.

6.9.1 Prepare stock standard solutions of 2,3,7,8–TCDD (mol wt 320) and either $^{37}C1_4$ 2,3,7,8–TCDD (mol wt 328) or $^{13}C1_{12}$ 2,3,7,8–TCDD (mol wt 332) in an isolated area by accurately weighing about 0.0100 g of pure material. Dissolve the material in pesticide quality solvent and dilute to volume in a 10-mL volumetric flask. When compound purity is assayed to be 96% or greater, the weight can be used without correction to calculate the concentration of the stock standard. Commercially prepared stock standards can be used at any concentration if they are certified by the manufacturer or by an independent source.

6.9.2 Transfer the stock standard solutions into Teflon-sealed screw-cap bottles. Store in an isolated refrigerator protected from light. Stock standard solutions should be checked frequently for signs of degradation or evaporation, especially just prior to preparing calibration standards or spiking solutions from them.

6.9.3 Stock standard solutions must be replaced after six months, or sooner if comparison with check standards indicates a problem.

6.10 Internal standard spiking solution (25 ng/mL)—Using stock standard solution, prepare a spiking solution in acetone of either $^{13}Cl_{12}$ or $^{37}Cl_4$ 2,3,7,8–TCDD at a concentration of 25 ng/mL. (See Section 10.2)

6.11 Quality control check sample concentrate—See Section 8.2.1.

7. Calibration

7.1 Establish gas chromatograhic operating conditions equivalent to those given in Table 1 and SIM conditions for the mass spectrometer as described in Section 12.2 The GC/MS system must be calibrated using the internal standard technique.

7.1.1 Using stock standards, prepare calibration standards that will allow measurement of relative response factors of at least three concentration ratios of 2,3,7,8–TCDD to internal standard. Each calibration standard must be prepared to contain the internal standard at a concentration of 25 ng/mL. If any interferences are contributed by the internal standard at m/z 320 and 322, its concentration may be reduced in the calibration standards and in the internal standard spiking solution (Section 6.10). One of the calibration standards should contain 2,3,7,8–TCDD at a concentration near, but above, the MDL and the other 2,3,7,8–TCDD concentrations should correspond to the expected range of concentrations found in real samples or should define the working range of the GC/MS system.

7.1.2 Using injections of 2 to 5 µL, analyze each calibration standardaccording to Section 12 and tabulate peak height or area response against the concentration of 2,3,7,8–TCDD and internal standard. Calculate response factors (RF) for 2,3,7,8–TCDD using Equation 1.

$$RF = \frac{(A_s)\,(C_{is})}{(A_{is})\,(C_s)}$$

Equation 1

where:

A_s=SIM response for 2,3,7,8–TCDD m/z 320.

A_{is}=SIM response for the internal standard, m/z 332 for $^{13}C_{12}$ 2,3,7,8–TCDD m/z 328 for $^{37}Cl_4$ 2,3,7,8–TCDD.

C_{is}=Concentration of the internal standard (µg/L).

C_s=Concentration of 2,3,7,8–TCDD (µg/L).

If the RF value over the working range is a constant (< 10% relative standard deviation, RSD), the RF can be assumed to be invariant and the average RF can be used for calculations. Alternatively, the results can be used to plot a calibration curve of response ratios, A_s/A_{is}, vs. RF.

7.1.3 The working calibration curve or RF must be verified on each working day by the measurement of one or more 2,3,7,8–TCDD calibration standards. If the response for 2,3,7,8–TCDD varies from the predicted response by more than ±15%, the test must be repeated using a fresh calibration standard. Alternatively, a new calibration curve must be prepared.

7.2 Before using any cleanup procedure, the analyst must process a series of calibration standards through the procedure to validate elution patterns and the absence of interferences from the reagents.

8. Quality Control

8.1 Each laboratory that uses this method is required to operate a formal quality control program. The minimum requirements of this program consist of an initial demonstration of laboratory capability and an ongoing analysis of spiked samples to evaluate and document data quality. The laboratory must maintain records to document the quality of data that is generated. Ongoing data quality checks are compared with established performance criteria to determine if the results of analyses meet the performance characteristics of the method. When results of sample spikes indicate atypical method performance, a quality control check standard must be analyzed to confirm that the measurements were performed in an in-control mode of operation.

8.1.1 The analyst must make an initial, one-time, demonstration of the ability to generate acceptable accuracy and precision with this method. This ability is established as described in Section 8.2.

8.1.2 In recognition of advances that are occurring in chromatography, the analyst is permitted certain options (detailed in Sections 10.5, 11.1, and 12.1) to improve the separations or lower the cost of measurements. Each time such a modification is made to the method, the analyst is required to repeat the procedure in Section 8.2

8.1.3 Before processing any samples, the analyst must analyze a reagent water blank to demonstrate that interferences from the analytical system and glassware are under control. Each time a set of samples is extracted or reagents are changed, a reagent water blank must be processed as a safeguard against laboratory contamination.

8.1.4 The laboratory must, on an ongoing basis, spike and analyze a minimum of 10% of all samples with native 2,3,7,8–TCDD to monitor and evaluate laboratory data quality. This procedure is described in Section 8.3.

8.1.5 The laboratory must, on an ongoing basis, demonstrate through the analyses of quality control check standards that the operation of the measurement system is in control. This procedure is described in Section 8.4. The frequency of the check standard analyses is equivalent to 10% of all samples analyzed but may be reduced if spike recoveries from samples (Section 8.3) meet all specified quality control criteria.

8.1.6 The laboratory must maintain performance records to document the quality of data that is generated. This procedure is described in Section 8.5.

8.2 To establish the ability to generate acceptable accuracy and precision, the analyst must perform the following operations.

8.2.1 A quality control (QC) check sample concentrate is required containing 2,3,7,8–TCDD at a concentration of 0.100 µg/mL in acetone. The QC check sample concentrate must be obtained from the U.S. Environmental Protection Agency, Environmental Monitoring and Support Laboratory in Cincinnati, Ohio, if available. If not available from that source, the QC check sample concentrate must be obtained from another external source. If not available from either source above, the QC check sample concentrate must be prepared by the laboratory using stock standards prepared independently from those used for calibration.

8.2.2 Using a pipet, prepare QC check samples at a concentration of 0.100 µg/L (100 ng/L) by adding 1.00 mL of QC check sample concentrate to each of four 1–L aliquots of reagent water.

8.2.3 Analyze the well-mixed QC check samples according to the method beginning in Section 10.

8.2.4 Calculate the average recovery ($\bar{X}$) in µg/L, and the standard deviation of the recovery (s) in µg/L, for 2,3,7,8–TCDD using the four results.

8.2.5 Compare s and ($\bar{X}$) with the corresponding acceptance criteria for precision and accuracy, respectively, found in Table 2. If s and $\bar{X}$ meet the acceptance criteria, the system performance is acceptable and analysis of actual samples can begin. If s exceeds the precision limit or $\bar{X}$ falls outside the range for accuracy, the system performance is unacceptable for 2,3,7,8–TCDD. Locate and correct the source of the problem and repeat the test beginning with Section 8.2.2.

8.3 The laboratory must, on an ongoing basis, spike at least 10% of the samples from each sample site being monitored to assess accuracy. For laboratories analyzing one to ten samples per month, at least one spiked sample per month is required.

8.3.1 The concentration of the spike in the sample should be determined as follows:

8.3.1.1 If, as in compliance monitoring, the concentration of 2,3,7,8–TCDD in the sample is being checked against a regulatory concentration limit, the spike should be at that limit or 1 to 5 times higher than the background concentration determined in Section 8.3.2, whichever concentration would be larger.

8.3.1.2 If the concentration of 2,3,7,8–TCDD in the sample is not being checked against a limit specific to that parameter, the spike should be at 0.100 µg/L or 1 to 5 times higher than the background concentration determined in Section 8.3.2, whichever concentration would be larger.

8.3.1.3 If it is impractical to determine background levels before spiking (e.g., maximum holding times will be exceeded), the

spike concentration should be (1) the regulatory concentration limit, if any; or, if none (2) the larger of either 5 times higher than the expected background concentration or 0.100 µg/L.

8.3.2 Analyze one sample aliquot to determine the background concentration (B) of 2,3,7,8–TCDD. If necessary, prepare a new QC check sample concentrate (Section 8.2.1) appropriate for the background concentration in the sample. Spike a second sample aliquot with 1.0 mL of the QC check sample concentrate and analyze it to determine the concentration after spiking (A) of 2,3,7,8–TCDD. Calculate percent recovery (P) as 100(A–B)%T, where T is the known true value of the spike.

8.3.3 Compare the percent recovery (P) for 2,3,7,8–TCDD with the corresponding QC acceptance criteria found in Table 2. These acceptance criteria were calculated to include an allowance for error in measurement of both the background and spike concentrations, assuming a spike to background ratio of 5:1. This error will be accounted for to the extent that the analyst's spike to background ratio approaches 5:1.[11] If spiking was performed at a concentration lower than 0.100 µg/L, the analyst must use either the QC acceptance criteria in Table 2, or optional QC acceptance criteria calculated for the specific spike concentration. To calculate optional acceptance criteria for the recovery of 2,3,7,8–TCDD: (1) Calculate accuracy (X′) using the equation in Table 3, substituting the spike concentration (T) for C; (2) calculate overall precision (S′) using the equation in Table 3, substituting X′ for X; (3) calculate the range for recovery at the spike concentration as (100 X′/T)±2.44(100 S′/T)%.[11]

8.3.4 If the recovery of 2,3,7,8–TCDD falls outside the designated range for recovery, a check standard must be analyzed as described in Section 8.4.

8.4 If the recovery of 2,3,7,8–TCDD fails the acceptance criteria for recovery in Section 8.3, a QC check standard must be prepared and analyzed.

NOTE: The frequency for the required analysis of a QC check standard will depend upon the complexity of the sample matrix and the performance of the laboratory.

8.4.1 Prepare the QC check standard by adding 1.0 mL of QC check sample concentrate (Section 8.2.1 or 8.3.2) to 1 L of reagent water.

8.4.2 Analyze the QC check standard to determine the concentration measured (A) of 2,3,7,8–TCDD. Calculate the percent recovery (P_s) as 100 (A/T)%, where T is the true value of the standard concentration.

8.4.3 Compare the percent recovery (P_s) with the corresponding QC acceptance criteria found in Table 2. If the recovery of 2,3,7,8–TCDD falls outside the designated range, the laboratory performance is judged to be out of control, and the problem must be immediately identified and corrected. The analytical result for 2,3,7,8–TCDD in the unspiked sample is suspect and may not be reported for regulatory compliance purposes.

8.5 As part of the QC program for the laboratory, method accuracy for wastewater samples must be assessed and records must be maintained. After the analysis of five spiked wastewater samples as in Section 8.3, calculate the average percent recovery ($\bar{P}$) and the spandard deviation of the percent recovery (s_p). Express the accuracy assessment as a percent recovery interval from $\bar{P}-2s_p$ to $\bar{P}+2s_p$. If $\bar{P}$=90% and s_p=10%, for example, the accuracy interval is expressed as 70–110%. Update the accuracy assessment on a regular basis (e.g. after each five to ten new accuracy measurements).

8.6 It is recommended that the laboratory adopt additional quality assurance practices for use with this method. The specific practices that are most productive depend upon the needs of the laboratory and the nature of the samples. Field duplicates may be analyzed to assess the precision of the environmental measurements. Whenever possible, the laboratory should analyze standard reference materials and participate in relevant performance evaluation studies.

9. Sample Collection, Preservation, and Handling

9.1 Grab samples must be collected in glass containers. Conventional sampling practices[12] should be followed, except that the bottle must not be prerinsed with sample before collection. Composite samples should be collected in refrigerated glass containers in accordance with the requirements of the program. Automatic sampling equipment must be as free as possible of Tygon tubing and other potential sources of contamination.

9.2 All samples must be iced or refrigerated at 4 °C and protected from light from the time of collection until extraction. Fill the sample bottles and, if residual chlorine is present, add 80 mg of sodium thiosulfate per liter of sample and mix well. EPA Methods 330.4 and 330.5 may be used for measurement of residual chlorine.[13] Field test kits are available for this purpose.

9.3 Label all samples and containers ''POISON'' and ship according to applicable U.S. Department of Transportation regulations.

9.4 All samples must be extracted within 7 days of collection and completely analyzed within 40 days of extraction.[2]

10. Sample Extraction

CAUTION: When using this method to analyze for 2,3,7,8–TCDD, all of the following operations must be performed in a limited-access laboratory with the analyst wearing full

protective covering for all exposed skin surfaces. See Section 4.2.

10.1 Mark the water meniscus on the side of the sample bottle for later determination of sample volume. Pour the entire sample into a 2–L separatory funnel.

10.2 Add 1.00 mL of internal standard spiking solution to the sample in the separatory funnel. If the final extract will be concentrated to a fixed volume below 1.00 mL (Section 12.3), only that volume of spiking solution should be added to the sample so that the final extract will contain 25 ng/mL of internal standard at the time of analysis.

10.3 Add 60 mL of methylene chloride to the sample bottle, seal, and shake 30 s to rinse the inner surface. Transfer the solvent to the separatory funnel and extract the sample by shaking the funnel for 2 min. with periodic venting to release excess pressure. Allow the organic layer to separate from the water phase for a minimum of 10 min. If the emulsion interface between layers is more than one-third the vmlume of the solvent layer, the analyst must employ mechanical techniques to complete the phase separation. The optimum technique depends upon the sample, but may include stirring, filtration of the emulsion through glass wool, centrifugation, or other physical methods. Collect the methylene chloride extract in a 250-mL Erlenmeyer flask.

10.4 Add a second 60-mL volume of methylene chloride to the sample bottle and repeat the extraction procedure a second time, combining the extracts in the Erlenmeyer flask. Perform a third extraction in the same manner.

10.5 Assemble a Kuderna-Danish (K–D) concentrator by attaching a 10-mL concentrator tube to a 500-mL evaporative flask. Other concentration devices or techniques may be used in place of the K–D concentrator if the requirements of Section 8.2 are met.

10.6 Pour the combined extract into the K–D concentrator. Rinse the Erlenmeyer flask with 20 to 30 mL of methylele chloride to complete the quantitative transfer.

10.7 Add one or two clean boiling chips to the evaporative flask and attach a three-ball Snyder column. Prewet the Snyder column by adding about 1 mL of methylene chloride to the top. Place the K–D apparatus on a hot water bath (60 to 65 °C) so that the concentrator tube is partially immersed in the hot water, and the entire lower rounded surface of the flask is bathed with hot vapor. Adjust the vertical position of the apparatus and the water temperature as required to complete the concentration in 15 to 20 min. At the proper rate of distillation the balls of the column will actively chatter but the chambers will not flood with condensed solvent. When the apparent volume of liquid reaches 1 mL, remove the K–D apparatus and allow it to drain and cool for at least 10 min.

10.8 Momentarily remove the Snyder column, add 50 mL of hexane and a new boiling chip, and reattach the Snyder column. Raise the temperature of the water bath to 85 to 90°C. Concentrate the extract as in Section 10.7, except use hexane to prewet the column. Remove the Snyder column and rinse the flask and its lower joint into the concentrator tube with 1 to 2 mL of hexane. A 5-mL syringe is recommended for this operation. Set aside the K–D glassware for reuse in Section 10.14.

10.9 Pour the hexane extract from the concentrator tube into a 125-mL separatory funnel. Rinse the concentrator tube four times with 10-mL aliquots of hexane. Combine all rinses in the 125-mL separatory funnel.

10.10 Add 50 mL of sodium hydroxide solution to the funnel and shake for 30 to 60 s. Discard the aqueous phase.

10.11 Perform a second wash of the organic layer with 50 mL of reagent water. Discard the aqueous phase.

10.12 Wash the hexane layer with a least two 50-mL aliquots of concentrated sulfuric acid. Continue washing the hexane layer with 50-mL aliquots of concentrated sulfuric acid until the acid layer remains colorless. Discard all acid fractions.

10.13 Wash the hexane layer with two 50-mL aliquots of reagent water. Discard the aqueous phases.

10.14 Transfer the hexane extract into a 125-mL Erlenmeyer flask containing 1 to 2 g of anhydrous sodium sulfate. Swirl the flask for 30 s and decant the hexane extract into the reassembled K–D apparatus. Complete the quantitative transfer with two 10-mL hexane rinses of the Erlenmeyer flask.

10.15 Replace the one or two clean boiling chips and concentrate the extract to 6 to 10 mL as in Section 10.8.

10.16 Add a clean boiling chip to the concentrator tube and attach a two-ball micro-Snyder column. Prewet the column by adding about 1 mL of hexane to the top. Place the micro-K–D apparatus on the water bath so that the concentrator tube is partially immersed in the hot water. Adjust the vertical position of the apparatus and the water temperature as required to complete the concentration in 5 to 10 min. At the proper rate of distillation the balls of the column will actively chatter but the chambers will not flood. When the apparent volume of liquid reaches about 0.5 mL, remove the K–D apparatus and allow it to drain and cool for at least 10 min. Remove the micro-Snyder column and rinse its lower joint into the concentrator tube with 0.2 mL of hexane.

Adjust the extract volume to 1.0 mL with hexane. Stopper the concentrator tube and store refrigerated and protected from light if further processing will not be performed immediately. If the extract will be stored

longer than two days, it should be transferred to a Teflon-sealed screw-cap vial. If the sample extract requires no further cleanup, proceed with GC/MS analysis (Section 12). If the sample requires further cleanup, proceed to Section 11.

10.17 Determine the original sample volume by refilling the sample bottle to the mark and transferring the liquid to a 1000-mL graduated cylinder. Record the sample volume to the nearest 5 mL.

11. Cleanup and Separation

11.1 Cleanup procedures may not be necessary for a relatively clean sample matrix. If particular circumstances demand the use of a cleanup procedure, the analyst may use either procedure below or any other appropriate procedure.[1,5–7] However, the analyst first must demonstrate that the requirements of Section 8.2 can be met using the method as revised to incorporate the cleanup procedure. Two cleanup column options are offered to the analyst in this section. The alumina column should be used first to overcome interferences. If background problems are still encountered, the silica gel column may be helpful.

11.2 Alumina column cleanup for 2,3,7,8–TCDD:

11.2.1 Fill a 300 mm long x 10 mm ID chromatographic column with activated alumina to the 150 mm level. Tap the column gently to settle the alumina and add 10 mm of anhydrous sodium sulfate to the top.

11.2.2 Preelute the column with 50 mL of hexane. Adjust the elution rate to 1 mL/min. Discard the eluate and just prior to exposure of the sodium sulfate layer to the air, quantitatively transfer the 1.0-mL sample extract onto the column using two 2-mL portions of hexane to complete the transfer.

11.2.3 Just prior to exposure of the sodium sulfate layer to the air, add 50 mL of 3% methylene chloride/95% hexane (V/V) and continue the elution of the column. Discard the eluate.

11.2.4 Next, elute the column with 50 mL of 20% methylene chloride/80% hexane (V/V) into a 500-mL K–D flask equipped with a 10-mL concentrator tube. Concentrate the collected fraction to 1.0 mL as in Section 10.16 and analyze by GC/MS (Section 12).

11.3 Silica gel column cleanup for 2,3,7,8–TCDD:

11.3.1 Fill a 400 mm long x 11 mm ID chromatmgraphic column with silica gel to the 300 mm level. Tap the column gently to settle the silica gel and add 10 mm of anhydrous sodium sulfate to the top.

11.3.2 Preelute the column with 50 mL of 20% benzene/80% hexane (V/V). Adjust the elution rate to 1 mL/min. Discard the eluate and just prior to exposure of the sodium sulfate layer to the air, quantitatively transfer the 1.0-mL sample extract onto the column using two 2-mL portions of 20% benzene/80% hexane to complete the transfer.

11.3.3 Just prior to exposure of the sodium sulfate layer to the air, add 40 mL of 20% benzene/80% hexane to the column. Collect the eluate in a clean 500-mL K–D flask equipped with a 10-mL concentrator tube. Concentrate the collected fraction to 1.0 mL as in Section 10.16 and analyze by GC/MS.

12. GC/MS Analysis

12.1 Table 1 summarizes the recommended operating conditions for the gas chromatograph. Included in this table are retention times and MDL that can be achieved under these conditions. Other capillary columns or chromatographic conditions may be used if the requirements of Sections 5.5.2 and 8.2 are met.

12.2 Analyze standards and samples with the mass spectrometer operating in the selected ion monitoring (SIM) mode using a dwell time to give at least seven points per peak. For LRMS, use masses at m/z 320, 322, and 257 for 2,3,7,8–TCDD and either m/z 328 for $^{37}Cl_4$ 2,3,7,8–TCDD or m/z 332 for $^{13}C_{12}$ 2,3,7,8–TCDD. For HRMS, use masses at m/z 319.8965 and 321.8936 for 2,3,7,8–TCDD and either m/z 327.8847 for $^{37}Cl_4$ 2,3,7,8–TCDD or m/z 331.9367 for $^{13}C_{12}$ 2,3,7,8–TCDD.

12.3 If lower detection limits are required, the extract may be carefully evaporated to dryness under a gentle stream of nitrogen with the concentrator tube in a water bath at about 40 °C. Conduct this operation immediately before GC/MS analysis. Redissolve the extract in the desired final volume of ortho-xylene or tetradecane.

12.4 Calibrate the system daily as described in Section 7.

12.5 Inject 2 to 5 µL of the sample extract into the gas chromatograph. The volume of calibration standard injected must be measured, or be the same as all sample injection volumes.

12.6 The presence of 2,3,7,8–TCDD is qualitatively confirmed if all of the following criteria are achieved:

12.6.1 The gas chromatographic column must resolve 2,3,7,8–TCDD from the other 21 TCDD isomers.

12.6.2 The masses for native 2,3,7,8–TCDD (LRMS-m/z 320, 322, and 257 and HRMS-m/z 320 and 322) and labeled 2,3,7,8–TCDD (m/z 328 or 332) must exhibit a simultaneous maximum at a retention time that matches that of native 2,3,7,8–TCDD in the calibration standard, with the performance specifications of the analytical system.

12.6.3 The chlorine isotope ratio at m/z 320 and m/z 322 must agree to within±10% of that in the calibration standard.

12.6.4 The signal of all peaks must be greater than 2.5 times the noise level.

12.7 For quantitation, measure the response of the m/z 320 peak for 2,3,7,8–TCDD

and the m/z 332 peak for $^{13}C_{12}$ 2,3,7,8–TCDD or the m/z 328 peak for $^{37}Cl_4$ 2,3,7,8–TCDD.

12.8 Co-eluting impurities are suspected if all criteria are achieved except those in Section 12.6.3. In this case, another SIM analysis using masses at m/z 257, 259, 320 and either m/a 328 or m/z 322 can be performed. The masses at m/z 257 and m/z 259 are indicative of the loss of one chlorine and one carbonyl group from 2,3,7,8–TCDD. If masses m/z 257 and m/z 259 give a chlorine isotope ratio that agrees to within ±10% of the same cluster in the calibration standards, then the presence of TCDD can be confirmed. Co-eluting DDD, DDE, and PCB residues can be confirmed, but will require another injection using the appropriate SIM masses or full repetitive mass scans. If the response for $^{37}Cl_4$ 2,3,7,8–TCDD at m/z 328 is too large, PCB contamination is suspected and can be confirmed by examining the response at both m/z 326 and m/z 328. The $^{37}Cl_4$ 2,3,7,8–TCDD internal standard gives negligible response at m/z 326. These pesticide residues can be removed using the alumina column cleanup procedure.

12.9 If broad background interference restricts the sensitivity of the GC/MS analysis, the analyst should employ additional cleanup procedures and reanalyze by GC/MS.

12.10 In those circumstances where these procedures do not yield a definitive conclusion, the use of high resolution mass spectrometry is suggested.[5]

13. Calculations

13.1 Calculate the concentration of 2,3,7,8–TCDD in the sample using the response factor (RF) determined in Section 7.1.2 and Equation 2.

$$\text{Concentration } (\mu g/L) = \frac{(A)(V_t)}{(V_i)(V_s)}$$

Equation 2

where:

A_s=SIM response for 2,3,7,8–TCDD at m/z 320.

A_{is}=SIM response for the internal standard at m/z 328 or 332.

I_s=Amount of internal standard added to each extract (µg).

V_o=Volume of water extracted (L).

13.2 For each sample, calculate the percent recovery of the internal standard by comparing the area of the m/z peak measured in the sample to the area of the same peak in the calibration standard. If the recovery is below 50%, the analyst should review all aspects of his analytical technique.

13.3 Report results in µg/L without correction for recovery data. All QC data obtained should be reported with the sample results.

14. Method Performance

14.1 The method detection limit (MDL) is defined as the minimum concentration of a substance that can be measured and reported with 99% confidence that the value is above zero.[1] The MDL concentration listed in Table 1 was obtained using reagent water.[14] The MDL actually achieved in a given analysis will vary depending on instrument sensitivity and matrix effects.

14.2 This method was tested by 11 laboratories using reagent water, drinking water, surface water, and three industrial wastewaters spiked at six concentrations over the range 0.02 to 0.20 µg/L.[15] Single operator precision, overall precision, and method accuracy were found to be directly related to the concentration of the parameter and essentially independent of the sample matrix. Linear equations to describe these relationships are presented in Table 3.

References

1. 40 CFR part 136, appendix B.

2. ''Determination of TCDD in Industrial and Municipal Wastewaters,'' EPA 600/4–82–028, National Technical Information Service, PB82–196882, Springfield, Virginia 22161, April 1982.

3. Buser, H.R., and Rappe, C. ''High Resolution Gas Chromatography of the 22 Tetrachlorodibenzo-p-dioxin Isomers,'' *Analytical Chemistry, 52,* 2257 (1980).

4. ASTM Annual Book of Standards, Part 31, D3694–78. ''Standard Practices for Preparation of Sample Containers and for Preservation of Organic Constituents,'' American Society for Testing and Materials, Philadelphia.

5. Harless, R. L., Oswald, E. O., and Wilkinson, M. K. ''Sample Preparation and Gas Chromatography/Mass Spectrometry Determination of 2,3,7,8-Tetrachlorodibenzo-p-dioxin,'' *Analytical Chemistry, 52,* 1239 (1980).

6. Lamparski, L. L., and Nestrick, T. J. ''Determination of Tetra-, Hepta-, and Octachlorodibenzo-p-dioxin Isomers in Particulate Samples at Parts per Trillion Levels,'' *Analytical Chemistry, 52,* 2045 (1980).

7. Longhorst, M. L., and Shadoff, L. A. ''Determination of Parts-per-Trillion Concentrations of Tetra-, Hexa-, and Octachlorodibenzo-p-dioxins in Human Milk,'' *Analytical Chemistry, 52,* 2037 (1980).

8. ''Carcinogens—Working with Carcinogens,'' Department of Health, Education, and Welfare, Public Health Service, Center for Disease Control, National Institute for Occupational Safety and Health, Publication No. 77–206, August 1977.

9. ''OSHA Safety and Health Standards, General Industry,'' (29 CFR part 1910), Occuptional Safety and Health Administration, OSHA 2206 (Revised, January 1976).

10. ''Safety in Academic Chemistry Laboratories,'' American Chemical Society Publication, Committee on Chemical Safety, 3rd Edition, 1979.

11. Provost, L. P., and Elder, R. S., ''Interpretation of Percent Recovery Data,'' *American Laboratory, 15,* 58–63 (1983). (The value 2.44 used in the equation in Section 8.3.3 is two times the value 1.22 derived in this report.)

12. ASTM Annual Book of Standards, Part 31, D3370–76, ''Standard Practices for Sampling Water,'' American Society for Testing and Materials, Philadelphia.

13. ''Methods, 330.4 (Titrimetric, DPD–FAS) and 330.5 (Spectrophotometric DPD) for Chlorine, Total Residual,'' Methods for Chemical Analysis of Water and Wastes, EPA–600/4–79–020, U.S. Environmental Protection Agency, Environmental Monitoring and Support Laboratory, Cincinnati, Ohio 45268, March 1979.

14. Wong, A.S. et al. ''The Determination of 2,3,7,8–TCDD in Industrial and Municipal Wastewaters, Method 613, Part 1—Development and Detection Limits,'' G. Choudhay, L. Keith, and C. Ruppe, ed., Butterworth Inc., (1983).

15. ''EPA Method Study 26, Method 613: 2,3,7,8–Tetrachlorodibenzo-p-dioxin,'' EPA 600/4–84–037, National Technical Information Service, PB84–188879, Springfield, Virginia 22161, May 1984.

TABLE 1—Chromatographic Conditions and Method Detection Limit

Parameter	Retention time (min)	Method detection limit (µg/L)
2,3,7,8–TCDD	13.1	0.002

Column conditions: SP–2330 coated on a 60 m long × 0.25 mm ID glass column with hydrogen carrier gas at 40 cm/sec linear velocity, splitless injection using tetradecane. Column temperature held isothermal at 200°C for 1 min, then programmed at 8°C/min to 250 °C and held. Use of helium carrier gas will approximately double the retention time.

TABLE 2—QC Acceptance Criteria—Method 613

Parameter	Test conc. (µg/L)	Limit for s (µg/L)	Range for X (µg/L)	Range for P, P_s (%)
2,3,7,8–TCDD	0.100	0.0276	0.0523–0.1226	45–129

s=Standard deviation of four recovery measurements, in µg/L (Section 8.2.4).

X=Average recovery for four recovery measurements, in µg/L (Section 8.2.4).

P, P_s=Percent recovery measured (Section 8.3.2, Section 8.4.2).

NOTE: These criteria are based directly upon the method performance data in Table 3. Where necessary, the limits for recovery have been broadened to assure applicability of the limits to concentrations below those used to develop Table 3.

TABLE 3—METHOD ACCURACY AND PRECISION AS FUNCTIONS OF CONCENTRATION—METHOD 613

Parameter	Accuracy, as recovery, X′ (µg/L)	Single analyst, precision, s_r' (µ/L)	Overall precision, S′ (µ/g/L)
2,3,7,8-TCDD	0.86C+0.00145	$0.13\bar{X}+0.00129$	$0.19\bar{X}+0.00028$

X′=Expected recovery for one or more measurements. of a sample containing a concentration of C, in µg/L.
s_r'=Expected single analyst standard deviation of measurements at an average concentration found of $\bar{X}$, in µg/L.
S′=Expected interlaboratory standard deviation of measurements at an average concentration found of X, in µg/L.
C=True value for the concentration, in µg/L.
X=Average recovery found for measurements of samples containing a concentration of C, in µg/L.

METHOD 624—PURGEABLES

1. Scope and Application

1.1 This method covers the determination of a number of purgeable organics. The following parameters may be determined by this method:

Parameter	STORET No.	CAS No.
Benzene	34030	71–43–2
Bromodichloromethane	32101	75–27–4
Bromoform	32104	75–25–2
Bromomethane	34413	74–83–9
Carbon tetrachloride	32102	56–23–5
Chlorobenzene	34301	108–90–7
Chloroethane	34311	75–00–3
2-Chloroethylvinyl ether	34576	110–75–8
Chloroform	32106	67–66–3
Chloromethane	34418	74–87–3
Dibromochloromethane	32105	124–48–1
1,2-Dichlorobenzene	34536	95–50–1
1,3-Dichlorobenzene	34566	541–73–1
1,4-Dichlorobenzene	34571	106–46–7
1,1-Dichloroethane	34496	75–34–3
1,2-Dichloroethane	34531	107–06–2
1,1-Dichloroethane	34501	75–35–4
trans-1,2-Dichloroethene	34546	156–60–5
1,2-Dichloropropane	34541	78–87–5
cis-1,3-Dichloropropene	34704	10061–01–5
trans-1,3-Dichloropropene	34699	10061–02–6
Ethyl benzene	34371	100–41–4
Methylene chloride	34423	75–09–2
1,1,2,2-Tetrachloroethane	34516	79–34–5
Tetrachloroethene	34475	127–18–4
Toluene	34010	108–88–3
1,1,1-Trichloroethene	34506	71–55–6
1,1,2-Trichloroethene	34511	79–00–5
Trichloroethane	39180	79–01–6
Trichlorofluoromethane	34488	75–69–4
Vinyl chloride	39175	75–01–4

1.2 The method may be extended to screen samples for acrolein (STORET No. 34210, CAS No. 107–02–8) and acrylonitrile (STORET No. 34215, CAS No. 107–13–1), however, the preferred method for these two compounds in Method 603.

1.3 This is a purge and trap gas chromatographic/mass spectrometer (GC/MS) method applicable to the determination of the compounds listed above in municipal and industrial discharges as provided under 40 CFR 136.1.

1.4 The method detection limit (MDL, defined in Section 14.1)[1] for each parameter is listed in Table 1. The MDL for a specific wastewater may differ from those listed, depending upon the nature of interferences in the sample matrix.

1.5 Any modification to this method, beyond those expressly permitted, shall be considered as a major modification subject to application and approval of alternate test procedures under 40 CFR 136.4 and 136.5. Depending upon the nature of the modification and the extent of intended use, the applicant may be required to demonstrate that the modifications will produce equivalent results when applied to relevant wastewaters.

1.6 This method is restricted to use by or under the supervision of analysts experienced in the operation of a purge and trap system and a gas chromatograph/mass spectrometer and in the interpretation of mass spectra. Each analyst must demonstrate the ability to generate acceptable results with this method using the procedure described in Section 8.2.

2. Summary of Method

2.1 An inert gas is bubbled through a 5-mL water sample contained in a specially-designed purging chamber at ambient temperature. The purgeables are efficiently transferred from the aqueous phase to the vapor phase. The vapor is swept through a sorbent trap where the purgeables are trapped. After purging is completed, the trap is heated and backflushed with the inert gas to desorb the purgeables onto a gas chromatographic column. The gas chromatograph is temperature programmed to separate the purgeables which are then detected with a mass spectrometer.[2,3]

3. Interferences

3.1 Impurities in the purge gas, organic compounds outgassing from the plumbing ahead of the trap, and solvent vapors in the laboratory account for the majority of contamination problems. The analytical system must be demonstated to be free from contamination under the conditions of the analysis by running laboratory reagent blanks as described in Section 8.1.3. The use of non-Teflon plastic tubing, non-Teflon thread sealants, or flow controllers with rubber components in the purge and trap system should be avoided.

3.2 Samples can be contaminated by diffusion of volatile organics (particularly fluorocarbons and methylene chloride) through the septum seal into the sample during shipment and storage. A field reagent blank prepared from reagent water and carried through the sampling and handling protocol can serve as a check on such contamination.

3.3 Contamination by carry-over can occur whenever high level and low level samples are sequentially analyzed. To reduce carry-over, the purging device and sample syringe must be rinsed with reagent water between sample analyses. Whenever an unusually concentrated sample is encountered, it should be followed by an analysis of reagent water to check for cross contamination. For samples containing large amounts of water-soluble materials, suspended solids, high boiling compounds or high pureeable levels, it may be necessary to wash the purging device with a detergent solution, rinse it with distilled water, and then dry it in a 105 ° C oven between analyses. The trap and other parts of the system are also subject to contamination; therefore, frequent bakeout and purging of the entire system may be required.

4. Safety

4.1 The toxicity or carcinogenicity of each reagent used in this method has not been precisely defined; however, each chemical compound should be treated as a potential health hazard. From this viewpoint, exposure to these chemicals must be reduced to the lowest possible level by whatever means available. The laboratory is responsible for maintaining a current awareness file of OSHA regulations regarding the safe handling of the chemicals specified in this methmd. A reference file of material data handling sheets should also be made available to all personnel involved in the chemical analysis. Additional references to laboratory safety are available and have been identified[4–6] for the information of the analyst.

4.2. The following parameters covered by this method have been tentatively classified as known or suspected, human or mammalian carcinogens: benzene, carbon tetrachloride, chloroform, 1,4-dichlorobenzene, and vinyl chloride. Primary standards of these toxic compounds should be prepared in a hood. A NIOSH/MESA approved toxic gas respirator should be worn when the analyst handles high concentrations of these toxic compounds.

5. Apparatus and Materials

5.1 Sampling equipment, for discrete sampling.

5.1.1 Vial—25-mL capacity or larger, equipped with a screw cap with a hole in the

center (Pierce #13075 or equivalent). Detergent wash, rinse with tap and distilled water, and dry at 105 °C before use.

5.1.2 Septum—Teflon-faced silicane (Pierce #12722 or equivalent). Detergent wash, rinse with tap and distilled water, and dry at 105 °C for 1 h before use.

5.2 Purge and trap system—The purge and trap system consists of three separate pieces of equipment: A purging device, trap, and desorber. Several complete systems are now commercially available.

5.2.1 The purging device must be designed to accept 5-mL samples with a water column at least 3 cm deep. The gaseous head space between the water column and the trap must have a total volume of less than 15 mL. The purge gas must pass though the water column as finely divided bubbles with a diameter of less than 3 mm at the origin. The purge gas must be introduced no more than 5 mm from the base of the water column. The purging device illustrated in Figure 1 meets these design criteria.

5.2.2 The trap must be at least 25 cm long and have an inside diameter of at least 0.105 in. The trap must be packed to contain the following minimum lengths of adsorbents: 1.0 cm of methyl silicone coated packing (Section 6.3.2), 15 cm of 2,6-dyphenylene oxide polymer (Section 6.3.1), and 8 cm of silica gel (Section 6.3.3). The minimum specifications for the trap are illustrated in Figure 2.

5.2.3 The desorber should be capable of rapidly heating the trap to 180 °C. The polymer section of the trap should not be heated higher than 180 °C and the remaining sections should not exceed 200 °C. The desorber illustrated in Figure 2 meets these design criteria.

5.2.4 The purge and trap system may be assembled as a separate unit or be coupled to a gas chromatograph as illustrated in Figures 3 and 4.

5.3 GC/MS system:

5.3.1 Gas chromatograph—An analytical system complete with a temperature programmable gas chromatograph suitable for on-column injection and all required accessories including syringes, analytical columns, and gases.

5.3.2 Column—6 ft long x 0.1 in ID stainless steel or glass, packed with 1% SP–1000 on Carbopack B (60/80 mesh) or equivalent. This column was used to develop the method performance statements in Section 14. Guidelines for the use of alternate column packings are provided in Section 11.1.

5.3.3 Mass spectrometer—Capable of scanning from 20 to 260 amu every 7 s or less, utilizing 70 V (nominal) electron energy in the electron impact ionization mode, and producing a mass spectrum which meets all the criteria in Table 2 when 50 ng of 4-bromofluorobenzene (BFB) is injected through the GC inlet.

5.3.4 GC/MS interface—Any GC to MS interface that gives acceptable calibration points at 50 ng or less per injection for each of the parameters of interest and achieves all acceptable performance criteria (Section 10) may be used. GC to MS interfaces constructed of all glass or glass-lined materials are recommended. Glass can be deactivated by silanizing with dichlorodimethylsilane.

5.3.5 Data system—A computer system must be interfaced to the mass spectrometer that allows the continuous acquisition and storage on machine-readable media of all mass spectra obtained throughout the duration of the chromatographic program. The computer must have software that allows searching any GC/MS data file for specific m/z (masses) and plotting such m/z abundances versus time or scan number. This type of plot is defined as an Extracted Ion Current Profile (EICP). Software must also be available that allows integrating the abundance in any EICP between specified time or scan number limits.

5.4 Syringes—5-mL, glass hypodermic with Luerlok tip (two each), if applicable to the purging device.

5.5 Micro syringes—25-µL, 0.006 in. ID needle.

5.6 Syringe valve—2-way, with Luer ends (three each).

5.7 Syringe—5-mL, gas-tight with shut-off valve.

5.8 Bottle—15-mL, screw-cap, with Teflon cap liner.

5.9 Balance—Analytical, capable of accurately weighing 0.0001 g.

6. Reagents

6.1 Reagent water—Reagent water is defined as a water in which an interferent is not observed at the MDL of the parameters of interest.

6.1.1 Reagent water can be generated by passing tap water through a carbon filter bed containing about 1 lb of activated carbon (Filtrasorb–300, Calgon Corp., or equivalent).

6.1.2 A water purification system (Millipore Super–Q or equivalent) may be used to generate reagent water.

6.1.3 Reagent water may also be prepared by boiling water for 15 min. Subsequently, while maintaining the temperature at 90 °C, bubble a contaminant-free inert gas through the water for 1 h. While still hot, transfer the water to a narrow mouth screw-cap bottle and seal with a Teflon-lined septum and cap.

6.2 Sodium thiosulfate—(ACS) Granular.

6.3 Trap materials:

6.3.1 2,6-Diphenylene oxide polymer—Tenax, (60/80 mesh), chromatographic grade or equivalent.

6.3.2 Methyl silicone packing—3% OV–1 on Chromosorb-W (60/80 mesh) or equivalent.

6.3.3 Silica gel—35/60 mesh, Davison, grade-15 or equivalent.

6.4 Methanol—Pesticide quality or equivalent.

6.5 Stock standard solutions—Stock standard solutions may be prepared from pure standard materials or purchased as certified solutions. Prepare stock standard solutions in methanol using assayed liquids or gases as appropriate. Because of the toxicity of some of the compounds, primary dilutions of these materials should be prepared in a hood. A NIOSH/MESA approved toxic gas respirator should be used when the analyst handles high concentrations of such materials.

6.5.1 Place about 9.8 mL of methanol into a 10-mL ground glass stoppered volumetric flask. Allow the flask to stand, unstoppered, for about 10 min or until all alcohol wetted surfaces have dried. Weigh the flask to the nearest 0.1 mg.

6.5.2 Add the assayed reference material:

6.5.2.1 Liquids—Using a 100-µL syringe, immediately add two or more drops of assayed reference material to the flask, then reweigh. Be sure that the drops fall directly into the alcohol without contacting the neck of the flask.

6.5.2.2 Gases—To prepare standards for any of the four halocarbons that boil below 30 °C (bromomethane, chloroethane, chloromethane, and vinyl chloride), fill a 5-mL valved gas-tight syringe with the reference standard to the 5.0-mL mark. Lower the needle to 5 mm above the methanol meniscus. Slowly introduce the reference standard above the surface of the liquid (the heavy gas will rapidly dissolve in the methanol).

6.5.3 Reweigh, dilute to volume, stopper, then mix by inverting the flask several times. Calculate the concentration in µg/µL from the net gain in weight. When compound purity is assayed to be 96% or greater, the weight may be used without correction to calculate the concentration of the stock standard. Commercially prepared stock standards may be used at any concentration if they are certified by the manufacturer or by an independent source.

6.5.4 Transfer the stock standard solution into a Teflon-sealed screw-cap bottle. Store, with minimal headspace, at −10 to −20 °C and protect from light.

6.5.5 Prepare fresh standards weekly for the four gases and 2-chloroethylvinyl ether. All other standards must be replaced after one month, or sooner if comparison with check standards indicates a problem.

6.6 Secondary dilution standards—Using stock solutions, prepare secondary dilution standards in methanol that contain the compounds of interest, either singly or mixed together. The secondary dilution standards should be prepared at concentrations such that the aqueous calibration standards prepared in Section 7.3 will bracket the working range of the analytical system. Secondary dilution standards should be stored with minimal headspace and should be checked frequently for signs of degradation or evaporation, especially just prior to preparing calibration standards from them.

6.7 Surrogate standard spiking solution—Select a minimum of three surrogate compounds from Table 3. Prepare stock standard solutions for each surrogate standard in methanol as described in Section 6.5. Prepare a surrogate standard spiking solution from these stock standards at a concentration of 15 µg/mL in water. Store the solutions at 4 °C in Teflon-sealed glass containers with a minimum of headspace. The solutions should be checked frequently for stability. The addition of 10 µL of this solution of 5 mL of sample or standard is equivalent to a concentration of 30 µg/L of each surrogate standard.

6.8 BFB Standard—Prepare a 25 µg/mL solution of BFB in methanol.

6.9 Quality control check sample concentrate—See Section 8.2.1.

7. Calibration

7.1 Assemble a purge and trap system that meets the specifications in Section 5.2. Condition the trap overnight at 180 °C by backflushing with an inert gas flow of at least 20 mL/min. Condition the trap for 10 min once daily prior to use.

7.2 Connect the purge and trap system to a gas chromatograph. The gas chromatograph must be operated using temperature and flow rate conditions equivalent to those given in Table 1.

7.3 Internal standard calibration procedure—To use this approach, the analyst must select three or more internal standards that are similar in analytical behavior to the compounds of interest. The analyst must further demonstrate that the measurement of the internal standard is not affected by method or matrix interferences. Some recommended internal standards are listed in Table 3.

7.3.1 Prepare calibration standards at a minimum of three concentration levels for each parameter by carefully adding 20.0 µL of one or more secondary dilution standards to 50, 250, or 500 mL of reagent water. A 25-µL syringe with a 0.006 in. ID needle should be used for this operation. One of the calibration standards should be at a concentration near, but above, the MDL (Table 1) and the other concentrations should correspond to the expected range of concentrations found in real samples or should define the working range of the GC/MS system. These aqueous standards can be stored up to 24 h, if held in sealed vials with zero headspace as described in Section 9.2. If not so stored, they must be discarded after 1 h.

7.3.2 Prepare a spiking solution containing each of the internal standards using the procedures described in Sections 6.5 and 6.6. It is recommended that the secondary dilution standard be prepared at a concentration

of 15 µg/mL of each internal standard compound. The addition of 10 µL of this standard to 5.0 mL of sample or calibration standard would be equivalent to 30 µg/L.

7.3.3 Analyze each calibration standard according to Section 11, adding 10 µL of internal standard spiking solution directly to the syringe (Section 11.4). Tabulate the area response of the characteristic m/z against concentration for each compound and internal standard, and calculate response factors (RF) for each compound using Equation 1.

$$RF = \frac{(A_s)(C_{is})}{(A_{is})(C_s)}$$

Equation 1

where:

A_s=Area of the characteristic m/z for the parameter to be measured.

A_{is}=Area of the characteristic m/z for the inernal standard.

C_{is}=Concentration of the internal standard.

C_s=Concentration of the parameter to be measured.

If the RF value over the working range is a constant (<35% RSD), the RF can be assumed to be invariant and the average RF can be used for calculations. Alternatively, the results can be used to plot a calibration curve of response ratios, A_s/A_{is}, vs. RF.

7.4 The working calibration curve or RF must be verified on each working day by the measurement of a QC check sample.

7.4.1 Prepare the QC check sample as described in Section 8.2.2.

7.4.2 Analyze the QC check sample according to the method beginning in Section 10.

7.4.3 For each parameter, compare the response (Q) with the corresponding calibration acceptance criteria found in Table 5. If the responses for all parameters of interest fall within the designated ranges, analysis of actual samples can begin. If any individual Q falls outside the range, proceed according to Section 7.4.4.

NOTE: The large number of parameters in Table 5 present a substantial probability that one or more will not meet the calibration acceptance criteria when all parameters are analyzed.

7.4.4 Repeat the test only for those parameters that failed to meet the calibration acceptance criteria. If the response for a parameter does not fall within the range in this second test, a new calibration curve or RF must be prepared for that parameter according to Section 7.3.

8. Quality Control

8.1 Each laboratory that uses this method is required to operate a formal quality control program. The minimum requirements of this program consist of an initial demonstration of laboratory capability and an ongoing analysis of spiked samples to evaluate and document data quality. The laboratory must maintain records to document the quality of data that is generated. Ongoing data quality checks are compared with established performance criteria to determine if the results of analyses meet the performance characteristics of the method. When results of sample spikes indicate atypical method performance, a quality control check standard must be analyzed to confirm that the measurements were performed in an in-control mode of operation.

8.1.1 The analyst must make an initial, one-time, demonstration of the ability to generate acceptable accuracy and precision with this method. This ability is established as described in Section 8.2.

8.1.2 In recognition of advances that are occurring in chromatography, the analyst is permitted certain options (detailed in Section 11.1) to improve the separations or lower the cost of measurements. Each time such a modification is made to the method, the analyst is required to repeat the procedure in Section 8.2.

8.1.3 Each day, the analyst must analyze a reagent water blank to demonstrate that interferences from the analytical system are under control.

8.1.4 The laboratory must, on an ongoing basis, spike and analyze a minimum of 5% of all samples to monitor and evaluate laboratory data quality. This procedure is described in Section 8.3.

8.1.5 The laboratory must, on an ongoing basis, demonstrate through the analyses of quality control check standards that the operation of the measurement system is in control. This procedure is described in Section 8.4. The frequency of the check standard analyses is equivalent to 5% of all samples analyzed but may be reduced if spike recoveries from samples (Section 8.3) meet all specified quality control criteria.

8.1.6 The laboratory must spike all samples with surrogate standards to monitor continuing laboratory performance. This procedure is described in Section 8.5.

8.1.7 The laboratory must maintain performance records to document the quality of data that is generated. This procedure is described in Section 8.6.

8.2 To establish the ability to generate acceptable accuracy and precision, the analyst must perform the following operations.

8.2.1 A quality control (QC) check sample concentrate is required containing each parameter of interest at a concentration of 10 µg/mL in methanol. The QC check sample concentrate must be obtained from the U.S. Environmental Protection Agency, Environmental Monitoring and Support Laboratory in Cincinnati, Ohio, if available. If not available from that source, the QC check sample concentrate must be obtained from another external source. If not available from either

source above, the QC check sample concentrate must be prepared by the laboratory using stock standards prepared independently from those used for calibration.

8.2.2 Prepare a QC check sample to contain 20 µg/L of each parameter by adding 200 µL of QC check sample concentrate to 100 mL of reagent water.

8.2.3 Analyze four 5-mL aliquots of the well-mixed QC check sample according to the method beginning in Section 10.

8.2.4 Calculate the average recovery ($\bar{X}$) in µg/L, and the standard deviation of the recovery (s) in µg/L, for each parameter of interest using the four results.

8.2.5 For each parameter compare s and $\bar{X}$ with the corresponding acceptance criteria for precision and accuracy, respectively, found in Table 5. If s and $\bar{X}$ for all parameters of interest meet the acceptance criteria, the system performance is acceptable and analysis of actual samples can begin. If any individual s exceeds the precision limit or any individual $\bar{X}$ falls outside the range for accuracy, the system performance is unacceptable for that parameter.

NOTE: The large number of parameters in Table 5 present a substantial probability that one or more will fail at least one of the acceptance criteria when all parameters are analyzed.

8.2.6 When one or more of the parameters tested fail at least one of the acceptance criteria, the analyst must proceed according to Section 8.2.6.1 or 8.2.6.2.

8.2.6.1 Locate and correct the source of the problem and repeat the test for all parameters of interest beginning with Section 8.2.3.

8.2.6.2 Beginning with Section 8.2.3, repeat the test only for those parameters that failed to meet criteria. Repeated failure, however, will confirm a general problem with the measurement system. If this occurs, locate and correct the source of the problem and repeat the test for all compounds of interest beginning with Section 8.2.3.

8.3 The laboratory must, on an ongoing basis, spike at least 5% of the samples from each sample site being monitored to assess accuracy. For laboratories analyzing 1 to 20 samples per month, at least one spiked sample per month is required.

8.3.1 The concentration of the spike in the sample should be determined as follows:

8.3.1.1 If, as in compliance monitoring, the concentration of a specific parameter in the sample is being checked against a regulatory concentration limit, the spike should be at that limit or 1 to 5 times higher than the background concentration determined in Section 8.3.2, whichever concentration would be larger.

8.3.1.2 If the concentration of a specific parameter in the sample is not being checked against a limit specific to that parameter, the spike should be at 20 µg/L or 1 to 5 times higher than the background concentration determined in Section 8.3.2, whichever concentration would be larger.

8.3.2 Analyze one 5-mL sample aliquot to determine the background concentration (B) of each parameter. If necessary, prepare a new QC check sample concentrate (Section 8.2.1) appropriate for the background concentrations in the sample. Spike a second 5-mL sample aliquot with 10 µL of the QC check sample concentrate and analyze it to determine the concentration after spiking (A) of each parameter. Calculate each percent recovery (P) as 100(A–B)%/T, where T is the known true value of the spike.

8.3.3 Compare the percent recovery (P) for each parameter with the corresponding QC acceptance criteria found in Table 5. These acceptance criteria wer calculated to include an allowance for error in measurement of both the background and spike concentrations, assuming a spike to background ratio of 5:1. This error will be accounted for to the extent that the analyst's spike to background ratio approaches 5:1.[7] If spiking was performed at a concentration lower than 20 µg/L, the analyst must use either the QC acceptance criteria in Table 5, or optional QC acceptance criteria calculated for the specific spike concentration. To calculate optional acceptance criteria for the recoveryof a parameter: (1) Calculate accuracy (X′) using the equation in Table 6, substituting the spike concentration (T) for C; (2) calculate overall precision (S′) using the equation in Table 6, substituting X′ for $\bar{X}$; (3) calculate the range for recovery at the spike concentration as (100 X′/T) (±2.44(100 S′/T)%.[7]

8.3.4 If any individual P falls outside the designated range for recovery, that parameter has failed the acceptance criteria. A check standard containing each parameter that failed the criteria must be analyzed as described in Section 8.4.

8.4 If any parameter fails the acceptance criteria for recovery in Section 8.3, a QC check standard containing each parameter that failed must be prepared and analyzed.

NOTE: The frequency for the required anlaysis of a QC check standard will depend upon the number of parameters being simultaneously tested, the complexity of the sample matrix, and the performance of the laboratory. If the entire list of parameters in Table 5 must be measured in the sample in Section 8.3, the probability that the analysis of a QC check standard will be required is high. In this case the QC check standard should be routinely analyzed with the spiked sample.

8.4.1 Prepare the QC check standard by adding 10 µL of QC check sample concentrate (Section 8.2.1 or 8.3.2) to 5 mL of reagent water. The QC check standard needs only to

contain the parameters that failed criteria in the test in Section 8.3.

8.4.2 Analyze the QC check standard to determine the concentration measured (A) of each parameter. Calculate each percent recovery (P_S) as 100 (A/T)%, where T is the true value of the standard concentration.

8.4.3 Compare the percent recovery (P_S) for each parameter with the corresponding QC acceptance criteria found in Table 5. Only parameters that failed the test in Section 8.3 need to be compared with these criteria. If the recovery of any such parameter falls outside the designated range, the laboratory performance for that parameter is judged to be out of control, and the problem must be immediately identified and corrected. The analytical result for that parameter in the unspiked sample is suspect and may not be reported for regulatory compliance purposes.

8.5 As a quality control check, the laboratory must spike all samples with the surrogate standard spiking solutions as described in Section 11.4, and calculate the percent recovery of each surrogate compound.

8.6 As part of the QC program for the laboratory, method accuracy for wastewater samples must be assessed and records must be maintained. After the analysis of five spiked wastewater samples as in Section 8.3, calculate the average percent recovery ($\bar{P}$) and the standard deviation of the percent recovery (s_p). Express the accuracy assessment as a percent recovery interval from $\bar{P}-2s_p$ to $\bar{P} + 2s_p$. If $\bar{P}$=90% and s_p=10%, for example, the accuracy interval is expressed as 70–110%. Update the accuracy assessment for each parameter a regular basis (e.g. after each five to ten new accuracy measurements).

8.7 It is recommended that the laboratory adopt additional quality assurance practices for use with this method. The specific practices that are most productive depend upon the needs of the laboratory and the nature of the samples. Field duplicates may be analyzed to assess the precision of the environmental measurements. Whenever possible, the laboratory should analyze standard reference materials and participate in relevant performance evaluation studies.

9. Sample Collection, Preservation, and Handling

9.1 All samples must be iced or refrigerated from the time of collection until analysis. If the sample contains residual chlorine, add sodium thiosulfate preservative (10 mg/40 mL is sufficient for up to 5 ppm Cl_2) to the empty sample bottle just prior to shipping to the sampling site. EPA Methods 330.4 and 330.5 may be used for measurement of residual chlorine.[8] Field test kits are available for this purpose.

9.2 Grab samples must be collected in glass containers having a total volume of at least 25 mL. Fill the sample bottle just to overflowing in such a manner that no air bubbles pass through the sample as the bottle is being filled. Seal the bottle so that no air bubbles are entrapped in it. If preservative has been added, shake vigorously for 1 min. Maintain the hermetic seal on the sample bottle until time of analysis.

9.3 Experimental evidence indicates that some aromatic compounds, notably benzene, toluene, and ethyl benzene are susceptible to rapid biological degradation under certain environmental conditions.[3] Refrigeration alone may not be adequate to preserve these compounds in wastewaters for more than seven days. For this reason, a separate sample should be collected, acidified, and analyzed when these aromatics are to be determined. Collect about 500 mL of sample in a clean container. Adjust the pH of the sample to about 2 by adding 1+1 HCl while stirring vigorously, Check pH with narrow range (1.4 to 2.8) pH paper. Fill a sample container as described in Section 9.2.

9.4 All samples must be analyzed within 14 days of collection.[3]

10. Daily GC/MS Performance Tests

10.1 At the beginning of each day that analyses are to be performed, the GC/MS system must be checked to see if acceptable performance criteria are achieved for BFB.[9] The performance test must be passed before any samples, blanks, or standards are analyzed, unless the instrument has met the DFTPP test described in Method 625 earlier in the day.[10]

10.2 These performance tests require the following instrumental parameters:

Electron Energy: 70 V (nominal)

Mass Range: 20 to 260 amu

Scan Time: To give at least 5 scans per peak but not to exceed 7 s per scan.

10.3 At the beginning of each day, inject 2 µL of BFB solution directly on the column. Alternatively, add 2 µL of BFB solution to 5.0 mL of reagent water or standard solution and analyze the solution according to section 11. Obtain a background-corrected mass spectrum of BFB and confirm that all the key m/z criteria in Table 2 are achieved. If all the criteria are not achieved, the analyst must retune the mass spectrometer and repeat the test until all criteria are achieved.

11. Sample Purging and Gas Chromatography

11.1 Table 1 summarizes the recommended operating conditions for the gas chromatograph. Included in this table are retention times and MDL that can be achieved under these conditions. An example of the separations achieved by this column is shown in Figure 5. Other packed columns or chromatographic conditions may be used if the requirements of Section 8.2 are met.

11.2 After achieving the key m/z abundance criteria in Section 10, calibrate the system daiy as described in Section 7.

11.3 Adjust the purge gas (helium) flow rate to 40 mL/min. Attach the trap inlet to the purging device, and set the purge and trap system to purge (Figure 3). Open the syringe valve located on the purging device sample introduction needle.

11.4 Allow the sample to come to ambient temperature prior to introducing it into the syringe. Remove the plunger from a 5-mL syringe and attach a closed syringe valve. Open the sample bottle (or standard) and carefully pour the sample into the syringe barrel to just short of overflowing. Replace the syringe plunger and compress the sample. Open the syringe valve and vent any residual air while adjusting the sample volume to 5.0 mL. Since this process of taking an aliquot destroys the validity of the sample for future analysis, the analyst should fill a second syringe at this time to protect against possible loss of data. Add 10.0 µL of the surrogate spiking solution (Section 6.7) and 10.0 µL of the internal standard spiking solution (Section 7.3.2) through the valve bore, then close the valve. The surrogate and internal standards may be mixed and added as a single spiking solution.

11.5 Attach the syringe-syringe valve assembly to the syringe valve on the purging device. Open the syringe valves and inject the sample into the purging chamber.

11.6 Close both valves and purge the sample for 11.0±0.1 min at ambient temperature.

11.7 After the 11-min purge time, attach the trap to the chromatograph, adjust the purge and trap system to the desorb mode (Figure 4), and begin to temperature program the gas chromatograph. Introduce the trapped materials to the GC column by rapidly heating the trap to 180 °C while backflushing the trap with an inert gas between 20 and 60 mL/min for 4 min. If rapid heating of the trap cannot be achieved, the GC cloumn must be used as a secondary trap by cooling it to 30 °C (subambient temperature, if problems persist) instead of the initial program temperature of 45 °C.

11.8 While the trap is being desorbed into the gas chromatograph, empty the purging chamber using the sample introduction syringe. Wash the chamber with two 5-mL flushes of reagent water.

11.9 After desorbing the sample for 4 min, recondition the trap by returning the purge and trap system to the purge mode. Wait 15 s then close the syringe valve on the purging device to begin gas flow through the trap. The trap temperature should be maintained at 180 °C. After approximately 7 min, turn off the trap heater and open the syringe valve to stop the gas flow through the trap. When the trap is cool, the next sample can be analyzed.

11.10 If the response for any m/z exceeds the working range of the system, prepare a dilution of the sample with reagent water from the aliquot in the second syringe and reanalyze.

12. Qualitative Identification

12.1 Obtain EICPs for the primary m/z (Table 4) and at least two secondary masses for each parameter of interest. The following criteria must be met to make a qualitative identification:

12.1.1 The characteristic masses of each parameter of interest must maximize in the same or within one scan of each other.

12.1.2 The retention time must fall within ±30 s of the retention time of the authentic compound.

12.1.3 The relative peak heights of the three characteristic masses in the EICPs must fall within ± 20% of the relative intensities of these masses in a reference mass spectrum. The reference mass spectrum can be obtained from a standard analyzed in the GC/MS system or from a reference library.

12.2 Structural isomers that have very similar mass spectra and less than 30 s difference in retention time, can be explicitly identified only if the resolution between authentic isomers in a standard mix is acceptable. Acceptable resolution is achieved if the baseline to valley height between the isomers is less than 25% of the sum of the two peak heights. Otherwise, structural isomers are identified as isomeric pairs.

13. Calculations

13.1 When a parameter has been identified, the quantitation of that parameter should be based on the integrated abundance from the EICP of the primary characteristic m/z given in Table 4. If the sample produces an interference for the primary m/z, use a secondary characteristic m/z to quantitate.

Calculate the concentration in the sample using the response factor (RF) determined in Section 7.3.3 and Equation 2.

$$\text{Concentration } (\mu g/L) = \frac{(A_s)(C_{is})}{(A_{is})(RF)}$$

Equation 2

where:

A_s=Area of the characteristic m/z for the parameter or surrogate standard to be measured.

A_{is}=Area of the characteristic m/z for the internal standard.

C_{is}=Concentration of the internal standard.

13.2 Report results in µg/L without correction for recovery data. All QC data obtained should be reported with the sample results.

14. Method Performance

14.1 The method detection limit (MDL) is defined as the minimum concentration of a substance that can be measured and reported with 99% confidence that the value is above zero.[1] The MDL concentrations listed in Table 1 were obtained using reagent water.[11] Similar results were achieved using representative wastewaters. The MDL actually achieved in a given analysis will vary depending on instrument sensitivity and matrix effects.

14.2 This method was tested by 15 laboratories using reagent water, drinking water, surface water, and industrial wastewaters spiked at six concentrations over the range 5–600 µg/L.[12] Single operator precision, overall precision, and method accuracy were found to be directly related to the concentration of the parameter and essentially independent of the sample matrix. Linear equations to describe these relationships are presented in Table 5.

REFERENCES

1. 40 CFR part 136, appendix B.

2. Bellar, T.A., and Lichtenberg, J.J. ''Determining Volatile Organics at Microgram-per-Litre Levels by Gas Chromatography,'' *Journal American Water Works Association, 66,* 739 (1974).

3. Bellar, T.A., and Lichtenberg, J.J. ''Semi-Automated Headspace Analysis of Drinking Waters and Industrial Waters for Purgeable Volatile Organic Compounds, '' Measurement of Organic Pollutants in Water and Wastewater, C.E. Van Hall, editor, American Society for Testing and Materials, Philadelphia, PA. Special Technical Publication 686, 1978.

4. ''Carcinogens—Working With Carcinogens,'' Department of Health, Education, and Welfare, Public Health Service, Center for Disease Control, National Institute for Occupational Safety and Health, Publication No. 77–206, August 1977.

5. ''OSHA Safety and Health Standards, General Industry,'' (29 CFR part 1910), Occupational Safety and Health Administration, OSHA 2206 (Revised, January 1976).

6. ''Safety in Academic Chemistry Laboratories,'' American Chemical Society Publication, Committee on Chemical Safety, 3rd Edition, 1979.

7. Provost, L.P., and Elder, R.S. ''Interpretation of Percent Recovery Data,'' *American Laboratory, 15,* 58–63 (1983). (The value 2.44 used in the equation in Section 8.2.3 is two times the value 1.22 derived in this report.)

8. ''Methods 330.4 (Titrimetric, DPD-FAS) and 330.5 (Spectrophotometric, DPD) for Chlorine, Total Residual,'' Methods for Chemical Analysis of Water and Wastes, EPA–600/4–79–020, U.S. Environmental Protection Agency, Environmental Monitoring and Support Laboratory, Cincinnati, Ohio 45268, March 1979.

9. Budde, W.L., and Eichelberger, J.W. ''Performance Tests for the Evaluation of Computerized Eas Chromatography/Mass Spectrometry Equipment and Laboratories,'' EPA–600/4–80–025, U.S. Environmental Protection Agency, Environmental Monitoring and Support Laboratory, Cincinnati, Ohio 45268, April 1980.

10. Eichelberger, J.W., Harris, L.E., and Budde, W.L. ''Reference Compound to Calibrate Ion Abundance Measurement in Gas Chromatography—Mass Spectrometry Systems,'' *Analytical Chemistry, 47,* 995–1000 (1975).

11. ''Method Detection Limit for Methods 624 and 625,'' Olynyk, P., Budde, W.L., and Eichelberger, J.W. Unpublished report, May 14, 1980.

12. ''EPA Method Study 29 EPA Method 624—Purgeables,'' EPA 600/4–84–054, National Technical Information Service, PB84–209915, Springfield, Virginia 22161, June 1984.

13.''Method Performance Data for Method 624,'' Memorandum from R. Slater and T. Pressley, U.S. Environmental Protection Agency, Environmental Monitoring and Support Laboratory, Cincinnati, Ohio 45268, January 17, 1984.

TABLE 1—CHROMATOGRAPHIC CONDITIONS AND METHOD DETECTION LIMITS

Parameter	Retention time (min)	Method detection limit (µg/L)
Chloromethane	2.3	nd
Bromomethane	3.1	nd
Vinyl chloride	3.8	nd
Chloroethane	4.6	nd
Methylene chloride	6.4	2.8
Trichlorofluoromethane	8.3	nd
1,1-Dichloroethene	9.0	2.8
1,1-Dichloroethane	10.1	4.7
trans-1,2-Dichloroethene	10.8	1.6
Chloroform	11.4	1.6
1,2-Dichloroethane	12.1	2.8
1,1,1-Trichloroethane	13.4	3.8
Carbon tetrachloride	13.7	2.8
Bromodichloromethane	14.3	2.2
1,2-Dichloroproane	15.7	6.0
cis-1,3-Dichloropropene	15.9	5.0
Trichloroethene	16.5	1.9
Benzene	17.0	4.4
Dibromochloromethane	17.1	3.1
1,1,2-Trichloroethane	17.2	5.0
trans-1,3-Dichloropropene	17.2	nd
2-Chloroethylvinlyl ether	18.6	nd
Bromoform	19.8	4.7
1,1,2,2-Tetrachloroethane	22.1	6.9
Tetrachloroethene	22.2	4.1
Toluene	23.5	6.0
Chlorobenzene	24.6	6.0
Ethyl benzene	26.4	7.2
1,3-Dichlorobenzene	33.9	nd
1,2-Dichlorobenzene	35.0	nd

TABLE 1—CHROMATOGRAPHIC CONDITIONS AND METHOD DETECTION LIMITS—Continued

Parameter	Retention time (min)	Method detection limit (μg/L)
1,4-Dichlorobenzene	35.4	nd

Column conditions: Carbopak B (60/80 mesh) coated with 1% SP–1000 packed in a 6 ft by 0.1 in. ID glass column with helium carrier gas at 30 mL/min. flow rate. Column temperature held at 45°C for 3 min., then programmed at 8°C/min. to 220°C and held for 15 min.
nd=not determined.

TABLE 2—BFB KEY M/Z ABUNDANCE CRITERIA

Mass	m/z Abundance criteria
50	15 to 40% of mass 95.
75	30 to 60% of mass 95.
95	Base Peak, 100% Relative Abundance.
96	5 to 9% of mass 95.
173	<2% of mass 174.
174	>50% of mass 95.
175	5 to 9% of mass 174.
176	>95% but < 101% of mass 174.
177	5 to 9% of mass 176.

TABLE 3—SUGGESTED SURROGATE AND INTERNAL STANDARDS

Compound	Retention time (min)[a]	Primary m/z	Secondary masses
Benzene d-6	17.0	84	
4-Bromofluorobenzene	28.3	95	174, 176
1,2-Dichloroethane d-4	12.1	102	
1,4-Difluorobenzene	19.6	114	63, 88
Ethylbenzene d-5	26.4	111	
Ethylbenzene d-10	26.4	98	
Fluorobenzene	18.4	96	70
Pentafluorobenzene	23.5	168	
Bromochloromethane	9.3	128	49, 130, 51
2-Bromo-1-chloropropane	19.2	77	79, 156
1, 4-Dichlorobutane	25.8	55	90, 92

[a]For chromatographic conditions, see Table 1.

TABLE 4—CHARACTERISTIC MASSES FOR PURGEABLE ORGANICS

Parameter	Primary	Secondary
Chloromethane	50	52.
Bromomethane	94	96.
Vinyl chloride	62	64.
Chloroethane	64	66.
Methylene chloride	84	49, 51, and 86.
Trichlorofluoromethane	101	103.
1,1-Dichloroethene	96	61 and 98.
1,1-Dichloroethane	63	65, 83, 85, 98, and 100.
trans-1,2-Dichloroethene	96	61 and 98.
Chloroform	83	85.
1,2-Dichloroethane	98	62, 64, and 100.
1,1,1-Trichloroethane	97	99, 117, and 119.
Carbon tetrachloride	117	119 and 121.
Bromodichloromethane	127	83, 85, and 129.
1,2-Dichloropropane	112	63, 65, and 114.
trans-1,3-Dichloropropene	75	77.
Trichloroethene	130	95, 97, and 132.
Benzene	78	
Dibromochloromethane	127	129, 208, and 206.
1,1,2-Trichloroethane	97	83, 85, 99, 132, and 134.
cis-1,3-Dichloropropene	75	77.
2-Chloroethylvinyl ether	106	63 and 65.
Bromoform	173	171, 175, 250, 252, 254, and 256.
1,1,2,2-Tetrachloroethane	168	83, 85, 131, 133, and 166.
Tetrachloroethene	164	129, 131, and 166.
Toluene	92	91.
Chlorobenzene	112	114.
Ethyl benzene	106	91.
1,3-Dichlorobenzene	146	148 and 113.
1,2-Dichlorobenzene	146	148 and 113.
1,4-Dichlorobenzene	146	148 and 113.

TABLE 5—CALIBRATION AND QC ACCEPTANCE CRITERIA—METHOD 624[a]

Parameter	Range for Q (μ/g/L)	Limit for s (μ/g/L)	Range for $\bar{X}$ (μ/g/L)	Range for P, P_s (%)
Benzene	12.8 – 27.2	6.9	15.2 – 26.0	37 – 151
Bromodichloromethane	13.1 – 26.9	6.4	10.1 – 28.0	35 – 155
Bromoform	14.2 – 25.8	5.4	11.4 – 31.1	45 – 169
Bromomethane	2.8 – 37.2	17.9	D – 41.2	D – 242
Carbon tetrachloride	14.6 – 25.4	5.2	17.2 – 23.5	70 – 140
Chlorobenzene	13.2 – 26.8	6.3	16.4 – 27.4	37 – 160
Chloroethane	7.6 – 32.4	11.4	8.4 – 40.4	14 – 230
2-Chloroethylvinyl ether	D – 44.8	25.9	D – 50.4	D – 305
Chloroform	13.5 – 26.5	6.1	13.7 – 24.2	51 – 138
Chloromethane	D – 40.8	19.8	D – 45.9	D – 273
Dibromochloromethane	13.5 – 26.5	6.1	13.8 – 26.6	53 – 149
1,2-Dichlorobenzene	12.6 – 27.4	7.1	11.8 – 34.7	18 – 190
1,3-Dichlorobenzene	14.6 – 25.4	5.5	17.0 – 28.8	59 – 156
1,4-Dichlorobenzene	12.6 – 27.4	7.1	11.8 – 34.7	18 – 190
1,1-Dichloroethane	14.5 – 25.5	5.1	14.2 – 28.5	59 – 155
1,2-Dichloroethane	13.6 – 26.4	6.0	14.3 – 27.4	49 – 155
1,1-Dichlorothene	10.1 – 29.9	9.1	3.7 – 42.3	D – 234
trans-1,2-Dichloroethene	13.9 – 26.1	5.7	13.6 – 28.5	54 – 156

TABLE 5—CALIBRATION AND QC ACCEPTANCE CRITERIA—METHOD 624[a]—Continued

Parameter	Range for Q (µ/g/L)	Limit for s (µ/g/L)	Range for $\bar{X}$ (µ/g/L)	Range for P, P_s (%)
1,2-Dichloropropane	6.8 – 33.2	13.8	3.8 – 36.2	D – 210
cis-1,3-Dichloropropene	4.8 – 35.2	15.8	1.0 – 39.0	D – 227
trans-1,3-Dichloropropene	10.0 – 30.0	10.4	7.6 – 32.4	17 – 183
Ethyl benzene	11.8 – 28.2	7.5	17.4 – 26.7	37 – 162
Methylene chloride	12.1 – 27.9	7.4	D – 41.0	D – 221
1,1,2,2-Tetrachloroethane	12.1 – 27.9	7.4	13.5 – 27.2	46 – 157
Tetrachloroethene	14.7 – 25.3	5.0	17.0 – 26.6	64 – 148
Toluene	14.9 – 25.1	4.8	16.6 – 26.7	47 – 150
1,1,1-Trichloroethane	15.0 – 25.0	4.6	13.7 – 30.1	52 – 162
1,1,2-Trichloroethane	14.2 – 25.8	5.5	14.3 – 27.1	52 – 150
Trichloroethene	13.3 – 26.7	6.6	18.6 – 27.6	71 – 157
Trichlorofluoromethane	9.6 – 30.4	10.0	8.9 – 31.5	17 – 181
Vinyl chloride	0.8 – 39.2	20.0	D – 43.5	D – 251

Q=Concentration measured in QC check sample, in µg/L (Section 7.5.3).
s=Standard deviation of four recovery measurements, in µg/L (Section 8.2.4).
$\bar{X}$=Average recovery of four recovery measurements, in µg/L (Section 8.2.4).
P, P_s=Percent recovery measured, (Section 8.3.2, Section 8.4.2).
D=Detected; result must be greater than zero.
[a]Criteria were calculated assuming a QC check sample concentration of 20 µg/L.

NOTE: These criteria are based directly upon the method performance data in Table 6. Where necessary, the limits for recovery have been broadened to assure applicability of the limits to concentrations below those used to develop Table 6.

TABLE 6—METHOD ACCURACY AND PRECISION AS FUNCTIONS OF CONCENTRATION—METHOD 624

Parameter	Accuracy, as recovery, $\bar{X}'$ (µg/L)	Single analyst precision, s_r' (µg/L)	Overall precision, S' (µg/L)
Benzene	0.93C+2.00	$0.26\bar{X}-1.74$	$0.25\bar{X}-1.33$
Bromodichloromethane	1.03C – 1.58	$0.15\bar{X}+0.59$	$0.20\bar{X}+1.13$
Bromoform	1.18C – 2.35	$0.12\bar{X}+0.36$	$0.17\bar{X}+1.38$
Bromomethane[a]	1.00C	$0.43\bar{X}$	$0.58\bar{X}$
Carbon tetrachloride	1.10C – 1.68	$0.12\bar{X}+0.25$	$0.11\bar{X}+0.37$
Chlorobenzene	0.98C+2.28	$0.16\bar{X}-0.09$	$0.26\bar{X}-1.92$
Chloroethane	1.18C+0.81	$0.14\bar{X}+2.78$	$0.29\bar{X}+1.75$
2–Chloroethylvinyl ether[a]	1.00C	$0.62\bar{X}$	$0.84\bar{X}$
Chloroform	0.93C+0.33	$0.16\bar{X}+0.22$	$0.18\bar{X}+0.16$
Chloromethane	1.03C+0.81	$0.37\bar{X}+2.14$	$0.58\bar{X}+0.43$
Dibromochloromethane	1.01C – 0.03	$0.17\bar{X}-0.18$	$0.17\bar{X}+0.49$
1,2–Dichlorobenzene[b]	0.94C+4.47	$0.22\bar{X}-1.45$	$0.30\bar{X}-1.20$
1,3–Dichlorobenzene	1.06C+1.68	$0.14\bar{X}-0.48$	$0.18\bar{X}-0.82$
1,4–Dichlorobenzene[b]	0.94C+4.47	$0.22\bar{X}-1.45$	$0.30\bar{X}-1.20$
1,1–Dichloroethane	1.05C+0.36	$0.13\bar{X}-0.05$	$0.16\bar{X}+0.47$
1,2–Dichloroethane	1.02C+0.45	$0.17\bar{X}-0.32$	$0.21\bar{X}-0.38$
1,1–Dichloroethene	1.12C+0.61	$0.17\bar{X}+1.06$	$0.43\bar{X}-0.22$
trans–1,2,–Dichloroethene	1.05C+0.03	$0.14\bar{X}+0.09$	$0.19\bar{X}+0.17$
1,2–Dichloropropane[a]	1.00C	$0.33\bar{X}$	$0.45\bar{X}$
cis–1,3–Dichloropropene[a]	1.00C	$0.38\bar{X}$	$0.52\bar{X}$
trans–1,3–Dichloropropene[a]	1.00C	$0.25\bar{X}$	$0.34\bar{X}$
Ethyl benzene	0.98C+2.48	$0.14\bar{X}+1.00$	$0.26\bar{X}-1.72$
Methylene chloride	0.87C+1.88	$0.15\bar{X}+1.07$	$0.32\bar{X}+4.00$
1,1,2,2–Tetrachloroethane	0.93C+1.76	$0.16\bar{X}+0.69$	$0.20\bar{X}+0.41$
Tetrachloroethene	1.06C+0.60	$0.13\bar{X}-0.18$	$0.16\bar{X}-0.45$
Toluene	0.98C+2.03	$0.15\bar{X}-0.71$	$0.22\bar{X}-1.71$
1,1,1–Trichloroethane	1.06C+0.73	$0.12\bar{X}-0.15$	$0.21\bar{X}-0.39$
1,1,2–Trichloroethane	0.95C+1.71	$0.14\bar{X}+0.02$	$0.18\bar{X}+0.00$
Trichloroethene	1.04C+2.27	$0.13\bar{X}+0.36$	$0.12\bar{X}+0.59$
Trichloroflouromethane	0.99C+0.39	$0.33\bar{X}-1.48$	$0.34\bar{X}-0.39$
Vinyl chloride	1.00C	$0.48\bar{X}$	$0.65\bar{X}$

$\bar{X}'$=Expected recovery for one or more measurements of a sample containing a concentration of C, in µg/L.
S_r=Expected single analyst standard deviation of measurements at an average concentration found of$\bar{X}$, in µg/L.
S'=Expected interlaboratory standard deviation of measurements at an average concentration found of$\bar{X}$, in µg/L.
C=True value for the concentration, in µg/L.
$\bar{X}$=Average recovery found for measurements of samples containing a concentration of C, in µg/L.
[a]Estimates based upon the performance in a single laboratory.[13]
[b]Due to chromatographic resolution problems, performance statements for these isomers are based upon the sums of their concentrations.

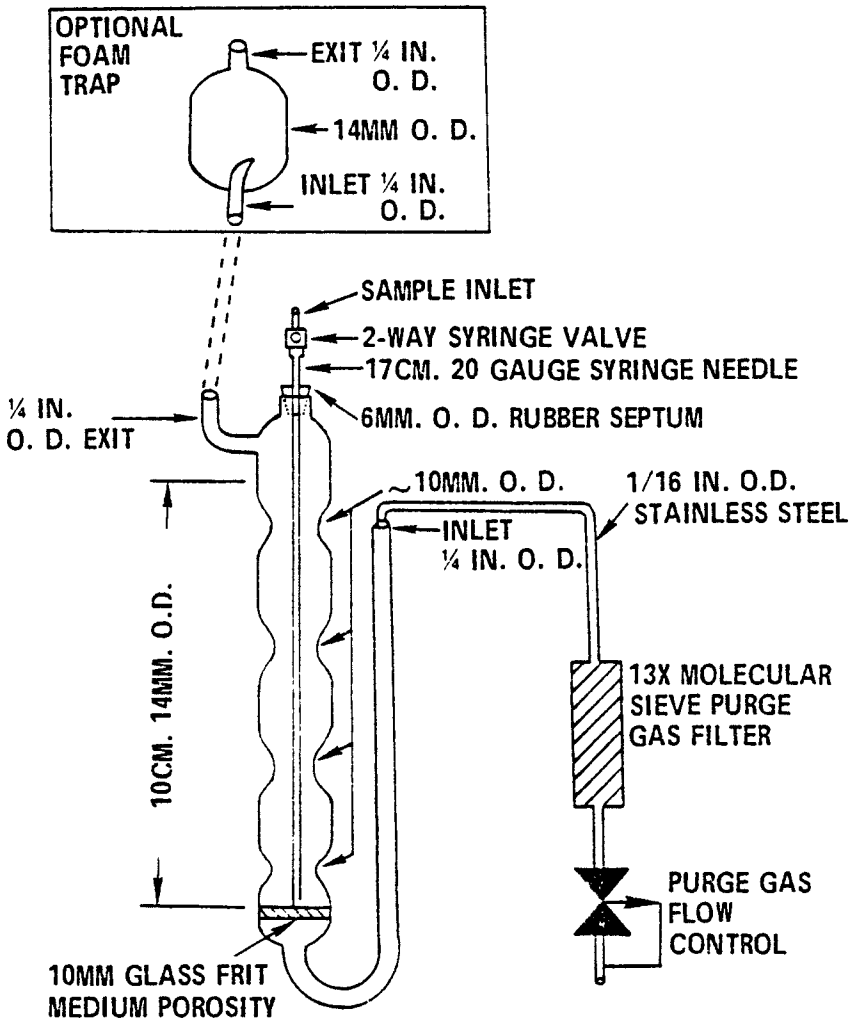


Figure 1. Purging device.

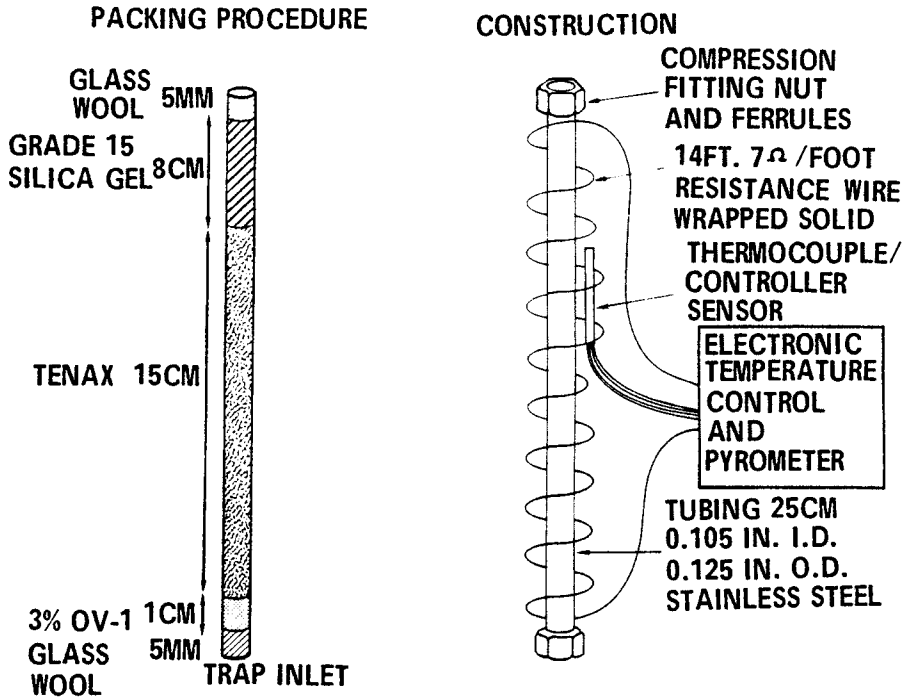


Figure 2. Trap packings and construction to include desorb capability.

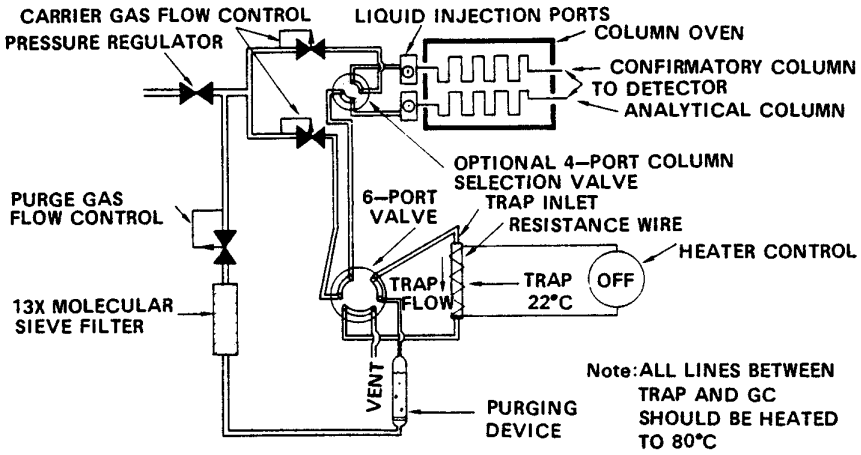


Figure 3. Purge and trap system - purge mode.

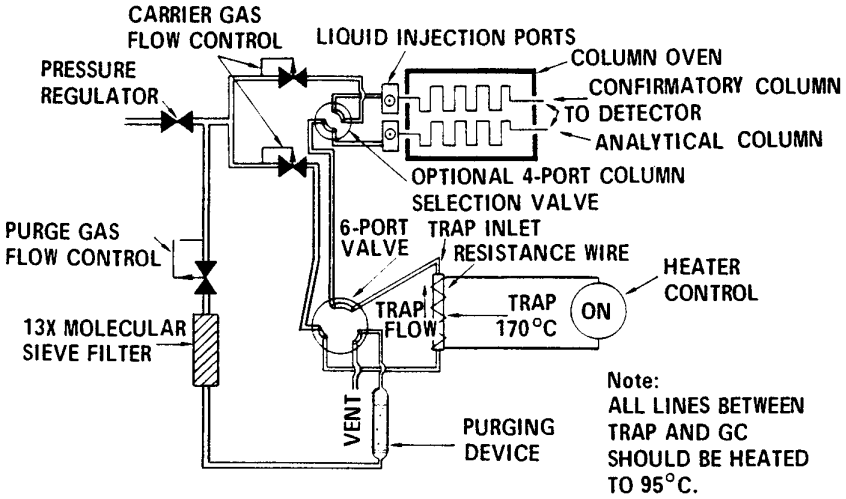


Figure 4. Purge and trap system - desorb mode.

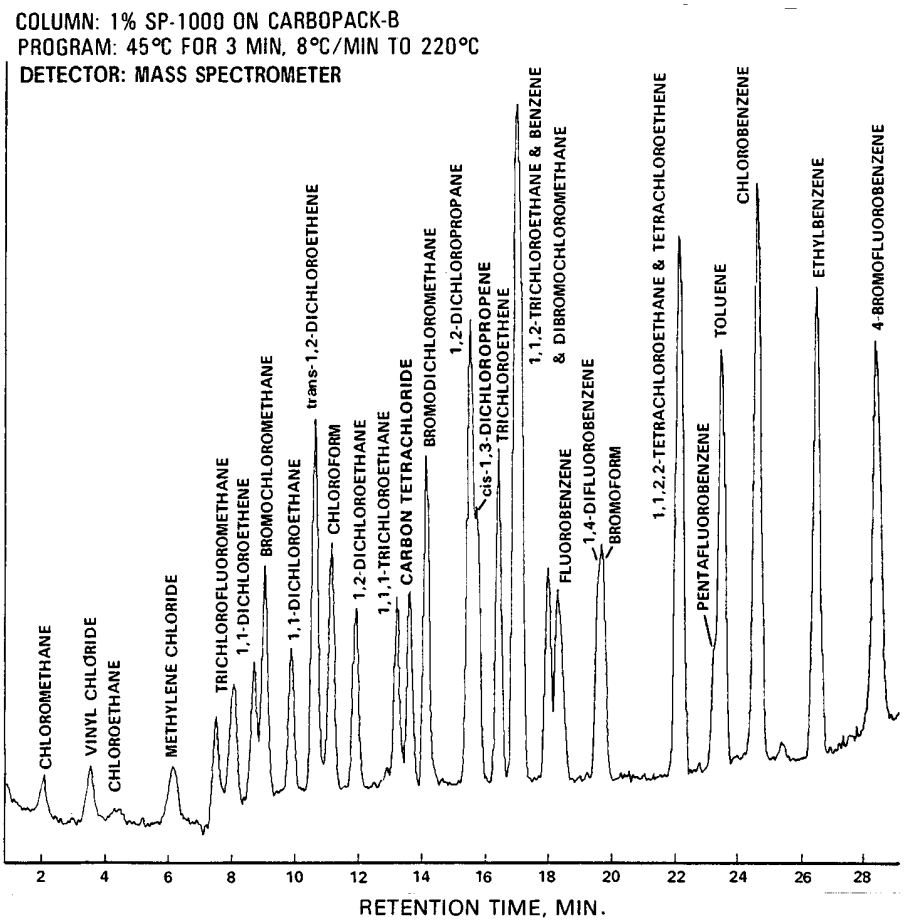


Figure 5. Gas chromatogram of volatile organics.

METHOD 625—BASE/NEUTRALS AND ACIDS

1. Scope and Application

1.1 This method covers the determination of a number of organic compounds that are partitioned into an organic solvent and are amenable to gas chromatography. The parameters listed in Tables 1 and 2 may be qualitatively and quantitatively determined using this method.

1.2 The method may be extended to include the parameters listed in Table 3. Benzidine can be subject to oxidative losses during solvent concentration. Under the alkaline conditions of the extraction step, α–BHC, γ–BHC, endosulfan I and II, and endrin are subject to decomposition. Hexachlorocyclopentadiene is subject to thermal decomposition in the inlet of the gas chromatograph, chemical reaction in acetone solution, and photochemical decomposition. N-nitrosodimethylamine is difficult to separate from the solvent under the chromatographic conditions described. N-nitrosodiphenylamine decomposes in the gas chromatographic inlet and cannot be separated from diphenylamine. The preferred method for each of these parameters is listed in Table 3.

1.3 This is a gas chromatographic/mass spectrometry (GC/MS) method[2, 14] applicable to the determination of the compounds listed in Tables 1, 2, and 3 in municipal and industrial discharges as provided under 40 CFR 136.1.

1.4 The method detection limit (MDL, defined in Section 16.1)[1] for each parameter is listed in Tables 4 and 5. The MDL for a specific wastewater may differ from those listed, depending upon the nature of interferences in the sample matrix.

1.5 Any modification to this method, beyond those expressly permitted, shall be considered as a major modification subject to application and approval of alternate test procedures under 40 CFR 136.4 and 136.5. Depending upon the nature of the modification and the extent of intended use, the applicant may be required to demonstrate that the modifications will produce equivalent results when applied to relevant wastewaters.

1.6 This method is restricted to use by or under the supervision of analysts experienced in the use of a gas chromatograph/mass spectrometer and in the interpretation of mass spectra. Each analyst must demonstrate the ability to generate acceptable results with this method using the procedure described in Section 8.2.

2. Summary of Method

2.1 A measured volume of sample, approximately 1–L, is serially extracted with methylene chloride at a pH greater than 11 and again at a pH less than 2 using a separatory funnel or a continuous extractor.[2] The methylene chloride extract is dried, concentrated to a volume of 1 mL, and analyzed by GC/MS. Qualitative identification of the parameters in the extract is performed using the retention time and the relative abundance of three characteristic masses (m/z). Quantitative analysis is performed using internal standard techniques with a single characteristic m/z.

3. Interferences

3.1 Method interferences may be caused by contaminants in solvents, reagents, glassware, and other sample processing hardware that lead to discrete artifacts and/or elevated baselines in the total ion current profiles. All of these materials must be routinely demonstrated to be free from interferences under the conditions of the analysis by running laboratory reagent blanks as described in Section 8.1.3.

3.1.1 Glassware must be scrupulously cleaned.[3] Clean all glassware as soon as possible after use by rinsing with the last solvent used in it. Solvent rinsing should be followed by detergent washing with hot water, and rinses with tap water and distilled water. The glassware should then be drained dry, and heated in a muffle furnace at 400 °C for 15 to 30 min. Some thermally stable materials, such as PCBs, may not be eliminated by this treatment. Solvent rinses with acetone and pesticide quality hexane may be substituted for the muffle furnace heating. Thmrough rinsing with such solvents usually eliminates PCB interference. Volumetric ware should not be heated in a muffle furnace. After drying and cooling, glassware should be sealed and stored in a clean environment to prevent any accumulation of dust or other contaminants. Store inverted or capped with aluminum foil.

3.1.2 The use of high purity reagents and solvents helps to minimize interference problems. Purification of solvents by distillation in all-glass systems may be required.

3.2 Matrix interferences may be caused by contaminants that are co-extracted from the sample. The extent of matrix interferences will vary considerably from source to source, depending upon the nature and diversity of the industrial complex or municipality being sampled.

3.3 The base-neutral extraction may cause significantly reduced recovery of phenol, 2-methylphenol, and 2,4-dimethylphenol. The analyst must recognize that results obtained under these conditions are minimum concentrations.

3.4 The packed gas chromatographic columns recommended for the basic fraction may not exhibit sufficient resolution for certain isomeric pairs including the following: anthracene and phenanthrene; chrysene and benzo(a)anthracene; and benzo(b)fluoranthene and benzo(k)fluoranthene. The gas

chromatographic retention time and mass spectra for these pairs of compounds are not sufficiently different to make an unambiguous identification. Alternative techniques should be used to identify and quantify these specific compounds, such as Method 610.

3.5 In samples that contain an inordinate number of interferences, the use of chemical ionization (CI) mass spectrometry may make identification easier. Tables 6 and 7 give characteristic CI ions for most of the compounds covered by this method. The use of CI mass spectrometry to support electron ionization (EI) mass spectrometry is encouraged but not required.

4. Safety

4.1 The toxicity or carcinogenicity of each reagent used in this method have not been precisely defined; however, each chemical compound should be treated as a potential health hazard. From this viewpoint, exposure to these chemicals must be reduced to the lowest possible level by whatever means available. The laboratory is responsible for maintaining a current awareness file of OSHA regulations regarding the safe handling of the chemicals specified in this method. A reference file of material data handling sheets should also be made available to all personnel involved in the chemical analysis. Additional references to laboratory safety are available and have been identified[4–6] for the information of the analyst.

4.2 The following parameters covered by this method have been tentatively classified as known or suspected, human or mammalian carcinogens: benzo(a)anthracene, benzidine, 3,3′-dichlorobenzidine, benzo(a)pyrene, α–BHC, β–BHC, δ–BHC, γ–BHC, dibenzo(a,h)anthracene, N-nitrosodimethylamine, 4,4′–DDT, and polychlorinated biphenyls (PCBs). Primary standards of these toxic compounds should be prepared in a hood. A NIOSH/MESA approved toxic gas respirator should be worn when the analyst handles high concentrations of these toxic compounds.

5. Apparatus and Materials

5.1 Sampling equipment, for discrete or composit sampling.

5.1.1 Grab sample bottle—1–L or 1-gt, amber glass, fitted with a screw cap lined with Teflon. Foil may be substituted for Teflon if the sample is not corrosive. If amber bottles are not available, protect samples from light. The bottle and cap liner must be washed, rinsed with acetone or methylene chloride, and dried before use to minimize contamination.

5.1.2 Automatic sampler (optional)—The sampler must incorporate glass sample containers for the collection of a minimum of 250 mL of sample. Sample containers must be kept refrigerated at 4 °C and protected from light during compositing. If the sampler uses a peristaltic pump, a minimum length of compressible silicone rubber tubing may be used. before use, however, the compressible tubing should be throughly rinsed with methanol, followed by repeated rinsings with distilled water to minimize the potential for contamination of the sample. An integrating flow meter is required to collect flow proportional composites.

5.2 Glassware (All specifications are suggested. Catalog numbers are included for illustration only.):

5.2.1 Separatory funnel—2–L, with Teflon stopcock.

5.2.2 Drying column—Chromatographic column, 19 mm ID, with coarse frit

5.2.3 Concentrator tube, Kuderna-Danish—10-mL, graduated (Kontes K–570050–1025 or equivalent). Calibration must be checked at the volumes employed in the test. Ground glass stopper is used to prevent evaporation of extracts.

5.2.4 Evaporative flask, Kuderna-Danish—500-mL (Kontes K–57001–0500 or equivalent). Attach to concentrator tube with springs.

5.2.5 Snyder column, Kuderna-Danish—Three all macro (Kontes K–503000–0121 or equivalent).

5.2.6 Snyder column, Kuderna-Danish—Two-ball macro (Kontes K–569001–0219 or equivalent).

5.2.7 Vials—10 to 15-mL, amber glass, with Teflon-lined screw cap.

5.2.8 Continuous liquid—liquid extractor—Equipped with Teflon or glass connecting joints and stopcocks requiring no lubrication. (Hershberg-Wolf Extractor, Ace Glass Company, Vineland, N.J., P/N 6841–10 or equivalent.)

5.3 Boiling chips—Approximately 10/40 mesh. Heat to 400 °C for 30 min of Soxhlet extract with methylene chloride.

5.4 Water bath—Heated, with concentric ring cover, capable of temperature control (±2°C). The bath should be used in a hood.

5.5 Balance—Analytical, capable of accurately weighing 0.0001 g.

5.6 GC/MS system:

5.6.1 Gas Chromatograph—An analytical system complete with a temperature programmable gas chromatograph and all required accessores including syringes, analytical columns, and gases. The injection port must be designed for on-column injection when using packed columns and for splitless injection when using capillary columns.

5.6.2 Column for base/neutrals—1.8 m long x 2 mm ID glass, packed with 3% SP–2250 on Supelcoport (100/120 mesh) or equivalent. This column was used to develop the method performance statements in Section 16. Guidelines for the use of alternate column packings are provided in Section 13.1.

5.6.3 Column for acids—1.8 m long x 2 mm ID glass, packed with 1% SP–1240DA on Supelcoport (100/120 mesh) or equivalent.

This column was used to develop the method performance statements in Section 16. Guidelines for the use of alternate column packings are given in Section 13.1.

5.6.4 Mass spectrometer—Capable of scanning from 35 to 450 amu every 7 s or less, utilizing a 70 V (nominal) electron energy in the electron impact ionization mode, and producing a mass spectrum which meets all the criteria in Table 9 when 50 ng of decafluorotriphenyl phosphine (DFTPP; bis(perfluorophenyl) phenyl phosphine) is injected through the GC inlet.

5.6.5 GC/MS interface—Any GC to MS interface that gives acceptable calibration points at 50 ng per injection for each of the parameters of interest and achieves all acceptable performance criteria (Section 12) may be used. GC to MS interfaces constructed of all glass or glass-lined materials are recommended. Glass can be deactivated by silanizing with dichlorodimethylsilane.

5.6.6 Data system—A computer system must be interfaced to the mass spectrometer that allows the contiluous acquisition and storage on machine-readable media of all mass spectra obtained throughout the duration of the chromatographic program. The computer must have software that allows searching any GC/MS data file for specific m/z and plotting such m/z abundances versus time or scan number. This type of plot is defined as an Extracted Ion Current Profile (EICP). Software must also be available that allows integrating the abundance in any EICP between specified time or scan number limits.

6. Reagents

6.1 Reagent water—Reagent water is defined as a water in which an interferent is not observed at the MDL of the parameters of interest.

6.2 Sodium hydroxide solution (10 N)—Dissolve 40 g of NaOH (ACS) in reagent water and dilute to 100 mL.

6.3 Sodium thiosulfate—(ACS) Granular.

6.4 Sulfuric acid (1+1)—Slowly, add 50 mL of H_2SO_4 (ACS, sp. gr. 1.84) to 50 mL of reagent water.

6.5 Acetone, methanol, methlylene chloride—Pesticide quality or equivalent.

6.6 Sodium sulfate—(ACS) Granular, anhydrous. Purify by heating at 400 °C for 4 h in a shallow tray.

6.7 Stock standard solutions (1.00 µg/µL)—standard solutions can be prepared from pure standard materials or purchased as certified solutions.

6.7.1 Prepare stock standard solutions by accurately weighing about 0.0100 g of pure material. Dissolve the material in pesticide quality acetone or other suitable solvent and dilute to volume in a 10-mL volumetric flask. Larger volumes can be used at the convenience of the analyst. When compound purity is assayed to be 96% or greater, the weight may be used without correction to calculate the concentration of the stock standard. Commercially prepared stock standards may be used at any concentration if they are certified by the manufacturer or by an independent source.

6.7.2 Transfer the stock standard solutions into Teflon-sealed screw-cap bottles. Store at 4 °C and protect from light. Stock standard solutions should be checked frequently for signs of degradation or evaporation, especially just prior to preparing calibration standards from them.

6.7.3 Stock standard solutions must be replaced after six months, or sooner if comparison with quality control check samples indicate a problem.

6.8 Surrogate standard spiking solution—Select a minimum of three surrogate compounds from Table 8. Prepare a surrogate standard spiking solution containing each selected surrogate compound at a concentration of 100 µg/mL in acetone. Addition of 1.00 mL of this solution to 1000 mL of sample is equivalent to a concentration of 100 µg/L of each surrogate standard. Store the spiking solution at 4 °C in Teflon-sealed glass container. The solution should be checked frequently for stability. The solution must be replaced after six months, or sooner if comparison with quality control check standards indicates a problem.

6.9 DFTPP standard—Prepare a 25 µg/mL solution of DFTPP in acetone.

6.10 Quality control check sample concentrate—See Section 8.2.1.

7. Calibration

7.1 Establish gas chromatographic operating parameters equivalent to those indicated in Table 4 or 5.

7.2 Internal standard calibration procedure—To use this approach, the analyst must select three or more internal standards that are similar in analytical behavior to the compounds of interest. The analyst must further demonstrate that the measurement of the internal standards is not affected by method or matrix interferences. Some recommended internal standards are listed in Table 8. Use the base peak m/z as the primary m/z for quantification of the standards. If interferences are noted, use one of the next two most intense m/z quantities for quantification.

7.2.1 Prepare calibration standards at a minimum of three concentration levels for each parameter of interest by adding appropriate volumes of one or more stock standards to a volumetric flask. To each calibration standard or standard mixture, add a known constant amount of one or more internal standards, and and dilute to volume with acetone. One of the calibration standards should be at a concentration near, but above, the MDL and the other concentrations should correspond to the expected

range of concentrations found in real samples or should define the working range of the GC/MS system.

7.2.2 Using injections of 2 to 5 µL, analyze each calibration standard according to Section 13 and tabulate the area of the primary characteristic m/z (Tables 4 and 5) against concentration for each compound and internal standard. Calculate response factors (RF) for each compound using Equation 1.

$$RF = \frac{(A_s)(C_{is})}{(A_{is})(C_s)}$$

Equation 1

where:

A_s=Area of the characteristic m/z for the parameter to be measured.

A_{is}=Area of the characteristic m/z for the internal standard.

C_{is}=Concentration of the internal standard (µg/L).

C_s=Concentration of the parameter to be measured (µg/L).

If the RF value over the working range is a constant (<35% RSD), the RF can be assumed to be invariant and the average RF can be used for calculations. Alternatively, the results can be used to plot a calibration curve of response ratios, A_s/A_{is}, vs. RF.

7.3 The working calibration curve or RF must be verified on each working day by the measurement of one or more calibration standards. If the response for any parameter varies from the predicted response by more than ±20%, the test must be repeated uning a fresh calibration standard. Alternatively, a new calibration curve must be prepared for that compound.

8. Quality Control

8.1 Each laboratory that uses this method is required to operate a formal quality control program. The minimum requirements of this program consist of an initial demonstration of laboratory capability and an ongoing analysis of spiked samples to evaluate and document data quality. The laboratory must maintain records to document the quality of data that is generated. Ongoing data quality checks are compared with established performance criteria to determine if the results of analyses meet the performance characteristics of the method. When results of sample spikes indicate atypical method performance, a quality control check standard must be analyzed to confirm that the measurements were performed in an in-control mode of operation.

8.1.1 The analyst must make an initial, one-time, demonstration of the ability to generate acceptable accuracy and precision with this method. This ability is established as described in Section 8.2.

8.1.2 In recognition of advances that are occuring in chromatography, the analyst is permitted certain options (detailed in Sections 10.6 and 13.1) to improve the separations or lower the cost of measurements. Each time such a modification is made to the method, the analyst is required to repeat the procedure in Section 8.2.

8.1.3 Before processing any samples, the analyst must analyze a reagent water blank to demonstrate that interferences from the analytical system and glassware are under control. Each time a set of samples is extracted or reagents are changed, a reagent water blank must be processed as a safeguard against laboratory contamination.

8.1.4 The laboratory must, on an ongoing basis, spike and analyze a minimum of 5% of all samples to monitor and evaluate laboratory data quality. This procedure is described in Section 8.3.

8.1.5 The laboratory must, on an ongoing basis, demonstrate through the analyses of quality control check standards that the operation of the measurement system is in control. This procedure is described in Section 8.4. The frequency of the check standard analyses is equivalent to 5% of all samples analyzed but may be reduced if spike recoveries from samples (Section 8.3) meet all specified quality control criteria.

8.1.6 The laboratory must maintain performance records to document the quality of data that is generated. This procedure is described in Section 8.5.

8.2 To establish the ability to generate acceptable accuracy and precision, the analyst must perform the following operations.

8.2.1 A quality control (QC) check sample concentrate is required containing each parameter of interest at a concentration of 100 µg/mL in acetone. Multiple solutions may be required. PCBs and multicomponent pesticides may be omitted from this test. The QC check sample concentrate must be obtained from the U.S. Environmental Protection Agency, Environmental Monitoring and Support Laboratory in Cincinnati, Ohio, if available. If not available from that source, the QC check sample concentrate must be obtained from another external source. If not available from either source above, the QC check sample concentrate must be prepared by the laboratory using stock standards prepared independently from those used for calibration.

8.2.2 Using a pipet, prepare QC check samples at a concentration of 100 µg/L by adding 1.00 mL of QC check sample concentrate to each of four 1–L aliquots of reagent water.

8.2.3 Analyze the well-mixed QC check samples according to the method beginning in Section 10 or 11.

8.2.4 Calculate the average recovery (X) in µg/L, and the standard deviation of the recovery (s) in µg/L, for each parameter using the four results.

8.2.5 For each parameter compare s and X with the corresponding acceptance criteria for precision and accuracy, respectively, found in Table 6. If s and X for all parameters of interest meet the acceptance criteria, the system performance is acceptable and analysis of actual samples can begin. If any individual s exceeds the precision limit or any individual X falls outside the range for accuracy, the system performance is unacceptable for that parameter.

NOTE: The large number of parameters in Table 6 present a substantial probability that one or more will fail at least one of the acceptance criteria when all parameters are analyzed.

8.2.6 When one or more of the parameters tested fail at least one of the acceptance criteria, the analyst must proceed according to Section 8.2.6.1 or 8.2.6.2.

8.2.6.1 Locate and correct the source of the problem and repeat the test for all parameters of interest beginning with Section 8.2.2.

8.2.6.2 Beginning with Section 8.2.2, repeat the test only for those parameters that failed to meet criteria. Repeated failure, however, will confirm a general problem with the measurement system. If this occurs, locate and correct the source of the problem and repeat the test for all compounds of interest beginning with Section 8.2.2.

8.3 The laboratory must, on an ongoing basis, spike at least 5% of the samples from each sample site being monitored to assess accuracy. For laboratories analyzing 1 to 20 samples per month, at least one spiked sample per month is required.

8.3.1. The concentration of the spike in the sample should be determined as follows:

8.3.1 If, as in compliance monitoring, the concentration of a specific parameter in the sample is being checked against a regulatory concentration limit, the spike should be at that limit or 1 to 5 times higher than the background concentration determined in Section 8.3.2, whichever concentration would be larger.

8.3.1.2 If the concentration of a specific parameter in the sample is not being checked against a limit specific to that parameter, the spike should be at 100 µg/L or 1 to 5 times higher than the background concentration determined in Section 8.3.2, whichever concentration would be larger.

8.3.1.3 If it is impractical to determine background levels before spiking (e.g., maximum holding times will be exceeded), the spike concentration should be (1) the regulatory concentration limit, if any; or, if none (2) the larger of either 5 times higher than the expected background concentration or 100 µg/L.

8.3.2 Analyze one sample aliquot to determine the background concentration (B) of each parameter. If necessary, prepare a new QC check sample concentrate (Section 8.2.1) appropriate for the background concentrations in the sample. Spike a second sample aliquot with 1.0 mL of the QC check sample concentrate and analyze it to determine the concentration after spiking (A) of each parameter. Calculate each percent recovery (P) as 100(A–B)%/T, where T is the known true value of the spike.

8.3.3 Compare the percent recovery (P) for each parameter with the corresponding QC acceptance criteria found in Table 6. These acceptance criteria were calculated to include an allowance for error in measurement of both the background and spike concentrations, assuming a spike to background ratio of 5:1. This error will be accounted for to the extent that the analyst's spike to background ratio approaches 5:1.[7] If spiking was performed at a concentration lower than 100 µg/L, the analyst must use either the QC acceptance criteria in Table 6, or optional QC acceptance criteria calculated for the specific spike concentration. To calculate optional acceptance criteria for the recovery of a parameter: (1) Calculate accuracy (X′) using the equation in Table 7, substituting the spike concentration (T) for C; (2) calculate overall precision (S′) using the equation in Table 7, substituting X′ for X; (3) calculate the range for recovery at the spike concentration as (100 X′/T)±2.44(100 S′/T)%[7]

8.3.4 If any individual P falls outside the designated range for recovery, that parameter has failed the acceptance criteria. A check standard containing each parameter that failed the criteria must be analyzed as described in Section 8.4.

8.4 If any parameter fails the acceptance criteria for recovery in Section 8.3, a QC check standard containing each parameter that failed must be prepared and analyzed.

NOTE: The frequency for the required analysis of a QC check standard will depend upon the number of parameters being simultaneously tested, the complexity of the sample matrix, and the performance of the laboratory. If the entire list of single-component parameters in Table 6 must be measured in the sample in Section 8.3, the probability that the analysis of a QC check standard will be required is high. In this case the QC check standard should be routinely analyzed with the spike sample.

8.4.1 Prepare the QC check standard by adding 1.0 mL of QC check sample concentrate (Section 8.2.1 or 8.3.2) to 1 L of reagent water. The QC check standard needs only to contain the parameters that failed criteria in the test in Section 8.3.

8.4.2 Analyze the QC check standard to determine the concentration measured (A) of each parameter. Calculate each percent recovery (P_S) as 100 (A/T)%, where T is the true value of the standard concentration.

8.4.3 Compare the percent recovery (P_s) for each parameter with the corresponding QC acceptance criteria found in Table 6. Only parameters that failed the test in Section 8.3 need to be compared with these criteria. If the recovery of any such parameter falls outside the designated range, the laboratory performance for that parameter is judged to be out of control, and the problem must be immediately identified and corrected. The analytical result for that parameter in the unspiked sample is suspect and may not be reported for regulatory compliance purposes.

8.5 As part of the QC program for the laboratory, method accuracy for wastewater samples must be assessed and records must be maintained. After the analysis of five spiked wastewater samples as in Section 8.3, calculate the average percent recovery ($\bar{P}$) and the standard deviation of the percent recovery (s_p). Express the accuracy assessment as a percent interval from $\bar{P}-2s_p$ to $\bar{P}+2s_p$. If $\bar{P}$=90% and s_p=10%, for example, the accuracy interval is expressed as 70−110%. Update the accuracy assessment for each parameter on a regular basis (e.g. after each five to ten new accuracy measurements).

8.6 As a quality control check, the laboratory must spike all samples with the surrogate standard spiking solution as described in Section 10.2, and calculate the percent recovery of each surrogate compound.

8.7 It is recommended that the laboratory adopt additional quality assurance practices for use with this method. The specific practices that are most productive depend upon the needs of the laboratory and the nature of the samples. Field duplicates may be analyzed to assess the precision of the environmental measurements. Whenever possible, the laboratory should analyze standard reference materials and participate in relevant performance evaluation studies.

9. Sample Collection, Preservation, and Handling

9.1 Grab samples must be collected in glass containers. Conventional sampling practices[8] should be followed, except that the bottle must not be prerinsed with sample before collection. Composite samples should be collected in refrigerated glass containers in accordance with the requirements of the program. Automatic sampling equipment must be as free as possible of Tygon tubing and other potential sources of contamination.

9.2 All sampling must be iced or refrigerated at 4 °C from the time of collection until extraction. Fill the sample bottles and, if residual chlorine is present, add 80 mg of sodium thiosulfate per liter of sample and mix well. EPA Methods 330.4 and 330.5 may be used for measurement of residual chlorine.[9] Field test kits are available for this purpose.

9.3 All samples must be extracted within 7 days of collection and completely analyzed within 40 days of extraction.

10. Separatory Funnel Extraction

10.1 Samples are usually extracted using separatory funnel techniques. If emulsions will prevent achieving acceptable solvent recovery with separatory funnel extractions, continuous extraction (Section 11) may be used. The separatory funnel extraction scheme described below assumes a sample volume of 1 L. When sample volumes of 2 L are to be extracted, use 250, 100, and 100-mL volumes of methylene chloride for the serial extraction of the base/neutrals and 200, 100, and 100-mL volumes of methylene chloride for the acids.

10.2 Mark the water meniscus on the side of the sample bottle for later determination of sample volume. Pour the entire sample into a 2–L separatory funnel. Pipet 1.00 mL of the surrogate standard spiking solution into the separatory funnel and mix well. Check the pH of the sample with wide-range pH paper and adjust to pH>11 with sodium hydroxide solution.

10.3 Add 60 mL of methylene chloride to the sample bottle, seal, and shake for 30 s to rinse the inner surface. Transfer the solvent to the separatory funnel and extract the sample by shaking the funnel for 2 min. with periodic venting to release excess pressure. Allow the organic layer to separate from the water phase for a minimum of 10 min. If the emulsion interface between layers is more than one-third the volume of the solvent layer, the analyst must employ mechanical techniques to complete the phase separation. The optimum technique depends upon the sample, but may include stirring, filtration of the emulsion through glass wool, centrifugation, or other physical methods. Collect the methylene chloride extract in a 250-mL Erlenmeyer flask. If the emulsion cannot be broken (recovery of less than 80% of the methylene chloride, corrected for the water solubility of methylene chloride), transfer the sample, solvent, and emulsion into the extraction chamber of a continuous extractor and proceed as described in Section 11.3.

10.4 Add a second 60-mL volume of methylene chloride to the sample bottle and repeat the extraction procedure a second time, combining the extracts in the Erlenmeyer flask. Perform a third extraction in the same manner. Label the combined extract as the base/neutral fraction.

10.5 Adjust the pH of the aqueous phase to less than 2 using sulfuric acid. Serially extract the acidified aqueous phase three times with 60-mL aliquots of methylene chloride. Collect and combine the extracts in a 250-mL Erlenmeyer flask and label the combined extracts as the acid fraction.

10.6 For each fraction, assemble a Kuderna-Danish (K–D) concentrator by attaching a 10-mL concentrator tube to a 500-mL evaporative flask. Other concentration devices or techniques may be used in place of the K–D concentrator if the requirements of Section 8.2 are met.

10.7 For each fraction, pour the combined extract through a solvent-rinsed drying column containing about 10 cm of anhydrous sodium sulfate, and collect the extract in the K–D concentrator. Rinse the Erlenmeyer flask and column with 20 to 30 mL of methylene chloride to complete the quantitative transfer.

10.8 Add one or two clean boiling chips and attach a three-ball Snyder column to the evaporative flask for each fraction. Prewet each Snyder column by adding about 1 mL of methylene chloride to the top. Place the K–D apparatus on a hot water bath (60 to 65 °C) so that the concentrator tube is partially immersed in the hot water, and the entire lower rounded surface of the flask is bathed with hot vapor. Adjust the vertical position of the apparatus and the water temperature as required to complete the concentration in 15 to 20 min. At the proper rate of distillation the balls of the column will actively chatter but the chambers will not flood with condensed solvent. When the apparent volume of liquid reaches 1 mL, remove the K–D apparatus from the water bath and allow it to drain and cool for at least 10 min. Remove the Snyder column and rinse the flask and its lower joint into the concentrator tube with 1 to 2 mL of methylene chloride. A 5-mL syringe is recommended for this operation.

10.9 Add another one or two clean boiling chips to the concentrator tube for each fraction and attach a two-ball micro-Snyder column. Prewet the Snyder column by adding about 0.5 mL of methylene chloride to the top. Place the K–D apparatus on a hot water bath (60 to 65 °C) so that the concentrator tube is partially immersed in hot water. Adjust the vertical position of the apparatus and the water temperature as required to complete the concentration in 5 to 10 min. At the proper rate of distillation the balls of the column will actively chatter but the chambers will not flood with condensed solvent. When the apparent volume of liquid reaches about 0.5 mL, remove the K–D apparatus from the water bath and allow it to drain and cool for at least 10 min. Remove the Snyder column and rinse the flask and its lower joint into the concentrator tube with approximately 0.2 mL of acetone or methylene chloride. Adjust the final volume to 1.0 mL with the solvent. Stopper the concentrator tube and store refrigerated if further processing will not be performed immediately. If the extracts will be stored longer than two days, they should be transferred to Teflon-sealed screw-cap vials and labeled base/neutral or acid fraction as appropriate.

10.10 Determine the original sample volume by refilling the sample bottle to the mark and transferring the liquid to a 1000-mL graduated cylinder. Record the sample volume to the nearest 5 mL.

11. Continuous Extraction

11.1 When experience with a sample from a given source indicates that a serious emulsion problem will result or an emulsion is encountered using a separatory funnel in Section 10.3, a continuous extractor should be used.

11.2 Mark the water meniscus on the side of the sample bottle for later determination of sample volume. Check the pH of the sample with wide-range pH paper and adjust to pH >11 with sodium hydroxide solution. Transfer the sample to the continuous extractor and using a pipet, add 1.00 mL of surrogate standard spiking solution and mix well. Add 60 mL of methylene chloride to the sample bottle, seal, and shake for 30 s to rinse the inner surface. Transfer the solvent to the extractor.

11.3 Repeat the sample bottle rinse with an additional 50 to 100-mL portion of methylene chloride and add the rinse to the extractor.

11.4 Add 200 to 500 mL of methylene chloride to the distilling flask, add sufficient reagent water to ensure proper operation, and extract for 24 h. Allow to cool, then detach the distilling flask. Dry, concentrate, and seal the extract as in Sections 10.6 through 10.9.

11.5 Charge a clean distilling flask with 500 mL of methylene chloride and attach it to the continuous extractor. Carefully, while stirring, adjust the pH of the aqueous phase to less than 2 using sulfuric acid. Extract for 24 h. Dry, concentrate, and seal the extract as in Sections 10.6 through 10.9.

12. Daily GC/MS Performance Tests

12.1 At the beginning of each day that analyses are to be performed, the GC/MS system must be checked to see if acceptable performance criteria are achieved for DFTPP.[10] Each day that benzidine is to be determined, the tailing factor criterion described in Section 12.4 must be achieved. Each day that the acids are to be determined, the tailing factor criterion in Section 12.5 must be achieved.

12.2 These performance tests require the following instrumental parameters:

Electron Energy: 70 V (nominal)
Mass Range: 35 to 450 amu
Scan Time: To give at least 5 scans per peak but not to exceed 7 s per scan.

12.3 DFTPP performance test—At the beginning of each day, inject 2 µL (50 ng) of

DFTPP standard solution. Obtain a background-corrected mass spectra of DFTPP and confirm that all the key m/z criteria in Table 9 are achieved. If all the criteria are not achieved, the analyst must retune the mass spectrometer and repeat the test until all criteria are achieved. The performance criteria must be achieved before any samples, blanks, or standards are analyzed. The taililg factor tests in Sections 12.4 and 12.5 may be performed simultaneously with the DFTPP test.

12.4 Column performance test for base/neutrals—At the beginning of each day that the base/neutral fraction is to be analyzed for benzidine, the benzidine tailing factor must be calculated. Inject 100 ng of benzidine either separately or as a part of a standard mixture that may contain DFTPP and calculate the tailing factor. The benzidine tailing factor must be less than 3.0. Calculation of the tailing factor is illustrated in Figure 13.[11] Replace the column packing if the tailing factor criterion cannot be achieved.

12.5 Column performance test for acids—At the beginning of each day that the acids are to be determined, inject 50 ng of pentachlorophenol either separately or as a part of a standard mix that may contain DFTPP. The tailing factor for pentachlorophenol must be less than 5. Calculation of the tailing factor is illustrated in Figure 13.[11] Replace the column packing if the tailing factor criterion cannot be achieved.

13. Gas Chromatography/Mass Spectrometry

13.1 Table 4 summarizes the recommended gas chromatographic operating conditions for the base/neutral fraction. Table 5 summarizes the recommended gas chromatographic operating conditions for the acid fraction. Included in these tables are retention times and MDL that can be achieved under these conditions. Examples of the separations achieved by these columns are shown in Figures 1 through 12. Other packed or capillary (open-tubular) columns or chromatographic conditions may be used if the requirements of Section 8.2 are met.

13.2 After conducting the GC/MS performance tests in Section 12, calibrate the system daily as described in Section 7.

13.3 The internal standard must be added to sample extract and mixed thoroughly immediately before it is injected into the instrument. This procedure minimizes losses due to adsorption, chemical reaction or evaporation.

13.4 Inject 2 to 5 µL of the sample extract or standard into the GC/MS system using the solvent-flush technique.[12] Smaller (1.0 µL) volumes may be injected if automatic devices are employed. Record the volume injected to the nearest 0.05 µL.

13.5 If the response for any m/z exceeds the working range of the GC/MS system, dilute the extract and reanalyze.

13.6 Perform all qualitative and quantitative measurements as described in Sections 14 and 15. When the extracts are not being used for analyses, store them refrigerated at 4°C, protected from light in screw-cap vials equipped with unpierced Teflon-lined septa.

14. Qualitative Identification

14.1 Obtain EICPs for the primary m/z and the two other masses listed in Tables 4 and 5. See Section 7.3 for masses to be used with internal and surrogate standards. The following criteria must be met to make a qualitative identification:

14.1.1 The characteristic masses of each parameter of interest must maximize in the same or within one scan of each other.

14.1.2 The retention time must fall within ±30 s of the retention time of the authentic compound.

14.1.3 The relative peak heights of the three characteristic masses in the EICPs must fall within ±20% of the relative intensities of these masses in a reference mass spectrum. The reference mass spectrum can be obtained from a standard analyzed in the GC/MS system or from a reference library.

14.2 Structural isomers that have very similar mass spectra and less than 30 s difference in retention time, can be explicitly identified only if the resolution between authentic isomers in a standard mix is acceptable. Acceptable resolution is achieved if the baseline to valley height between the isomers is less than 25% of the sum of the two peak heights. Otherwise, structural isomers are identified as isomeric pairs.

15. Calculations

15.1 When a parameter has been identified, the quantitation of that parameter will be based on the integrated abundance from the EICP of the primary characteristic m/z in Tables 4 and 5. Use the base peak m/z for internal and surrogate standards. If the sample produces an interference for the primary m/z, use a secondary characteristic m/z to quantitate.

Calculate the concentration in the sample using the response factor (RF) determined in Section 7.2.2 and Equation 3.

$$\text{Concentration } (\mu g/L) = \frac{(A_s)(I_s)}{(A_{is})(RF)(V_o)}$$

Equation 3

where:

A_s=Area of the characteristic m/z for the parameter or surrogate standard to be measured.

A_{is}=Area of the characteristic m/z for the internal standard.

I_s=Amount of internal standard added to each extract (µg).

V_o=Volume of water extracted (L).

15.2 Report results in µg/L without correction for recovery data. All QC data obtained should be reported with the sample results.

16. Method Performance

16.1 The method detection limit (MDL) is defined as the minimum concentration of a substance that can be measured and reported with 99% confidence that the value is above zero.[1] The MDL concentrations listed in Tables 4 and 5 were obtained using reagent water.[13] The MDL actually achieved in a given analysis will vary depending on instrument sensitivity and matrix effects.

16.2 This method was tested by 15 laboratories using reagent water, drinking water, surface water, and industrial wastewaters spiked at six concentrations over the range 5 to 1300 µg/L.[14] Single operator precision, overall precision, and method accuracy were found to be directly related to the concentration of the parameter and essentially independent of the sample matrix. Linear equations to describe these relationships are presented in Table 7.

17. Screening Procedure for 2,3,7,8-Tetrachlorodibenzo-p-dioxin (2,3,7,8–TCDD)

17.1 If the sample must be screened for the presence of 2,3,7,8–TCDD, it is recommended that the reference material not be handled in the laboratory unless extensive safety precautions are employed. It is sufficient to analyze the base/neutral extract by selected ion monitoring (SIM) GC/MS techniques, as follows:

17.1.1 Concentrate the base/neutral extract to a final volume of 0.2 ml.

17.1.2 Adjust the temperature of the base/neutral column (Section 5.6.2) to 220 °C.

17.1.3 Operate the mass spectrometer to acquire data in the SIM mode using the ions at m/z 257, 320 and 322 and a dwell time no greater than 333 milliseconds per mass.

17.1.4 Inject 5 to 7 µL of the base/neutral extract. Collect SIM data for a total of 10 min.

17.1.5 The possible presence of 2,3,7,8–TCDD is indicated if all three masses exhibit simultaneous peaks at any point in the selected ion current profiles.

17.1.6 For each occurrence where the possible presence of 2,3,7,8–TCDD is indicated, calculate and retain the relative abundances of each of the three masses.

17.2 False positives to this test may be caused by the presence of single or coeluting combinations of compounds whose mass spectra contain all of these masses.

17.3 Conclusive results of the presence and concentration level of 2,3,7,8–TCDD can be obtained only from a properly equipped laboratory through the use of EPA Method 613 or other approved alternate test procedures.

REFERENCES

1. 40 CFR part 136, appendix B.

2. ''Sampling and Analysis Procedures for Screening of Industrial Effluents for Priority Pollutants,'' U.S. Environmental Protection Agency, Environmental Monitoring and Support Laboratory, Cincinnati, Ohio 45268, March 1977, Revised April 1977. Available from Effluent Guidelines Division, Washington, DC 20460.

3. ASTM Annual Book of Standards, Part 31, D3694–78. ''Standard Practices for Preparation of Sample Containers and for Preservation of Organic Constituents,'' American Society for Testing and Materials, Philadelphia.

4. ''Carcinogens—Working With Carcinogens,'' Department of Health, Education, and Welfare, Public Health Service, Center for Disease Control, National Institute for Occupational Safety and Health, Publication No. 77–206, August 1977.

5. ''OSHA Safety and Health Standards, General Industry,'' (29 CFR part 1910), Occupational Safety and Health Administration, OSHA 2206 (Revised, January 1976).

6. ''Safety in Academic Chemistry Laboratories,''American Chemical Society Publication, Committee on Chemical Safety, 3rd Edition, 1979.

7. Provost, L.P., and Elder, R.S. ''Interpretation of Percent Recovery Data,'' *American Laboratory*, 15, 58–63 (1983). (The value 2.44 used in the equation in Section 8.3.3 is two times the value 1.22 derived in this report.)

8. ASTM Annual Book of Standards, Part 31, D3370–76. ''Standard Practices for Sampling Water,'' American Society for Testing and Materials, Philadelphia.

9. ''Methods 330.4 (Titrimetric, DPD–FAS) and 330.5 (Spectrophotometric, DPD) for Chlorine, Total Residual,'' Methods for Chemical Analysis of Water and Wastes, EPA–600/4–79–020, U.S. Environmental Protection Agency, Environmental Monitoring and Support Laboratory, Cincinnati, Ohio 45268, March 1979.

10. Eichelberger, J.W., Harris, L.E., and Budde, W.L. ''Reference Compound to Calibrate Ion Abundance Measurement in Gas Chromatography-Mass Spectometry,'' *Analytical Chemistry*, 47, 995 (1975).

11. McNair, N.M. and Bonelli, E.J. ''Basic Chromatography,'' Consolidated Printing, Berkeley, California, p. 52, 1969.

12. Burke, J.A. ''Gas Chromatography for Pesticide Residue Analysis; Some Practical

Aspects,'' *Journal of the Association of Official Analytical Chemists, 48,* 1037 (1965).

13. Olynyk, P., Budde, W.L., and Eichelberger, J.W. ''Method Detection Limit for Methods 624 and 625,'' Unpublished report, May 14, 1980.

14. ''EPA Method Study 30, Method 625, Base/Neutrals, Acids, and Pesticides,'' EPA 600/4–84–053, National Technical Information Service, PB84–206572, Springfield, Virginia 22161, June 1984.

TABLE 1—BASE/NEUTRAL EXTRACTABLES

Parameter	STORET No.	CAS No.
Acenaphthene	34205	83–32–9
Acenaphthylene	34200	208–96–8
Anthracene	34220	120–12–7
Aldrin	39330	309–00–2
Benzo(a)anthracene	34526	56–55–3
Benzo(b)fluoranthene	34230	205–99–2
Benzo(k)fluoranthene	34242	207–08–9
Benzo(a)pyrene	34247	50–32–8
Benzo(ghi)perylene	34521	191–24–2
Benzyl butyl phthalate	34292	85–68–7
β-BHC	39338	319–85–7
δ-BHC	34259	319–86–8
Bis(2-chloroethyl) ether	34273	111–44–4
Bis(2-chloroethoxy)methane	34278	111–91–1
Bis(2-ethylhexyl) phthalate	39100	117–81–7
Bis(2-chloroisopropyl) ether[a]	34283	108–60–1
4-Bromophenyl phenyl ether[a]	34636	101–55–3
Chlordane	39350	57–74–9
2-Chloronaphthalele	34581	91–58–7
4-Chlorophenyl phenyl ether	34641	7005–72–3
Chrysene	34320	218–01–9
4,4′-DDD	39310	72–54–8
4,4′-DDE	39320	72–55–9
4,4′-DDT	39300	50–29–3
Dibenzo(a,h)anthracene	34556	53–70–3
Di-n-butylphthalate	39110	84–74–2
1,3-Dichlorobenzene	34566	541–73–1
1,2-Dichlorobenzene	34536	95–50–1
1,4-Dichlorobenzene	34571	106–46–7
3,3′-Dichlorobenzidine	34631	91–94–1
Dieldrin	39380	60–57–1
Diethyl phthalate	34336	84–66–2
Dimethyl phthalate	34341	131–11–3
2,4-Dinitrotoluene	34611	121–14–2
2,6-Dinitrotoluene	34626	606–20–2
Di-n-octylphthalate	34596	117–84–0
Endosulfan sulfate	34351	1031–07–8
Endrin aldehyde	34366	7421–93–4
Fluoranthene	34376	206–44–0
Fluorene	34381	86–73–7
Heptachlor	39410	76–44–8
Heptchlor epoxide	39420	1024–57–3
Hexachlorobenzene	39700	118–74–1
Hexachlorobutadiene	34391	87–68–3

TABLE 1—BASE/NEUTRAL EXTRACTABLES—Continued

Parameter	STORET No.	CAS No.
Hexachloroethane	34396	67–72–1
Indeno(1,2,3-cd)pyrene	34403	193–39–5
Isophorone	34408	78–59–1
Naphthalene	34696	91–20–3
Nitrobenzene	34447	98–95–3
N-Nitrosodi-n-propylamine	34428	621–64–7
PCB-1016	34671	12674–11–2
PCB-1221	39488	11104–28–2
PCB-1232	39492	11141–16–5
PCB-1242	39496	53469–21–9
PCB-1248	39500	12672–29–6
PCB-1254	39504	11097–69–1
PCB-1260	39508	11096–82–5
Phenanthrene	34461	85–01–8
Pyrene	34469	129–00–0
Toxaphene	39400	8001–35–2
1,2,4-Trichlorobenzene	34551	120–82–1

[a] The proper chemical name is 2,2′-oxybis(1-chloropropane).

TABLE 2—ACID EXTRACTABLES

Parameter	STORET No.	CAS No.
4-Chloro-3-methylphenol	34452	59–50–7
2-Chlorophenol	34586	95–57–8
2,4-Dichlorophenol	34601	120–83–2
2,4-Dimethylphenol	34606	105–67–9
2,4-Dinitrophenol	34616	51–28–5
2-Methyl-4,6-dinitrophenol	34657	534–52–1
2-Nitrophenol	34591	88–75–5
4-Nitrophenol	34646	100–02–7
Pentachlorophenol	39032	87–86–5
Phenol	34694	108–95–2
2,4,6-Trichlorophenol	34621	88–06–2

TABLE 3—ADDITIONAL EXTRACTABLE PARAMETERS[a]

Parameter	STORET No.	CAS No.	Method
Benzidine	39120	92–87–5	605
β-BHC	39337	319–84–6	608
δ-BHC	39340	58–89–8	608
Endosulfan I	34361	959–98–8	608
Endosulfan II	34356	33213–65–9	608
Endrin	39390	72–20–8	608
Hexachlorocylopentadiene	34386	77–47–4	612
N-Nitrosodimethylamine	34438	62–75–9	607
N-Nitrosodiphenylamine	34433	86–30–6	607

[a] See Section 1.2.

TABLE 4—CHROMATOGRAPHIC CONDITIONS, METHOD DETECTION LIMITS, AND CHARACTERISTIC MASSES FOR BASE/NEUTRAL EXTRACTABLES

Parameter	Retention time (min)	Method detection limit (µg/L)	Characteristic masses					
			Electron impact			Chemical ionization		
			Primary	Secondary	Secondary	Methane	Methane	Methane
1,3-Dichlorobenzene	7.4	1.9	146	148	113	146	148	150
1,4-Dichlorobenzene	7.8	4.4	146	148	113	146	148	150
Hexachloroethane	8.4	1.6	117	201	199	199	201	203

TABLE 4—CHROMATOGRAPHIC CONDITIONS, METHOD DETECTION LIMITS, AND CHARACTERISTIC MASSES FOR BASE/NEUTRAL EXTRACTABLES—Continued

Parameter	Retention time (min)	Method detection limit (µg/L)	Characteristic masses					
			Electron impact			Chemical ionization		
			Primary	Secondary	Secondary	Methane	Methane	Methane
Bis(2-chloroethyl) ether[a]	8.4	5.7	93	63	95	63	107	109
1,2-Dichlorobenzene	8.4	1.9	146	148	113	146	148	150
Bis(2-chloroisopropyl) ether[a]	9.3	5.7	45	77	79	77	135	137
N-Nitrosodi-n-propylamine	.....	.....	130	42	101	.....	.....	.....
Nitrobenzene	11.1	1.9	77	123	65	124	152	164
Hexachlorobutadiene	11.4	0.9	225	223	227	223	225	227
1,2,4-Trichlorobenzene	11.6	1.9	180	182	145	181	183	209
Isophorone	11.9	2.2	82	95	138	139	167	178
Naphthalene	12.1	1.6	128	129	127	129	157	169
Bis(2-chloroethoxy) methane	12.2	5.3	93	95	123	65	107	137
Hexachlorocyclopentadiene[a]	13.9	.....	237	235	272	235	237	239
2-Chloronaphthalene	15.9	1.9	162	164	127	163	191	203
Acenaphthylene	17.4	3.5	152	151	153	152	153	181
Acenaphthene	17.8	1.9	154	153	152	154	155	183
Dimethyl phthalate	18.3	1.6	163	194	164	151	163	164
2,6-Dinitrotoluene	18.7	1.9	165	89	121	183	211	223
Fluorene	19.5	1.9	166	165	167	166	167	195
4-Chlorophenyl phenyl ether	19.5	4.2	204	206	141	.....	.....	.....
2,4-Dinitrotoluene	19.8	5.7	165	63	182	183	211	223
Diethyl phthalate	20.1	1.9	149	177	150	177	223	251
N-Nitrosodiphenylamine[b]	20.5	1.9	169	168	167	169	170	198
Hexachlorobenzene	21.0	1.9	284	142	249	284	286	288
β-BHC[b]	21.1	.....	183	181	109	.....	.....	.....
4-Bromophenyl phenyl ether	21.2	1.9	248	250	141	249	251	277
δ-BHC[b]	22.4	.....	183	181	109	.....	.....	.....
Phenanthrene	22.8	5.4	178	179	176	178	179	207
Anthracene	22.8	1.9	178	179	176	178	179	207
β-BHC	23.4	4.2	181	183	109	.....	.....	.....
Heptachlor	23.4	1.9	100	272	274	.....	.....	.....
δ-BHC	23.7	3.1	183	109	181	.....	.....	.....
Aldrin	24.0	1.9	66	263	220	.....	.....	.....
Dibutyl phthalate	24.7	2.5	149	150	104	149	205	279
Heptachlor epoxide	25.6	2.2	353	355	351	.....	.....	.....
Endosulfan I[b]	26.4	.....	237	339	341	.....	.....	.....
Fluoranthene	26.5	2.2	202	101	100	203	231	243
Dieldrin	27.2	2.5	79	263	279	.....	.....	.....
4,4′-DDE	27.2	5.6	246	248	176	.....	.....	.....
Pyrene	27.3	1.9	202	101	100	203	231	243
Endrin[b]	27.9	.....	81	263	82	.....	.....	.....
Endosulfan II[b]	28.6	.....	237	339	341	.....	.....	.....
4,4′-DDD	28.6	2.8	235	237	165	.....	.....	.....
Benzidine[b]	28.8	44	184	92	185	185	213	225
4,4′-DDT	29.3	4.7	235	237	165	.....	.....	.....
Endosulfan sulfate	29.8	5.6	272	387	422	.....	.....	.....
Endrin aldehyde	.....	.....	67	345	250	.....	.....	.....
Butyl benzyl phthalate	29.9	2.5	149	91	206	149	299	327
Bis(2-ethylhexyl) phthalate	30.6	2.5	149	167	279	149	.....	.....
Chrysene	31.5	2.5	228	226	229	228	229	257
Benzo(a)anthracene	31.5	7.8	228	229	226	228	229	257
3,3′-Dichlorobenzidine	32.2	16.5	252	254	126	.....	.....	.....
Di-n-octyl phthalate	32.5	2.5	149	.....	.....	.....	.....	.....
Benzo(b)fluoranthene	34.9	4.8	252	253	125	252	253	281
Benzo(k)fluoranthene	34.9	2.5	252	253	125	252	253	281
Benzo(a)pyrene	36.4	2.5	252	253	125	252	253	281
Indeno(1,2,3-cd) pyrene	42.7	3.7	276	138	277	276	277	305
Dibenzo(a,h)anthracene	43.2	2.5	278	139	279	278	279	307
Benzo(ghi)perylene	45.1	4.1	276	138	277	276	277	305
N-Nitrosodimethylamine[b]	.....	.....	42	74	44	.....	.....	.....
Chlordane[c]	19–30	.....	373	375	377	.....	.....	.....
Toxaphene[c]	25–34	.....	159	231	233	.....	.....	.....
PCB 1016[c]	18–30	.....	224	260	294	.....	.....	.....
PCB 1221[c]	15–30	30	190	224	260	.....	.....	.....
PCB 1232[c]	15–32	.....	190	224	260	.....	.....	.....
PCB 1242[c]	15–32	.....	224	260	294	.....	.....	.....
PCB 1248[c]	12–34	.....	294	330	262	.....	.....	.....
PCB 1254[c]	22–34	36	294	330	362	.....	.....	.....

TABLE 4—CHROMATOGRAPHIC CONDITIONS, METHOD DETECTION LIMITS, AND CHARACTERISTIC MASSES FOR BASE/NEUTRAL EXTRACTABLES—Continued

Parameter	Retention time (min)	Method detection limit (µg/L)	Characteristic masses					
			Electron impact			Chemical ionization		
			Primary	Secondary	Secondary	Methane	Methane	Methane
PCB 1260[c]	23–32	.....	330	362	394	.....	.....	.....

[a] The proper chemical name is 2,2′-bisoxy(1-chloropropane).
[b] See Section 1.2.
[c] These compounds are mixtures of various isomers (See Figures 2 through 12). Column conditions: Supelcoport (100/120 mesh) coated with 3% SP–2250 packed in a 1.8 m long x 2 mm ID glass column with helium carrier gas at 30 mL/min. flow rate. Column temperature held isothermal at 50 °C for 4 min., then programmed at 8 °C/min. to 270 °C and held for 30 min.

TABLE 5—CHROMATOGRAPHIC CONDITIONS, METHOD DETECTION LIMITS, AND CHARACTERISTIC MASSES FOR ACID EXTRACTABLES

Parameter	Retention time (min)	Method detection limit (µg/L)	Characteristic masses					
			Electron Impact			Chemical ionization		
			Primary	Secondary	Secondary	Methane	Methane	Methane
2-Chlorophenol	5.9	3.3	128	64	130	129	131	157
2-Nitrophenol	6.5	3.6	139	65	109	140	168	122
Phenol	8.0	1.5	94	65	66	95	123	135
2,4-Dimethylphenol	9.4	2.7	122	107	121	123	151	163
2,4-Dichlorophenol	9.8	2.7	162	164	98	163	165	167
2,4,6-Trichlorophenol	11.8	2.7	196	198	200	197	199	201
4-Chloro-3-methylphenol	13.2	3.0	142	107	144	143	171	183
2,4-Dinitrophenol	15.9	42	184	63	154	185	213	225
2-Methyl-4,6-dinitrophenol	16.2	24	198	182	77	199	227	239
Pentachlorophenol	17.5	3.6	266	264	268	267	265	269
4-Nitrophenol	20.3	2.4	65	139	109	140	168	122

Column conditions: Supelcoport (100/120 mesh) coated with 1% SP–1240DA packed in a 1.8 m long x 2mm ID glass column with helium carrier gas at 30 mL/min. flow rate. Column temperature held isothermal at 70 °C for 2 min. then programmed at 8 °C/min. to 200 °C.

TABLE 6—QC ACCEPTANCE CRITERIA—METHOD 625

Parameter	Test conclusion (µg/L)	Limits for s (µg/L)	Range for X(µg/L)	Range for P, P_s (Percent)
Acenaphthene	100	27.6	60.1–132.3	47–145
Acenaphthylene	100	40.2	53.5–126.0	33–145
Aldrin	100	39.0	7.2–152.2	D–166
Anthracene	100	32.0	43.4–118.0	27–133
Benzo(a)anthracene	100	27.6	41.8–133.0	33–143
Benzo(b)fluoranthene	100	38.8	42.0–140.4	24–159
Benzo(k)fluoranthene	100	32.3	25.2–145.7	11–162
Benzo(a)pyrene	100	39.0	31.7–148.0	17–163
Benzo(ghi)perylene	100	58.9	D–195.0	D–219
Benzyl butyl phthalate	100	23.4	D–139.9	D–152
β-BHC	100	31.5	41.5–130.6	24–149
δ-BHC	100	21.6	D–100.0	D–110
Bis(2-chloroethyl) ether	100	55.0	42.9–126.0	12–158
Bis(2-chloroethoxy)methane	100	34.5	49.2–164.7	33–184
Bis(2-chloroisopropyl) ether[a]	100	46.3	62.8–138.6	36–166
Bis(2-ethylhexyl) phthalate	100	41.1	28.9–136.8	8–158
4-Bromophenyl phenyl ether	100	23.0	64.9–114.4	53–127
2-Chloronaphthalene	100	13.0	64.5–113.5	60–118
4-Chlorophenyl phenyl ether	100	33.4	38.4–144.7	25–158
Chrysene	100	48.3	44.1–139.9	17–168
4,4′-DDD	100	31.0	D–134.5	D–145
4,4′-DDE	100	32.0	19.2–119.7	4–136
4,4′-DDT	100	61.6	D–170.6	D–203
Dibenzo(a,h)anthracene	100	70.0	D–199.7	D–227
Di-n-butyl phthalate	100	16.7	8.4–111.0	1–118
1,2-Dichlorobenzene	100	30.9	48.6–112.0	32–129
1,3-Dichlorobenzene	100	41.7	16.7–153.9	D–172
1,4,-Dichlorobenzene	100	32.1	37.3–105.7	20–124

TABLE 6—QC ACCEPTANCE CRITERIA—METHOD 625—Continued

Parameter	Test conclusion (μg/L)	Limits for s (μg/L)	Range for X(μg/L)	Range for P, P_s (Percent)
3,3′-Dhlorobenzidine	100	71.4	8.2–212.5	D–262
Dieldrin	100	30.7	44.3–119.3	29–136
Diethyl phthalate	100	26.5	D–100.0	D–114
Dimethyl phthalate	100	23.2	D–100.0	D–112
2,4-Dinitrotoluene	100	21.8	47.5–126.9	39–139
2,6-Dinitrotoluene	100	29.6	68.1–136.7	50–158
Di-n-octyl phthalate	100	31.4	18.6–131.8	4–146
Endosulfan sulfate	100	16.7	D–103.5	D–107
Endrin aldehyde	100	32.5	D–188.8	D–209
Fluoranthene	100	32.8	42.9–121.3	26–137
Fluorene	100	20.7	71.6–108.4	59–121
Heptachlor	100	37.2	D–172.2	D–192
Heptachlor epoxide	100	54.7	70.9–109.4	26–155
Hexachlorobenzene	100	24.9	7.8–141.5	D–152
Hexachlorobutadiene	100	26.3	37.8–102.2	24–116
Hexachloroethane	100	24.5	55.2–100.0	40–113
Indeno(1,2,3-cd)pyrene	100	44.6	D–150.9	D–171
Isophorone	100	63.3	46.6–180.2	21–196
Naphthalene	100	30.1	35.6–119.6	21–133
Nitrobenzene	100	39.3	54.3–157.6	35–180
N-Nitrosodi-n-propylamine	100	55.4	13.6–197.9	D–230
PCB-1260	100	54.2	19.3–121.0	D–164
Phenanthrene	100	20.6	65.2–108.7	54–120
Pyrene	100	25.2	69.6–100.0	52–115
1,2,4-Trichlorobenzene	100	28.1	57.3–129.2	44–142
4-Chloro-3-methylphenol	100	37.2	40.8–127.9	22–147
2-Chlorophenol	100	28.7	36.2–120.4	23–134
2,4-Dichlorophenol	100	26.4	52.5–121.7	39–135
2,4-Dimethylphenol	100	26.1	41.8–109.0	32–119
2,4-Dinitrophenol	100	49.8	D–172.9	D–191
2-Methyl-4,6-dinitrophenol	100	93.2	53.0–100.0	D–181
2-Nitrophenol	100	35.2	45.0–166.7	29–182
4-Nitrophenol	100	47.2	13.0–106.5	D–132
Pentachlorophenol	100	48.9	38.1–151.8	14–176
Phenol	100	22.6	16.6–100.0	5–112
2,4,6-Trichlorophenol	100	31.7	52.4–129.2	37–144

s=Standard deviation for four recovery measurements, in μg/L (Section 8.2.4).
X=Average recovery for four recovery measurements, in μg/L (Section 8.2.4).
P, P_s=Percent recovery measured (Section 8.3.2, Section 8.4.2).
D=Detected; result must be greater than zero.

NOTE: These criteria are based directly upon the method performance data in Table 7. Where necessary, the limits for recovery have been broadened to assure applicability of the limts to concentrations below those used to develop Table 7.

[a] The proper chemical name is 2,2′oxybis(1-chloropropane).

TABLE 7—METHOD ACCURACY AND PRECISION AS FUNCTIONS OF CONCENTRATION—METHOD 625

Parameter	Accuracy, as recovery, X′ (μg/L)	Single analyst precision, S_r' (μg/L)	Overall precision, S′ (μg/L)
Acenaphthene	$0.96C+0.19$	$0.15\bar{X}-0.12$	$0.21\bar{X}-0.67$
Acenaphthylene	$0.89C+0.74$	$0.24\bar{X}-1.06$	$0.26\bar{X}-0.54$
Aldrin	$0.78C+1.66$	$0.27\bar{X}-1.28$	$0.43\bar{X}+1.13$
Anthracene	$0.80C+0.68$	$0.21\bar{X}-0.32$	$0.27\bar{X}-0.64$
Benzo(a)anthracene	$0.88C-0.60$	$0.15\bar{X}+0.93$	$0.26\bar{X}-0.28$
Benzo(b)fluoranthene	$0.93C-1.80$	$0.22\bar{X}+0.43$	$0.29\bar{X}+0.96$
Benzo(k)fluoranthene	$0.87C-1.56$	$0.19\bar{X}+1.03$	$0.35\bar{X}+0.40$
Benzo(a)pyrene	$0.90C-0.13$	$0.22\bar{X}+0.48$	$0.32\bar{X}+1.35$
Benzo(ghi)perylene	$0.98C-0.86$	$0.29\bar{X}+2.40$	$0.51\bar{X}-0.44$
Benzyl butyl phthalate	$0.66C-1.68$	$0.18\bar{X}+0.94$	$0.53\bar{X}+0.92$
β-BHC	$0.87C-0.94$	$0.20\bar{X}-0.58$	$0.30\bar{X}-1.94$
δ-BHC	$0.29C-1.09$	$0.34\bar{X}+0.86$	$0.93\bar{X}-0.17$
Bis(2-chloroethyl) ether	$0.86C-1.54$	$0.35\bar{X}-0.99$	$0.35\bar{X}+0.10$
Bis(2-chloroethoxy)methane	$1.12C-5.04$	$0.16\bar{X}+1.34$	$0.26\bar{X}+2.01$
Bis(2-chloroisopropyl) ether[a]	$1.03C-2.31$	$0.24\bar{X}+0.28$	$0.25\bar{X}+1.04$
Bis(2-ethylhexyl) phthalate	$0.84C-1.18$	$0.26\bar{X}+0.73$	$0.36\bar{X}+0.67$
4-Bromophenyl phenyl ether	$0.91C-1.34$	$0.13\bar{X}+0.66$	$0.16\bar{X}+0.66$
2-Chloronaphthalene	$0.89C+0.01$	$0.07\bar{X}+0.52$	$0.13\bar{X}+0.34$
4-Chlorophenyl phenyl ether	$0.91C+0.53$	$0.20\bar{X}-0.94$	$0.30\bar{X}-0.46$
Chrysene	$0.93C-1.00$	$0.28\bar{X}+0.13$	$0.33\bar{X}-0.09$

TABLE 7—METHOD ACCURACY AND PRECISION AS FUNCTIONS OF CONCENTRATION—METHOD 625—Continued

Parameter	Accuracy, as recovery, X′ (μg/L)	Single analyst precision, s_r' (μg/L)	Overall precision, S′ (μg/L)
4,4′-DDD	0.56C − 0.40	0.29$\bar{X}$ − 0.32	0.66$\bar{X}$ − 0.96
4,4′-DDE	0.70C − 0.54	0.26$\bar{X}$ − 1.17	0.39$\bar{X}$ − 1.04
4,4′-DDT	0.79C − 3.28	0.42$\bar{X}$ + 0.19	0.65$\bar{X}$ − 0.58
Dibenzo(a,h)anthracene	0.88C + 4.72	0.30$\bar{X}$ + 8.51	0.59$\bar{X}$ + 0.25
Di-n-butyl phthalate	0.59C + 0.71	0.13$\bar{X}$ + 1.16	0.39$\bar{X}$ + 0.60
1,2-Dichlorobenzene	0.80C + 0.28	0.20$\bar{X}$ + 0.47	0.24$\bar{X}$ + 0.39
1,3-Dichlorobenzene	0.86C − 0.70	0.25$\bar{X}$ + 0.68	0.41$\bar{X}$ + 0.11
1,4-Dichlorobenzene	0.73C − 1.47	0.24$\bar{X}$ + 0.23	0.29$\bar{X}$ + 0.36
3,3′-Dichlorobenzidine	1.23C − 12.65	0.28$\bar{X}$ + 7.33	0.47$\bar{X}$ + 3.45
Dieldrin	0.82C − 0.16	0.20$\bar{X}$ − 0.16	0.26$\bar{X}$ − 0.07
Diethyl phthalate	0.43C + 1.00	0.28$\bar{X}$ + 1.44	0.52$\bar{X}$ + 0.22
Dimethyl phthalate	0.20C + 1.03	0.54$\bar{X}$ + 0.19	1.05$\bar{X}$ − 0.92
2,4-Dinitrotoluene	0.92C − 4.81	0.12$\bar{X}$ + 1.06	0.21$\bar{X}$ + 1.50
2,6-Dinitrotoluene	1.06C − 3.60	0.14$\bar{X}$ + 1.26	0.19$\bar{X}$ + 0.35
Di-n-octyl phthalate	0.76C − 0.79	0.21$\bar{X}$ + 1.19	0.37$\bar{X}$ + 1.19
Endosulfan sulfate	0.39C + 0.41	0.12$\bar{X}$ + 2.47	0.63$\bar{X}$ − 1.03
Endrin aldehyde	0.76C − 3.86	0.18$\bar{X}$ + 3.91	0.73$\bar{X}$ − 0.62
Fluoranthene	0.81C + 1.10	0.22$\bar{X}$ − 0.73	0.28$\bar{X}$ − 0.60
Fluorene	0.90C − 0.00	0.12$\bar{X}$ + 0.26	0.13$\bar{X}$ + 0.61
Heptachlor	0.87C − 2.97	0.24$\bar{X}$ − 0.56	0.50$\bar{X}$ − 0.23
Heptachlor epoxide	0.92C − 1.87	0.33$\bar{X}$ − 0.46	0.28$\bar{X}$ + 0.64
Hexachlorobenzene	0.74C + 0.66	0.18$\bar{X}$ − 0.10	0.43$\bar{X}$ − 0.52
Hexachlorobutadiene	0.71C − 1.01	0.19$\bar{X}$ + 0.92	0.26$\bar{X}$ + 0.49
Hexachloroethane	0.73C − 0.83	0.17$\bar{X}$ + 0.67	0.17$\bar{X}$ + 0.80
Indeno(1,2,3-cd)pyrene	0.78C − 3.10	0.29$\bar{X}$ + 1.46	0.50$\bar{X}$ + 0.44
Isophorone	1.12C + 1.41	0.27$\bar{X}$ + 0.77	0.33$\bar{X}$ + 0.26
Naphthalene	0.76C + 1.58	0.21$\bar{X}$ − 0.41	0.30$\bar{X}$ − 0.68
Nitrobenzene	1.09C − 3.05	0.19$\bar{X}$ + 0.92	0.27$\bar{X}$ + 0.21
N-Nitrosodi-n-propylamine	1.12C − 6.22	0.27$\bar{X}$ + 0.68	0.44$\bar{X}$ + 0.47
PCB–1260	0.81C − 10.86	0.35$\bar{X}$ + 3.61	0.43$\bar{X}$ + 1.82
Phenanthrene	0.87C − 0.06	0.12$\bar{X}$ + 0.57	0.15$\bar{X}$ + 0.25
Pyrene	0.84C − 0.16	0.16$\bar{X}$ + 0.06	0.15$\bar{X}$ + 0.31
1,2,4-Trichlorobenzene	0.94C − 0.79	0.15$\bar{X}$ + 0.85	0.21$\bar{X}$ + 0.39
4-Chloro-3-methylphenol	0.84C + 0.35	0.23$\bar{X}$ + 0.75	0.29$\bar{X}$ + 1.31
2-Chlorophenol	0.78C + 0.29	0.18$\bar{X}$ + 1.46	0.28$\bar{X}$ + 0.97
2,4-Dichlorophenol	0.87C + 0.13	0.15$\bar{X}$ + 1.25	0.21$\bar{X}$ + 1.28
2,4-Dimethylphenol	0.71C + 4.41	0.16$\bar{X}$ + 1.21	0.22$\bar{X}$ + 1.31
2,4-Dinitrophenol	0.81C − 18.04	0.38$\bar{X}$ + 2.36	0.42$\bar{X}$ + 26.29
2-Methyl-4,6-Dinitrophenol	1.04C − 28.04	0.05$\bar{X}$ + 42.29	0.26$\bar{X}$ + 23.10
2-Nitrophenol	1.07C − 1.15	0.16$\bar{X}$ + 1.94	0.27$\bar{X}$ + 2.60
4-Nitrophenol	0.61C − 1.22	0.38$\bar{X}$ + 2.57	0.44$\bar{X}$ + 3.24
Pentachlorophenol	0.93C + 1.99	0.24$\bar{X}$ + 3.03	0.30$\bar{X}$ + 4.33
Phenol	0.43C + 1.26	0.26$\bar{X}$ + 0.73	0.35$\bar{X}$ + 0.58
2,4,6-Trichlorophenol	0.91C − 0.18	0.16$\bar{X}$ + 2.22	0.22$\bar{X}$ + 1.81

X′=Expected recovery for one or more measurements of a sample containing a concentration of C, in μg/L.
s_r'=Expected single analyst standard deviation of measurements at an average concentration found of $\bar{X}$, in μg/L.
S′=Expected interlaboratory standard deviation of measurements at an average concentration found of $\bar{X}$, in μg/L.
C=True value for the concentration, in μg/L.
$\bar{X}$=Average recovery found for measurements of samples containing a concentration of C, in μg/L.
[a] The proper chemical name is 2,2′oxybis(1-chloropropane).

TABLE 8—SUGGESTED INTERNAL AND SURROGATE STANDARDS

Base/neutral fraction	Acid fraction
Aniline-d_5	2-Fluorophenol.
Anthracene-d_{10}	Pentafluorophenol.
Benzo(a)anthracene-d_{12}	Phenol-d_5
4,4′-Dibromobiphenyl	2-Perfluoromethyl phenol.
4,4′-Dibromooctafluorobiphenyl.	
Decafluorobiphenyl	
2,2[1]-Difluorobiphenyl	
4-Fluoroaniline	
1-Fluoronaphthalene	
2-Fluoronaphthalene	
Naphthalene-d_8	

TABLE 8—SUGGESTED INTERNAL AND SURROGATE STANDARDS—Continued

Base/neutral fraction	Acid fraction
Nitrobenzene-d_5	
2,3,4,5,6-Pentafluorobiphenyl	
Phenanthrene-d_{10}	
Pyridine-d_5	

TABLE 9—DFTPP KEY MASSES AND ABUNDANCE CRITERIA

Mass	m/z Abundance criteria
51	30–60 percent of mass 198.

TABLE 9—DFTPP KEY MASSES AND ABUNDANCE CRITERIA—Continued

Mass	m/z Abundance criteria
68	Less than 2 percent of mass 69.
70	Less than 2 percent of mass 69.
127	40–60 percent of mass 198.
197	Less than 1 percent of mass 198.
198	Base peak, 100 percent relative abundance.
199	5–9 percent of mass 198.

TABLE 9—DFTPP KEY MASSES AND ABUNDANCE CRITERIA—Continued

Mass	m/z Abundance criteria
275	10–30 percent of mass 198.
365	Greater than 1 percent of mass 198.
441	Present but less than mass 443.
442	Greater than 40 percent of mass 198.
443	17–23 percent of mass 442.

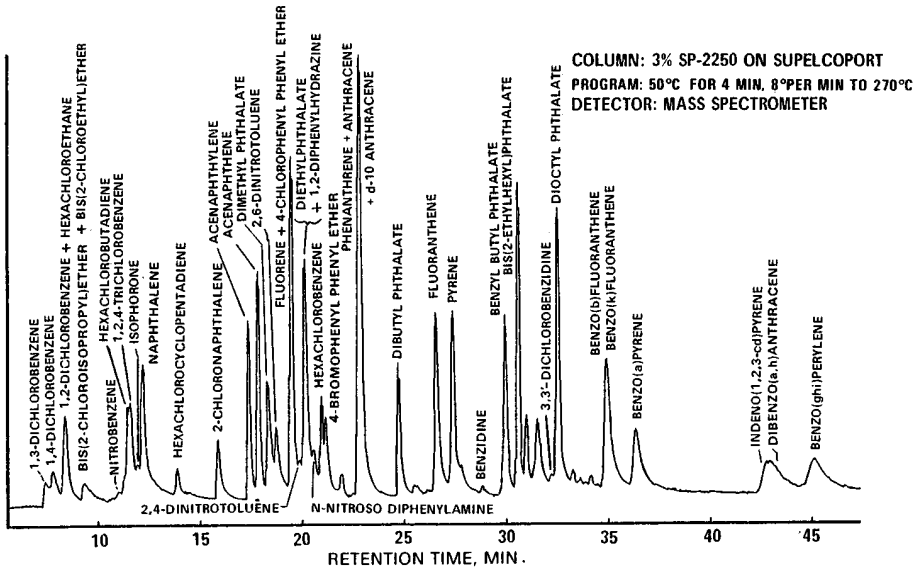


Figure 1. Gas chromatogram of base/neutral fraction.

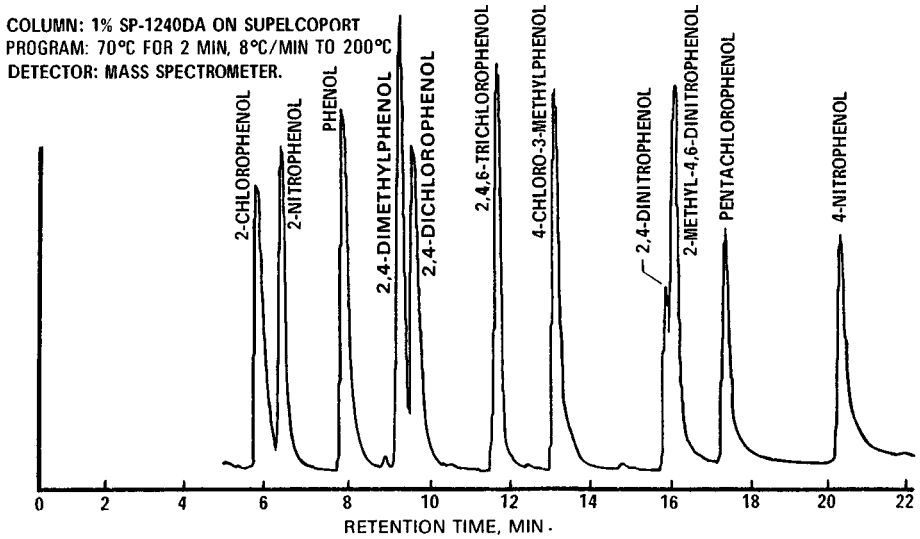


Figure 2. Gas chromatogram of acid fraction.

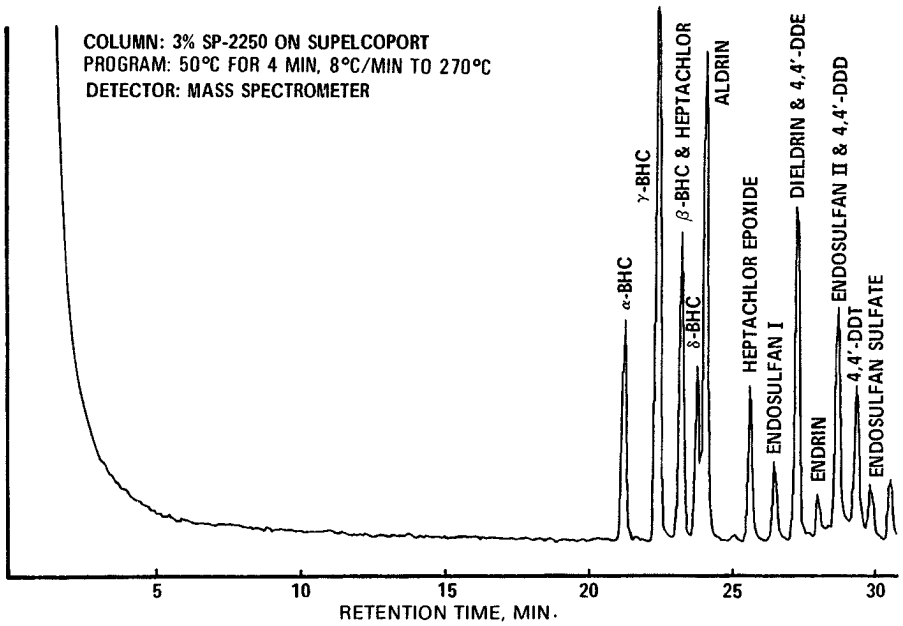


Figure 3. Gas chromatogram of pesticide fraction.

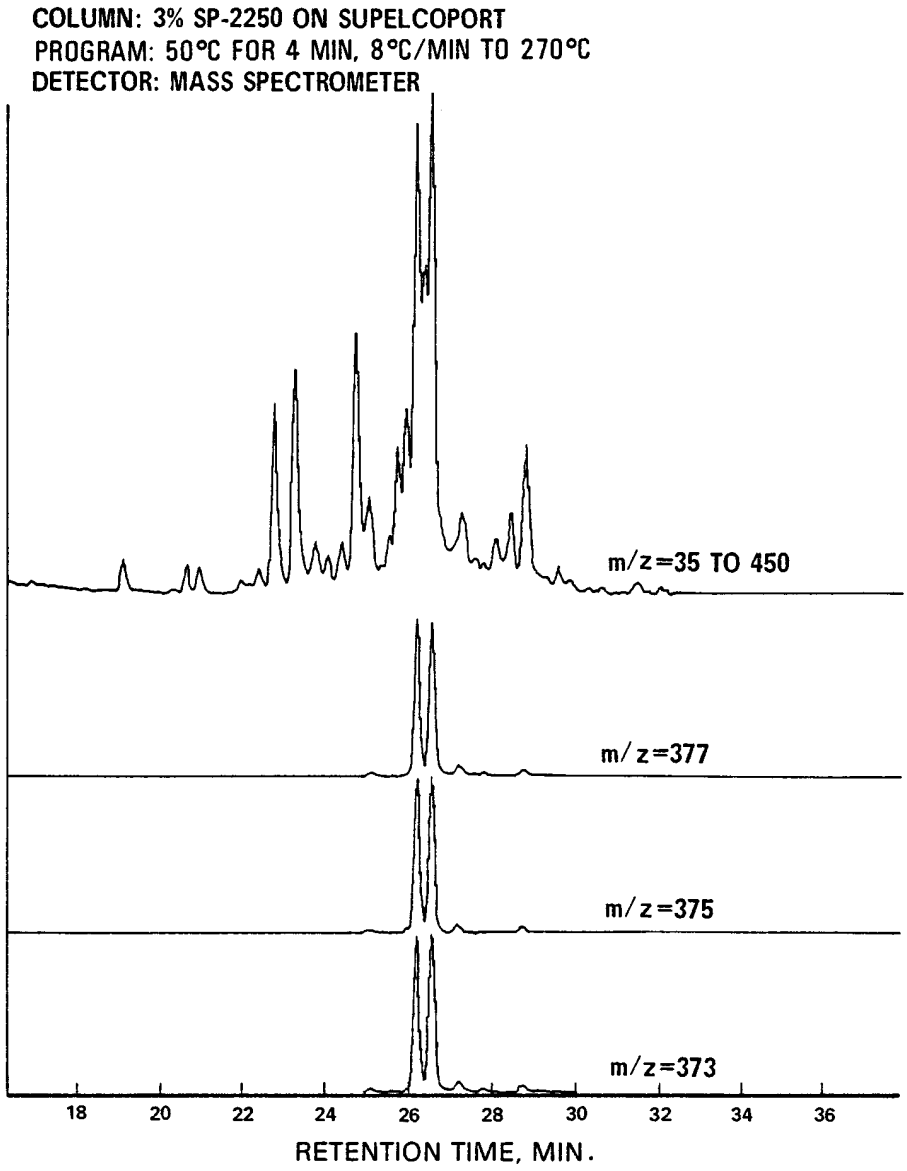


Figure 4. Gas chromatogram of chlordane.

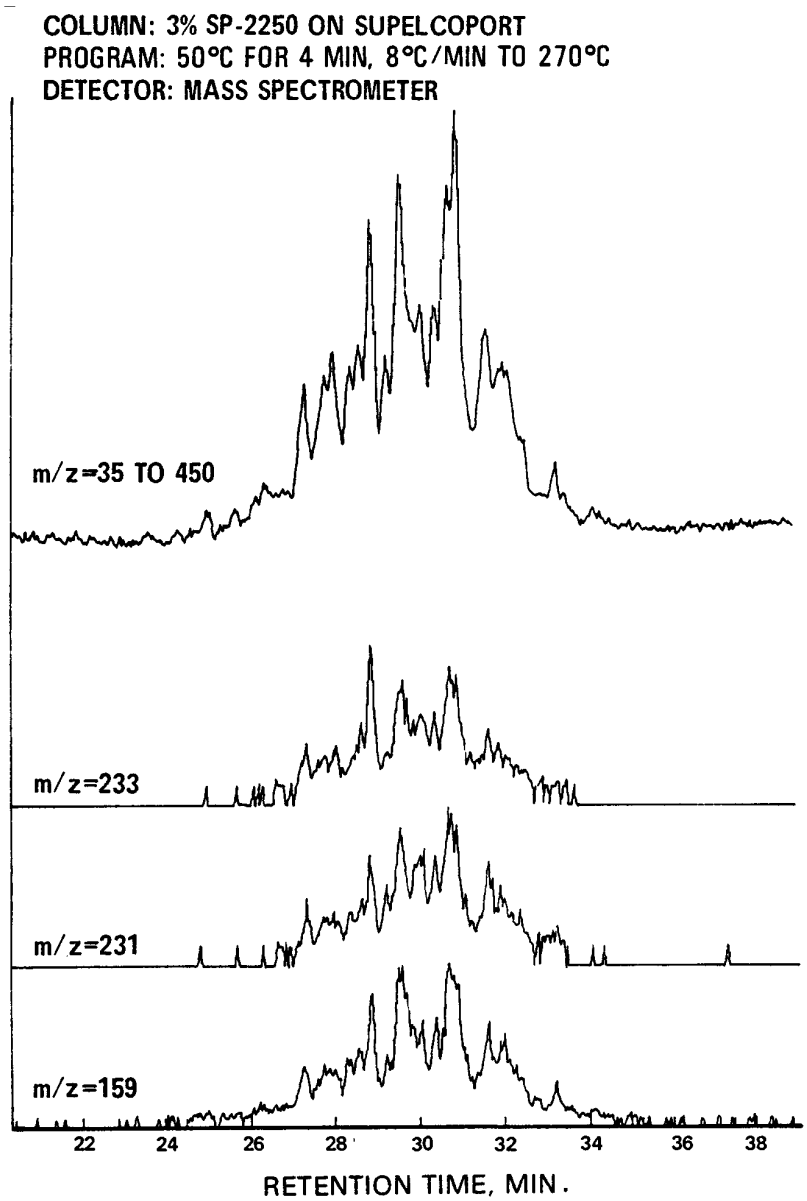


Figure 5. Gas chromatogram of toxaphene.

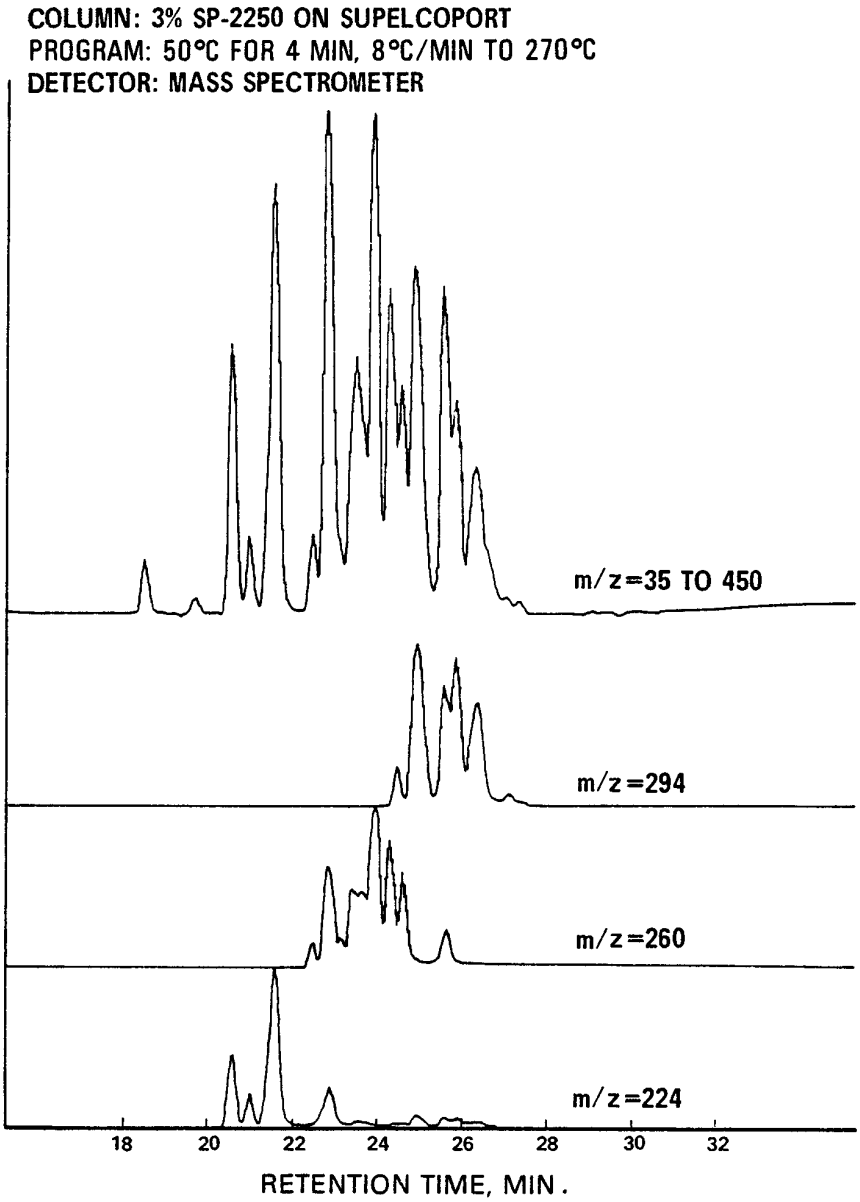


Figure 6. Gas chromatogram of PCB-1016.

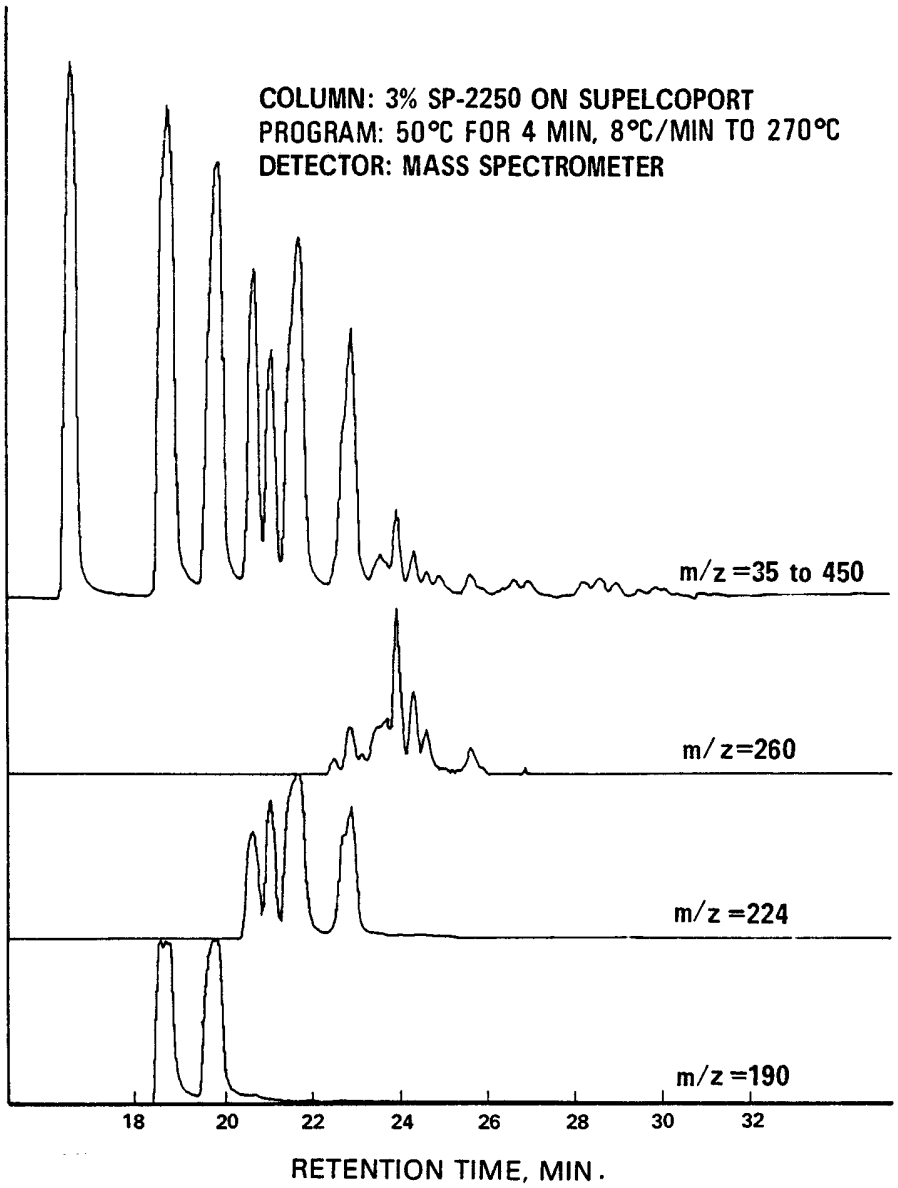


Figure 7. Gas chromatogram of PCB-1221.

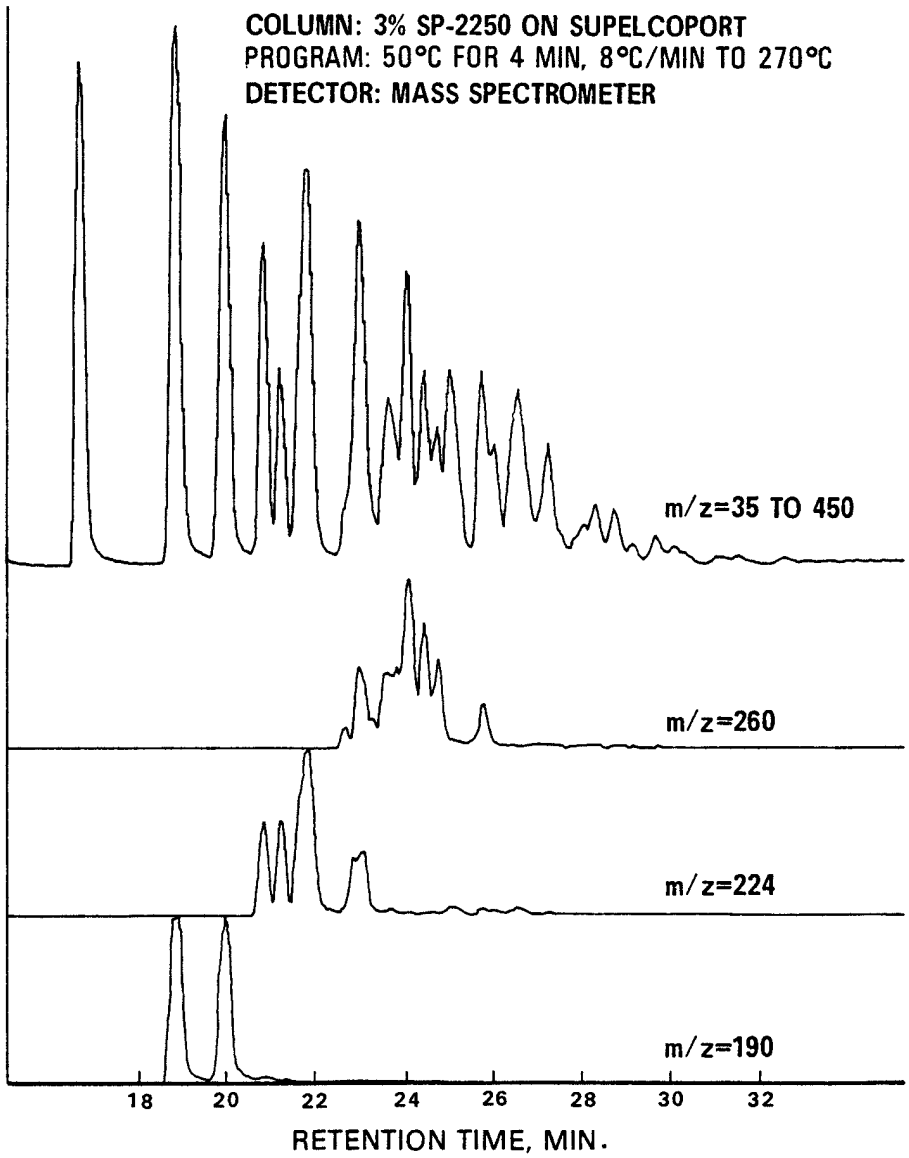


Figure 8. Gas chromatogram of PCB-1232.

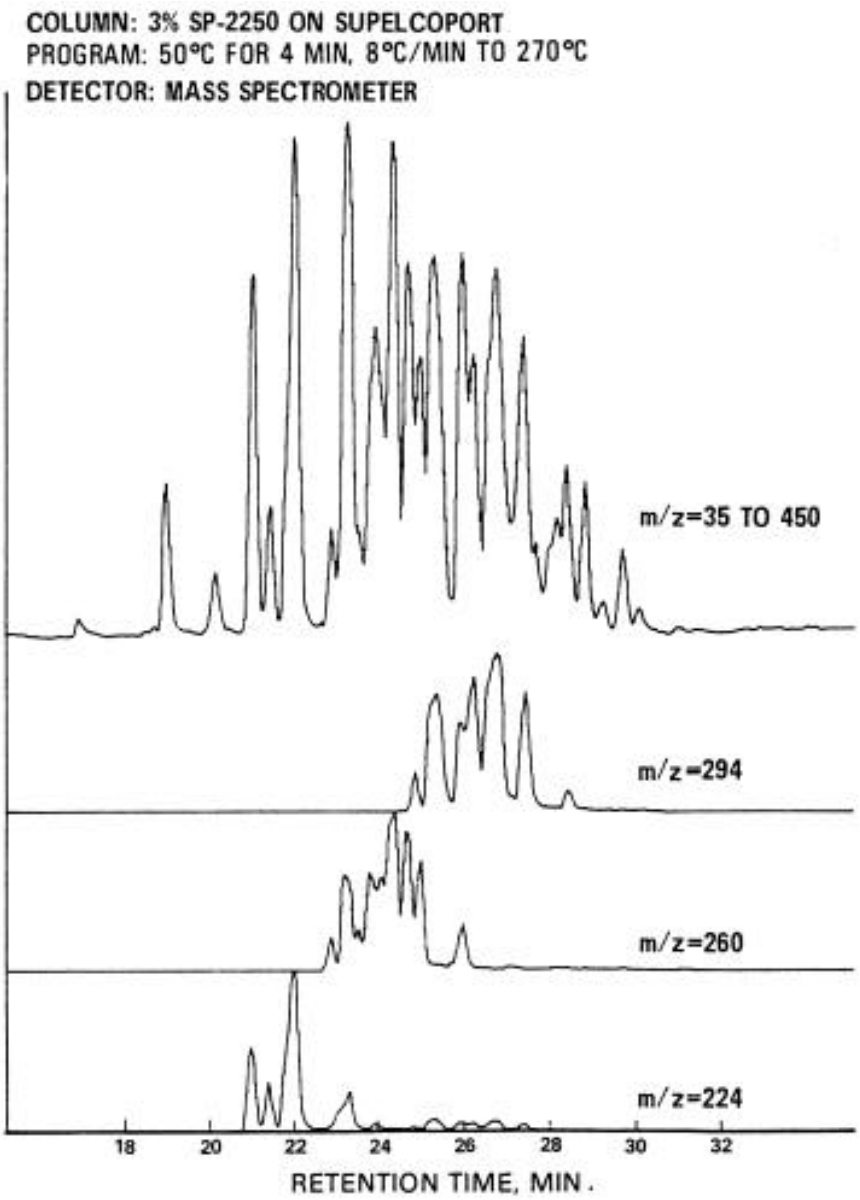


Figure 9. Gas chromatogram of PCB-1242.

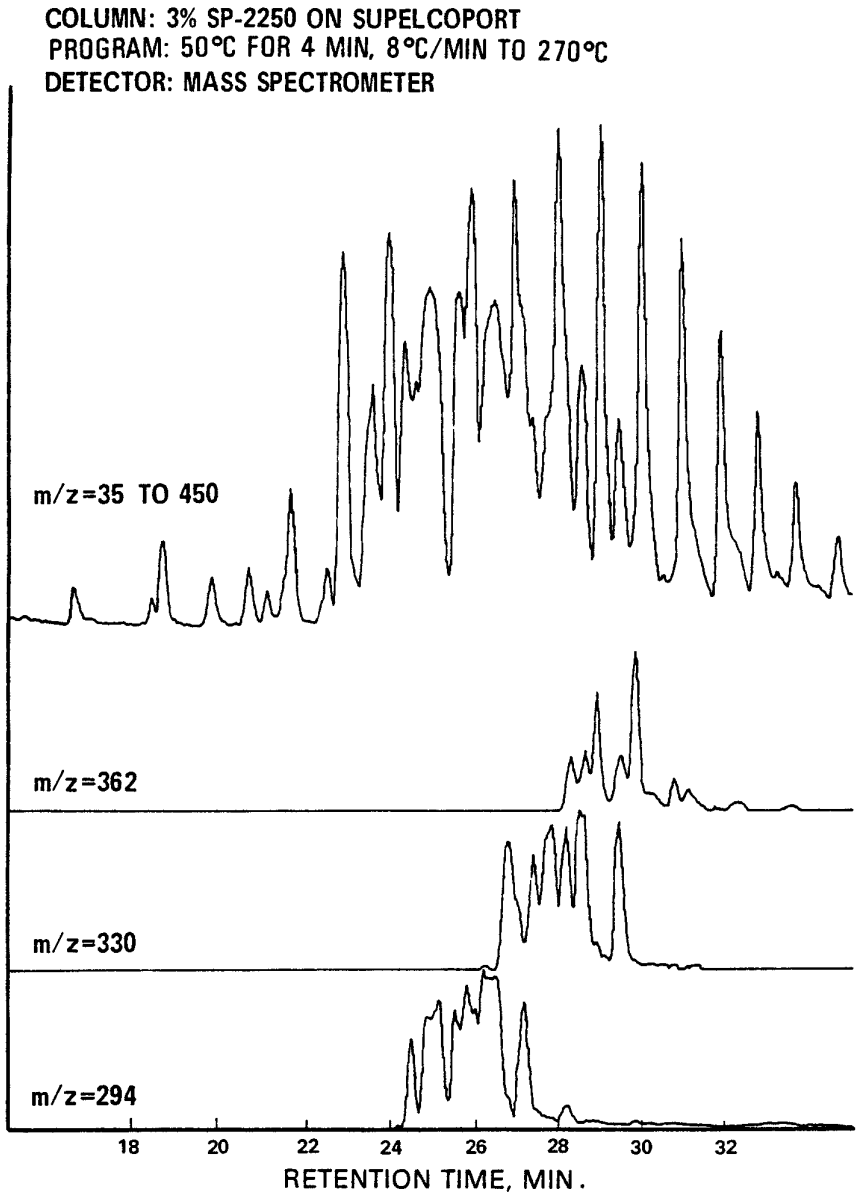


Figure 10. Gas chromatogram of PCB-1248.

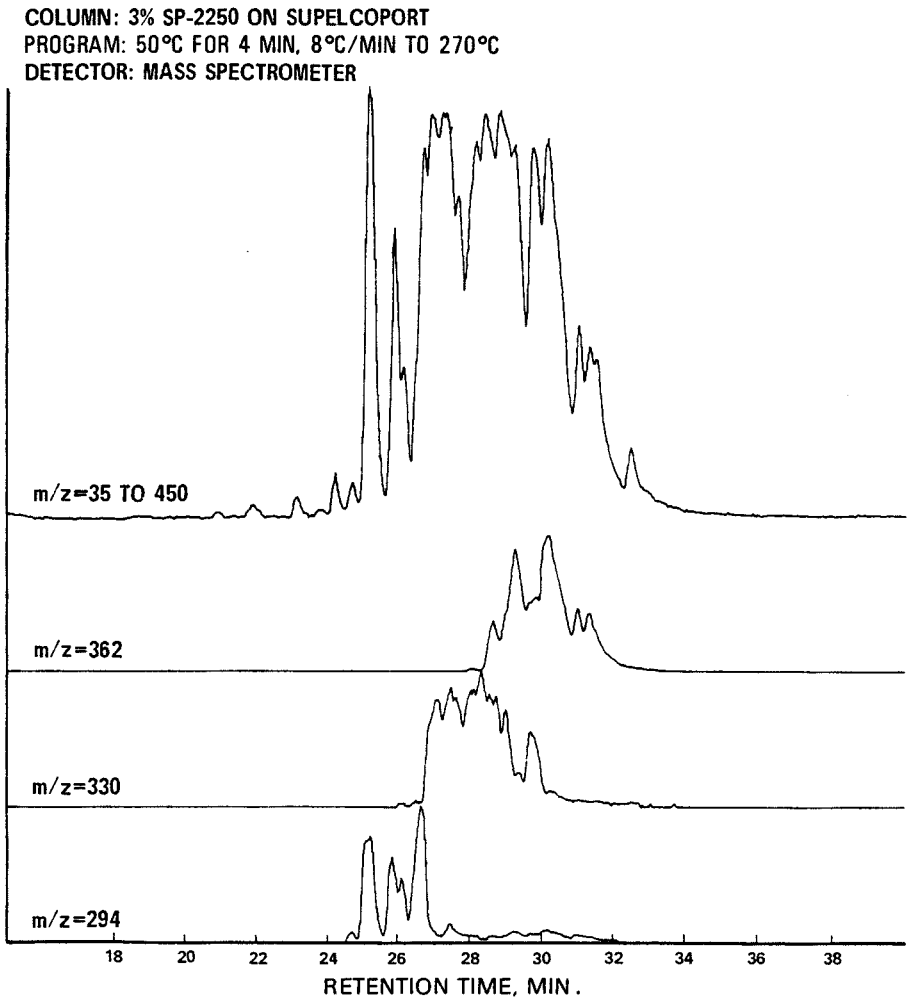


Figure 11. Gas chromatogram of PCB-1254.

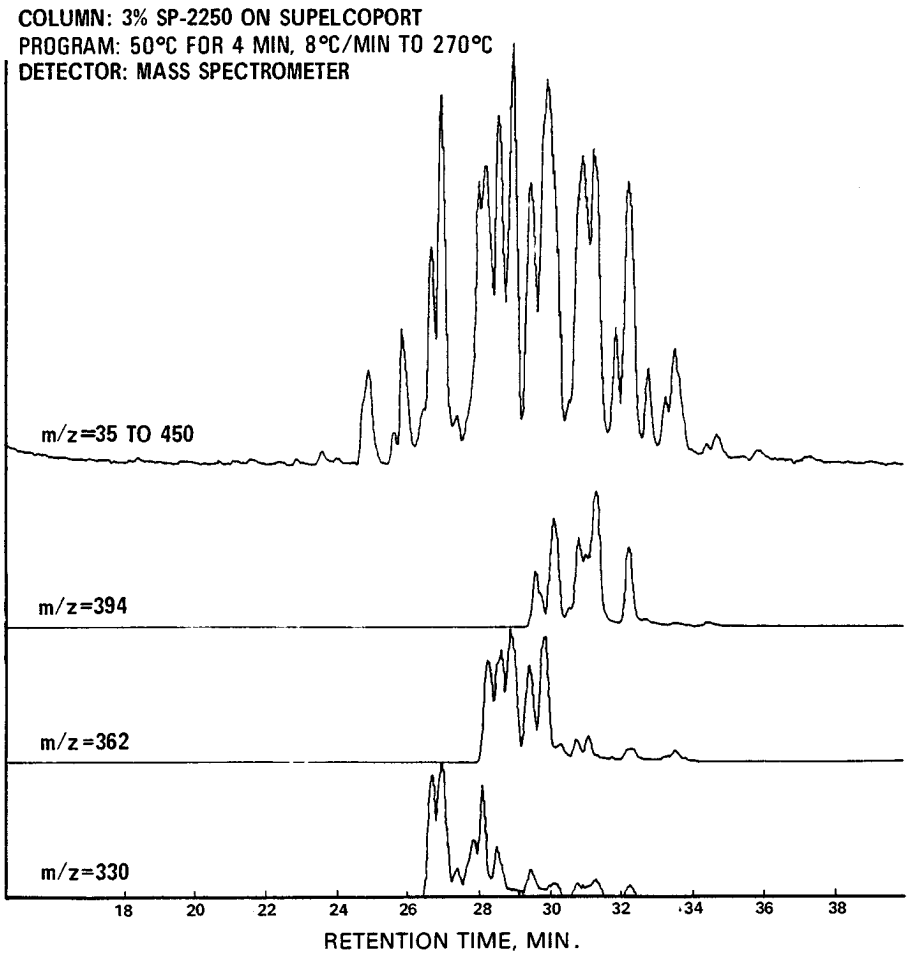
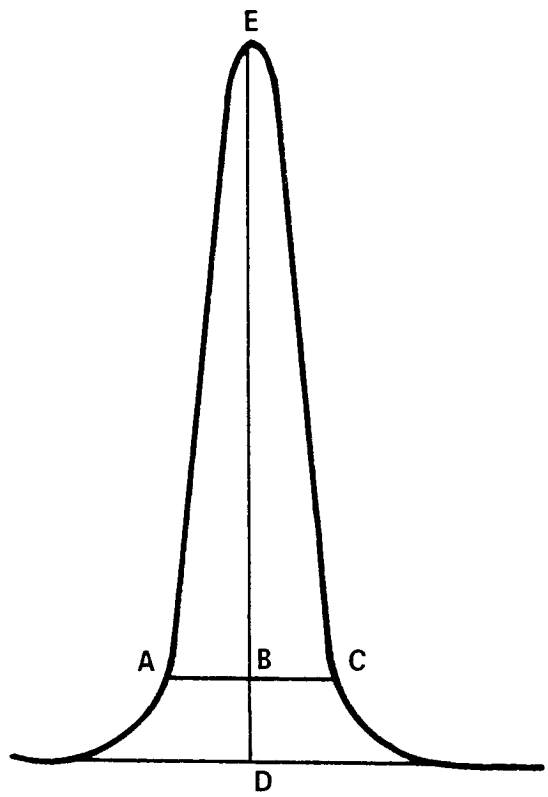


Figure 12. Gas chromatogram of PCB-1260.



$$\text{TAILING FACTOR} = \frac{BC}{AB}$$

Example calculation: Peak Height = DE = 100 mm
10% Peak Height = BD = 10 mm
Peak Width at 10% Peak Height = AC = 23 mm
AB = 11 mm
BC = 12 mm

Therefore: Tailing Factor $= \frac{12}{11} = 1.1$

Figure 13. Tailing factor calculation.

METHOD 1624 REVISION B—VOLATILE ORGANIC COMPOUNDS BY ISOTOPE DILUTION GC/MS

1. Scope and Application

1.1 This method is designed to determine the volatile toxic organic pollutants associated with the 1976 Consent Decree and additional compounds amenable to purge and trap gas chromatography-mass spectrometry (GC/MS).

1.2 The chemical compounds listed in table 1 may be determined in municipal and industrial discharges by this method. The methmd is designed to meet the survey requirements of Effluent Guidelines Division (EGD) and the National Pollutants Discharge Elimination System (NPDES) under 40 CFR 136.1 and 136.5. Any modifications of this method, beyond those expressly permitted, shall be considered as major modifications subject to application and approval of alternate test procedures under 40 CFR 136.4 and 136.5.

1.3 The detection limit of this method is usually dependent on the level of interferences rather than instrumental limitations. The limits in table 2 represent the minimum quantity that can be detected with no interferences present.

1.4 The GC/MS portions of this method are for use only by analysts experienced with GC/MS or under the close supervision of such qualified persons. Laboratories unfamiliar with the analyses of environmental samples by GC/MS should run the performance tests in reference 1 before beginning.

2. Summary of Method

2.1 Stable isotopically labeled analogs of the compounds of interest are added to a 5 mL water sample. The sample is purged at 20–25 °C with an inert gas in a specially designed chamber. The volatile organic compounds are transferred from the aqueous phase into the gaseous phase where they are passed into a sorbent column and trapped. After purging is completed, the trap is backflushed and heated rapidly to desorb the compounds into a gas chromatograph (GC). The compounds are separated by the GC and detected by a mass spectrometer (MS) (references 2 and 3). The labeled compounds serve to correct the variability of the analytical technique.

2.2 Identification of a compound (qualitative analysis) is performed by comparing the GC retention time and the background corrected characteristic spectral masses with those of authentic standards.

2.3 Quantitative analysis is performed by GC/MS using extracted ion current profile (EICP) areas. Isotope dilution is used when labeled compounds are available; otherwise, an internal standard method is used.

2.4 Quality is assured through reproducible calibration and testing of the purge and trap and GC/MS systems.

3. Contamination and Interferences

3.1 Impurities in the purge gas, organic compounds out-gassing from the plumbing upstream of the trap, and solvent vapors in the laboratory account for the majority of contamination problems. The analytical system is demonstrated to be free from interferences under conditions of the analysis by analyzing blanks initially and with each sample lot (samples analyzed on the same 8 hr shift), as described in Section 8.5.

3.2 Samples can be contaminated by diffusion of volatile organic compounds (particularly methylene chloride) through the bottle seal during shipment and storage. A field blank prepared from reagent water and carried through the sampling and handling protocol serves as a check on such contamination.

3.3 Contamination by carry-over can occur when high level and low level samples are analyzed sequentially. To reduce carry-over, the purging device and sample syringe are rinsed between samples with reagent water. When an unusually concentrated sample is encountered, it is followed by analysis of a reagent water blank to check for carry-over. For samples containing large amounts of water soluble materials, suspended solids, high boiling compounds, or high levels or purgeable compounds, the purge device is washed with soap solution, rinsed with tap and distilled water, and dried in an oven at 100–125 °C. The trap and other parts of the system are also subject to contamination; therefore, frequent bakeout and purging of the entire system may be required.

3.4 Interferences resulting from samples will vary considerably from source to source, depending on the diversity of the industrial complex or municipality being sampled.

4. Safety

4.1 The toxicity or carcinogenicity of each compound or reagent used in this method has not been precisely determined; however, each chemical compound should be treated as a potential health hazard. Exposure to these compounds should be reduced to the lowest possible level. The laboratory is responsible for maintaining a current awareness file of OSHA regulations regarding the safe handling of the chemicals specified in this method. A reference file of data handling sheets should also be made available to all personnel involved in these analyses. Additional information on laboratory safety can be found in references 4–6.

4.2 The following compounds covered by this method have been tentatively classified as known or suspected human or mammalian carcinogens: benzene, carbon tetrachloride,

chloroform, and vinyl chloride. Primary standards of these toxic compounds should be prepared in a hood, and a NIOSH/MESA approved toxic gas respirator should be worn when high concentrations are handled.

5. Apparatus and Materials

5.1 Sample bottles for discrete sampling.

5.1.1 Bottle—25 to 40 mL with screw cap (Pierce 13075, or equivalent). Detergent wash, rinse with tap and distilled water, and dry at >105 °C for one hr minimum before use.

5.1.2 Septum—Teflon-faced silicone (Pierce 12722, or equivalent), cleaned as above and baked at 100–200 °C, for one hour minimum.

5.2 Purge and trap device—consists of purging device, trap, and desorber. Complete devices are commercially available.

5.2.1 Purging device—designed to accept 5 mL samples with water column at least 3 cm deep. The volume of the gaseous head space between the water and trap shall be less than 15 mL. The purge gas shall be introduced less than 5 mm from the base of the water column and shall pass through the water as bubbles with a diameter less than 3 mm. The purging device shown in Figure 1 meets these criteria.

5.2.2 Trap—25 to 30 cm x 2.5 mm i.d. minimum, containing the following:

5.2.2.1 Methyl silicone packing—one ± 0.2 cm, 3 percent OV–1 on 60/80 mesh Chromosorb W, or equivalent.

5.2.2.2 Porous polymer—15 ± 1.0 cm, Tenax GC (2,6-diphenylene oxide polymer), 60/80 mesh, chromatographic grade, or equivalent.

5.2.2.3 Silica gel—8 ± 1.0 cm, Davison Chemical, 35/60 mesh, grade 15, or equivalent. The trap shown in Figure 2 meets these specifications.

5.2.3 Desorber—shall heat the trap to 175 ± 5 °C in 45 seconds or less. The polymer section of the trap shall not exceed 180 °C, and the remaining sections shall not exceed 220 °C. The desorber shown in Figure 2 meets these specifications.

5.2.4 The purge and trap device may be a separate unit or coupled to a GC as shown in Figures 3 and 4.

5.3 Gas chromatograph—shall be linearly temperature programmable with initial and final holds, shall contain a glass jet separator as the MS interface, and shall produce results which meet the calibration (Section 7), quality assurance (Section 8), and performance tests (Section 11) of this method.

5.3.1 Column—2.8 ± 0.4 m x 2 ± 0.5 mm i. d. glass, packekd with one percent SP–1000 on Carbopak B, 60/80 mesh, or equivalent.

5.4 Mass spectrometer—70 eV electron impact ionization; shall repetitively scan from 20 to 250 amu every 2–3 seconds, and produce a unit resolution (valleys between m/z 174–176 less than 10 percent of the height of the m/z 175 peak), background corrected mass spectrum from 50 ng 4-bromo-fluorobenzene (BFB) injected into the GC. The BFB spectrum shall meet the mass-intensity criteria in Table 3. All portions of the GC column, transfer lines, and separator which connect the GC column to the ion source shall remain at or above the column temperature during analysis to preclude condensation of less volatile compounds.

5.5 Data system—shall collect and record MS data, store mass intensity data in spectral libraries, process GC/MS data and generate reports, and shall calculate and record response factors.

5.5.1 Data acquisition—mass spectra shall be collected continuously throughout the analysis and stored on a mass storage device.

5.5.2 Mass spectral libraries—user created libraries containing mass spectra obtained from analysis of authentic standards shall be employed to reverse search GC/MS runs for the compounds of interest (Section 7.2).

5.5.3 Data processing—the data system shall be used to search, locate, identify, and quantify the compounds of interest in each GC/MS analysis. Software routines shall be employed to compute retention times and EICP areas. Displays of spectra, mass chromatograms, and library comparisons are required to verify results.

5.5.4 Response factors and multipoint calibrations—the data system shall be used to record and maintain lists of response factors (response ratios for isotope dilution) and generate multi-point calibration curves (Section 7). Computations of relative standard deviation (coefficient of variation) are useful for testing calibration linearity. Statistics on initial and on-going performance shall be maintained (Sections 8 and 11).

5.6 Syringes—5 mL glass hypodermic, with Luer-lok tips.

5.7 Micro syringes—10, 25, and 100 uL.

5.8 Syringe valves—2-way, with Luer ends (Telfon or Kel-F).

5.9 Syringe—5 mL, gas-tight, with shut-off valve.

5.10 Bottles—15 mL., screw-cap with Telfon liner.

5.11 Balance—analytical, capable of weighing 0.1 mg.

6. Reagents and Standards

6.1 Reagent water—water in which the compounds of interest and interfering compounds are not detected by this method (Section 11.7). It may be generated by any of the following methods:

6.1.1 Activated carbon—pass tap water through a carbon bed (Calgon Filtrasorb-300, or equivalent).

6.1.2 Water purifier—pass tap water through a purifier (Millipore Super Q, or equivalent).

6.1.3 Boil and purge—heat tap water to 90–100 °C and bubble contaminant free inert gas through it for approx one hour. While still

hot, transfer the water to screw-cap bottles and seal with a Teflon-lined cap.

6.2 Sodium thiosulfate—ACS granular.

6.3 Methanol—pesticide quality or equivalent.

6.4 Standard solutions—purchased as solution or mixtures with certification to their purity, concentration, and authenticity, or prepared from materials of known purity and composition. If compound purity is 96 percent or greater, the weight may be used without correction to calculate the concentration of the standard.

6.5 Preparation of stock solutions—prepare in methanol using liquid or gaseous standards per the steps below. Observe the safety precautions given in Section 4.

6.5.1 Place approx 9.8 mL of methanol in a 10 mL ground glass stoppered volumetric flask. Allow the flask to stand unstoppered for approximately 10 minutes or until all methanol wetted surfaces have dried. In each case, weigh the flask, immediately add the compound, then immediately reweigh to prevent evaporation losses from affecting the measurement.

6.5.1.1 Liquids—using a 100 µL syringe, permit 2 drops of liquid to fall into the methanol without contacting the leck of the flask. Alternatively, inject a known volume of the compound into the methanol in the flask using a micro-syringe.

6.5.1.2 Gases (chloromethane, bromomethane, chloroethane, vinyl chloride)—fill a valved 5 mL gas-tight syringe with the compound. Lower the needle to approximately 5 mm above the methanol meniscus. Slowly introduce the compound above the surface of the meniscus. The gas will dissolve rapidly in the methanol.

6.5.2 Fill the flask to volume, stopper, then mix by inverting several times. Calculate the concentration in mg/mL (µg/µL) from the weight gain (or density if a known volume was injected).

6.5.3 Transfer the stock solution to a Teflon sealed screw-cap–bottle. Store, with minimal headspace, in the dark at −10 to −20 °C.

6.5.4 Prepare fresh standards weekly for the gases and 2-chloroethylvinyl ether. All other standards are replaced after one month, or sooner if comparison with check standards indicate a change in concentration. Quality control check standards that can be used to determine the accuracy of calibration standards are available from the US Environmental Protection Agency, Environmental Monitoring and Support Laboratory, Cincinnati, Ohio.

6.6 Labeled compound spiking solution—from stock standard solutions prepared as above, or from mixtures, prepare the spiking solution to contain a concentration such that a 5–10 µL spike into each 5 mL sample, blank, or aqueous standard analyzed will result in a concentration of 20 µg/L of each labeled compound. For the gases and for the water soluble compounds (acrolein, acrylonitrile, acetone, diethyl ether, and MEK), a concentration of 100 µg/L may be used. Include the internal standards (Section 7.5) in this solution so that a concentration of 20 µg/L in each sample, blank, or aqueous standard will be produced.

6.7 Secondary standards—using stock solutions, prepare a secondary standard in methanol to contain each pollutant at a concentration of 500 µg/mL For the gases and water soluble compounds (Section 6.6), a concentration of 2.5 mg/mL may be used.

6.7.1 Aqueous calibration standards—using a 25 µL syringe, add 20 µL of the secondary standard (Section 6.7) to 50, 100, 200, 500, and 1000 mL of reagent water to produce concentrations of 200, 100, 50, 20, and 10 µg/L, respectively. If the higher concentration standard for the gases and water soluble compounds was chosen (Section 6.6), these compounds will be at concentrations of 1000, 500, 250, 100, and 50 µg/L in the aqueous calibration standards.

6.7.2 Aqueous performance standard—an aqueous standard containing all pollutants, internal standards, labeled compounds, and BFB is prepared daily, and analyzed each shift to demonstrate performance (Section 11). This standard shall contain either 20 or 100 µg/L of the labeled and pollutant gases and water soluble compounds, 10 µg/L BFB, and 20 µg/L of all other pollutants, labeled compounds, and internal standards. It may be the nominal 20 µg/L aqueous calibration standard (Section 6.7.1).

6.7.3 A methanolic standard containing all pollutants and internal standards is prepared to demonstrate recovery of these compounds when syringe injection and purge and trap analyses are compared. This standard shall contain either 100 µg/mL or 500 µg/mL of the gases and water soluble compounds, and 100 µg/mL of the remaining pollutants and internal standards (consistent with the amounts in the aqueous performance standard in 6.7.2).

6.7.4 Other standards which may be needed are those for test of BFB performance (Section 7.1) and for collection of mass spectra for storage in spectral libraries (Section 7.2).

7. Calibration

7.1 Assemble the gas chromatographic apparatus and establish operating conditions given in table 2. By injecting standards into the GC, demonstrate that the analytical system meets the detection limits in table 2 and the mass-intensity criteria in table 3 for 50 ng BFB.

7.2 Mass spectral libraries—detection and identification of the compound of interest are dependent upon the spectra stored in user created libraries.

7.2.1 Obtain a mass spectrum of each pollutant and labeled compound and each internal standard by analyzing an authentic standard either singly or as part of a mixture in which there is no interference between closely eluted components. That only a single compound is present is determined by examination of the spectrum. Fragments not attributable to the compound under study indicate the presence of an interfering compound. Adjust the analytical conditions and scan rate (for this test only) to produce an undistorted spectrum at the GC peak maximum. An undistorted spectrum will usually be obtained if five complete spectra are collected across the upper half of the GC peak. Software algorithms designed to ''enhance'' the spectrum may eliminate distortion, but may also eliminate authentic m/z's or introduce other distortion.

7.2.3 The authentic reference spectrum is obtained under BFB tuning conditions (Section 7.1 and table 3) to normalize it to spectra from other instruments.

7.2.4 The spectrum is edited by saving the 5 most intense mass spectral peaks and all other mass spectral peaks greater than 10 percent of the base peak. This spectrum is stored for reverse search and for compound confirmation.

7.3 Assemble the purge and trap device. Pack the trap as shown in Figure 2 and condition overnight at 170–180 °C by backflushing with an inert gas at a flow rate of 20–30 mL/min. Condition traps daily for a minimum of 10 minutes prior to use.

7.3.1 Analyze the aqueous performance standard (Section 6.7.2) according to the purge and trap procedure in Section 10. Compute the area at the primary m/z (table 4) for each compound. Compare these areas to those obtained by injecting one µL of the methanolic standard (Section 6.7.3) to determine compound recovery. The recovery shall be greater than 20 percent for the water soluble compounds, and 60–110 percent for all other compounds. This recovery is demonstrated initially for each purge and trap GC/MS system. The test is repeated only if the purge and trap or GC/MS systems are modified in any way that might result in a change in recovery.

7.3.2 Demonstrate that 100 ng toluene (or toluene-d8) produces an area at m/z 91 (or 99) approx one-tenth that required to exceed the linear range of the system. The exact value must be determined by experience for each instrument. It is used to match the calibration range of the instrument to the analytical range and detection limits required.

7.4 Calibration by isotope dilution—the isotope dilution approach is used for the purgeable organic compounds when appropriate labeled compounds are available and when interferences do not preclude the analysis. If labeled compounds are not available, or interferences are present, internal standard methods (Section 7.5 or 7.6) are used. A calibration curve encompassing the concentration range of interest is prepared for each compound determined. The relative response (RR) vs concentration (µg/L) is plotted or computed using a linear regression. An example of a calibration curve for toluene using toluene-d8 is given in figure 5. Also shown are the ±10 percent error limits (dotted lines). Relative response is determined according to the procedures described below. A minimum of five data points are required for calibration (Section 7.4.4).

7.4.1 The relative response (RR) of pollutant to labeled compound is determined from isotope ratio values calculated from acquired data. Three isotope ratios are used in this process:

R_x=the isotope ratio measured in the pure pollutant (figure 6A).

R_y=the isotope ratio of pure labeled compound (figure 6B).

R_m=the isotope ratio measured in the analytical mixture of the pollutant and labeled compounds (figure 6C).

The correct way to calculate RR is: $RR=(R_y-R_m)\ (R_x+1)/(R_m-R_x)(R_y+1)$ If R_m is not between $2R_y$ and $0.5R_x$, the method does not apply and the sample is analyzed by internal or external standard methods (Section 7.5 or 7.6).

7.4.2 In most cases, the retention times of the pollutant and labeled compound are the same and isotope ratios (R's) can be calculated from the EICP areas, where: R=(area at m_1/z)/(area at m_2/z) If either of the areas is zero, it is assigned a value of one in the calculations; that is, if: area of m_1/z=50721, and area of m_2/z=0, then R=50721/1=50720. The m/z's are always selected such that $R_x>R_y$. When there is a difference in retention times (RT) between the pollutant and labeled compounds, special precautions are required to determine the isotope ratios.

R_x, R_y, and R_m are defined as follows:

R_x=[area m_1/z (at RT_1)]/1

R_y=1/[area m_2/z (at RT_2)]

R_m=[area m_1/z (at RT_1)]/[area m_2/z (at RT_2)]

7.4.3 An example of the above calculations can be taken from the data plotted in figure 6 for toluene and toluene-d8. For these data, R_x=168920/1=168900, R_y=1/60960=0.00001640, and R_m=96868/82508=1.174. The RR for the above data is then calculated using the equation given in Section 7.4.1. For the example, RR=1.174.

NOTE: Not all labeled compounds elute before their pollutant analogs.

7.4.4 To calibrate the analytical system by isotope dilution, analyze a 5 mL aliquot of each of the aqueous calibration standards (Section 6.7.1) spiked with an appropriate constant amount of the labeled compound spiking solution (Section 6.6), using the purge and trap procedure in section 10. Compute the RR at each concentration.

7.4.5 Linearity—if the ratio of relative response to concentration for any compound is constant (less than 20 percent coefficient of variation) over the 5 point calibration range, an averaged relative response/concentration ratio may be used for that compound; otherwise, the complete calibration curve for that compound shall be used over the 5 point calibration range.

7.5 Calibration by internal standard—used when criteria for isotope dilution (Section 7.4) cannot be met. The method is applied to pollutants having no labeled analog and to the labeled compounds. The internal standards used for volatiles analyses are bromochloromethane, 2-bromo-1-chloropropane, and 1,4-dichlorobutane. Concentrations of the labeled compounds and pollutants without labeled analogs are computed relative to the nearest eluted internal standard, as shown in table 2.

7.5.1 Response factors—calibration requires the determination of response factors (RF) which are defined by the following equation:

$RF=(A_s x C_{is})/(A_{is} x C_s)$, where A_s is the EICP area at the characteristic m/z for the compound in the daily standard. A_{is} is the EICP area at the characteristic m/z for the internal standard.

C_{is} is the concentration (ug/L) of the internal standard

C_s is the concentration of the pollutant in the daily standard.

7.5.2 The response factor is determined at 10, 20, 50, 100, and 200 ug/L for the pollutants (optionally at five times these concentrations for gases and water soluble pollutants—see Section 6.7), in a way analogous to that for calibration by isotope dilution (Section 7.4.4). The RF is plotted against concentration for each compound in the standard (C_s) to produce a calibration curve.

7.5.3 Linearity—if the response factor (RF) for any compound is constant (less than 35 percent coefficient of variation) over the 5 point calibration range, an averaged response factor may be used for that compound; otherwise, the complete calibration curve for that compound shall be used over the 5 point range.

7.6 Combined calibration—by adding the isotopically labeled compounds and internal standards (Section 6.6) to the aqueous calibration standards (Section 6.7.1), a single set of analyses can be used to produce calibration curves for the isotope dilution and internal standard methods. These curves are verified each shift (Section 11.5) by purging the aqueous performance standard (Section 6.7.2). Recalibration is required only if calibration and on-going performance (Section 11.5) criteria cannot be met.

8. Quality Assurance/Quality Control

8.1 Each laboratory that uses this method is required to operate a formal quality assurance program. The minimum requirements of this program consist of an initial demonstration of laboratory capability, analysis of samples spiked with labeled compounds to evaluate and document data quality, and analysis of standards and blanks as tests of continued performance. Laboratory performance is compared to established performance criteria to determine if the results of analyses meet the performance characteristics of the method.

8.1.1 The analyst shall make an initial demonstration of the ability to generate acceptable accuracy and precision with this method. This ability is established as described in Section 8.2.

8.1.2 The analyst is permitted to modify this method to improve separations or lower the costs of measurements, provided all performance specifications are met. Each time a modification is made to the method, the analyst is required to repeat the procedure in Section 8.2 to demonstrate method performance.

8.1.3 Analyses of blanks are required to demonstrate freedom from contamination and that the compounds of interest and interfering compounds have not been carried over from a previous analysis (Section 3). The procedures and criteria for analysis of a blank are described in Sections 8.5 and 11.7.

8.1.4 The laboratory shall spike all samples with labeled compounds to monitor method performance. This test is described in Section 8.3. When results of these spikes indicate atypical method performance for samples, the samples are diluted to bring method performance within acceptable limits (Section 14.2).

8.1.5 The laboratory shall, on an on-going basis, demonstrate through the analysis of the aqueous performance standard (Section 6.7.2) that the analysis system is in control. This procedure is described in Sections 11.1 and 11.5.

8.1.6 The laboratory shall maintain records to define the quality of data that is generated. Development of accuracy statements is described in Sections 8.4 and 11.5.2.

8.2 Initial precision and accuracy—to establish the ability to generate acceptable precision and accuracy, the analyst shall perform the following operations:

8.2.1 Analyze two sets of four 5–mL aliquots (8 aliquots total) of the aqueous performance standard (Section 6.7.2) according to the method beginning in Section 10.

8.2.2 Using results of the first set of four analyses in Section 8.2.1, compute the average recovery (X) in µg/L and the standard deviation of the recovery (s) in µg/L for each compound, by isotope dilution for pollutants with a labeled analog, and by internal standard for labeled compounds and pollutants with no labeled analog.

8.2.3 For each compound, compare s and $\bar{X}$ with the corresponding limits for initial precision and accuracy found in table 5. If s and $\bar{X}$ for all compounds meet the acceptance criteria, system performance is acceptable and analysis of blanks and samples may begin. If individual $\bar{X}$ falls outside the range for accuracy, system performance is unacceptable for that compound.

NOTE: The large number of compounds in table 5 present a substantial probability that one or more will fail one of the acceptance criteria when all compoulds are analyzed. To determine if the analytical system is out of control, or if the failure can be attributed to probability, proceed as follows:

8.2.4 Using the results of the second set of four analyses, compute s and $\bar{X}$ for only those compounds which failed the test of the first set of four analyses (Section 8.2.3). If these compounds now pass, system performance is acceptable for all compounds and analysis of blanks and samples may begin. If, however, any of the same compounds fail again, the analysis system is not performing properly for the compound(s) in question. In this event, correct the problem and repeat the entire test (Section 8.2.1).

8.3 The laboratory shall spike all samples with labeled compounds to assess method performance on the sample matrix.

8.3.1 Spike and analyze each sample according to the method beginning in Section 10.

8.3.2 Compute the percent recovery (P) of the labeled compounds using the internal standard method (Section 7.5).

8.3.3 Compare the percent recovery for each compound with the corresponding labeled compound recovery limit in table 5. If the recovery of any compound falls outside its warning limit, method performance is unacceptable for that compound in that sample. Therefore, the sample matrix is complex and the sample is to be diluted and reanalyzed, per Section 14.2.

8.4 As part of the QA program for the laboratory, method accuracy for wastewater samples shall be assessed and records shall be maintained. After the analysis of five wastewater samples for which the labeled compounds pass the tests in Section 8.3.3, compute the average percent recovery (P) and the standard deviation of the percent recovery (s_p) for the labeled compounds only. Express the accuracy assessment as a percent recovery interval from $P-2s_p$ to $P+2s_p$. For example, if P=90% and s_p=10%, the accuracy interval is expressed as 70–110%. Update the accuracy assessment for each compound on a regular basis (e.g. after each 5–10 new accuracy measurements).

8.5 Blanks—reagent water blanks are analyzed to demonstrate freedom from carry-over (Section 3) and contamination.

8.5.1 The level at which the purge and trap system will carry greater than 5 µg/L of a pollutant of interest (table 1) into a succeeding blank shall be determined by analyzing successively larger concentrations of these compounds. When a sample contains this concentration or more, a blank shall be analyzed immediately following this sample to demonstrate no carry-over at the 5 µg/L level.

8.5.2 With each sample lot (samples analyzed on the same 8 hr shift), a blank shall be analyzed immediately after analysis of the aqueous performance standard (Section 11.1) to demonstrate freedom from contamination. If any of the compounds of interest (table 1) or any potentially interfering compound is found in a blank at greater than 10 µg/L (assuming a response factor of 1 relative to the nearest eluted internal standard for compounds not listed in table 1), analysis of samples is halted until the source of contamination is eliminated and a blank shows no evidence of contamination at this level.

8.6 The specifications contained in this method can be met if the apparatus used is calibrated properly, then maintained in a calibrated state.

The standards used for calibration (Section 7), calibration verification (Section 11.5) and for initial (Section 8.2) and on-going (Section 11.5) precision and accuracy should be identical, so that the most precise results will be obtained. The GC/MS instrument in particular will provide the most reproducible results if dedicated to the settings and conditions required for the analyses of volatiles by this method.

8.7 Depending on specific program requirements, field replicates may be collected to determine the precision of the sampling technique, and spiked samples may be required to determine the accuracy of the analysis when internal or external standard methods are used.

9. Sample Collection, Preservation, and Handling

9.1 Grab samples are collected in glass containers having a total volume greater than 20 mL. Fill sample bottles so that no air bubbles pass through the sample as the bottle is filled. Seal each bottle so that no air bubbles are entrapped. Maintain the hermetic seal on the sample bottle until time of analysis.

9.2 Samples are maintained at 0–4 °C from the time of collection until analysis. If the sample contains residual chlorine, add sodium thiosulfate preservative (10 mg/40 mL) to the empty sample bottles just prior to shipment to the sample site. EPA Methods 330.4 and 330.5 may be used for measurement of residual chlorine (Reference 8). If preservative has been added, shake bottle vigorously for one minute immediately after filling.

9.3 Experimental evidence indicates that some aromatic compounds, notably benzene, toluene, and ethyl benzene are susceptible to rapid biological degradation under certain environmental conditions. Refrigeration alone may not be adequate to preserve these compounds in wastewaters for more than seven days. For this reason, a separate sample should be collected, acidified, and analyzed when these aromatics are to be determined. Collect about 500 mL of sample in a clean container.

Adjust the pH of the sample to about 2 by adding HCl (1+1) while stirring. Check pH with narrow range (1.4 to 2.8) pH paper. Fill a sample container as described in Section 9.1. If residual chlorine is present, add sodium thiosulfate to a separate sample container and fill as in Section 9.1.

9.4 All samples shall be analyzed within 14 days of collection.

10. Purge, Trap, and GC/MS Analysis

10.1 Remove standards and samples from cold storage and bring to 20–25 °.

10.2 Adjust the purge gas flow rate to 40 ±4 mL/min. Attach the trap inlet to the purging device and set the valve to the purge mode (figure 3). Open the syringe valve located on the purging device sample introduction needle (figure 1).

10.3 Remove the plunger from a 5–mL syringe and attach a closed syringe valve. Open the sample bottle and carefully pour the sample into the syringe barrel until it overflows. Replace the plunger and compress the sample. Open the syringe valve and vent any residual air while adjusting the sample volume to 5.0 mL. Because this process of taking an aliquot destroys the validity of the sample for future analysis, fill a second syringe at this time to protect against possible loss of data. Add an appropriate amount of the labeled compound spiking solution (Section 6.6) through the valve bore, then close the valve.

10.4 Attach the syringe valve assembly to the syringe valve on the purging device. Open both syringe valves and inject the sample into the purging chamber.

10.5 Close both valves and purge the sample for 11.0 ± 0.1 minutes at 20–25 °C.

10.6 After the 11 minute purge time, attach the trap to the chromatograph and set the purge and trap apparatus to the desorb mode (figure 4). Desorb the trapped compounds into the GC column by heating the trap to 170–180 °C while backflushing with carrier gas at 20–60 mL/min for four minutes. Start MS data acquisition upon start of the desorb cycle, and start the GC column temperature program 3 minutes later. Table 1 summarizes the recommended operating conditions for the gas chromatograph. Included in this table are retention times and detection limits that were achieved under these conditions. Other columns may be used provided the requirements in Section 8 can be met. If the priority pollutant gases produce GC peaks so broad that the precision and recovery specifications (Section 8.2) cannot be met, the column may be cooled to ambient or sub-ambient temperatures to sharpen these peaks.

10.7 While analysis of the desorbed compounds proceeds, empty the purging chamber using the sample introduction syringe. Wash the chamber with two 5-mL portions of reagent water. After the purging device has been emptied, allow the purge gas to vent through the chamber until the frit is dry, so that it is ready for the next sample.

10.8 After desorbing the sample for four minutes, recondition the trap by returning to the purge mode. Wait 15 seconds, then close the syringe valve on the purging device to begin gas flow through the trap. Maintain the trap temperature at 170–180 °C. After approximately seven minutes, turn off the trap heater and open the syringe valve to stop the gas flow through the trap. When cool, the trap is ready for the next sample.

11. System Performance

11.1 At the beginning of each 8 hr shift during which analyses are performed, system calibration and performance shall be verified for all pollutants and labeled compounds. For these tests, analysis of the aqueous performance standard (Section 6.7.2) shall be used to verify all performance criteria. Adjustment and/or recalibration (per Section 7) shall be performed until all performance criteria are met. Only after all performance criteria are met may blanks and samples be analyzed.

11.2 BFB spectrum validity—the criteria in table 3 shall be met.

11.3 Retention times—the absolute retention times of all compounds shall approximate those given in Table 2.

11.4 GC resolution—the valley height between toluene and toluene-d8 (at m/z 91 and 99 plotted on the same graph) shall be less than 10 percent of the taller of the two peaks.

11.5 Calibration verification and on-going precision and accuracy—compute the concentration of each polutant (Table 1) by isotope dilution (Section 7.4) for those compmunds which have labeled analogs. Compute the concentration of each pollutant (Table 1) which has no labeled analog by the internal standard method (Section 7.5). Compute the concentration of the labeled compounds by the internal standard method. These concentrations are computed based on the calibration data determined in Section 7.

11.5.1 For each pollutant and labeled compound, compare the concentration with the corresponding limit for on-going accuracy in Table 5. If all compmunds meet the acceptance criteria, system performance is

acceptable and analysis of blanks and samples may continue. If any individual value falls outside the range given, system performance is unacceptable for that compound.

NOTE: The large number of compounds in Table 5 present a substantial probability that one or more will fail the acceptance criteria when all compounds are analyzed. To determine if the analytical system is out of control, or if the failure may be attributed to probability, proceed as follows:

11.5.1.1 Analyze a second aliquot of the aqueous performance standard (Section 6.7.2).

11.5.1.2 Compute the concentration for only those compounds which failed the first test (Section 11.5.1). If these compounds now pass, system performance is acceptable for all compounds and analyses of blanks and samples may proceed. If, however, any of the compounds fail again, the measurement system is not performing properly for these compounds. In this event, locate and correct the problem or recalibrate the system (Section 7), and repeat the entire test (Section 11.1) for all compounds.

11.5.2 Add results which pass the specification in 11.5.1.2 to initial (Section 8.2) and previous on-going data. Update QC charts to form a graphic representation of laboratory performance (Figure 7). Develop a statement of accuracy for each pollutant and labeled compound by calculating the average percentage recovery (R) and the standard deviation of percent recovery (s_r). Express the accuracy as a recovery interval from $R-2s_r$ to $R+2s_r$. For example, if R=95% and s_r=5%, the accuracy is 85–105 percent.

12. Qualitative Determination—Accomplished by Comparison of Data from Analysis of a Sample or Blank with Data from Analysis of the Shift Standard (Section 11.1). Identification is Confirmed When Spectra and Retention Times Agree Per the Criteria Below

12.1 Labeled compounds and pollutants having no labeled analog:

12.1.1 The signals for all characteristic masses stored in the spectral library (Section 7.2.4) shall be present and shall maximize within the same two consecutive scans.

12.1.2 Either (1) the background corrected EICP areas, or (2) the corrected relative intensities of the mass spectral peaks at the GC peak maximum shall agree within a factor of two (0.5 to 2 times) for all masses stored in the library.

12.1.3 The retention time relative to the nearest eluted internal standard shall be within ±7 scans or ±20 seconds, whichever is greater.

12.2 Pollutants having a labeled analog:

12.2.1 The signals for all characteristic masses stored in the spectral library (Section 7.2.4) shall be present and shall maximize within the same two consecutive scans.

12.2.2 Either (1) the background corrected EICP areas, or (2) the corrected relative intensities of the mass spectral peaks at the GC peak maximum shall agree within a factor of two for all masses stored in the spectral library.

12.2.3 The retention time difference between the pollutant and its labeled analog shall agree within ±2 scans or ±6 seconds (whichever is greater) of this difference in the shift standard (Section 11.1).

12.3 Masses present in the experimental mass spectrum that are not present in the reference mass spectrum shall be accounted for by contaminant or background ions. If the experimental mass spectrum is contaminated, an experienced spectrometrist (Section 1.4) is to determine the presence or absence of the compound.

13. Quantitative Determination

13.1 Isotope dilution—by adding a known amount of a labeled compound to every sample prior to purging, correction for recovery of the pollutant can be made because the pollutant and its labeled analog exhibit the same effects upon purging, desorption, and gas chromatography. Relative response (RR) values for sample mixtures are used in conjunction with calibration curves described in Section 7.4 to determine concentrations directly, so long as labeled compound spiking levels are constant. For the toluene example given in Figure 6 (Section 7.4.3), RR would be equal to 1.174. For this RR value, the toluene calibration curve given in Figure 5 indicates a concentration of 31.8 µg/L.

13.2 Internal standard—calculate the concentration using the response factor determined from calibration data (Section 7.5) and the following equation:

Concentration $=(A_s \times C_{is})/(A_{is} \times RF)$ where the terms are as defined in Section 7.5.1.

13.3 If the EICP area at the quantitation mass for any compound exceeds the calibration range of the system, the sample is diluted by successive factors of 10 and these dilutions are analyzed until the area is within the calibration range.

13.4 Report results for all pollutants and labeled compounds (Table 1) found in all standards, blanks, and samples, in µg/L to three significant figures. Results for samples which have been diluted are reported at the least dilute level at which the area at the quantitation mass is within the calibration range (Section 13.3) and the labeled compound recovery is within the normal range for the Method (Section 14.2).

14. Analysis of Complex Samples

14.1 Untreated effluents and other samples frequently contain high levels (>1000 µg/L) of the compounds of interest and of interfering compounds. Some samples will foam

excessively when purged; others will overload the trap/or GC column.

14.2 Dilute 0.5 mL of sample with 4.5 mL of reagent water and analyze this diluted sample when labeled compound recovery is outside the range given in Table 5. If the recovery remains outside of the range for this diluted sample, the aqueous performance standard shall be analyzed (Section 11) and calibration verified (Section 11.5). If the recovery for the labeled compmund in the aqueous performance standard is outside the range given in Table 5, the analytical system is out of control. In this case, the instrumelt shall be repaired, the performance specifications in Section 11 shall be met, and the analysis of the undiluted sample shall be repeated. If the recovery for the aqueous performance standard is within the range given in Table 5, the method does not work on the sample being analyzed and the result may not be reported for regulatory compliance purposes.

14.3 Reverse search computer programs can misinterpret the spectrum of chromatographically unresolved pollutant and labeled compound pairs with overlapping spectra when a high level of the pollutant is present. Examine each chromatogram for peaks greater than the height of the internal standard peaks. These peaks can obscure the compounds of interest.

15. Method Performance

15.1 The specifications for this method were taken from the inter-laboratory validation of EPA Method 624 (reference 9). Method 1624 has been shown to yield slightly better performance on treated effluents than Method 624. Additional method performance data can be found in Reference 10.

References

1. ''Performance Tests for the Evaluation of Computerized Gas Chromatography/Mass Spectrometry Equipment and Laboratories,'' USEPA, EMSL/Cincinnati, OH 45268, EPA–600/4–80–025 (April 1980).

2. Bellar, T.A. and Lichtenberg, J.J., ''Journal American Water Works Association,'' *66*, 739 (1974).

3. Bellar, T.A. and Lichtenberg, J.J., ''Semi-automated Headspace Analysis of Drinking Waters and Industrial Waters for Purgeable Volatile Organic Compounds,'' in *Measurement of Organic Pollutants Water and Wastewater*, C.E. VanHall, ed., American Society for Testing Materials, Philadelphia, PA, Special Technical Publication 686, (1978).

4. ''Working with Carcinogens,'' DHEW, PHS, NIOSH, Publication 77–206 (1977).

5. ''OSHA Safety and Health Standards, General Industry,'' 29 CFR part 1910, OSHA 2206, (1976).

6. ''Safety in Academic Chemistry Laboratories,'' American Chemical Society Publication, Committee on Chemical Safety (1979).

7. ''Handbook of Analytical Quality Control in Water and Wastewater Laboratories,'' USEPA, EMSL/Cincinnati, OH 45268, EPA–4–79–019 (March 1979).

8. ''Methods 330.4 and 330.5 for Total Residual Chlorine,'' USEPA, EMSL/Cincinnati, OH 45268, EPA–4–79–020 (March 1979).

9. ''EPA Method Study 29 EPA Method 624—Purgeables,'' EPA 600/4–84–054, National Technical Information Service, PB84–209915, Springfield, Virginia 22161, June 1984.

10. ''Colby, B.N., Beimer, R.G., Rushneck, D.R., and Telliard, W.A., ''Isotope Dilution Gas Chromatography-Mass Spectrometry for the Determination of Priority Pollutants in Industrial Effluents,'' USEPA, Effluent Guidelines Division, Washington, DC 20460 (1980).

TABLE 1—VOLATILE ORGANIC COMPOUNDS ANALYZED BY ISOTOPE DILUTION GC/MS

Compound	Storet	CAS registry	EPA–EGD	NPDES
Acetone	81552	67–64–1	516 V	.....
Acrolein	34210	107–02–8	002 V	001 V
Acrylonitrile	34215	107–13–1	003 V	002 V
Benzene	34030	71–43–2	004 V	003 V
Bromodichloromethane	32101	75–27–4	048 V	012 V
Bromoform	32104	75–25–2	047 V	005 V
Bromomethane	34413	74–83–9	046 V	020 V
Carbon tetrachloride	32102	56–23–5	006 V	006 V
Chlorobenzene	34301	108–90–7	007 V	007 V
Chloroethane	34311	75–00–3	016 V	009 V
2-chloroethylvinyl ether	34576	110–75–8	019 V	010 V
Chloroform	32106	67–66–1	023 V	011 V
Chloromethane	34418	74–87–3	045 V	021 V
Dibromochloromethane	32105	124–48–1	051 V	008 V
1,1-dichloroethane	34496	75–34–3	013 V	014 V
1,2-dichloroethane	34536	107–06–2	010 V	015 V
1,1-dichloroethene	34501	75–35–4	029 V	016 V
Trans-1,2-dichloroethane	34546	156–60–5	030 V	026 V
1,2-dichloropropane	34541	78–87–5	032 V	017 V
Cis-1,3-dichloropropene	34704	10061–01–5	.....	.....

TABLE 1—VOLATILE ORGANIC COMPOUNDS ANALYZED BY ISOTOPE DILUTION GC/MS—Continued

Compound	Storet	CAS registry	EPA–EGD	NPDES
Trans-1,3-dichloropropene	34699	10061–02–6	033 V	.....
Diethyl ether	81576	60–29–7	515 V	.....
P-dioxane	81582	123–91–1	527 V	.....
Ethylbenzene	34371	100–41–4	038 V	019 V
Methylene chloride	34423	75–09–2	044 V	022 V
Methyl ethyl ketone	81595	78–93–3	514 V	.....
1,1,2,2-tetrachloroethane	34516	79–34–5	015 V	023 V
Tetrachlorethene	34475	127–18–4	085 V	024 V
Toluene	34010	108–88–3	086 V	025 V
1,1,1-trichloroethane	34506	71–55–6	011 V	027 V
1,1,2-trichloroethane	34511	79–00–5	014 V	028 V
Trichloroethene	39180	79–01–6	087 V	029 V
Vinyl chloride	39175	75–01–4	088 V	031 V

TABLE 2—GAS CHROMATOGRAPHY OF PURGEABLE ORGANIC COMPOUNDS BY ISOTOPE DILUTION GC/MS

EGD No. (1)	Compound	Ref EGD No.	Mean retention time (sec)	Minimum level (2) (µg/L)
181	Bromochloromethane (I.S.)	181	730	10
245	Chloromethane-d3	181	147	50
345	Chloromethane	245	148	50
246	Bromomethane-d3	181	243	50
346	Bromomethane	246	246	50
288	Vinyl chloride-d3	181	301	50
388	Vinyl chloride	288	304	10
216	Chloroethane-d5	181	378	50
316	Chloroethane	216	386	50
244	Methylene chloride-d2	181	512	10
344	Methylene chloride	244	517	10
616	Acetone-d6	181	554	50
716	Acetone	616	565	50
002	Acrolein	181	566	50
203	Acrylonitrile-d3	181	606	50
303	Acrylonitrile	203	612	50
229	1,1-dichloroethene-d2	181	696	10
329	1,1-dichloroethene	229	696	10
213	1,1-dichloroethane-d3	181	778	10
313	1,1-dichloroethane	213	786	10
615	Diethyl ether-d10	181	804	50
715	Diethyl ether	615	820	50
230	Trans-1,2-dichloroethene-d2	181	821	10
330	Trans-1,2-dichloroethene	230	821	10
614	Methyl ethyl ketone-d3	181	840	50
714	Methyl ethyl ketone	614	848	50
223	Chloroform-13C1	181	861	10
323	Chloroform	223	861	10
210	1,2-dichloroethane-d4	181	901	10
310	1,2-dichloroethane	210	910	10
211	1,1,1-trichloroethane-13C2	181	989	10
311	1,1,1-trichloroethane	211	999	10
527	p-dioxane	181	1001	10
206	Carbon tetrachloride-13C1	182	1018	10
306	Carbon tetrachloride	206	1018	10
248	Bromodichloromethane-13C1	182	1045	10
348	Bromodichloromethane	248	1045	10
232	1,2-dichloropropane-d6	182	1123	10
332	1.2-dichloropropane	232	1134	10
233	Trans-1,3-dichloropropene-d4	182	1138	10
333	Trans-1,3-dichloropropene	233	1138	10
287	Trichloroethene-13C1	182	1172	10
387	Trichloroethene	287	1187	10
204	Benzene-d6	182	1200	10
304	Benzene	204	1212	10
251	Chlorodibromemethane-13C1	182	1222	10

TABLE 2—GAS CHROMATOGRAPHY OF PURGEABLE ORGANIC COMPOUNDS BY ISOTOPE DILUTION GC/MS—Continued

EGD No. (1)	Compound	Ref EGD No.	Mean retention time (sec)	Minimum level (2) (µg/L)
351	Chlorodibromomethane	251	1222	10
214	1,1,2-trichloroethane-13C2	182	1224	10
314	1,1,2-trichloroethane	214	1224	10
019	2-chloroethylvinyl ether	182	1278	10
182	2-bromo-1-chloropropane (I.S.)	182	1306	10
247	Bromoform-13C1	182	1386	10
347	Bromoform	247	1386	10
215	1,1,2,2-tetrachloroethane-d2	183	1525	10
315	1,1,2,2-tetrachloroethane	215	1525	10
285	Tetrachloroethene-13C2	183	1528	10
385	Tetrachloroethene	285	1528	10
183	1,4-dichlorobutale (int std)	183	1555	10
286	Toluene-d8	183	1603	10
386	Toluene	286	1619	10
207	Chlorobenzene-d5	183	1679	10
307	Chlorobenzene	207	1679	10
238	Ethylbenzene-d10	183	1802	10
338	Ethylbenzene	238	1820	10
185	Bromofluorobenzene	183	1985	10

(1) Reference numbers beginning with 0, 1 or 5 indicate a pollutant quantified by the internal standard method; reference numbers beginning with 2 or 6 indicate a labeled compound quantified by the internal standard method; reference numbers beginning with 3 or 7 indicate a pollutant quantified by isotope dilution.

(2) This is a minimum level at which the analytical system shall give recognizable mass spectra (background corrected) and acceptable calibration points. Column: 2.4m (8 ft) x 2 mm i.d. glass, packed with one percent SP–1000 coated on 60/80 Carbopak B. Carrier gas: helium at 40 mL/min. Temperature program: 3 min at 45 °C, 8 °C per min to 240 °C, hold at 240 °C for 15 minutes.

NOTE: The specifications in this table were developed from data collected from three wastewater laboratories.

TABLE 3—BFB Mass-Intensity Specifications

Mass	Intensity required
50	15 to 40 percent of mass 95.
75	30 to 60 percent of mass 95.
95	base peak, 100 percent.
96	5 to 9 percent of mass 95.
173	<2 percent of mass 174.
174	>50 percent of mass 95.
175	5 to 9 percent of mass 174
176	95 to 101 percent of mass 174

TABLE 3—BFB Mass-Intensity Specifications—Continued

Mass	Intensity required
177	5 to 9 percent of mass 176.

TABLE 4—VOLATILE ORGANIC COMPOUND CHARACTERISTIC MASSES

Labeled compound	Analog	Primary m/z's
Acetone	d6	58/64
Acrolein	d2	56/58
Acrylonitrile	d3	53/56
Benzene	d6	78/84
Bromodichloromethane	13C	83/86
Bromoform	13C	173/176
Bromomethale	d3	96/99
Carbon tetrachloride	13C	47/48
Chlorobenzene	d5	112/117
Chloroethane	d5	64/71
2-chloroethylvinyl ether	d7	106/113
Chloroform	13C	85/86
Chloromethane	d3	50/53

TABLE 4—VOLATILE ORGANIC COMPOUND CHARACTERISTIC MASSES—Continued

Labeled compound	Analog	Primary m/z's
Dibromochloromethane	13C	129/130
1,1-dichloroethane	d3	63/66
1,2-dichloroethane	d4	62/67
1,1-dichloroethene	d2	61/65
Trans-1,2-dichloroethene	d2	61/65
1,2-dichloropropane	d6	63/67
Cis-1,3-dichloropropene	d4	75/79
Trans-1,3-dichloropropene	d4	75/79
Diethyl ether	d10	74/84
p-dioxane	d8	88/96
Ethylbenzene	d10	106/116
Methylene chloride	d2	84/88
Methyl ethyl ketone	d3	72/75
1,1,2,2-tetrachloroethane	d2	83/84
Tetrachloroethene	13C2	166/172
Toluene	d8	92/99
1,1,1-trichloroethane	d3	97/102
1,1,2-trichloroethane	13C2	83/84
Trichloroethene	13C	95/133
Vinyl chloride	d3	62/65

TABLE 5—ACCEPTANCE CRITERIA FOR PERFORMANCE TESTS

Compound	Acceptance criteria at 20 µg/L			
	Initial precision and accuracy section 8.2.3		Labeled compound recovery sec. 8.3 and 14.2	On-going accuracy sec. 11.5
	s (µg/L)	$\bar{X}$ (µg/L)	P (percent)	R (µg/L)
Acetone		Note 1		
Acrolein		Note 2		
Acrylonitrile		Note 2		
Benzene	9.0	13.0–28.2	ns–196	4–33
Bromodichloromethane	8.2	6.5–31.5	ns–199	4–34
Bromoform	7.0	7.4–35.1	ns–214	6–36
Bromomethane	25.0	d–54.3	ns–414	d–61
Carbon tetrachloride	6.9	15.9–24.8	42–165	12–30
Chlorobenzene	8.2	14.2–29.6	ns–205	4–35
Chloroethane	14.8	2.1–46.7	ns–308	d–51
2–chloroethylvinyl ether	36.0	d–69.8	ns–554	d–79
Chloroform	7.9	11.6–26.3	18–172	8–30
Chloromethane	26.0	d–55.5	ns–410	d–64
Dibromochloromethane	7.9	11.2–29.1	16–185	8–32
1,1-dichloroethane	6.7	11.4–31.4	23–191	9–33
1,2-dichloroethane	7.7	11.6–30.1	12–192	8–33
1,1-dichloroethene	11.7	d–49.8	ns–315	d–52
Trans-1,2–dichloroethene	7.4	10.5–31.5	15–195	8–34
1,2-dichloropropane	19.2	d–46.8	ns–343	d–51
Cis–1,3–dichloropropene	22.1	d–51.0	ns–381	d–56
Trans–1,3–dichloropropene	14.5	d–40.2	ns–284	d–44
Diethyl ether		Note 1		
P–dioxane		Note 1		
Ethyl benzene	9.6	15.6–28.5	ns–203	5–35
Methylene chloride	9.7	d–49.8	ns–316	d–50
Methyl ethyl ketone		note 1		
1,1,2,2-tetrachloroethane	9.6	10.7–30.0	5–199	7–34
Tetrachloroethene	6.6	15.1–28.5	31–181	11–32
Toluene	6.3	14.5–28.7	4–193	6–33
1,1,1-trichloroethane	5.9	10.5–33.4	12–200	8–35
1,1,2-trichloroethane	7.1	11.8–29.7	21–184	9–32
Trichloroethene	8.9	16.6–29.5	35–196	12–34
Vinyl chloride	27.9	d–58.5	ns–452	d–65

d=detected; result must be greater than zero.
ns=no specification; limit would be below detection limit.

NOTE 1: Specifications not available for these compounds at time of release of this method.
NOTE 2: Specifications not developed for these compounds; use method 603.

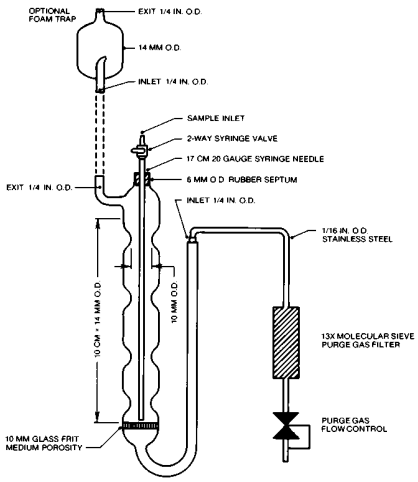


FIGURE 1 Purging Device.

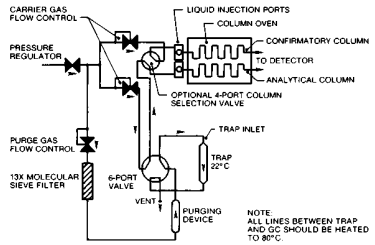


FIGURE 3 Schematic of Purge and Trap Device—Purge Mode.

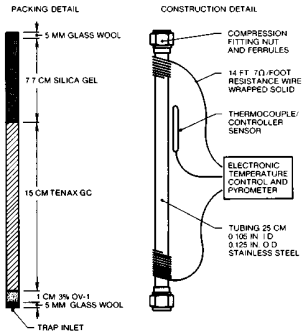


FIGURE 2 Trap Packings and Construction to Include Desorb Capability.

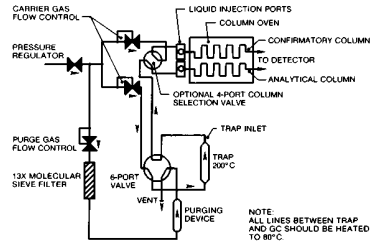


FIGURE 4 Schematic of Purge and Trap Device—Desorb Mode.

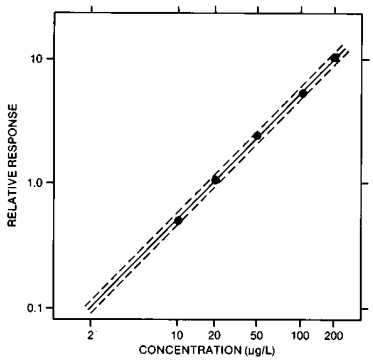


FIGURE 5 Relative Response Calibration Curve for Toluene. The Dotted Lines Enclose a ±10 Percent Error Window.

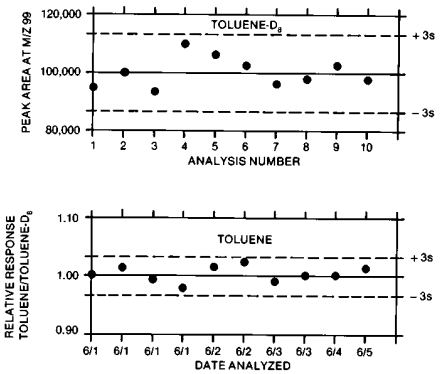


FIGURE 7 Quality Control Charts Showing Area (top graph) and Relative Response of Toluene to Toluene-d_8 (lower graph) Plotted as a Function of Time or Analysis Number.

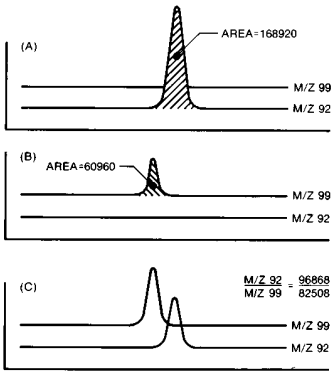


FIGURE 6 Extracted Ion Current Profiles for (A) Toluene, (B) Toluene-d_8, and a Mixture of Toluene and Toluene-d_8.

METHOD 1625 REVISION B—SEMIVOLATILE ORGANIC COMPOUNDS BY ISOTOPE DILUTION GC/MS

1. Scope and Application

1.1 This method is designed to determine the semivolatile toxic organic pollutants associated with the 1976 Consent Decree and additional compounds amenable to extraction and analysis by capillary column gas chromatography-mass spectrometry (GC/MS).

1.2 The chemical compounds listed in Tables 1 and 2 may be determined in municipal and industrial discharges by this method. The method is designed to meet the survey requirements of Effluent Guidelines Division (EGD) and the National Pollutants Discharge Elimination System (NPDES) under 40 CFR 136.1. Any modifications of this method, beyond those expressly permitted, shall be considered as major modifications subject to application and approval of alternate test procedures under 40 CFR 136.4 and 136.5.

1.3 The detection limit of this method is usually dependent on the level of interferences rather than instrumental limitations. The limits listed in Tables 3 and 4 represent the minimum quantity that can be detected with no interferences present.

1.4 The GC/MS portions of this method are for use only by analysts experienced with GC/MS or under the close supervision of such qualified persons. Laboratories unfamiliar with analyses of environmental samples by GC/MS should run the performance tests in reference 1 before beginning.

2. Summary of Method

2.1 Stable isotopically labeled analogs of the compounds of interest are added to a one liter wastewater sample. The sample is extracted at pH 12–13, then at pH <2 with methylene chloride using continuous extraction techniques. The extract is dried over sodium sulfate and concentrated to a volume of one mL. An internal standard is added to the extract, and the extract is injected into the gas chromatograph (GC). The compounds are separated by GC and detected by a mass spectrometer (MS). The labeled compounds serve to correct the variability of the analytical technique.

2.2 Identification of a compound (qualitative analysis) is performed by comparing the GC retention time and background corrected characteristic spectral masses with those of authentic standards.

2.3 Quantitative analysis is performed by GC/MS using extracted ion current profile (EICP) areas. Isotope dilution is used when labeled compounds are available; otherwise, an internal standard method is used.

2.4 Quality is assured through reproducible calibration and testing of the extraction and GC/MS systems.

3. Contamination and Interferences

3.1 Solvents, reagents, glassware, and other sample processing hardware may yield artifacts and/or elevated baselines causing misinterpretation of chromatograms and spectra. All materials shall be demonstrated to be free from interferences under the conditions of analysis by running method blanks initially and with each sample lot (samples started through the extraction process on a given 8 hr shift, to a maximum of 20). Specific selection of reagents and purification of solvents by distillation in all-glass systems may be required. Glassware and, where possible, reagents are cleaned by solvent rinse and baking at 450 °C for one hour minimum.

3.2 Interferences coextracted from samples will vary considerably from source to source, depending on the diversity of the industrial complex or municipality being samples.

4. Safety

4.1 The toxicity or carcinogenicity of each compound or reagent used in this method has not been precisely determined; however, each chemical compound should be treated as a potential health hazard. Exposure to these compounds should be reduced to the lowest possible level. The laboratory is responsible for maintaining a current awareness file of OSHA regulations regarding the safe handling of the chemicals specified in this method. A reference file of data handling sheets should also be made available to all personnel involved in these analyses. Additional information on laboratory safety can be found in references 2–4.

4.2 The following compounds covered by this method have been tentatively classified as known or suspected human or mammalian carcinogens: benzidine benzo(a)anthracene, 3,3′-dichlorobenzidine, benzo(a)pyrene, dibenzo(a,h)anthracene, N-nitrosodimethylamine, and β-naphtylamine. Primary standards of these compounds shall be prepared in a hood, and a NIOSH/MESA approved toxic gas respirator should be worn when high concentrations are handled.

5. Apparatus and Materials

5.1 Sampling equipment for discrete or composite sampling.

5.1.1 Sample bottle, amber glass, 1.1 liters minimum. If amber bottles are not available, samples shall be protected from light. Bottles are detergent water washed, then solvent rinsed or baked at 450 °C for one hour minimum before use.

5.1.2 Bottle caps—threaded to fit sample bottles. Caps are lined with Teflon. Aluminum foil may be substituted if the sample is not corrosive. Liners are detergent water washed, then reagent water (Section 6.5) and solvent rinsed, and baked at approximately 200 °C for one hour minimum before use.

5.1.3 Compositing equipment—automatic or manual compositing system incorporating glass containers for collection of a minimum 1.1 liters. Sample containers are kept at 0 to 4 °C during sampling. Glass or Teflon tubing only shall be used. If the sampler uses a peristaltic pump, a minimum length of compressible silicone rubber tubing may be used in the pump only. Before use, the tubing is thoroughly rinsed with methanol, followed by repeated rinsings with reagent water (Section 6.5) to minimize sample contamination. An integrating flow meter is used to collect proportional composite samples.

5.2 Continuous liquid-liquid extractor—Teflon or glass conncecting joints and stopcocks without lubrication (Hershberg-Wolf Extractor) one liter capacity, Ace Glass 6841–10, or equivalent.

5.3 Drying column—15 to 20 mm i.d. Pyrex chromatographic column equipped with coarse glass frit or glass wool plug.

5.4 Kuderna-Danish (K–D) apparatus

5.4.1 Concentrator tube—10mL, graduated (Kontes K–570050–1025, or equivalent) with calibration verified. Ground glass stopper (size 19/22 joint) is used to prevent evaporation of extracts.

5.4.2 Evaporation flask—500 mL (Kontes K–570001–0500, or equivalent), attached to concentrator tube with springs (Kontes K–662750–0012).

5.4.3 Snyder column—three ball macro (Kontes K–503000–0232, or equivalent).

5.4.4 Snyder column—two ball micro (Kontes K–469002–0219, or equivalent).

5.4.5 Boiling chips—approx 10/40 mesh, extracted with methylene chloride and baked at 450 °C for one hr minimum.

5.5 Water bath—heated, with concentric ring cover, capable of temperature control ±2 °C, installed in a fume hood.

5.6 Sample vials—amber glass, 2–5 mL with Teflon-lined screw cap.

5.7 Analytical balance—capable of weighing 0.1 mg.

5.8 Gas chromatograph—shall have splitless or on-column injection port for capillary column, temperature program with 30 °C hold, and shall meet all of the performance specifications in Section 12.

5.8.1 Column—30±5 m×0.25±0.02 mm i.d. 5% phenyl, 94% methyl, 1% vinyl silicone bonded phase fused silica capillary column (J & W DB–5, or equivalent).

5.9 Mass spectrometer—70 eV electron impact ionization, shall repetitively scan from 35 to 450 amu in 0.95 to 1.00 second, and shall produce a unit resolution (valleys between m/z 441–442 less than 10 percent of the height of the 441 peak), backgound corrected mass spectrum from 50 ng decafluorotriphenylphosphine (DFTPP) introduced through the GC inlet. The spectrum shall meet the mass-intensity criteria in Table 5 (reference 5). The mass spectrometer shall be interfaced to the GC such that the end of the capillary column terminates within one centimeter of the ion source but does not intercept the electron or ion beams. All portions of the column which connect the GC to the ion source shall remain at or above the column temperature during analysis to preclude condensation of less volatile compounds.

5.10 Data system—shall collect and record MS data, store mass-intensity data in spectral libraries, process GC/MS data, generate reports, and shall compute and record response factors.

5.10.1 Data acquisition—mass spectra shall be collected continuously throughout the analysis and stored on a mass storage device.

5.10.2 Mass spectral libraries—user created libraries containing mass spectra obtained from analysis of authentic standards shall be employed to reverse search GC/MS runs for the compounds of interest (Section 7.2).

5.10.3 Data processing—the data system shall be used to search, locate, identify, and quantify the compounds of interest in each GC/MS analysis. Software routines shall be employed to compute retention times and peak areas. Displays of spectra, mass chromatograms, and library comparisons are required to verify results.

5.10.4 Response factors and multipoint calibrations—the data system shall be used to record and maintain lists of response factors (response ratios for isotope dilution) and multipoint calibration curves (Section 7). Computations of relative standard deviation (coefficient of variation) are useful for testing calibration linearity. Statistics on initial (Section 8.2) and on-going (Section 12.7) performance shall be computed and maintained.

6. Reagents and Standards

6.1 Sodium hydroxide—reagent grade, 6N in reagent water.

6.2 Sulfuric acid—reagent grade, 6N in reagent water.

6.3 Sodium sulfate—reagent grade, granular anhydrous, rinsed with methylene chloride (20 mL/g) and conditioned at 450 °C for one hour minimum.

6.4 Methylene chloride—distilled in glass (Burdick and Jackson, or equivalent).

6.5 Reagent water—water in which the compounds of interest and interfering compounds are not detected by this method.

6.6 Standard solutions—purchased as solutions or mixtures with certification to their purity, concentration, and authenticity, or prepared from materials of known purity and composition. If compound purity is 96 percent or greater, the weight may be used without correction to compute the concentration of the standard. When not being used, standards are stored in the dark at −20 to −10 °C in screw-capped vials with Teflon-lined lids. A mark is placed on the vial at the

level of the solution so that solvent evaporation loss can be detected. The vials are brought to room temperature prior to use. Any precipitate is redissolved and solvent is added if solvent loss has occurred.

6.7 Preparation of stock solutions—prepare in methylene chloride, benzene, p-dioxane, or a mixture of these solvents per the steps below. Observe the safety precautions in Section 4. The large number of labeled and unlabeled acid, base/neutral, and Appendix C compounds used for combined calibration (Section 7) and calibration verification (12.5) require high concentratimns (approx 40 mg/mL) when individual stock solutions are prepared, so that dilutions of mixtures will permit calibration with all compounds in a single set of solutions. The working range for most compounds is 10–200 µg/mL. Compounds with a reduced MS response may be prepared at higher concentrations.

6.7.1 Dissolve an appropriate amount of assayed reference material in a suitable solvent. For example, weigh 400 mg naphthalene in a 10 mL ground glass stoppered volumetric flask and fill to the mark with benzene. After the naphthalene is completely dissolved, transfer the solution to a 15 mL vial with Teflon-lined cap.

6.7.2 Stock standard solutions should be checked for signs of degradation prior to the preparation of calibration or performance test standards. Quality control check samples that can be used to determine the accuracy of calibration standards are available from the US Environmental Protection Agency, Environmental Monitoring and Support Laboratory, Cincinnati, Ohio 45268.

6.7.3 Stock standard solutions shall be replaced after six months, or sooner if comparison with quality control check samples indicates a change in concentration.

6.8 Labeled compound spiking solution—from stock standard solutions prepared as above, or from mixtures, prepare the spiking solution at a concentration of 200 µg/mL, or at a concentration appropriate to the MS response of each compound.

6.9 Secondary standard—using stock solutions (Section 6.7), prepare a secondary standard containing all of the compounds in Tables 1 and 2 at a concentration of 400 µg/mL, or higher concentration appropriate to the MS response of the compound.

6.10 Internal standard solution—prepare 2,2′-difluorobiphenyl (DFB) at a concentration of 10 mg/mL in benzene.

6.11 DFTPP solution—prepare at 50 µg/mL in acetone.

6.12 Solutions for obtaining authentic mass spectra (Section 7.2)—prepare mixtures of compounds at concentrations which will assure authentic spectra are obtained for storage in libraries.

6.13 Calibration solutions—combine 0.5 mL of the solution in Section 6.8 with 25, 50, 125, 250, and 500 uL of the solution in section 6.9 and bring to 1.00 mL total volume each. This will produce calibration solutions of nominal 10, 20, 50, 100, and 200 µg/mL of the pollutants and a constant nominal 100 µg/mL of the labeled compounds. Spike each solution with 10 µL of the internal standard solution (Section 6.10). These solutions permit the relative response (labeled to unlabeled) to be measured as a function of concentration (Section 7.4).

6.14 Precision and recovery standard—used for determination of initial (Section 8.2) and on-going (Section 12.7) precision and recovery. This solution shall contain the pollutants and labeled compounds at a nominal concentration of 100 µg/mL.

6.15 Stability of solutions—all standard solutions (Sections 6.8–6.14) shall be analyzed within 48 hours of preparation and on a monthly basis thereafter for signs of degradation. Standards will remain acceptable if the peak area at the quantitation mass relative to the DFB internal standard remains within ±15 percent of the area obtained in the initial analysis of the standard.

7. Calibration

7.1 Assemble the GC/MS and establish the operating conditions in Table 3. Analyze standards per the procedure in Section 11 to demonstrate that the analytical system meets the detection limits in Tables 3 and 4, and the mass-intensity criteria in Table 5 for 50 ng DFTPP.

7.2 Mass spectral libraries—detection and identification of compounds of interest are dependent upon spectra stored in user created libraries.

7.2.1 Obtain a mass spectrum of each pollutant, labeled compound, and the internal standard by analyzing an authentic standard either singly or as part of a mixture in which there is no interference between closely eluted components. That only a single compound is present is determined by examination of the spectrum. Fragments not attributable to the compound under study indicate the presence of an interfering compound.

7.2.2 Adjust the analytical conditions and scan rate (for this test only) to produce an undistorted spectrum at the GC peak maximum. An undistorted spectrum will usually be obtained if five complete spectra are collected across the upper half of the GC peak. Software algorithms designed to ''enhance'' the spectrum may eliminate distortion, but may also eliminate authentic masses or introduce other distortion.

7.2.3 The authentic reference spectrum is obtained under DFTPP tuning conditions (Section 7.1 and Table 5) to normalize it to spectra from other instruments.

7.2.4 The spectrum is edited by saving the 5 most intense mass spectral peaks and all other mass spectral peaks greater than 10

percent of the base peak. This edited spectrum is stored for reverse search and for compound confirmation.

7.3 Analytical range—demonstrate that 20 ng anthracene or phenanthrene produces an area at m/z 178 approx one-tenth that required to exceed the linear range of the system. The exact value must be determined by experience for each instrument. It is used to match the calibration range of the instrument to the analytical range and detection limits required, and to diagnose instrument sensitivity problems (Section 15.4). The 20 ug/mL calibration standard (Section 6.13) can be used to demonstrate this performance.

7.3.1 Polar compound detection—demonstrate that unlabeled pentachlorophenol and benzidine are detectable at the 50 µg/mL level (per all criteria in Section 13). The 50 µg/mL calibration standard (Section 6.13) can be used to demonstrate this performance.

7.4 Calibration with isotope dilution—isotope dilution is used when (1) labeled compounds are available, (2) interferences do not preclude its use, and (3) the quantitation mass extracted ion current profile (EICP) area for the compound is in the calibration range. If any of these conditions preclude isotope dilution, internal standard methods (Section 7.5 or 7.6) are used.

7.4.1 A calibration curve encompassing the concentration range is prepared for each compound to be determined. The relative response (pollutant to labeled) vs concentration in standard solutions is plotted or computed using a linear regression. The example in Figure 1 shows a calibration curve for phenol using phenol-d5 as the isotopic diluent. Also shown are the ± 10 percent error limits (dotted lines). Relative Reponse (RR) is determined according to the procedures described below. A minimum of five data points are employed for calibration.

7.4.2 The relative response of a pollutant to its labeled analog is determined from isotope ratio values computed from acquired data. Three isotope ratios are used in this process:

R_x = the isotope ratio measured for the pure pollutant.

R_y = the isotope ratio measured for the labeled compound.

R_m = the isotope ratio of an analytical mixture of pollutant and labeled compounds.

The m/z's are selected such that $R_x > R_y$. If R_m is not between $2R_y$ and $0.5R_x$, the method does not apply and the sample is analyzed by internal or external standard methods.

7.4.3 Capillary columns usually separate the pollutant-labeled pair, with the labeled compound eluted first (Figure 2). For this case, R_x = [area m_1/z]/1, at the retention time of the pollutant (RT_2). R_y = 1/[area m_2/z, at the retention time of the labeled compound RT_1). R_m = [area at m_1/z (at RT_2)]/[area at RT_1)], as measured in the mixture of the pollutant and labeled compounds (Figure 2), and RR = R_m.

7.4.4 Special precautions are taken when the pollutant-labeled pair is not separated, or when another labeled compound with interfering spectral masses overlaps the pollutant (a case which can occur with isomeric compounds). In this case, it is necessary to determine the respective contributions of the pollutant and labeled compounds to the respective EICP areas. If the peaks are separated well enough to permit the data system or operator to remove the contributions of the compounds to each other, the equations in Section 7.4.3 apply. This usually occurs when the height of the valley between the two GC peaks at the same m/z is less than 10 percent of the height of the shorter of the two peaks. If significant GC and spectral overlap occur, RR is computed using the following equation:

$RR = (R_y - R_m)(R_x + 1)/(R_m - R_x)(R_y + 1)$, where R_x is measured as shown in Figure 3A, R_y is measured as shown in Figure 3B, and R_m is measured as shown in Figure 3C. For example, R_x = 46100/4780 = 9.644, R_y = 2650/43600 = 0.0608, R_m = 49200/48300 = 1.019. amd RR = 1.114.

7.4.5 To calibrate the analytical system by isotope dilution, analyze a 1.0 µL aliquot of each of the calibration standards (Section 6.13) using the procedure in Section 11. Compute the RR at each concentration.

7.4.6 Linearity—if the ratio of relative response to concentration for any compound is constant (less than 20 percent coefficient of variation) over the 5 point calibration range, and averaged relative response/concentration ratio may be used for that compound; otherwise, the complete calibration curve for that compound shall be used over the 5 point calibration range.

7.5 Calibration by internal standard—used when criteria for istope dilution (Section 7.4) cannot be met. The internal standard to be used for both acid and base/neutral analyses is 2,2'–difluorobiphenyl. The internal standard method is also applied to determination of compounds having no labeled analog, and to measurement of labeled compounds for intra-laboratory statistics (Sections 8.4 and 12.7.4).

7.5.1 Response factors—calibration requires the determination of response factors (RF) which are defined by the following equation:

$RF = (A_s \times C_{is})/(A_{is} \times C_s)$, where

A_s is the area of the characteristic mass for the compmund in the daily standard

A_{is} is the area of the characteristic mass for the internal standard

C_{is} is the concentration of the internal standard (µg/mL)

C_s is the concentration of the compound in the daily standard (µg/mL)

7.5.1.1 The response factor is determined for at least five concentrations appropriate to the response of each compound (Section 6.13); nominally, 10, 20, 50, 100, and 200 µg/mL. The amount of internal standard added to each extract is the same (100 µg/mL) so that C_{is} remains constant. The RF is plotted vs concentration for each compound in the standard (C_s) to produce a calibration curve.

7.5.1.2 Linearity—if the response factor (RF) for any compound is constant (less than 35 percent coefficient of variation) over the 5 point calibration range, an averaged response factor may be used for that compound; otherwise, the complete calibration curve for that compound shall be used over the 5 point range.

7.6 Combined calibration—by using calibration solutions (Section 6.13) containing the pollutants, labeled compounds, and the internal standard, a single set of analyses can be used to produce calibration curves for the isotope dilution and internal standard methods. These curves are verified each shift (Section 12.5) by analyzing the 100 θg/mL calibration standard (Section 6.13). Recalibration is required only if calibration verification (Section 12.5) criteria cannot be met.

8. Quality Assurance/Quality Control

8.1 Each laboratory that uses this method is required to operate a formal quality assurance program. The minimum requirements of this program consist of an initial demonstration of laboratory capability, analysis of samples spiked with labeled compounds to evaluate and document data quality, and analysis of standards and blanks as tests of continued performance. Laboratory performance is compared to established performance criteria to determine if the results of analyses meet the performance characteristics of the method.

8.1.1 The analyst shall make an initial demonstration of the ability to generate acceptable accuracy and precision with this method. This ability is established as described in Section 8.2.

8.1.2 The analyst is permitted to modify this method to improve separations or lower the costs of measurements, provided all performance specifications are met. Each time a modification is made to the method, the analyst is required to repeat the procedure in Section 8.2 to demonstrate method performance.

8.1.3 Analyses of blanks are required to demonstrate freedom from contamination. The procedures and criteria for analysis of a blank are described in Section 8.5.

8.1.4 The laboratory shall spike all samples with labeled compounds to monitor method performance. This test is described in Section 8.3. When results of these spikes indicate atypical method performance for samples, the samples are diluted to bring method performance within acceptable limits (Section 15).

8.1.5 The laboratory shall, on an on-going basis, demonstrate through calibration verification and the analysis of the precision and recovery standard (Section 6.14) that the analysis system is in control. These procedures are described in Sections 12.1, 12.5, and 12.7.

8.1.6 The laboratory shall maintain records to define the quality of data that is generated. Development of accuracy statements is described in Section 8.4.

8.2 Initial precision and accuracy—to establish the ability to generate acceptable precision and accuracy, the analyst shall perform the following operations:

8.2.1 Extract, concentrate, and analyze two sets of four one-liter aliquots (8 aliquots total) of the precision and recovery standard (Section 6.14) according to the procedure in Section 10.

8.2.2 Using results of the first set of four analyses, compute the average recovery ($\bar{X}$) in µg/mL and the standard deviation of the recovery (s) in θg/µL for each compound, by isotope dilution for pollutants with a labeled analog, and by internal standard for labeled compounds and pollutants with no labeled analog.

8.2.3 For each compound, compare s and $\bar{X}$ with the corresponding limits for initial precision and accuracy in Table 8. If s and $\bar{X}$ for all compounds meet the acceptance criteria, system performance is acceptable and analysis of blanks and samples may begin. If, however, any individual s exceeds the precision limit or any individual $\bar{X}$ falls outside the range for accuracy, system performance is unacceptable for that compound.

NOTE: The large number of compounds in Table 8 present a substantial probability that one or more will fail the acceptance criteria when all compounds are analyzed. To determine if the analytical system is out of control, or if the failure can be attributed to probability, proceed as follows:

8.2.4 Using the results of the second set of four analyses, compute s and $\bar{X}$ for only those compounds which failed the test of the first set of four analyses (Section 8.2.3). If these compounds now pass, system performance is acceptable for all compounds and analysis of blanks and samples may begin. If, however, any of the same compoulds fail again, the analysis system is not performing properly for these compounds. In this event, correct the problem and repeat the entire test (Section 8.2.1).

8.3 The laboratory shall spike all samples with labeled compounds to assess method performance on the sample matrix.

8.3.1 Analyze each sample according to the method in Section 10.

8.3.2 Compute the percent recovery (P) of the labeled compounds using the internal standard methmd (Section 7.5).

8.3.3 Compare the labeled compound recovery for each compound with the corresponding limits in Table 8. If the recovery of any compounds falls outside its warning limit, method performance is unacceptable for that compound in that sample, Therefore, the sample is complex and is to be diluted and reanalyzed per Section 15.4.

8.4 As part of the QA program for the laboratory, method accuracy for wastewater samples shall be assessed and records shall be maintained. After the analysis of five wastewater samples for which the labeled compounds pass the tests in Section 8.3, compute the average percent recovery (P) and the standard deviation of the percent recovery (s_p) for the labeled compounds only. Express the accuracy assessment as a percent recovery interval from $P—2_{sp}$ to $P+2_{sp}$. For example, if P=90% and s_p=10%, the accuracy interval is expressed as 70–100%. Update the accuracy assessment for each compound on a regular basis (e.g. after each 5–10 new accuracy measurements).

8.5 Blanks—reagent water blanks are analyzed to demonstrate freedom from contamination.

8.5.1 Extract and concentrate a blank with each sample lot (samples started through the extraction process on the same 8 hr shift, to a maximum of 20 samples). Analyze the blank immediately after analysis of the precision and recovery standard (Section 6.14) to demonstrate freedom from contamination.

8.5.2 If any of the compounds of interest (Tables 1 and 2) or any potentially interfering compound is found in a blank at greater than 10 µg/L (assuming a response factor of 1 relative to the internal standard for compounds not listed in Tables 1 and 2), analysis of samples is halted until the source of contamination is eliminated and a blank shows no evidence of contamination at this level.

8.6 The specifications contained in this method can be met if the apparatus used is calibrated properly, then maintained in a calibrated state. The standards used for calibration (Section 7), calibration verification (Section 12.5), and for initial (Section 8.2) and on-going (Section 12.7) precision and recovery should be identical, so that the most precise results will be obtained. The GC/MS instrument in particular will provide the most reproducible results if dedicated to the settings and conditions required for the analysis of semi-volatiles by this method.

8.7 Depending on specific program requirements, field replicates may be collected to determine the precision of the sampling technique, and spiked samples may be required to determine the accuracy of the analysis when internal or external standard methods are used.

9. Sample Collection, Preservation, and Handling

9.1 Collect samples in glass containers following conventional sampling practices (Reference 7). Composite samples are collected in refrigerated glass containers (Section 5.1.3) in accordance with the requirements of the sampling program.

9.2 Maintain samples at 0–4 °C from the time collectimn until extraction. If residual chlorine is present, add 80 mg sodium thiosulfate per liter of water. EPA Methods 330.4 and 330.5 may be used to measure residual chlorine (Reference 8).

9.3 Begin sample extraction within seven days of collection, and analyze all extracts within 40 days of extraction.

10. Sample Extraction and Concentration (See Figure 4)

10.1 Labeled compound spiking—measure 1.00 ± 0.01 liter of sample into a glass container. For untreated effluents, and samples which are expected to be difficult to extract and/or concentrate, measure an additional 10.0 ± 0.1 mL and dilute to a final volume of 1.00 ± 0.01 liter with reagent water in a glass container.

10.1.1 For each sample or sample lot (to a maximum of 20) to be extracted at the same time, place three 1.00 ± 0.10 liter aliquots of reagent water in glass containers.

10.1.2 Spike 0.5 mL of the labeled compound spiking solution (Section 6.8) into all samples and one reagant water aliquot.

10.1.3 Spike 1.0 mL of the precision and recovery standard (Section 6.14) into the two remaining reagent water aliquots.

10.1.4 Stir and equilibrate all solutions for 1–2 hr.

10.2 Base/neutral extraction—place 100–150 mL methylene chloride in each continuous extractor and 200–300 in each distilling flask.

10.2.1 Pour the sample(s), blank, and standard aliquots into the extractors. Rinse the glass containers with 50–100 mL methylene chloride and add to the respective extractor.

10.2.2 Adjust the pH of the waters in the extractors to 12–13 with 6N NaOH while monitoring with a pH meter. Begin the extraction by heating the flask until the methylene chloride is boiling. When properly adjusted, 1–2 drops of methylene chloride per second will fall from the condensor tip into the water. After 1–2 hours of extraction, test the pH and readjust to 12–13 if required. Extract for 18–24 hours.

10.2.3 Remove the distilling flask, estimate and record the volume of extract (to the nearest 100 mL), and pour the contents through a drying column containing 7 to 10 cm anhydrous sodium sulfate. Rinse the distilling flask with 30–50 mL of methylene chloride and pour through the drying column. Collect the solution in a 500 mL K–D

evaporator flask equipped with a 10 mL concentrator tube. Seal, label as the base/neutral fraction, and concentrate per Sections 10.4 to 10.5.

10.3 Acid extraction—adjust the pH of the waters in the extractors to 2 or less using 6N sulfuric acid. Charge clean distilling flasks with 300–400 mL of methylene chloride. Test and adjust the pH of the waters after the first 1–2 hr of extraction. Extract for 18–24 hours.

10.3.1 Repeat Section 10.2.3, except label as the acid fraction.

10.4 Concentration—concentrate the extracts in separate 500 mL K–D flasks equipped with 10 mL concentrator tubes.

10.4.1 Add 1 to 2 clean boiling chips to the flask and attach a three-ball macro Snyder column. Prewet the column by adding approximately one mL of methylene chloride through the top. Place the K–D apparatus in a hot water bath so that the entire lower rounded surface of the flask is bathed with steam. Adjust the vertical position of the apparatus and the water temperature as required to complete the concentration in 15 to 20 minutes. At the proper rate of distillation, the balls of the column will actively chatter but the chambers will not flood. When the liquid has reached an apparent volume of 1 mL, remove the K–D apparatus from the bath and allow the solvent to drain and cool for at least 10 minutes. Remove the Snyder column and rinse the flask and its lower joint into the concentrator tube with 1–2 mL of methylene chloride. A 5-mL syringe is recommended for this operation.

10.4.2 For performance standards (Sections 8.2 and 12.7) and for blanks (Section 8.5), combine the acid and base/neutral extracts for each at this point. Do not combine the acid and base/neutral extracts for samples.

10.5 Add a clean boiling chip and attach a two ball micro Snyder column to the concentrator tube. Prewet the column by adding approx 0.5 mL methylene chloride through the top. Place the apparatus in the hot water bath. Adjust the vertical position and the water temperature as required to complete the concentration in 5–10 minutes. At the proper rate of distillation, the balls of the column will actively chatter but the chambers will not flood. When the liquid reaches an apparent volume of approx 0.5 mL, remove the apparatus from the water bath and allow to drain and cool for at least 10 minutes. Remove the micro Snyder column and rinse its lower joint into the concentrator tube with approx 0.2 mL of methylene chloride. Adjust the final volume to 1.0 mL.

10.6 Transfer the concentrated extract to a clean screw-cap vial. Seal the vial with a Teflon-lined lid, and mark the level on the vial. Label with the sample number and fraction, and store in the dark at −20 to −10 °C until ready for analysis.

11. GC/MS Analysis

11.1 Establish the operating conditions given in Table 3 or 4 for analysis of the base/neutral or acid extracts, respectively. For analysis of combined extracts (Section 10.4.2), use the operating conditions in Table 3.

11.2 Bring the concentrated extract (Section 10.6) or standard (Sections 6.13 through 6.14) to room temperature and verify that any precipitate has redissolved. Verify the level on the extract (Sections 6.6 and 10.6) and bring to the mark with solvent if required.

11.3 Add the internal standard solution (Section 6.10) to the extract (use 1.0 uL of solution per 0.1 mL of extract) immediately prior to injection to minimize the possibility of loss by evaporation, adsorption, or reaction. Mix thoroughly.

11.4 Inject a volume of the standard solution or extract such that 100 ng of the internal standard will be injected, using on-column or splitless injection. For 1 mL extracts, this volume will be 1.0 uL. Start the GC column initial isothermal hold upon injection. Start MS data collection after the solvent peak elutes. Stop data collection after the benzo (ghi) perylene or pentachlorophenol peak elutes for the base/neutral or acid fraction, respectively. Return the column to the initial temperature for analysis of the next sample.

12. System and Laboratory Performance

12.1 At the beginning of each 8 hr shift during which analyses are performed, GC/MS system performance and calibration are verified for all pollutants and labeled compounds. For these tests, analysis of the 100 µg/mL calibration standard (Section 6.13) shall be used to verify all performance criteria. Adjustment and/or recalibration (per Section 7) shall be performed until all performance criteria are met. Only after all performance criteria are met may samples, blanks, and precision and recovery standards be analyzed.

12.2 DFTPP spectrum validity—inject 1 µL of the DFTPP solution (Section 6.11) either separately or within a few seconds of injection of the standard (Section 12.1) analyzed at the beginning of each shift. The criteria in Table 5 shall be met.

12.3 Retention times—the absolute retention time of 2,2′-difluorobiphenyl shall be within the range of 1078 to 1248 seconds and the relative retention times of all pollutants and labeled compounds shall fall within the limits given in Tables 3 and 4.

12.4 GC resolution—the valley height between anthracene and phenanthrene at m/z 178 (or the analogs at m/z 188) shall not exceed 10 percent of the taller of the two peaks.

12.5 Calibration verification—compute the concentration of each pollutant (Tables 1

and 2) by isotope dilution (Section 7.4) for those compounds which have labeled analogs. Compute the concentration of each pollutant which has no labeled analog by the internal standard method (Section 7.5). Compute the concentration of the labeled compounds by the internal standard method. These concentrations are computed based on the calibration data determined in Section 7.

12.5.1 For each pollutant and labeled compound being tested, compare the concentration with the calibration verification limit in Table 8. If all compounds meet the acceptance criteria, calibration has been verified and analysis of blanks, samples, and precision and recovery standards may proceed. If, however, any compound fails, the measurement system is not performing properly for that compound. In this event, prepare a fresh calibration standard or correct the problem causing the failure and repeat the test (Section 12.1), or recalibrate (Section 7).

12.6 Multiple peaks—each compound injected shall give a single, distinct GC peak.

12.7 On-going precision and accuracy.

12.7.1 Analyze the extract of one of the pair of precision and recovery standards (Section 10.1.3) prior to analysis of samples from the same lot.

12.7.2 Compute the concentration of each pollutant (Tables 1 and 2) by isotope dilution (Section 7.4) for those compounds which have labeled analogs. Compute the concentration of each pollutant which has no labeled analog by the internal standard method (Section 7.5). Compute the concentration of the labeled compounds by the internal standard method.

12.7.3 For each pollutant and labeled compound, compare the concentration with the limits for on-going accuracy in Table 8. If all compounds meet the acceptance criteria, system performance is acceptable and analysis of blanks and samples may proceed. If, however, any individual concentration falls outside of the range given, system performance is unacceptable for that compound.

NOTE: The large number of compounds in Table 8 present a substantial probability that one or more will fail when all compounds are analyzed. To determine if the extraction/concentration system is out of control or if the failure is caused by probability, proceed as follows:

12.7.3.1 Analyze the second aliquot of the pair of precision and recovery standard (Section 10.1.3).

12.7.3.2 Compute the concentration of only those pollutants or labeled compounds that failed the previous test (Section 12.7.3). If these compounds now pass, the extraction/concentration processes are in control and analysis of blanks and samples may proceed. If, however, any of the same compounds fail again, the extraction/concentration processes are not being performed properly for these compounds. In this event, correct the problem, re-extract the sample lot (Section 10) and repeat the on-going precision and recovery test (Section 12.7).

12.7.4 Add results which pass the specifications in Section 12.7.2 to initial and previous on-going data. Update QC charts to perform a graphic representation of continued laboratory performance (Figure 5). Develop a statement of laboratory accuracy for each pollutant and labeled compound by calculating the average percent recovery (R) and the standard deviation of percent recovery (s_r). Express the accuracy as a recovery interval from $R-2s_r$ to $R+2s_r$. For example, if R=95% and s_r=5%, the accuracy is 85−105%.

13. Qualitative Determination

13.1 Qualititative determination is accomplished by comparison of data from analysis of a sample or blank with data from analysis of the shift standard (Section 12.1) and with data stored in the spectral libraries (Section 7.2.4). Identification is confirmed when spectra and retention times agree per the criteria below.

13.2 Labeled compounds and pollutants having no labeled analog:

13.2.1 The signals for all characteristic masses stored in the spectral library (Section 7.2.4) shall be present and shall maximize within the same two consecutive scans.

13.2.2 Either (1) the background corrected EICP areas, or (2) the corrected relative intensities of the mass spectral peaks at the GC peak maximum shall agree within a factor of two (0.5 to 2 times) for all masses stored in the library.

13.2.3 The retention time relative to the nearest eluted internal standard shall be within ±15 scans or ±15 seconds, whichever is greater of this difference in the shift standard (Section 12.1).

13.3 Pollutants having a labled analog:

13.3.1 The signals for all characteristic masses stored in the spectral library (Section 7.2.4) shall be present and shall maximize within the same two consecutive scans.

13.3.2. Either (1) the background corrected EICP areas, or (2) the corrected relative intensities of the mass spectral peaks at the GC peak maximum shall agree within a factor of two for all masses stored in the spectral library.

13.3.3. The retention time difference between the pollutant and its labeled analog shall agree within ± 6 scans or ± 6 seconds (whichever is greater) of this difference in the shift standard (Section 12.1).

13.4 Masses present in the experimental mass spectrum that are not present in the reference mass spectrum shall be accounted for by contaminant or background ions. If

the experimental mass spectrum is contaminated, an experienced spectrometrist (Section 1.4) is to determine the presence or absence of the cmmpound.

14. Quantitative Determination

14.1 Isotope dilution—by adding a known amount of a labeled compound to every sample prior to extraction, correction for recovery of the pollutant can be made because the pollutant and its labeled analog exhibit the same effects upon extraction, concentration, and gas chromatography. Relative response (RR) values for mixtures are used in conjunction with calibration curves described in Section 7.4 to determine concentrations directly, so long as labeled compound spiking levels are constant. For the phenml example given in Figure 1 (Section 7.4.1), RR would be equal to 1.114. For this RR value, the phenol calibration curve given in Figure 1 indicates a concentration of 27 µg/mL in the sample extract (C_{ex}).

14.2 Internal standard—compute the concentration in the extract using the response factor determined from calibration data (Section 7.5) and the following equation: C_{ex}(µg/mL)=(A_s x C_{is}/(A_{is} x RF) where C_{ex} is the concentration of the compound in the extract, and the other terms are as defined in Section 7.5.1.

14.3 The concentration of the pollutant in water is computed using the volumes of the original water sample (Section 10.1) and the final extract volume (Section 10.5), as follows: Concentration in water (µg/L)=(C_{ex} x V_{ex})/V_s where V_{ex} is the extract volume in mL, and V_s is the sample volume in liters.

14.4 If the EICP area at the quantitiation mass for any compound exceeds the calibration range of the system, the extract of the dilute aliquot (Section 10.1) is analyzed by isotope dilution; otherwise, the extract is diluted by a factor of 10, 9 µL of internal standard solution (Section 6.10) are added to a 1.0 mL aliquot, and this diluted extract is analyzed by the internal standard method (Section 14.2). Quantify each compound at the highest concentration level within the calibration range.

14.5 Report results for all pollutants and labeled compounds (Tables 1 and 2) found in all standards, blanks, and samples in µg/L, to three significant figures. Results for samples which have been diluted are reported at the least dilute level at which the area at the quantitation mass is within the calibration range (Section 14.4) and the labeled compound recovery is within the normal range for the method (Section 15.4).

15. Analysis of Complex Samples

15.1 Untreated effluents and other samples frequently contain high levels (>1000 µg/L) of the compounds of interest, interfering compounds, and/or polymeric materials. Some samples will not concentrate to one mL (Section 10.5); others will overload the GC column and/or mass spectrometer.

15.2 Analyze the dilute aliquot (Section 10.1) when the sample will not concentrate to 1.0 mL. If a dilute aliquot was not extracted, and the sample holding time (Section 9.3) has not been exceeded, dilute an aliquot of the sample with reagent water and re-extract (Section 10.1); otherwise, dilute the extract (Section 14.4) and analyze by the internal standard method (Section 14.2).

15.3 Recovery of internal standard— the EICP area of the internal standard should be within a factor of two of the area in the shift standard (Section 12.1). If the absolute areas of the labeled compounds are within a factor of two of the respective areas in the shift standard, and the internal standard area is less than one-half of its respective area, then internal standard loss in the extract has occurred. In this case, use one of the labeled compounds (perferably a polynuclear aromatic hydrocarbon) to compute the concentration of a pollutant with no labeled analog.

15.4 Recovery of labeled compounds— in most samples, labeled compound recoveries will be similar to those from reagent water (Section 12.7). If the labeled compound recovery is outside the limits given in Table 8, the dilute extract (Section 10.1) is analyzed as in Section 14.4. If the recoveries of all labeled compounds and the internal staldard are low (per the criteria above), then a loss in instrument sensitivity is the most likely cause. In this case, the 100 µg/mL calibration standard (Section 12.1) shall be analyzed and calibration verified (Section 12.5). If a loss in sensitivity has occurred, the instrument shall be repaired, the performance specifications in Section 12 shall be met, and the extract reanalyzed. If a loss in instrument sensitivity has not occurred, the method does not work on the sample being analyzed and the result may not be reported for regulatory compliance purposes.

16. Method Performance

16.1 Interlaboratory performance for this method is detailed in references 9 and 10.

16.2 A chromatogram of the 100 µg/mL acid/base/neutral calibration standard (Section 6.13) is shown in Figure 6.

References

1. ''Performance Tests for the Evaluation of Computerized Gas Chromatography/Mass Spectrometry Equipment and Laboratories'' USEPA, EMSL/Cincinnati, OH 45268, EPA–600/4–80–025 (April 1980).

2. ''Working with Carcinogens,'' DHEW, PHS, CDC, NIOSH, Publication 77–206, (August 1977).

3. ''OSHA Safety and Health Standards, General Industry'' OSHA 2206, 29 CFR part 1910 (January 1976).

4. ''Safety in Academic Chemistry Laboratories, '' ACS Committee on Chemical Safety (1979).

5. ''Reference Compound to Calibrate Ion Abundance Measurement in Gas Chromatography-Mass Spectrometry Systems,'' J.W. Eichelberger, L.E. Harris, and W.L. Budde, Anal. Chem., *47*, 955 (1975).

6. ''Handbook of Analytical Quality Control in Water and Wastewater Laboratories,'' USEPA, EMSL/Cincinnati, OH 45268, EPA–600/4–79–019 (March 1979).

7. ''Standard Practice for Sampling Water,'' ASTM Annual Book of Standards, ASTM, Philadelphia, PA, 76 (1980).

8. ''Methods 330.4 and 330.5 for Total Residual Chlorine,'' USEPA, EMSL/ Cincinnati, OH 45268, EPA 600/4–70–020 (March 1979).

9. Colby, B.N., Beimer, R.G., Rushneck, D.R., and Telliard, W.A., ''Isotope Dilution Gas Chromatography-Mass Spectrometry for the determination of Priority Pollutants in Industrial Effluents.'' USEPA, Effluent Guidelines Division, Washington, DC 20460 (1980).

10. ''Inter-laboratory Validation of US Environmental Protection Agency Method 1625,'' USEPA, Effluent Guidelines Division, Washington, DC 20460 (June 15, 1984).

TABLE 1—Base/Neutral Extractable Compounds

Compound	STORET	CAS registry	EPA-EGD	NPDES
Acenaphthene	34205	83–32–9	001 B	001 B
Acenaphthylene	34200	208–96–8	077 B	002 B
Anthracene	34220	120–12–7	078 B	003 B
Benzidine	39120	92–87–5	005 B	004 B
Benzo(a)anthracene	34526	56–55–3	072 B	005 B
Benzo(b)fluoranthene	34230	205–99–2	074 B	007 B
Benzo(k)fluoranthene	34242	207–08–9	075 B	009 B
Benzo(a)pyrene	34247	50–32–8	073 B	006 B
Benzo(ghi)perylene	34521	191–24–2	079 B	008 B
Biphenyl (Appendix C)	81513	92–52–4	512 B	.....
Bis(2-chloroethyl) ether	34273	111–44–4	018 B	011 B
Bis(2-chloroethyoxy)methane	34278	111–91–1	043 B	010 B
Bis(2-chloroisopropyl) ether	34283	108–60–1	042 B	012 B
Bis(2-ethylhexyl) phthalate	39100	117–81–7	066 B	013 B
4-bromophenyl phenyl ether	34636	101–55–3	041 B	014 B
Butyl benzyl phthalate	34292	85–68–7	067 B	015 B
n-C10 (Appendix C)	77427	124–18–5	517 B	.....
n-C12 (Appendix C)	77588	112–40–2	506 B	.....
n-C14 (Appendix C)	77691	629–59–4	518 B	.....
n-C16 (Appendix C)	77757	544–76–3	519 B	.....
n-C18 (Appendix C)	77804	593–45–3	520 B	.....
n-C20 (Appendix C)	77830	112–95–8	521 B	.....
n-C22 (Appendix C)	77859	629–97–0	522 B	.....
n-C24 (Appendix C)	77886	646–31–1	523 B	.....
n-C26 (Appendix C)	77901	630–01–3	524 B	.....
n-C28 (Appendix C)	78116	630–02–4	525 B	.....
n-C30 (Appendix C)	78117	638–68–6	526 B	.....
Carbazole (4c)	77571	86–74–8	528 B	.....
2-chloronaphthalene	34581	91–58–7	020 B	016 B
4-chlorophenyl phenyl ether	34641	7005–72–3	040 B	017 B
Chrysene	34320	218–01–9	076 B	018 B
P-cymene (Appendix C)	77356	99–87–6	513 B	.....
Dibenzo(a,h)anthracene	34556	53–70–3	082 B	019 B
Dibenzofuran (Appendix C and 4c)	81302	132–64–9	505 B	.....
Dibenzothiophene (Synfuel)	77639	132–65–0	504 B	.....
Di-n-butyl phthalate	39110	84–74–2	068 B	026 B
1,2-dichlorobenzene	34536	95–50–1	025 B	020 B
1,3-dichlorobenzene	34566	541–73–1	026 B	021 B
1,4-dichlorobenzene	34571	106–46–7	027 B	022 B
3,3′-dichlorobenzidine	34631	91–94–1	028 B	023 B
Diethyl phthalate	34336	84–66–2	070 B	024 B
2,4-dimethylphenol	34606	105–67–9	034 A	003 A
Dimethyl phthalate	34341	131–11–3	071 B	025 B
2,4-dinitrotoluene	34611	121–14–2	035 B	027 B
2,6-dinitrotoluene	34626	606–20–2	036 B	028 B
Di-n-octyl phthalate	34596	117–84–0	069 B	029 B
Diphenylamine (Appendix C)	77579	122–39–4	507 B	.....
Diphenyl ether (Appendix C)	77587	101–84–8	508 B	.....
1,2-diphenylhydrazine	34346	122–66–7	037 B	030 B
Fluoranthene	34376	206–44–0	039 B	031 B
Fluorene	34381	86–73–7	080 B	032 B

TABLE 1—Base/Neutral Extractable Compounds—Continued

Compound	STORET	CAS registry	EPA-EGD	NPDES
Hexachlorobenzene	39700	118–74–1	009 B	033 B
Hexachlorobutadiene	34391	87–68–3	052 B	034 B
Hexachloroethane	34396	67–72–1	012 B	036 B
Hexachlorocyclopentadiene	34386	77–47–4	053 B	035 B
Indeno(1,2,3-cd)pyrene	34403	193–39–5	083 B	037 B
Isophorone	34408	78–59–1	054 B	038 B
Naphthalene	34696	91–20–3	055 B	039 B
B-naphthylamine (Appendix C)	82553	91–59–8	502 B	.....
Nitrobenzene	34447	98–95–3	056 B	040 B
N-nitrosodimethylamine	34438	62–75–9	061 B	041 B
N-nitrosodi-n-propylamine	34428	621–64–7	063 B	042 B
N-nitrosodiphenylamine	34433	86–30–3	062 B	043 B
Phenanthrene	34461	85–01–8	081 B	044 B
Phenol	34694	108–95–2	065 A	010 A
a-Picoline (Synfuel)	77088	109–06–89	503 B	.....
Pyrene	34469	129–00–0	084 B	045 B
styrene (Appendix C)	77128	100–42–5	510 B	.....
a-terpineol (Appendix C)	77493	98–55–5	509 B	.....
1,2,3-trichlorobenzene (4c)	77613	87–61–6	529 B	.....
1,2,4-trichlorobenzene	34551	120–82–1	008 B	046 B

TABLE 2—Acid Extractable Compounds

Compound	STORET	CAS registry	EPA-EGD	NPDES
4-chloro-3-methylphenol	34452	59–50–7	022 A	008 A
2-chlorophenol	34586	95–57–8	024 A	001 A
2,4-dichlorophenol	34601	120–83–2	031 A	002 A
2,4-dinitrophenol	34616	51–28–5	059 A	005 A
2-methyl-4,6-dinitrophenol	34657	534–52–1	060 A	004 A
2-nitrophenol	34591	88–75–5	057 A	006 A
4-nitrophenol	34646	100–02–7	058 A	007 A
Pentachlorophenol	39032	87–86–5	064 A	009 A
2,3,6-trichlorophenol (4c)	77688	93–37–55	530 A	.....
2,4,5-trichlorophenol (4c)	.....	95–95–4	531 A	.....
2,4,6-trichlorophenol	34621	88–06–2	021 A	011 A

TABLE 3—GAS CHROMATOGRAPHY OF BASE/NEUTRAL EXTRACTABLE COMPOUNDS

EGD No.[1]	Compound	Retention time			Detection limit[2] (µg/L)
		Mean (sec)	EGD Ref	Relative	
164	2,2′-difluorobiphenyl (int std)	1163	164	1.000–1.000	10
061	N-nitrosodimethylamine	385	164	ns	50
603	alpha picoline-d7	417	164	0.326–0.393	50
703	alpha picoline	426	603	1.006–1.028	50
610	styrene-d5	546	164	0.450–0.488	10
710	styrene	549	610	1.002–1.009	10
613	p-cymene-d14	742	164	0.624–0.652	10
713	p-cymene	755	613	1.008–1.023	10
265	phenol-d5	696	164	0.584–0.613	10
365	phenol	700	265	0.995–1.010	10
218	bis(2-chloroethyl) ether-d8	696	164	0.584–0.607	10
318	bis(2-chloroethyl) ether	704	218	1.007–1.016	10
617	n-decane-d22	698	164	0.585–0.615	10
717	n-decane	720	617	1.022–1.038	10
226	1,3-dichlorobenzene-d4	722	164	0.605–0.636	10
326	1,3-dichlorobenzene	724	226	0.998–1.008	10
227	1,4-dichlorobenzene-d4	737	164	0.601–0.666	10
327	1,4-dichlorobenzene	740	227	0.997–1.009	10
225	1,2-dichlorobenzene-d4	758	164	0.632–0.667	10
325	1,2-dichlorobenzene	760	225	0.995–1.008	10
242	bis(2-chloroisopropyl) ether-d12	788	164	0.664–0.691	10
342	bis(2-chloroisopropyl) ether	799	242	1.010–1.016	10
212	hexachloroethane-13C	819	164	0.690–0.717	10
312	hexachloroethane	823	212	0.999–1.001	10
063	N-nitrosodi-n-propylamine	830	164	ns	20

TABLE 3—GAS CHROMATOGRAPHY OF BASE/NEUTRAL EXTRACTABLE COMPOUNDS—Continued

EGD No.[1]	Compound	Retention time			Detection limit[2] (µg/L)
		Mean (sec)	EGD Ref	Relative	
256	nitrobenzene-d5	845	164	0.706–0.727	10
356	nitrobenzene	849	256	1.002–1.007	10
254	isophorone-d8	881	164	0.747–0.767	10
354	isophorone	889	254	0.999–1.017	10
234	2,4-dimethyl phenol-d3	921	164	0.781–0.803	10
334	2,4-dimethylphenol	924	234	0.999–1.003	10
043	bis(2-chloroethoxy) methane	939	164	ns	10
208	1,2,4-trichlorobenzene-d3	955	164	0.813–0.830	10
308	1,2,4-trichlorobenzene	958	208	1.000–1.005	10
255	naphthalene-d8	963	164	0.819–0.836	10
355	naphthalene	967	255	1.001–1.006	10
609	alpha-terpineol-d3	973	164	0.829–0.844	10
709	alpha-terpineol	975	609	0.998–1.008	10
606	n-dodecane-d26	953	164	0.730–0.908	10
706	n-dodecane	981	606	0.986–1.051	10
529	1,2,3-trichlorobenzene	1003	164	ns	10
252	hexachlorobutadiene-13C4	1005	164	0.856–0.871	10
352	hexachlorobutadiene	1006	252	0.999–1.002	10
253	hexachlorocyclopentadiene-13C4	1147	164	0.976–0.986	10
353	hexachlorocyclopentadiene	1142	253	0.999–1.001	10
220	2-chloronaphthalene-d7	1185	164	1.014–1.024	10
320	2-chloronaphthalene	1200	220	0.997–1.007	10
518	n-tetradecane	1203	164	ns	10
612	Biphenyl-d10	1205	164	1.016–1.027	10
712	Biphenyl	1195	612	1.001–1.006	10
608	Diphenyl ether-d10	1211	164	1.036–1.047	10
708	Diphenyl ether	1216	608	0.997–1.009	10
277	Acenaphthylene-d8	1265	164	1.080–1.095	10
377	Acenaphthylene	1247	277	1.000–1.004	10
271	Dimethyl phthalate-d4	1269	164	1.083–1.102	10
371	Dimethyl phthalate	1273	271	0.998–1.005	10
236	2,6-dinitrotoluene-d3	1283	164	1.090–1.112	10
336	2,6-dinitrotoluene	1300	236	1.001–1.005	10
201	Acenaphthene-d10	1298	164	1.107–1.125	10
301	Acenaphthene	1304	201	0.999–1.009	10
605	Dibenzofuran-d8	1331	164	1.134–1.155	10
705	Dibenzofuran	1335	605	0.998–1.007	10
602	Beta-naphthylamine-d7	1368	164	1.163–1.189	50
702	Beta-naphthylamine	1371	602	0.996–1.007	50
280	Fluorene-d10	1395	164	1.185–1.214	10
380	Fluorene	1401	281	0.999–1.008	10
240	4-chlorophenyl phenyl ether-d5	1406	164	1.194–1.223	10
340	4-chlorophenyl phenyl ether	1409	240	0.990–1.015	10
270	Diethyl phthalate-d4	1409	164	1.197–1.229	10
370	Diethyl phthalate	1414	270	0.996–1.006	10
619	n-hexadecane-d34	1447	164	1.010–1.478	10
719	n-hexadecane	1469	619	1.013–1.020	10
235	2,4-dinitrotoluene-d3	1359	164	1.152–1.181	10
335	2,4-dinitrotoluene	1344	235	1.000–1.002	10
237	1,2-diphenylhydrazine-d8	1433	164	1.216–1.248	20
337	1,2-diphenylhydrazine ([3])	1439	237	0.999–1.009	20
607	Diphenylamine–d10	1437	164	1.213–1.249	20
707	Diphenylamine	1439	607	1.000–1.007	20
262	N-nitrosodiphenylamine-d6	1447	164	1.225–1.252	20
362	N-nitrosodiphenylamine ([4])	1464	262	1.000–1.002	20
041	4-bromophenyl phenyl ether	1498	164	1.271–1.307	10
209	Hexachlorobenzene-13C6	1521	164	1.288–1.327	10
309	Hexachlorobenzene	1522	209	0.999–1.001	10
281	Phenanthrene-d10	1578	164	1.334–1.380	10
520	n-octadecane	1580	164	ns	10
381	Phenanthrene	1583	281	1.000–1.005	10
278	Anthracene-d10	1588	164	1.342–1.388	10
378	Anthracene	1592	278	0.998–1.006	10
604	Dibenzothiophene-d8	1559	164	1.314–1.361	10
704	Dibenzothiophene	1564	604	1.000–1.006	10
528	Carbazole	1650	164	ns	20
621	n-eicosane-d42	1655	164	1.184–1.662	10
721	n-eicosane	1677	621	1.010–1.021	10
268	Di-n-butyl phthalate-d4	1719	164	1.446–1.510	10
368	Di-n-butyl phthalate	1723	268	1.000–1.003	10

TABLE 3—GAS CHROMATOGRAPHY OF BASE/NEUTRAL EXTRACTABLE COMPOUNDS—Continued

EGD No.[1]	Compound	Retention time			Detection limit[2] (µg/L)
		Mean (sec)	EGD Ref	Relative	
239	Fluoranthene-d10	1813	164	1.522–1.596	10
339	Fluoranthene	1817	239	1.000–1.004	10
284	Pyrene-d10	1844	164	1.523–1.644	10
384	Pyrene	1852	284	1.001–1.003	10
205	Benzidine-d8	1854	164	1.549–1.632	50
305	Benzidine	1853	205	1.000–1.002	50
522	n-docosane	1889	164	ns	10
623	n-tetracosane-d50	1997	164	1.671–1.764	10
723	n-tetracosane	2025	612	1.012–1.015	10
067	Butylbenzyl phthalate	2060	164	ns	10
276	Chrysene-d12	2081	164	1.743–1.837	10
376	Chrysene	2083	276	1.000–1.004	10
272	Benzo(a)anthracene-d12	2082	164	1.735–1.846	10
372	Benzo(a)anthracene	2090	272	0.999–1.007	10
228	3,3′-dichlorobenzidine-d6	2088	164	1.744–1.848	50
328	3,3′-dichlorobenzidine	2086	228	1.000–1.001	50
266	Bis(2-ethylhexyl) phthalate-d4	2123	164	1.771–1.880	10
366	Bis(2-ethylhexyl) phthalate	2124	266	1.000–1.002	10
524	n-hexacosane	2147	164	ns	10
269	di-n-octyl phthalate-d4	2239	164	1.867–1.982	10
369	di-n-octyl phthalate	2240	269	1.000–1.002	10
525	n-octacosane	2272	164	ns	10
274	Benzo(b)fluoranthene-d12	2281	164	1.902–2.025	10
354	Benzo(b)fluoranthene	2293	274	1.000–1.005	10
275	Benzo(k)fluoranthene-d12	2287	164	1.906–2.033	10
375	Benzo(k)fluoranthene	2293	275	1.000–1.005	10
273	Benzo(a)pyrene-d12	2351	164	1.954–2.088	10
373	Benzo(a)pyrene	2350	273	1.000–1.004	10
626	N-triacontane-d62	2384	164	1.972–2.127	10
726	N-triacontane	2429	626	1.011–1.028	10
083	Indeno(1,2,3-cd)pyrene	2650	164	ns	20
082	Dibenzo(a,h)anthracene	2660	164	ns	20
279	Benzo(ghi)perylene-d12	2741	164	2.187–2.524	20
379	Benzo(ghi)perylene	2750	279	1.001–1.006	20

[1] Reference numbers beginning with 0, 1 or 5 indicate a pollutant quantified by the internal standard method; reference numbers beginning with 2 or 6 indicate a labeled compound quantified by the internal standard method; reference numbers beginning with 3 or 7 indicate a pollutant quantified by isotope dilution.

[2] This is a minimum level at which the entire GC/MS system must give recognizable mass spectra (background corrected) and acceptable calibration points.

[3] Detected as azobenzene.

[4] Detected as diphenylamine.

ns = specification not available at time of release of method.

Column: 30 ±2 m × 0.25 ±0.02 mm i.d. 94% methyl, 4% phenyl, 1% vinyl bonded phase fused silica capillary.

Temperature program: 5 min at 30 °C; 30 – 280 °C at 8 °C per min; isothermal at 280 °C until benzo(ghi)perylene elutes.

Gas velocity: 30 ±5 cm/sec.

TABLE 4—GAS CHROMATOGRAPHY OF ACID EXTRACTABLE COMPOUNDS

EGD No. [1]	Compound	Retention time			Detection limit [2] (µg/L)
		Mean (sec)	EGD Ref	Relative	
164	2,2′-difluorobiphenyl (int std)	1163	164	1.000–1.000	10
224	2-chlorophenol-d4	701	164	0.587–0.618	10
324	2-chlorophenol	705	224	0.997–1.010	10
257	2-nitrophenol-d4	898	164	0.761–0.783	20
357	2-nitrophenol	900	257	0.994–1.009	20
231	2,4-dichlorophenol-d3	944	164	0.802–0.822	10
331	2,4-dichlorophenol	947	231	0.997–1.006	10
222	4-chloro-3-methylphenol-d2	1086	164	0.930–0.943	10
322	4-chloro-3-methylphenol	1091	222	0.998–1.003	10
221	2,4,6-trichlorophenol-d2	1162	164	0.994–1.005	10
321	2,4,6-trichlorophenol	1165	221	0.998–1.004	10
531	2,4,5-trichlorophenol	1170	164	ns	10
530	2,3,6-trichlorophenol	1195	164	ns	10
259	2,4-dinitrophenol-d3	1323	164	1.127–1.149	50
359	2,4-dinitrophenol	1325	259	1.000–1.005	50
258	4-nitrophenol-d4	1349	164	1.147–1.175	50
358	4-nitrophenol	1354	258	0.997–1.006	50

TABLE 4—GAS CHROMATOGRAPHY OF ACID EXTRACTABLE COMPOUNDS—Continued

EGD No. [1]	Compound	Retention time			Detection limit [2] (μg/L)
		Mean (sec)	EGD Ref	Relative	
260	2-methyl-4,6-dinitrophenol-d2	1433	164	1.216–1.249	20
360	2-methyl-4,6-dinitrophenol	1435	260	1.000–1.002	20
264	Pentachlorophenol-13C6	1559	164	1.320–1.363	50
364	Pentachlorophenol	1561	264	0.998–1.002	50

[1] Reference numbers beginning with 0, 1 or 5 indicate a pollutant quantified by the internal standard method; reference numbers beginning with 2 or 6 indicate a labeled compound quantified by the internal standard method; reference numbers beginning with 3 or 7 indicate a pollutant quantified by isotope dilution.

[2] This is a minimum level at which the entire GC/MS system must give recognizable mass spectra (background corrected) and acceptable calibration points.

ns=specification not available at time of release of method.
Column: 30±2m×0.25±0.02mm i.d. 94% methyl, 4% phenyl, 1% vinyl bonded phase fused silica capillary.
Temperature program: 5 min at 30 °C; 8°C/min. to 250°C or until pentachlorophenol elutes.
Gas velocity: 30±5 cm/sec.

TABLE 5—DFTPP MASS INTENSITY SPECIFICATIONS

Mass	Intensity required
51	30–60 percent of mass 198.
68	Less than 2 percent of mass 69.
70	Less than 2 percent of mass 69.
127	40–60 percent of mass 198.
197	Less than 1 percent of mass 198.
199	5–9 percent of mass 198.
275	10–30 percent of mass 198.
365	greater than 1 percent of mass 198
441	present and less than mass 443
442	40–100 percent of mass 198.
443	17–23 percent of mass 442.

TABLE 6—BASE/NEUTRAL EXTRACTABLE COMPOUND CHARACTERISTIC MASSES

Compound	Labeled analog	Primary m/z
Acenaphthene	d10	154/164
Acenaphthylene	d8	152/160
Anthracene	d10	178/188
Benzidine	d8	184/192
Benzo(a)anthracene	d12	228/240
Benzo(b)fluoranthene	d12	252/264
Benzo(k)fluoranthene	d12	252/264
Benzo(a)pyrene	d12	252/264
Benzo(ghi)perylene	d12	276/288
Biphenyl	d10	154/164
Bis(2-chloroethyl) ether	d8	93/101
Bis(2-chloroethoxy)methane	.....	93
Bis(2-chloroisopropyl) ether	d12	121/131
Bis(2-ethylhexyl) phthalate	d4	149/153
4-bromophenyl phenyl ether	.....	248
Butyl benzyl phthalate	.....	149
n-C10	d22	55/66
n-C12	d26	55/66
n-C14	.....	55
n-C16	d34	55/66
n-C18	.....	55
n-C20	d42	55/66
n-C22	.....	55
n-C24	d50	55/66
n-C26	.....	55
n-C28	.....	55
n-C30	d62	55/66
Carbazole	d8	167/175
2-chloronaphthalene	d7	162/169
4-chlorophenyl phenyl ether	d5	204/209
Chrysene	d12	228/240
p-cymene	d14	114/130

TABLE 6—BASE/NEUTRAL EXTRACTABLE COMPOUND CHARACTERISTIC MASSES—Continued

Compound	Labeled analog	Primary m/z
Dibenzo(a,h)anthracene	.....	278
Dibenzofuran	d8	168/176
Dibenzothiophene	d8	184/192
Di-n-butyl phthalate	d4	149/153
1,2-dichlorobenzene	d4	146/152
1,3-dichlorobenzene	d4	146/152
1,4-dichlorobenzene	d4	146/152
3,3′-dichlorobenzidine	d6	252/258
Diethyl phthalate	d4	149/153
2,4-dimethylphenol	d3	122/125
Dimethyl phthalate	d4	163/167
2,4-dinitrotoluene	d3	164/168
2,6-dinitrotoluene	d3	165/167
Di-n-octyl phthalate	d4	149/153
Diphenylamine	d10	169/179
Diphenyl ether	d10	170/180
1,2-diphenylhydrazine [1]	d10	77/82
Fluoranthene	d10	202/212
Fluorene	d10	166/176
Hexachlorobenzene	13C6	284/292
Hexachlorobutadiene	13C4	225/231
Hexachloroethane	13C	201/204
Hexachlorocyclopentadiene	13C4	237/241
Ideno(1,2,3-cd)pyrene	.....	276
Isophorone	d8	82/88
Naphthalene	d8	128/136
B-naphthylamine	d7	143/150
Nitrobenzene	d5	123/128
N-nitrosodimethylamine	.....	74
N-nitrosodi-n-propylamine	.....	70
N-nitrosodiphenylamile [2]	d6	169/175
Phenanthrene	d10	178/188
Phenol	d5	94/71
a-picoline	d7	93/100
Pyrene	d10	202/212
Styrene	d5	104/109
a-terpineol	d3	59/62
1,2,3-trichlorobenzene	d3	180/183
1,2,4-trichlorobenzene	d3	180/183

[1] Detected as azobenzene.
[2] Detected as diphenylamine.

TABLE 77—ACID EXTRACTABLE COMPOUND CHARACTERISTIC MASSES

Compound	Labeled analog	Primary m/z
4-chloro-3-methylphenol	d2	107/109
2-chlorophenol	d4	128/132
2,4-dichlorophenol	d3	162/167
2,4-dinitrophenol	d3	184/187
2-methyl-4,6-dinitrophenol	d2	198/200
2-nitrophenol	d4	139/143

TABLE 77—ACID EXTRACTABLE COMPOUND CHARACTERISTIC MASSES—Continued

Compound	Labeled analog	Primary m/z
4-nitrophenol	d4	139/143
Pentachlorophenol	13C6	266/272
2,3,6-trichlorophenol	d2	196/200
2,4,5-trichlorophenol	d2	196/200
2,4,6-trichlorophenol	d2	196/200

TABLE 8—ACCEPTANCE CRITERIA FOR PERFORMANCE TESTS

EGD No.[1]	Compound	Acceptance criteria				
		Initial precision and accuracy section 8.2.3 (µg/L)		Labeled compound recovery sec. 8.3 and 14.2 P (percent)	Calibration verification sec. 12.5 (µg/mL)	On-going accuracy sec. 11.6 R (µg/L)
		s	X			
301	Acenaphthene	21	79–134		80–125	72–144
201	Acenaphthene-d10	38	38–147	20–270	71–141	30–180
377	Acenaphtylene	38	69–186		60–166	61–207
277	Acenaphthylene-d8	31	38–146	23–239	66–152	33–168
378	Anthracene	41	58–174		60–168	50–199
278	Anthracene-d10	49	31–194	14–419	58–171	23–242
305	Benzidine	119	16–518		34–296	11–672
205	Benzidine-d8	269	ns–ns	ns–ns	ns–ns	ns–ns
372	Benzo(a)anthracene	20	65–168		70–142	62–176
272	Benzo(a)anthracene-d12	41	25–298	12–605	28–357	22–329
374	Benzo(b)fluoranthene	183	32–545		61–164	20–ns
274	Benzo(b)fluoranthene-d12	168	11–577	ns–ns	14–ns	ns–ns
375	Benzo(k)fluoranthene	26	59–143		13–ns	53–155
275	Benzo(k)fluoranthene-d12	114	15–514	ns–ns	13–ns	ns–685
373	Benzo(a)pyrene	26	62–195		78–129	59–206
273	Benzo(a)pyrene-d12	24	35–181	21–290	12–ns	32–194
379	Benzo(ghi)perylene	21	72–160		69–145	58–168
279	Benzo(ghi)perylene-d12	45	29–268	14–529	13–ns	25–303
712	Biphenyl (Appendix C)	41	75–148		58–171	62–176
612	Biphenyl-d12	43	28–165	ns–ns	52–192	17–267
318	Bis(2-chloroethyl) ether	34	55–196		61–164	50–213
218	Bis(2-chloroethyl) ether-d8	33	29–196	15–372	52–194	25–222
043	Bis(2-chloroethoxy)methane*	27	43–153		44–228	39–166
342	Bis(2-chloroisopropyl) ether	17	81–138		67–148	77–145
242	Bis(2-chloroisopropyl)ether-d12	27	35–149	20–260	44–229	30–169
366	Bis(2-ethylhexyl) phthalate	31	69–220		76–131	64–232
266	Bis(2-ethylhexyl) phthalate-d4	29	32–205	18–364	43–232	28–224
041	4-bromophenyl phenyl ether*	44	44–140		52–193	35–172
067	Butyl benzyl phthalate*	31	19–233		22–450	35–170
717	n-C10 (Appendix C)	51	24–195		42–235	19–237
617	n-C10-d22	70	ns–298	ns–ns	44–227	ns–504
706	n-C12 (Appendix C)	74	35–369		60–166	29–424
606	n-C12-d26	53	ns–331	ns–ns	41–242	ns–408
518	n-C14 (Appendix C)*	109	ns–985		37–268	ns–ns
719	n-C16 (Appendix C)	33	80–162		72–138	71–181
619	n-C16-d34	46	37–162	18–308	54–186	28–202
520	n-C18 (Appendix C)*	39	42–131		40–249	35–167
721	n-C20 (Appendix C)	59	53–263		54–184	46–301
621	n-C20-d42	34	34–172	19–306	62–162	29–198
522	n-C22 (Appendix C)*	31	45–152		40–249	39–195
723	n-C24 (Appendix C)	11	80–139		65–154	78–142
623	n-C24-d50	28	27–211	15–376	50–199	25–229
524	n-C26 (Appendix C)*	35	35–193		26–392	31–212
525	n-C28 (Appendix C)*	35	35–193		26–392	31–212
726	n-C30 (Appendix C)	32	61–200		66–152	56–215
626	n-C30-d62	41	27–242	13–479	24–423	23–274
528	Carbazole (4c)*	38	36–165		44–227	31–188
320	2-chloronaphthalene	100	46–357		58–171	35–442
220	2-chloronaphthalene-d7	41	30–168	15–324	72–139	24–204
322	4-chloro-3-methylphenol	37	76–131		85–115	62–159
222	4-chloro-3-methylphenol-d2	111	30–174	ns–613	68–147	14–314
324	2-chlorophenol	13	79–135		78–129	76–138
224	2-chlorophenol-d4	24	36–162	23–255	55–180	33–176
340	4-chlorophenyl phenyl ether	42	75–166		71–142	63–194

TABLE 8—ACCEPTANCE CRITERIA FOR PERFORMANCE TESTS—Continued

EGD No.[1]	Compound	Acceptance criteria				
		Initial precision and accuracy section 8.2.3 (µg/L)		Labeled compound recovery sec. 8.3 and 14.2 P (percent)	Calibration verification sec. 12.5 (µg/mL)	On-going accuracy sec. 11.6 R (µg/L)
		s	X			
240	4-chlorophenyl phenyl ether-d5	52	40–161	19–325	57–175	29–212
376	Chrysene	51	59–186	.....	70–142	48–221
276	Chrysene-d12	69	33–219	13–512	24–411	23–290
713	p-cymene (Appendix C)	18	76–140	.....	79–127	72–147
613	p-cymene-d14	67	ns–359	ns–ns	66–152	ns–468
082	Dibenzo(a,h)anthracene*	55	23–299	.....	13–761	19–340
705	Dibenzofuran (Appendix C)	20	85–136	.....	73–136	79–146
605	Dibenzofuran-d8	31	47–136	28–220	66–150	39–160
704	Dibenzothiophene (Synfuel)	31	79–150	.....	72–140	70–168
604	Dibenzothiophene-d8	31	48–130	29–215	69–145	40–156
368	Di-n-butyl phthalate	15	76–165	.....	71–142	74–169
268	Di-n-butyl phthalate-d4	23	23–195	13–346	52–192	22–209
325	1,2-dichlorobenzene	17	73–146	.....	74–135	70–152
225	1,2-dichlorobenzene-d4	35	14–212	ns–494	61–164	11–247
326	1,3-dichlorobenzene	43	63–201	.....	65–154	55–225
226	1,3-dichlorobenzene-d4	48	13–203	ns–550	52–192	ns–260
327	1,4-dichlorobenzene	42	61–194	.....	62–161	53–219
227	1,4-dichlorobenzene-d4	48	15–193	ns–474	65–153	11–245
328	3,3′-dichlorobenzidine	26	68–174	.....	77–130	64–185
228	3,3′-dichlorobenzidine-d6	80	ns–562	ns–ns	18–558	ns–ns
331	2,4-dichlorophenol	12	85–131	.....	67–149	83–135
231	2,4-dichlorophenol-d3	28	38–164	24–260	64–157	34–182
370	Diethyl phthalate	44	75–196	.....	74–135	65–222
270	Diethyl phthalate-d4	78	ns–260	ns–ns	47–211	ns–ns
334	2,4-dimethylphenol	13	62–153	.....	67–150	60–156
234	2,4-dimethylphenol-d3	22	15–228	ns–449	58–172	14–242
371	Dimethyl phthalate	36	74–188	.....	73–137	67–207
271	Dimethyl phthalate-d4	108	ns–640	ns–ns	50–201	ns–ns
359	2,4-dinitrophenol	18	72–134	.....	75–133	68–141
259	2,4-dinitrophenol-d3	66	22–308	ns–ns	39–256	17–378
335	2,4-dinitrotoluene	18	75–158	.....	79–127	72–164
235	2,4-dinitrotoluene-d3	37	22–245	10–514	53–187	19–275
336	2,6-dinitrotoluene	30	80–141	.....	55–183	70–159
236	2,6-dinitrotoluene-d3	59	44–184	17–442	36–278	31–250
369	Di-n-octyl phthalate	16	77–161	.....	71–140	74–166
269	Di-n-octyl phthalate-d4	46	12–383	ns–ns	21–467	10–433
707	Diphenylamine (Appendix C)	45	58–205	.....	57–176	51–231
607	Diphenylamine-d10	42	27–206	11–488	59–169	21–249
708	Diphenyl ether (Appendix C)	19	82–136	.....	83–120	77–144
608	Diphenyl ether-d10	37	36–155	19–281	77–129	29–186
337	1,2-diphenylhydrazine	73	49–308	.....	75–134	40–360
237	1,2-diphenylhydrazine-d10	35	31–173	17–316	58–174	26–200
339	Fluoranthene	33	71–177	.....	67–149	64–194
239	Fluoranthene-d10	35	36–161	20–278	47–215	30–187
380	Fluorene	29	81–132	.....	74–135	70–151
280	Fluorene-d10	43	51–131	27–238	61–164	38–172
309	Hexachlorobenzene	16	90–124	.....	78–128	85–132
209	Hexachlorobenzene-13C6	81	36–228	13–595	38–265	23–321
352	hexachlorobutadiene	56	51–251	.....	74–135	43–287
252	hexachlorobutadiene-13C4	63	ns–316	ns–ns	68–148	ns–413
312	hexachloroethane	227	21–ns	.....	71–141	13–ns
212	hexachloroethane-13C1	77	ns–400	ns–ns	47–212	ns–563
353	hexachlorocyclopentadiene	15	69–144	.....	77–129	67–148
253	hexachlorocyclopentadiene-13C4	60	ns–ns	ns–ns	47–211	ns–ns
083	ideno(1,2,3-cd)pyrene*	55	23–299	.....	13–761	19–340
354	isophorone	25	76–156	.....	70–142	70–168
254	isophorone-d8	23	49–133	33–193	52–194	44–147
360	2-methyl-4,6-dinitrophenol	19	77–133	.....	69–145	72–142
260	2-methyl-4,6-dinitrophenol-d2	64	36–247	16–527	56–177	28–307
355	naphthalene	20	80–139	.....	73–137	75–149
255	naphthalene-d8	39	28–157	14–305	71–141	22–192
702	B-naphthylamine (Appendix C)	49	10–ns	.....	39–256	ns–ns
602	B-naphthylamine-d7	33	ns–ns	ns–ns	44–230	ns–ns
356	nitrobenzene	25	69–161	.....	85–115	65–169
256	nitrobenzene-d5	28	18–265	ns–ns	46–219	15–314
357	2-nitrophenol	15	78–140	.....	77–129	75–145
257	2-nitrophenol-d4	23	41–145	27–217	61–163	37–158

TABLE 8—ACCEPTANCE CRITERIA FOR PERFORMANCE TESTS—Continued

EGD No.[1]	Compound	Acceptance criteria				
		Initial precision and accuracy section 8.2.3 (µg/L)		Labeled compound recovery sec. 8.3 and 14.2 P (percent)	Calibration verification sec. 12.5 (µg/mL)	On-going accuracy sec. 11.6 R (µg/L)
		s	X			
358	4-nitrophenol	42	62–146	.....	55–183	51–175
258	4-nitrophenol-d4	188	14–398	ns–ns	35–287	ns–ns
061	N-nitrosodimethylamile*	198	21–472	.....	40–249	12–807
063	N-nitrosodi-n-proplyamine*	198	21–472	.....	40–249	12–807
362	N-nitrosodiphenylamine	45	65–142	.....	68–148	53–173
262	N-nitrosodiphenylamine-d6	37	54–126	26–256	59–170	40–166
364	pentachlorophenol	21	76–140	.....	77–130	71–150
264	pentachlorophenol-13C6	49	37–212	18–412	42–237	29–254
381	phenanthrene	13	93–119	.....	75–133	87–126
281	phenanthrene-d10	40	45–130	24–241	67–149	34–168
365	phenol	36	77–127	.....	65–155	62–154
265	phenol-d5	161	21–210	ns–ns	48–208	ns–ns
703	a-picoline (Synfuel)	38	59–149	.....	60–165	50–174
603	a-picoline-d7	138	11–380	ns–ns	31–324	ns–608
384	pyrene	19	76–152	.....	76–132	72–159
284	pyrene-d10	29	32–176	18–303	48–210	28–196
710	styrene (Appendix C)	42	53–221	.....	65–153	48–244
610	styrene-d5	49	ns–281	ns–ns	44–228	ns–348
709	a-terpineol (Appendix C)	44	42–234	.....	54–186	38–258
609	a-terpineol-d3	48	22–292	ns–672	20–502	18–339
529	1,2,3-trichlorobenzene (4c)*	69	15–229	.....	60–167	11–297
308	1,2,4-trichlorobenzene	19	82–136	.....	78–128	77–144
208	1,2,4-trichlorobenzene-d3	57	15–212	ns–592	61–163	10–282
530	2,3,6-trichlorophenol (4c)*	30	58–137	.....	56–180	51–153
531	2,4,5-trichlorophenol (4c)*	30	58–137	.....	56–180	51–153
321	2,4,6-trichlorophenol	57	59–205	.....	81–123	48–244
221	2,4,6-trichlorophenol-d2	47	43–183	21–363	69–144	34–226

[1] Reference numbers beginning with 0, 1 or 5 indicate a pollutant quantified by the internal standard method; reference numbers beginning with 2 or 6 indicate a labeled compound quantified by the internal standard method; reference numbers beginning with 3 or 7 indicate a pollutant quantified by isotope dilution.

* Measured by internal standard; specification derived from related compound.
ns=no specification; limit is outside the range that can be measured reliably.

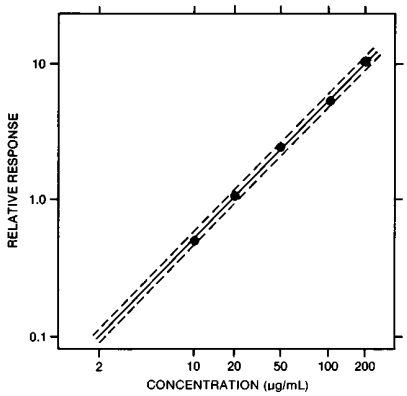


FIGURE 1 Relative Response Calibration Curve for Phenol. The Dotted Lines Enclose a ±10 Percent Error Window.

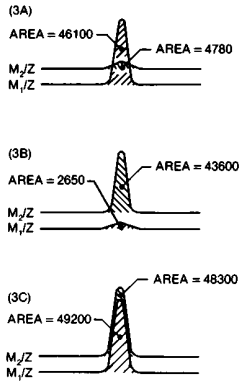


FIGURE 3 Extracted Ion Current Profiles for (3A) Unlabeled Compound, (3B) Labeled Compound, and (3C) Equal Mixture of Unlabeled and Labeled Compounds.

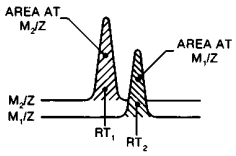


FIGURE 2 Extracted Ion Current Profiles for Chromatographically Resolved Labeled (m_2/z) and Unlabeled (m_1/z) Pairs.

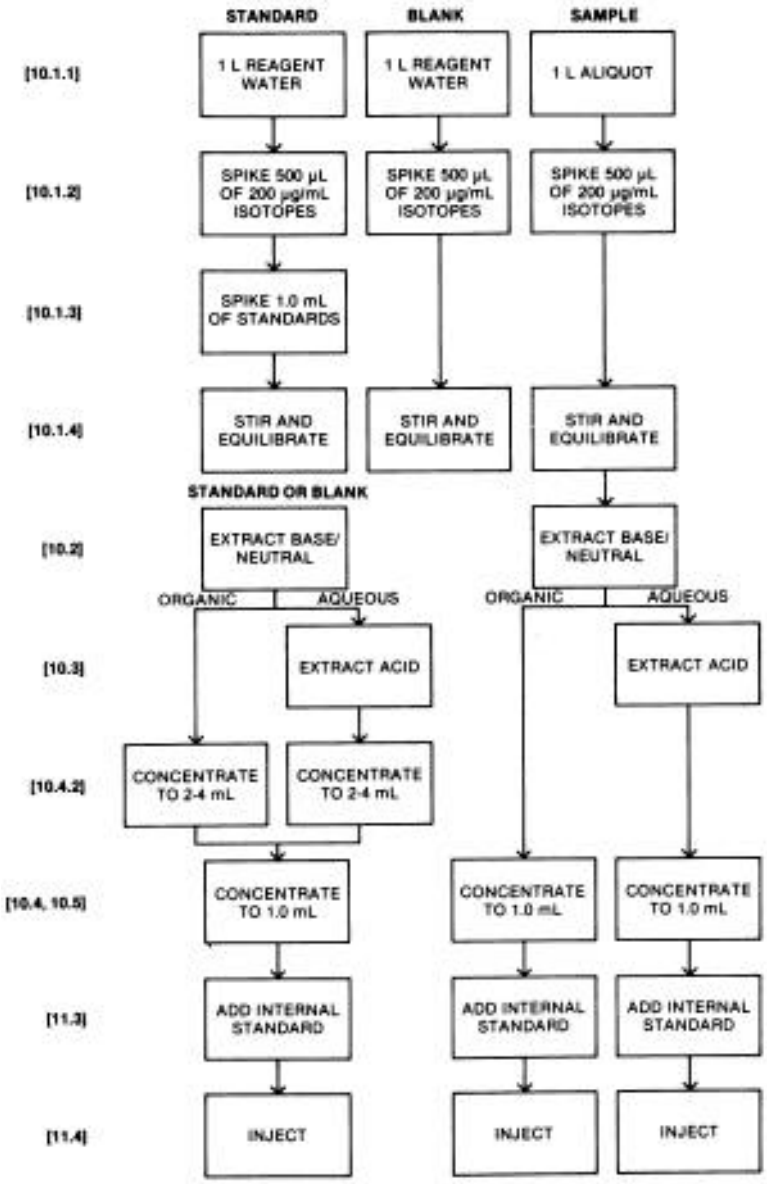


FIGURE 4 Flow Chart for Extraction/Concentration of Precision and Recovery Standard, Blank, and Sample by Method 1625. Numbers in Brackets [ ] Refer to Section Numbers in the Method.

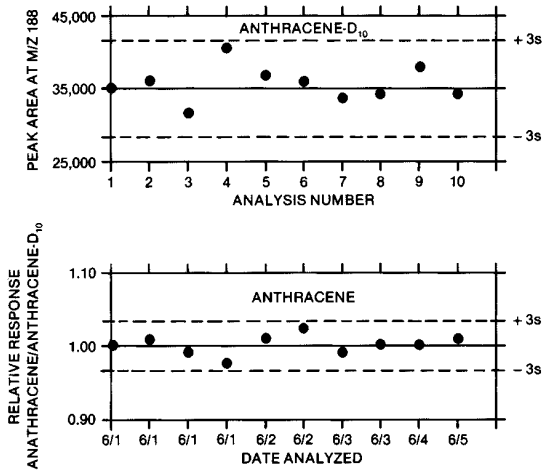


FIGURE 5 Quality Control Charts Showing Area (top graph) and Relative Response of Anthracene to Anthracene-d_{10} (lower graph) Plotted as a Function of Time or Analysis Number.

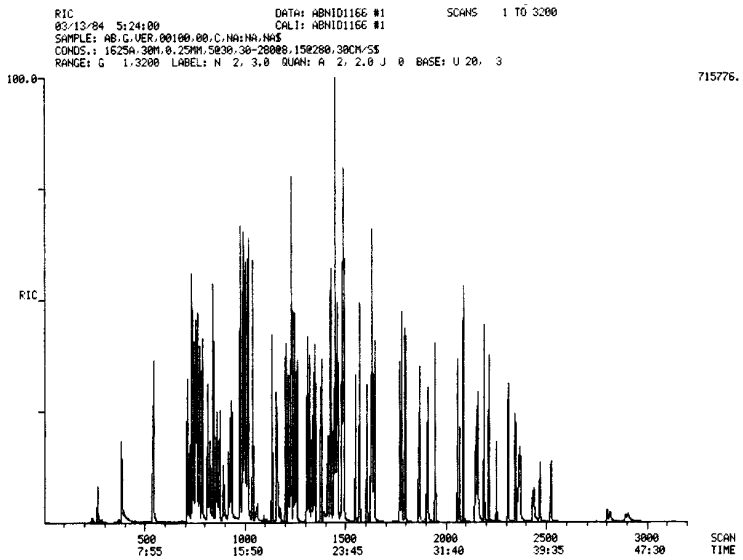


FIGURE 6 Chromatogram of Combined Acid/base/neutral Standard.

[49 FR 43261, Oct. 26, 1984; 50 FR 692, 695, Jan. 4, 1985, as amended at 51 FR 23702, June 30, 1986]

APPENDIX B TO PART 136—DEFINITION AND PROCEDURE FOR THE DETERMINATION OF THE METHOD DETECTION LIMIT—REVISION 1.11

Definition

The method detection limit (MDL) is defined as the minimum concentration of a substance that can be measured and reported with 99% confidence that the analyte concentration is greater than zero and is determined from analysis of a sample in a given matrix containing the analyte.

Scope and Application

This procedure is designed for applicability to a wide variety of sample types ranging from reagent (blank) water containing analyte to wastewater containing analyte. The MDL for an analytical procedure may vary as a function of sample type. The procedure requires a complete, specific, and well defined analytical method. It is essential that all sample processing steps of the analytical method be included in the determination of the method detection limit.

The MDL obtained by this procedure is used to judge the significance of a single measurement of a future sample.

The MDL procedure was designed for applicability to a broad variety of physical and chemical methods. To accomplish this, the procedure was made device- or instrument-independent.

Procedure

1. Make an estimate of the detection limit using one of the following:

(a) The concentration value that corresponds to an instrument signal/noise in the range of 2.5 to 5.

(b) The concentration equivalent of three times the standard deviation of replicate instrumental measurements of the analyte in reagent water.

(c) That region of the standard curve where there is a significant change in sensitivity, i.e., a break in the slope of the standard curve.

(d) Instrumental limitations.

It is recognized that the experience of the analyst is important to this process. However, the analyst must include the above considerations in the initial estimate of the detection limit.

2. Prepare reagent (blank) water that is as free of analyte as possible. Reagent or interference free water is defined as a water sample in which analyte and interferent concentrations are not detected at the method detection limit of each analyte of interest. Interferences are defined as systematic errors in the measured analytical signal of an established procedure caused by the presence of interfering species (interferent). The interferent concentration is presupposed to be normally distributed in representative samples of a given matrix.

3. (a) If the MDL is to be determined in reagent (blank) water, prepare a laboratory standard (analyte in reagent water) at a concentration which is at least equal to or in the same concentration range as the estimated method detection limit. (Recommend between 1 and 5 times the estimated method detection limit.) Proceed to Step 4.

(b) If the MDL is to be determined in another sample matrix, analyze the sample. If the measured level of the analyte is in the recommended range of one to five times the estimated detection limit, proceed to Step 4.

If the measured level of analyte is less than the estimated detection limit, add a known amount of analyte to bring the level of analyte between one and five times the estimated detection limit.

If the measured level of analyte is greater than five times the estimated detection limit, there are two options.

(1) Obtain another sample with a lower level of analyte in the same matrix if possible.

(2) The sample may be used as is for determining the method detection limit if the analyte level does not exceed 10 times the MDL of the analyte in reagent water. The variance of the analytical method changes as the analyte concentration increases from the MDL, hence the MDL determined under these circumstances may not truly reflect method variance at lower analyte concentrations.

4. (a) Take a minimum of seven aliquots of the sample to be used to calculate the method detection limit and process each through the entire analytical method. Make all computations according to the defined method with final results in the method reporting units. If a blank measurement is required to calculate the measured level of analyte, obtain a separate blank measurement for each sample aliquot analyzed. The average blank measurement is subtracted from the respective sample measurements.

(b) It may be economically and technically desirable to evaluate the estimated method detection limit before proceeding with 4a. This will: (1) Prevent repeating this entire procedure when the costs of analyses are high and (2) insure that the procedure is being conducted at the correct concentration. It is quite possible that an inflated MDL will be calculated from data obtained at many times the real MDL even though the level of analyte is less than five times the calculated method detection limit. To insure that the estimate of the method detection limit is a good estimate, it is necessary to determine that a lower concentration of analyte will not result in a significantly lower method detection limit. Take two aliquots of the sample to be used to calculate the method detection limit and process each

through the entire method, including blank measurements as described above in 4a. Evaluate these data:

(1) If these measurements indicate the sample is in desirable range for determination of the MDL, take five additional aliquots and proceed. Use all seven measurements for calculation of the MDL.

(2) If these measurements indicate the sample is not in correct range, reestimate the MDL, obtain new sample as in 3 and repeat either 4a or 4b.

5. Calculate the variance (S^2) and standard deviation (S) of the replicate measurements, as follows:

$$S^2 = \frac{1}{n-1}\left[\sum_{i=1}^{n} x_i^2 - \frac{\left(\sum_{i=1}^{n} X_i\right)^2}{n}\right] S = \left(S^2\right)^{\frac{1}{2}}$$

where:

X_i; i=1 to n, are the analytical results in the final method reporting units obtained from the n sample aliquots and Σ refers to the sum of the X values from i=l to n.

6. (a) Compute the MDL as follows:

$$MDL = t_{(n-1,1-\alpha = 0.99)} \quad (S)$$

where:

MDL = the method detection limit

$t_{(n-1,1-\alpha = .99)}$ = the students' t value appropriate for a 99% confidence level and a standard deviation estimate with n-1 degrees of freedom. See Table.

S = standard deviation of the replicate analyses.

(b) The 95% confidence interval estimates for the MDL derived in 6a are computed according to the following equations derived from percentiles of the chi square over degrees of freedom distribution (χ^2/df).

LCL = 0.64 MDL

UCL = 2.20 MDL

where: LCL and UCL are the lower and upper 95% confidence limits respectively based on seven aliquots.

7. Optional iterative procedure to verify the reasonableness of the estimate of the MDL and subsequent MDL determinations.

(a) If this is the initial attempt to compute MDL based on the estimate of MDL formulated in Step 1, take the MDL as calculated in Step 6, spike the matrix at this calculated MDL and proceed through the procedure starting with Step 4.

(b) If this is the second or later iteration of the MDL calculation, use S^2 from the current MDL calculation and S^2 from the previous MDL calculation to compute the F-ratio. The F-ratio is calculated by substituting the larger S^2 into the numerator S^2_A and the other into the denominator S^2_B. The computed F-ratio is then compared with the F-ratio found in the table which is 3.05 as follows: if $S^2_A/S^2_B<3.05$, then compute the pooled standard deviation by the following equation:

$$S_{pooled} = \left[\frac{6S_A^2 + 6S_B^2}{12}\right]^{\frac{1}{2}}$$

if $S^2_A/S^2_B>3.05$, respike at the most recent calculated MDL and process the samples through the procedure starting with Step 4. If the most recent calculated MDL does not permit qualitative identification when samples are spiked at that level, report the MDL as a concentration between the current and previous MDL which permits qualitative identification.

(c) Use the S_{pooled} as calculated in 7b to compute the final MDL according to the following equation:

$$MDL = 2.681\ (S_{pooled})$$

where 2.681 is equal to $t_{(12,\ 1-\alpha\ =.99)}$.

(d) The 95% confidence limits for MDL derived in 7c are computed according to the following equations derived from precentiles of the chi squared over degrees of freedom distribution.

LCL=0.72 MDL

UCL=1.65 MDL

where LCL and UCL are the lower and upper 95% confidence limits respectively based on 14 aliquots.

TABLES OF STUDENTS' t VALUES AT THE 99 PERCENT CONFIDENCE LEVEL

Number of replicates	Degrees of freedom (n-1)	$t_{cn-1,\ .99}$)
7	6	3.143
8	7	2.998
9	8	2.896
10	9	2.821
11	10	2.764
16	15	2.602
21	20	2.528
26	25	2.485
31	30	2.457
61	60	2.390
00	00	2.326

Reporting

The analytical method used must be specifically identified by number or title ald the MDL for each analyte expressed in the appropriate method reporting units. If the analytical method permits options which affect the method detection limit, these conditions must be specified with the MDL value. The sample matrix used to determine the MDL must also be identified with MDL value. Report the mean analyte level with the MDL

and indicate if the MDL procedure was iterated. If a laboratory standard or a sample that contained a known amount analyte was used for this determination, also report the mean recovery.

If the level of analyte in the sample was below the determined MDL or exceeds 10 times the MDL of the analyte in reagent water, do not report a value for the MDL.

[49 FR 43430, Oct. 26, 1984; 50 FR 694, 696, Jan. 4, 1985, as amended at 51 FR 23703, June 30, 1986]

APPENDIX C TO PART 136—INDUCTIVELY COUPLED PLASMA—ATOMIC EMISSION SPECTROMETRIC METHOD FOR TRACE ELEMENT ANALYSIS OF WATER AND WASTES METHOD 200.7

1. Scope and Application

1.1 This method may be used for the determination of dissolved, suspended, or total elements in drinking water, surface water, and domestic and industrial wastewaters.

1.2 Dissolved elements are determined in filtered and acidified samples. Appropriate steps must be taken in all analyses to ensure that potential interferences are taken into account. This is especially true when dissolved solids exceed 1500 mg/L. (See Section 5.)

1.3 Total elements are determined after appropriate digestion procedures are performed. Since digestion techniques increase the dissolved solids content of the samples, appropriate steps must be taken to correct for potential interference effects. (See Section 5.)

1.4 Table 1 lists elements for which this method applies along with recommended wavelengths and typical estimated instrumental detection limits using conventional pneumatic nebulization. Actual working detection limits are sample dependent and as the sample matrix varies, these concentrations may also vary. In time, other elements may be added as more information becomes available and as required.

1.5 Because of the differences between various makes and models of satisfactory instruments, no detailed instrumental operating instructions can be provided. Instead, the analyst is referred to the instruction provided by the manufacturer of the particular instrument.

2. Summary of Method

2.1 The method describes a technique for the simultaneous or sequential multielement determination of trace elements in solution. The basis of the method is the measurement of atomic emission by an optical spectroscopic technique. Samples are nebulized and the aerosol that is produced is transported to the plasma torch where excitation occurs. Characteristic atomic-line emission spectra are produced by a radio-frequency inductively coupled plasma (ICP). The spectra are dispersed by a grating spectrometer and the intensities of the lines are monitored by photomultiplier tubes. The photocurrents from the photomultiplier tubes are processed and controlled by a computer system. A background correction technique is required to compensate for variable background contribution to the determination of trace elements. Background must be measured adjacent to analyte lines on samples during analysis. The position selected for the background intensity measurement, on either or both sides of the analytical line, will be determined by the complexity of the spectrum adjacent to the analyte line. The position used must be free of spectral interference and reflect the same change in background intensity as occurs at the analyte wavelength measured. Background correction is not required in cases of line broadening where a background correction measurement would actually degrade the analytical result. The possibility of additional interferences named in 5.1 (and tests for their presence as described in 5.2) should also be recognized and appropriate corrections made.

3. Definitions

3.1 *Dissolved*—Those elements which will pass through a 0.45 µm membrane filter.

3.2 *Suspended*—Those elements which are retained by a 0.45 µm membrane filter.

3.3 *Total*—The concentration determined on an unfiltered sample following vigorous digestion (Section 9.3), or the sum of the dissolved plus suspended concentrations. (Section 9.1 plus 9.2).

3.4 *Total recoverable*—The concentration determined on an unfiltered sample following treatment with hot, dilute mineral acid (Section 9.4).

3.5 *Instrumental detection limit*—The concentration equivalent to a signal, due to the analyte, which is equal to three times the standard deviation of a series of ten replicate measurements of a reagent blank signal at the same wavelength.

3.6 *Sensitivity*—The slope of the analytical curve, i.e. functional relationship between emission intensity and concentration.

3.7 *Instrument check standard*—A multielement standard of known concentrations prepared by the analyst to monitor and verify instrument performance on a daily basis. (See 7.6.1)

3.8 *Interference check sample*—A solution containing both interfering and analyte elemelts of known concentration that can be used to verify background and interelement correction factors. (See 7.6.2.)

3.9 *Quality control sample*—A solution obtained from an outside source having known, concentration values to be used to verify the calibration standards. (See 7.6.3)

3.10 *Calibration standards*—A series of known standard solutions used by the analyst for calibration of the instrument (i.e., preparation of the analytical curve). (See 7.4)

3.11 *Linear dynamic range*—The concentration range over which the analytical curve remains linear.

3.12 *Reagent blank*—A volume of deionized, distilled water containing the same acid matrix as the calibration standards carried through the entire analytical scheme. (See 7.5.2)

3.13 *Calibration blank*—A volume of deionized, distilled water acidified with HNO_3 and HCl. (See 7.5.1)

3.14 *Methmd of standard addition*—The standard addition technique involves the use of the unknown and the unknown plus a known amount of standard. (See 10.6.1.)

4. Safety

4.1 The toxicity of carcinogenicity of each reagent used in this method has not been precisely defined; however, each chemical compound should be treated as a potential health hazard. From this viewpoint, exposure to these chemicals must be reduced to the lowest possible level by whatever means available. The laboratory is repsonsible for maintaining a current awareness file of OSHA regulations regarding the safe handling of the chemicals specified in this method. A reference file of material data handling sheets should also be made available to all personnel involved in the chemical analysis. Additional references to laboratory safety are available and have been identified [14.7, 14.8 and 14.9] for the information of the analyst.

5. Interferences

5.1 Several types of interference effects may contribute to inaccuracies in the determination of trace elements. They can be summarized as follows:

5.1.1 *Spectral interferences* can be categorized as (1) overlap of a spectral line from another element; (2) unresolved overlap of molecular band spectra; (3) background contribution from continuous or recombination phenomena; and (4) background contribution from stray light from the line emission of high concentration elements. The first of these effects can be compensated by utilizing a computer correction of the raw data, requiring the monitoring and measurement of the interfering element. The second effect may require selection of an alternate wavelength. The third and fourth effects can usually be compensated by a background correction adjacent to the analyte line. In addition, users of simultaneous multi-element instrumentation must assume the responsibility of verifying the absence of spectral interference from an element that could occur in a sample but for which there is no channel in the instrument array. Listed in Table 2 are some interference effects for the recommended wavelengths given in Table 1. The data in Table 2 are intended for use only as a rudimentary guide for the indication of potential spectral interferences. For this purpose, linear relations between concentration and intensity for the analytes and the interferents can be assumed. The Interference information, which was collected at the Ames Laboratory,[1] is expressed as analyte concentration equivalents (i.e. false analyte concentrations) arising from 100 mg/L of the interferent element. The suggested use of this information is as follows: Assume that arsenic (at 193.696 nm) is to be determined in a sample containing approximately 10 mg/L of aluminum. According to Table 2, 100 mg/L of aluminum would yield a false signal for arsenic equivalent to approximately 1.3 mg/L. Therefore, 10 mg/L of aluminum would result in a false signal for arsenic equivalent to approximately 0.13 mg/L. The reader is cautioned that other analytical systems may exhibit somewhat different levels of interference than those shown in Table 2, and that the interference effects must be evaluated for each individual system.

Only those interferents listed were investigated and the blank spaces in Table 2 indicate that measurable interferences were not observed for the interferent concentrations listed in Table 3. Generally, interferences were discernible if they produced peaks or background shifts corresponding to 2–5% of the peaks generated by the analyte concentrations also listed in Table 3.

At present, information on the listed silver and potassium wavelengths are not available but it has been reported that second order energy from the magnesium 383.231 nm wavelength interferes with the listed potassium line at 766.491 nm.

5.1.2 *Physical interferences* are generally considered to be effects associated with the sample nebulization and transport processes. Such properties as change in viscosity and surface tension can cause significant inaccuracies especially in samples which may contain high dissolved solids and/or acid concentrations. The use of a peristaltic pump may lessen these interferences. If these types of interferences are operative, they must be reduced by dilution of the sample and/or utilization of standard addition techniques. Another problem which can occur from high dissolved solids is salt buildup at the tip of the nebulizer. This affects aersol flow rate causing instrumental drift. Wetting the argon prior to nebulization, the use of a tip washer, or sample dilution have been used to control this problem. Also, it has been reported that better control of the argon flow rate improves instrument performance. This

[1] Ames Laboratory, USDOE, Iowa State University, Ames Iowa 50011.

is accomplished with the use of mass flow controllers.

5.1.3 *Chemical Interferences* are characterized by molecular compound formation, ionization effects and solute vaporization effects. Normally these effects are not pronounced with the ICP technique, however, if observed they can be minimized by careful selection of operating conditions (that is, incident power, observation position, and so forth), by buffering of the sample, by matrix matching, and by standard addition procedures. These types of interferences can be highly dependent on matrix type and the specific analyte element.

5.2 It is recommended that whenever a new or unusual sample matrix is encountered, a series of tests be performed prior to reporting concentration data for analyte elements. These tests, as outlined in 5.2.1 through 5.2.4, will ensure the analyst that neither positive nor negative interference effects are operative on any of the analyte elements thereby distorting the accuracy of the reported values.

5.2.1 *Serial dilution.* If the analyte concentration is sufficiently high (minimally a factor of 10 above the instrumental detection limit after dilution), an analysis of a dilution should agree within 5 percent of the original determination (or within some acceptable control limit (14.3) that has been established for that matrix.). If not, a chemical or physical interference effect should be suspected.

5.2.2 *Spike addition.* The recovery of a spike addition added at a minimum level of 10X the instrumental detection limit (maximum 100X) to the original determination should be recovered to within 90 to 110 percent or within the established control limit for that matrix. If not, a matrix effect should be suspected. The use of a standard addition analysis procedure can usually compensate for this effect.

Caution: The standard addition technique does not detect coincident spectral overlap. If suspected, use of computerized compensation, an alternate wavelength, or comparison with an alternate method is recommended (See 5.2.3).

5.2.3 *Comparison with alternate method of analysis.* When investigating a new sample matrix, comparison tests may be performed with other analytical techniques such as atomic absorption spectrometry, or other approved methodology.

5.2.4 *Wavelength scanning of analyte line region.* If the appropriate equipment is available, wavelength scanning can be performed to detect potential spectral interferences.

6. Apparatus

6.1 Inductively Coupled Plasma-Atomic Emission Spectrometer.

6.1.1 Computer controlled atomic emission spectrometer with background correction.

6.1.2 Radiofrequency generator.

6.1.3 Argon gas supply, welding grade or better.

6.2 Operating conditions—Because of the differences between various makes and models of satisfactory instruments, no detailed operating instructions can be provided. Instead, the analyst should follow the instructions provided by the manufacturer of the particular instrument. Sensitivity, instrumental detection limit, precision, linear dynamic range, and interference effects must be investigated and established for each individual analyte line on that particular instrument. It is the responsibility of the analyst to verify that the instrument configuration and operating conditions used satisfy the analytical requirements and to maintain quality control data confirming instrument performance and analytical results.

7. Reagents and Standards

7.1 Acids used in the preparation of standards and for sample processing must be ultra-high purity grade or equivalent. Redistilled acids are acceptable.

7.1.1 *Acetic acid,* conc. (sp gr 1.06).

7.1.2 *Hydrochloric acid,* conc. (sp gr 1.19).

7.1.3 *Hydrochloric acid,* (1+1): Add 500 mL conc. HCl (sp gr 1.19) to 400 mL deionized, distilled water and dilute to 1 liter.

7.1.4 *Nitric acid,* conc. (sp gr 1.41).

7.1.5 *Nitric acid,* (1+1): Add 500 mL conc. HNO_3 (sp gr 1.41) to 400 mL deionized, distilled water and dilute to 1 liter.

7.2 *Deionized, distilled water:* Prepare by passing distilled water through a mixed bed of cation and anion exchange resins. Use deionized, distilled water for the preparation of all reagents, calibration standards and as dilution water. The purity of this water must be equivalent to ASTM Type II reagent water of Specification D 1193 (14.6).

7.3 *Standard stock solutions* may be purchased or prepared from ultra high purity grade chemicals or metals. All salts must be dried for 1 h at 105°C unless otherwise specified.

(CAUTION: Many metal salts are extremely toxic and may be fatal if swallowed. Wash hands thoroughly after handling.)

Typical stock solution preparation procedures follow:

7.3.1 *Aluminum solution, stock,* 1 mL=100µg Al: Dissolve 0.100 g of aluminum metal in an acid mixture of 4 mL of (1+1) HCl and 1 mL of conc. HNO_3 in a beaker. Warm gently to effect solution. When solution is complete, transfer quantitatively to a liter flask add an additional 10 mL of (1+1) HCl and dilute to 1,000 mL with deionized, distilled water.

7.3.2 *Antimony solution stock,* 1 mL=100 µg Sb: Dissolve 0.2669 g $K(SbO)C_4H_4O_6$ in deionized distilled water, add 10 mL (1+1) HCl

and dilute to 1,000 mL with deionized, distilled water.

7.3.3 *Arsenic solution, stock,* 1 mL=100 µg As: Dissolve 0.1320 g of As_2O_3 in 100 mL of deionized, distilled water containing 0.4 g NaOH. Acidify the solution with 2 mL conc. HNO_3 and dilute to 1,000 mL with deionized, distilled water.

7.3.4 *Barium solution, stock,* 1 mL=100 µg Ba: Dissolve 0.1516 g $BaCl_2$ (dried at 250°C for 2 hrs) in 10 mL deionized, distilled water with 1 mL (1+1) HCl. Add 10.0 mL (1+1) HCl and dilute to 1,000 with mL deionized, distilled water.

7.3.5 *Beryllium solution, stock,* 1 mL=100 µg Be: *Do not dry*. Dissolve 1.966 g $BeSO_4 \cdot 4H_2O$, in deionized, distilled water, add 10.0 mL conc. HNO_3 and dilute to 1,000 mL with deionized, distilled water.

7.3.6 *Boron solution, stock,* 1 mL=100 µg B: *Do not dry*. Dissolve 0.5716 g anhydrous H_3BO_3 in deionized, distilled water and dilute to 1,000 mL. Use a reagent meeting ACS specifications, keep the bottle tightly stoppered and store in a desiccator to prevent the entrance of atmospheric moisture.

7.3.7 *Cadmium solution, stock,* 1 mL=100 µg Cd: Dissolve 0.1142 g CdO in a minimum amount of (1+1) HNO_3. Heat to increase rate of dissolution. Add 10.0 mL conc. HNO_3 and dilute to 1,000 mL with deionized, distilled water.

7.3.8 *Calcium solution, stock,* 1 mL=100 µg Ca: Suspend 0.2498 g $CaCO_3$ dried at 180 °C for 1 h before weighing in deionized, distilled water and dissolve cautiously with a minimum amount of (1+1) HNO_3. Add 10.0 mL conc. HNO_3 and dilute to 1,000 mL with deionized, distilled water.

7.3.9 *Chromium solution, stock,* 1 mL=100 µg Cr: Dissolve 0.1923 g of CrO_3 in deionized, distilled water. When solution is complete, acidify with 10 mL conc. HNO_3 and dilute to 1,000 mL with deionized, distilled water.

7.3.10 *Cobalt solution, stock,* 1 mL=100 µg Co: Dissolve 0.1000 g of cobalt metal in a minimum amount of (1+1) HNO_3. Add 10.0 mL (1+1) HCl and dilute to 1,000 mL with deionized, distilled water.

7.3.11 *Copper solution, stock,* 1 mL=100 µg Cu: Dissolve 0.1252 g CuO in a minimum amount of (1+1) HNO_3. Add 10.0 mL conc. HNO_3 and dilute to 1,000 mL with deionized, distilled water.

7.3.12 *Iron solution, stock,* 1 mL=100 µg Fe: Dissolve 0.1430 g Fe_2O_3 in a warm mixture of 20 mL (1+1) HCl and 2 mL of conc. HNO_3. Cool, add an additional 5 mL of conc. HNO_3 and dilute to 1,000 mL with deionized, distilled water.

7.3.13 *Lead solution, stock,* 1 mL=100 µg Pb: Dissolve 0.1599 g $Pb(NO_3)_2$ in a minimum amount of (1+1) HNO_3. Add 10.0 mL conc. HNO_3 and dilute to 1,000 mL with deionized, distilled water.

7.3.14 *Magnesium solution, stock,* 1 mL=100 µg Mg: Dissolve 0.1658 g MgO in a minimum amount of (1+1) HNO_3. Add 10.0 mL conc. HNO_3 and dilute to 1,000 mL with deionized, distilled water.

7.3.15 *Manganese solution, stock,* 1 mL=100 µg Mn: Dissolve 0.1000 g of manganese metal in the acid mixture 10 mL conc. HCl and 1 mL conc. HNO_3, and dilute to 1,000 mL with deionized, distilled water.

7.3.16 *Molybdenum solution, stock,* 1 mL=100 µg Mo: Dissolve 0.2043 g $(NH_4)_2MoO_4$ in deionized, distilled water and dilute to 1,000 mL.

7.3.17 *Nickel solution, stock,* 1 mL=100 µg Ni: Dissolve 0.1000 g of nickel metal in 10 mL hot conc. HNO_3, cool and dilute to 1,000 mL with deionized, distilled water.

7.3.18 *Potassium solution, stock,* 1 mL=100 µg K: Dissolve 0.1907 g KCl, dried at 110 °C, in deionized, distilled water and dilute to 1,000 mL.

7.3.19 *Selenium solution, stock,* 1 mL=100 µg Se: *Do not dry*. Dissolve 0.1727 g H_2SeO_3 (actual assay 94.6%) in deionized, distilled water and dilute to 1,000 mL.

7.3.20 *Silica solution, stock,* 1 mL=100 µg SiO_2: *Do not dry*. Dissolve 0.4730 g $Na_2SiO_3 \cdot 9H_2O$ in deionized, distilled water. Add 10.0 mL conc. HNO_3 and dilute to 1,000 mL with deionized, distilled water.

7.3.21 *Silver solution, stock,* 1 mL=100 µg Ag: Dissolve 0.1575 g $AgNO_3$ in 100 mL of deionized, distilled water and 10 mL conc. HNO_3. Dilute to 1,000 mL with deionized, distilled water.

7.3.22 *Sodium solution, stock,* 1 mL=100 µg Na: Dissolve 0.2542 g NaCl in deionized, distilled water. Add 10.0 mL conc. HNO_3 and dilute to 1,000 mL with deionized, distilled water.

7.3.23 *Thallium solution, stock,* 1 mL=100 µg Tl: Dissolve 0.1303 g $TlNO_3$ in deionized, distilled water. Add 10.0 mL conc. HNO_3 and dilute to 1,000 mL with deionized, distilled water.

7.3.24 *Vanadium solution, stock,* 1 mL=100 µg V: Dissolve 0.2297 NH_4VO_3 in a minimum amount of conc. HNO_3. Heat to increase rate of dissolution. Add 10.0 mL conc. HNO_3 and dilute to 1,000 mL with deionized, distilled water.

7.3.25 *Zinc solution, stock,* 1 mL=100 µg Zn: Dissolve 0.1245 g ZnO in a minimum amount of dilute HNO_3. Add 10.0 mL conc. HNO_3 and dilute to 1,000 mL deionized, distilled water.

7.4 *Mixed calibration standard solutions*—Prepare mixed calibration standard solutions by combining appropriate volumes of the stock solutions in volumetric flasks. (See 7.4.1 thru 7.4.5) Add 2 mL of (1+1) HNO_3 and 10 mL of (1+1) HC1 and dilute to 100 mL with deionized, distilled water. (See Notes 1 and 6.) Prior to preparing the mixed standards, each stock solution should be analyzed separately to determine possible spectral interference or the presence of impurities. Care should be taken when preparing the

mixed standards that the elemelts are compatible and stable. Transfer the mixed standard solutions to a FEP fluorocarbon or unused polyethylene bottle for storage. Fresh mixed standards should be prepared as needed with the realization that concentration can change on aging. Calibration standards must be initially verified using a quality control sample and monitored weekly for stability (See 7.6.3). Although not specifically required, some typical calibration standard combinations follow when using those specific wavelengths listed in Table 1.

7.4.1 *Mixed standard solution I*—Manganese, beryllium, cadmium, lead, and zinc.

7.4.2 *Mixed standard solution II*—Barium, copper, iron, vanadium, and cobalt.

7.4.3 *Mixed standard solution III*—Molybdenum, silica, arsenic, and selenium.

7.4.4 *Mixed standard solution IV*—Calcium, sodium, potassium, aluminum, chromium and nickel.

7.4.5 *Mixed standard solution V*—Antimony, boron, magnesium, silver, and thallium.

NOTE: 1. If the addition of silver to the recommended acid combination results in an initial precipitation, add 15 mL of deionized distilled water and warm the flask until the solution clears. Cool and dilute to 100 mL with deionized, distilled water. For this acid combination the silver concentration should be limited to 2 mg/L. Silver under these conditions is stable in a tap water matrix for 30 days. Higher concentrations of silver require additional HCl.

7.5 Two types of blanks are required for the analysis. The calibration blank (3.13) is used in establishing the analytical curve while the reagent blank (3.12) is used to correct for possible contamination resulting from varying amounts of the acids used in the sample processing.

7.5.1 *The calibration blank* is prepared by diluting 2 mL of (1+1) HNO_3 and 10 mL of (1+1) HCl to 100 mL with deionized, distilled water. (See Note 6.) Prepare a sufficient quantity to be used to flush the system between standards and samples.

7.5.2 *The reagent blank* must contain all the reagents and in the same volumes as used in the processing of the samples. The reagent blank must be carried through the complete procedure and contain the same acid concentration in the final solution as the sample solution used for analysis.

7.6 In addition to the calibration standards, an instrument check standard (3.7), an interference check sample (3.8) and a quality control sample (3.9) are also required for the analyses.

7.6.1 The *instrument check standard* is prepared by the analyst by combining compatible elements at a concentration equivalent to the midpoint of their respective calibration curves. (See 12.1.1.)

7.6.2 The *interference check sample* is prepared by the analyst in the following manner. Select a representative sample which contains minimal concentrations of the analytes of interest but known concentration of interfering elements that will provide an adequate test of the correction factors. Spike the sample with the elements of interest at the approximate concentration of either 100 µg/L or 5 times the estimated detection limits given in Table 1. (For effluent samples of expected high concentrations, spike at an appropriate level.) If the type of samples analyzed are varied, a synthetically prepared sample may be used if the above criteria and intent are met.

7.6.3 The *quality control sample* should be prepared in the same acid matrix as the calibration standards at a concentration near 1 mg/L and in accordance with the instructions provided by the supplier. The Quality Assurance Branch of EMSL-Cincinnati will either supply a quality control sample or information where one of equal quality can be procured. (See 12.1.3.)

8. Sample Handling and Preservation

8.1 For the determination of trace elements, contamination and loss are of prime concern. Dust in the laboratory environment, impurities in reagents and impurities on laboratory apparatus which the sample contacts are all sources of potential contamination. Sample containers can introduce either positive or negative errors in the measurement of trace elements by (a) contributing contaminants through leaching or surface desorption and (b) by depleting concentrations through adsorption. Thus the collection and treatment of the sample prior to analysis requires particular attention. Laboratory glassware including the sample bottle (whether polyethylene, polyproplyene or FEP-fluorocarbon) should be thoroughly washed with detergent and tap water; rinsed with (1+1) nitric acid, tap water, (1+1) hydrochloric acid, tap and finally deionized, distilled water in that order (See Notes 2 and 3).

NOTE: 2. Chromic acid may be useful to remove organic deposits from glassware; however, the analyst should be cautioned that the glassware must be thoroughly rinsed with water to remove the last traces of chromium. This is especially important if chromium is to be included in the analytical scheme. A commercial product, NOCHROMIX, available from Godax Laboratories, 6 Varick St., New York, NY 10013, may be used in place of chromic acid. Chromic acid should not be used with plastic bottles.

NOTE: 3. If it can be documented through an active analytical quality control program using spiked samples and reagent blanks, that certain steps in the cleaning procedure

are not required for routine samples, those steps may be eliminated from the procedure.

8.2 Before collection of the sample a decision must be made as to the type of data desired, that is dissolved, suspended or total, so that the appropriate preservation and pretreatment steps may be accomplished. Filtration, acid preservation, etc., are to be performed at the time the sample is collected or as soon as possible thereafter.

8.2.1 For the determination of dissolved elements the sample must be filtered through a 0.45-µm membrane filter as soon as practical after collection. (Glass or plastic filtering apparatus are recommended to avoid possible contamination.) Use the first 50–100 mL to rinse the filter flask. Discard this portion and collect the required volume of filtrate. Acidify the filtrate with (1+1) HNO_3 to a pH of 2 or less. Normally, 3 mL of (1+1) acid per liter should be sufficient to preserve the sample.

8.2.2 For the determination of suspended elements a measured volume of unpreserved sample must be filtered through a 0.45-µm membrane filter as soon as practical after collection. The filter plus suspended material should be transferred to a suitable container for storage and/or shipment. No preservative is required.

8.2.3 For the determination of total or total recoverable elements, the sample is acidified with (1+1) HNO_3 to pH 2 or less as soon as possible, preferably at the time of collection. The sample is not filtered before processing.

9. Sample Preparation

9.1 For the determinations of dissolved elements, the filtered, preserved sample may often be analyzed as received. The acid matrix and concentration of the samples and calibration standards must be the same. (See Note 6.) If a precipitate formed upon acidification of the sample or during transit or storage, it must be redissolved before the analysis by adding additional acid and/or by heat as described in 9.3.

9.2 For the determination of suspended elements, transfer the membrane filter containing the insoluble material to a 150-mL Griffin beaker and add 4 mL conc. HNO_3. Cover the beaker with a watch glass and heat gently. The warm acid will soon dissolve the membrane. Increase the temperature of the hot plate and digest the material. When the acid has nearly evaporated, cool the beaker and watch glass and add another 3 mL of conc. HNO_3. Cover and continue heating until the digestion is complete, generally indicated by a light colored digestate. Evaporate to near dryness (2 mL), cool, and 10 mL HCl (1+1) and 15 mL deionized, distilled water per 100 mL dilution and warm the beaker gently for 15 min. to dissolve any precipitated or residue material. Allow to cool, wash down the watch glass and beaker walls with deionized distilled water and filter the sample to remove insoluble material that could clog the nebulizer. (See Note 4.) Adjust the volume based on the expected concentrations of elements present. This volume will vary depending on the elements to be determined (See Note 6). The sample is now ready for analysis. Concentrations so determined shall be reported as ''suspended.''

NOTE: 4. In place of filtering, the sample after diluting and mixing may be centrifuged or allowed to settle by gravity overnight to remove insoluble material.

9.3 For the determination of total elements, choose a measured volume of the well mixed acid preserved sample appropriate for the expected level of elements and transfer to a Griffin beaker. (See Note 5.) Add 3 mL of conc. HNO_3. Place the beaker on a hot plate and evaporate to near dryness cautiously, making certain that the sample does not boil and that no area of the bottom of the beaker is allowed to go dry. Cool the beaker and add another 5 mL portion of conc. HNO_3. Cover the beaker with a watch glass and return to the hot plate. Increase the temperature of the hot plate so that a gently reflux action occurs. Continue heating, adding additional acid as necessary, until the digestion is complete (generally indicated when the digestate is light in color or does not change in appearance with continued refluxing.) Again, evaporate to near dryness and cool the beaker. Add 10 mL of 1+1 HCl and 15 mL of deionized, distilled water per 100 mL of final solution and warm the beaker gently for 15 min. to dissolve any precipitate or residue resulting from evaporation. Allow to cool, wash down the beaker walls and watch glass with deionized distilled water and filter the sample to remove insoluble material that could clog the nebulizer. (See Note 4.) Adjust the sample to a predetermined volume based on the expected concentrations of elements present. The sample is now ready for analysis (See Note 6). Concentrations so determined shall be reported as ''total.''

NOTE: 5. If low determinations of boron are critical, quartz glassware should be used.

NOTE: 6. If the sample analysis solution has a different acid concentration from that given in 9.4, but does not introduce a physical interference or affect the analytical result, the same calibration standards may be used.

9.4 For the determination of total recoverable elements, choose a measured volume of a well mixed, acid preserved sample appropriate for the expected level of elements and transfer to a Griffin beaker. (See Note 5.) Add 2 mL of (1+1) HNO_3 and 10 mL of (1+1) HCl to the sample and heat on a steam bath

or hot plate until the volume has been reduced to near 25 mL making certain the sample does not boil. After this treatment, cool the sample and filter to remove insoluble material that could clog the nebulizer. (See Note 4.) Adjust the volume to 100 mL and mix. The sample is now ready for analysis. Concentrations so determined shall be reported as ''total.''

10. Procedure

10.1 Set up instrument with proper operating parameters established in Section 6.2. The instrument must be allowed to become thermally stable before beginning. This usually requires at least 30 min. of operation prior to calibration.

10.2 Initiate appropriate operating configuration of computer.

10.3 Profile and calibrate instrument according to instrument manufacturer's recommended procedures, using the typical mixed calibration standard solutions described in Section 7.4. Flush the system with the calibration blank (7.5.1) between each standard. (See Note 7.) (The use of the average intensity of multiple exposures for both standardization and sample analysis has been found to reduce random error.)

NOTE: 7. For boron concentrations greater than 500 µg/L extended flush times of 1 to 2 minutes may be required.

10.4 Before beginning the sample run, reanalyze the highest mixed calibration standard as if it were a sample. Concentration values obtained should not deviate from the actual values by more than ±5 percent (or the established control limits whichever is lower). If they do, follow the recommendations of the instrument manufacturer to correct for this condition.

10.5 Begin the sample run flushing the system with the calibration blank solution (7.5.1) between each sample. (See Note 7.) Analyze the instrument check standard (7.6.1) and the calibration blank (7.5.1) each 10 samples.

10.6 If it has been found that methods of standard addition are required, the following procedure is recommended.

10.6.1 The standard addition technique (14.2) involves preparing new standards in the sample matrix by adding known amounts of standard to one or more aliquots of the processed sample solution. This technique compensates for a sample constitutent that enhances or depresses the analyte signal thus producing a different slope from that of the calibration standards. It will not correct for additive interference which causes a baseline shift. The simplest version of this technique is the single-addition method. The procedure is as follows. Two identical aliquots of the sample solution, each of volume V_x, are taken. To the first (labeled A) is added a small volume V_s of a standard analyte solution of concentration c_s. To the second (labeled B) is added the same volume V_s of the solvent. The analytical signals of A and B are measured and corrected for nonanalyte signals. The unknown sample concentration c_x is calculated:

$$c_x = \frac{S_B V_s c_s}{(S_A - S_B)\ V_x}$$

where S_A and S_B are the analytical signals (corrected for the blank) of solutions A and B, respectively. V_s and c_s should be chosen so that S_A is roughly twice S_B on the average. It is best if V_s is made much less than V_x, and thus c_s is much greater than c_x, to avoid excess dilution of the sample matrix. If a separation or concentration step is used, the additions are best made first and carried through the entire procedure. For the results from this technique to be valid, the following limitations must be taken into consideration:

1. The analytical curve must be linear.
2. The chemical form of the analyte added must respond the same as the analyte in the sample.
3. The interference effect must be constant over the working range of concern.
4. The signal must be corrected for any additive interference.

11. Calculation

11.1 Reagent blanks (7.5.2) should be subtracted from all samples. This is particularly important for digested samples requiring large quantities of acids to complete the digestion.

11.2 If dilutions were performed, the appropriate factor must be applied to sample values.

11.3 Data should be rounded to the thousandth place and all results should be reported in mg/L up to three significant figures.

12. Quality Control (Instrumental)

12.1 Check the instrument standardization by analyzing appropriate quality control check standards as follow:

12.1.1 Analyze and appropriate instrument check standard (7.6.1) containing the elements of interest at a frequency of 10%. This check standard is used to determine instrument drift. If agreement is not within ±5% of the expected values or within the established control limits, whichever is lower, the analysis is out of control. The analysis should be terminated, the problem corrected, and the instrument recalibrated.

Analyze the calibration blank (7.5.1) at a frequency of 10%. The result should be within the established control limits of 2 standard deviations of the meal value. If not, repeat the analysis two more times and average the three results. If the average is not wihin the control limit, terminate the analysis, correct the problem and recalibrate the instrument.

12.1.2 To verify interelement and background correction factors analyze the interference check sample (7.6.2) at the beginning, end, and at periodic intervals throughout the sample run. Results should fall within the established control limits of 1.5 times the standard deviation of the mean value. If not, terminate the analysis, correct the problem and recalibrate the instrument.

12.1.3 A quality control sample (7.6.3) obtained from an outside source must first be used for the initial verification of the calibration standards. A fresh dilution of this sample shall be analyzed every week thereafter to monitor their stability. If the results are not within ±5% of the true value listed for the control sample, prepare a new calibration standard and recalibrate the instrument. If this does not correct the problem, prepare a new stock standard and a new calibration standard and repeat the calibration.

13. Precision and Accuracy

13.1 An interlaboratory study of metal analyses by this method was conducted by the Quality Assurance Branch (QAB) of the Environmental Monitoring Systems Laboratory—Cincinnati (EMSL–CI). Synthetic concentrates containing various levels of the twenty-five elements listed in Table 4 were added to reagent water, surface water, drinking water and three effluents. These samples were digested by both the total digestion procedure (9.3) and the total recoverable procedure (9.4). Results for both digestions for the twenty-five elements in reagent water are given in Table 4; results for the other matrices can be found in Reference 14.10.

14. References

14.1 Winge, R.K., V.J. Peterson, and V.A. Fassel, ''Inductively Coupled Plasma-Atomic Emission Spectroscopy: Prominent Lines, EPA–600/4–79–017.

14.2 Winefordner, J.D., ''Trace Analysis: Spectroscopic Methods for Elements,'' *Chemical Analysis,* Vol, *46,* pp. 41–42.

14.3 Handbook for Analytical Quality Control in Water and Wastewater Laboratories, EPA–600/4–79–019.

14.4 Garbarino, J.R. and Taylor, H.E., ''An Inductively-Coupled Plasma Atomic Emission Spectrometric Method for Routine Water Quality Testing,'' Applied Spectroscopy *33,* No. 3 (1979).

14.5 ''Methods for Chemical Analysis of Water and Wastes,'' EPA–600/4–79–020.

14.6 Annual Book of ASTM Standards, Part 31.

14.7 ''Carcinogens—Working With Carcinogens,'' Department of Health, Education, and Welfare, Public Health Service, Center for Disease Control, National Institute for Occupational Safety and Health, Publication No. 77–206, August 1977.

14.8 ''OSHA Safety and Health Standards, General Industry,'' (29 CFR Part 1910), Occupational Safety and Health Administration, OSHA 2206, (Revised, January 1976).

14.9 ''Safety in Academic Chemistry Laboratories, American Chemical Society Publication, Committee on Chemical Safety, 3rd Edition, 1979.

14.10 Maxfield R. and Minak B., ''EPA Method Study 27, Method 200.7 Trace Metals by ICP,'' National Technical Information Service, Order No. PB 85–248–656, November 1983.

TABLE 1—RECOMMENDED WAVELENGTHS[1] AND ESTIMATED INSTRUMENTAL DETECTION LIMITS

Element	Wavelength, nm	Estimated detection limit, µg/L[2]
Aluminum	308.215	45
Arsenic	193.696	53
Antimony	206.833	32
Barium	455.403	2
Beryllium	313.042	0.3
Boron	249.773	5
Cadmium	226.502	4
Calcium	317.933	10
Chromium	267.716	7
Cobalt	228.616	7
Copper	324.754	6
Iron	259.940	7
Lead	220.353	42
Magnesium	279.079	30
Manganese	257.610	2
Molybdenum	202.030	8
Nickel	231.604	15
Potassium	766.491	[3]
Selenium	196.026	75
Silica (SiO_2)	288.158	58
Silver	328.068	7
Sodium	588.995	29
Thallium	190.864	40
Vanadium	292.402	8
Zinc	213.856	2

[1]The wavelengths listed are recommended because of their sensitivity and overall acceptance. Other wavelengths may be substituted if they can provide the needed sensitivity and are treated with the same corrective techniques for spectral interference. (See 5.1.1).

[2]The estimated instrumental detection limits as shown are taken from ''Inductively Coupled Plasma-Atomic Emission Spectroscopy-Prominent Lines,'' EPA–600/4–79–017. They are given as a guide for an instrumental limit. The actual method detection limits are sample dependent and may vary as the sample matrix varies.

[3]Highly dependent on operating conditions and plasma position.

TABLE 1—ANALYTE CONCENTRATION EQUIVALENTS (MG/L) ARISING FROM INTERFERENTS AT THE 100 MG/L LEVEL

Analyte	Wave-length, nm	Interferent—									
		A1	Ca	Cr	Cu	Fe	Mg	Mn	Ni	Ti	V
Aluminum	308.214	.....	.....	.....	.....	.....	.....	0.21	.....	.....	1.4
Antimony	206.833	0.47	.....	2.9	.....	0.08	.....	.....	.....	0.25	0.45
Arsenic	193.696	1.3	.....	0.44	.....	.....	.....	.....	.....	.....	1.1
Barium	455.403	.....	.....	.....	.....	.....	.....	.....	.....	.....	.....
Beryllium	313.042	.....	.....	.....	.....	.....	.....	.....	.....	0.04	0.05
Boron	249.773	0.04	.....	.....	.....	0.32	.....	.....	.....	.....	.....
Cadmium	226.502	.....	.....	.....	.....	0.03	.....	.....	0.02	.....	.....
Calcium	317.933	.....	.....	0.08	.....	0.01	0.01	0.04	.....	0.03	0.03
Chromium	267.716	.....	.....	.....	.....	0.003	.....	0.04	.....	.....	0.04
Cobalt	228.616	.....	.....	0.03	.....	0.005	.....	.....	0.03	0.15	.....
Copper	324.754	.....	.....	.....	.....	0.003	.....	.....	.....	0.05	0.02
Iron	259.940	.....	.....	.....	.....	.....	.....	0.12	.....	.....	.....
Lead	220.353	0.17	.....	.....	.....	.....	.....	.....	.....	.....	.....
Magnesium	279.079	.....	0.02	0.11	.....	0.13	.....	0.25	.....	0.07	0.12
Manganese	257.610	0.005	.....	0.01	.....	0.002	0.002	.....	.....	.....	.....
Molybdenum	202.030	0.05	.....	.....	.....	0.03	.....	.....	.....	.....	.....
Nickel	231.604	.....	.....	.....	.....	.....	.....	.....	.....	.....	.....
Selenium	196.026	0.23	.....	.....	.....	0.09	.....	.....	.....	.....	.....
Silicon	288.158	.....	.....	0.07	.....	.....	.....	.....	.....	.....	0.01
Sodium	588.995	.....	.....	.....	.....	.....	.....	.....	.....	0.08	.....
Thallium	190.864	0.30	.....	.....	.....	.....	.....	.....	.....	.....	.....
Vanadium	292.402	.....	.....	0.05	.....	0.005	.....	.....	.....	0.02	.....
Zinc	213.856	.....	.....	.....	0.14	.....	.....	.....	0.29	.....	.....

TABLE 3—INTERFERENT AND ANALYTE ELEMENTAL CONCENTRATIONS USED FOR INTERFERENCE MEASUREMELTS IN TABLE 2

	Analytes	(mg/L)		Interferents	(mg/L)	
	Al	10		Al	1,000	
	AS	10		Ca	1,000	
	B	10		Cr	200	
	Ba	1		Cu	200	
	Be	1		Fe	1,000	
	Ca	1		Mg	1,000	
	Cd	10		Mn	200	
	Co	1		Ni	200	
	Cr	1		Ti	200	
	Cu	1		V	200	
	Fe	1				
	Mg	1				
	Mn	1				
	Mo	10				
	Na	10				
	Ni	10				
	Pb	10				
	Sb	10				
	Se	10				
	Si	1				
	Tl	10				
	V	1				
	Zn	10				

TABLE 4—ICP PRECISION AND RECOVERY DATA

Analyte	Concentration µg/L	Total digestion (9.3) µg/L	Recoverable digestion (9.4) µg/L
Aluminum	69–4792	X=0.9273(C)+3.6 S=0.0559(X)+18.6 SR=0.0507(X)+3.5	X=0.9380(C)+22.1 S=0.0873(X)+31.7 SR=0.0481(X)+18.8
Antimony	77–1406	X=0.7940(C) − 17.0 S=0.1556(X) − 0.6 SR=0.1081(X)+3.9	X=0.8908(C)+0.9 S=0.0982(X)+8.3 SR=0.0682(X)+2.5
Arsenic	69–1887	X=1.0437(C) − 12.2 S=0.1239(X)+2.4 SR=0.0874(X)+6.4	X=1.0175(C)+3.9 S=0.1288(X)+6.1 SR=0.0643(X)+10.3
Barium	9–377	X=0.7683(C)+0.47 S=0.1819(X)+2.78 SR=0.1285(X)+2.55	X=0.8380(C)+1.68 S=0.2540(X)+0.30 SR=0.0826(X)+3.54
Beryllium	3–1906	X=0.9629(C)+0.05 S=0.0136(X)+0.95 SR=0.0203(X) − 0.07	X=1.0177(C) − 0.55 S=0.0359(X)+0.90 SR=0.0445(X) − 0.10
Boron	19–5189	X=0.8807(C)+9.0 S=0.1150(X)+14.1 SR=0.0742(X)+23.2	X=0.9676(C)+18.7 S=0.1320(X)+16.0 SR=0.0743(X)+21.1
Cadmium	9–1943	X=0.9874(C) − 0.18 S=0.557(X)+2.02 SR=0.0300(X)+0.94	X=1.0137(C) − 0.65 S=0.0585(X)+1.15 SR=0.332(X)+0.90
Calcium	17–47170	X=0.9182(C) − 2.6 S=0.1228(X)+10.1 SR=0.0189(X)+3.7	X=0.9658(C)+0.8 S=0.0917(X)+6.9 SR=0.0327(X)+10.1
Chromium	13–1406	X=0.9544(C)+3.1 S=0.0499(X)+4.4 SR=0.0009(X)+7.9	X=1.0049(C) − 1.2 S=0.0698(X)+2.8 SR=0.0571(X)+1.0
Cobalt	17–2340	X=0.9209(C) − 4.5 S=0.0436(X)+3.8 SR=0.0428(X)+0.5	X=0.9278(C) − 1.5 S=0.0498(X)+2.6 SR=0.0407(X)+0.4
Copper	8–1887	X=0.9297(C) − 0.30 S=0.0442(X)+2.85 SR=0.0128(X)+2.53	X=0.9647(C) − 3.64 S=0.0497(X)+2.28 SR=0.0406(X)+0.96
Iron	13–9359	X=0.8829(C)+7.0 S=0.0683(X)+11.5 SR=0.0046(X)+10.0	X=0.9830(C)+5.7 S=0.1024(X)+13.0 SR=0.0790(X)+11.5
Lead	42–4717	X=0.9699(C) − 2.2 S=0.0558(X)+7.0 SR=0.0353(X)+3.6	X=1.0056(C)+4.1 S=0.0779(X)+4.6 SR=0.0448(X)+3.5
Magnesium	34–13868	X=0.9881(C) − 1.1 S=0.0607(C)+11.6	X=0.9879(C)+2.2 S=0.0564(X)+13.2

TABLE 4—ICP PRECISION AND RECOVERY DATA—Continued

Analyte	Concentration µg/L	Total digestion (9.3) µg/L	Recoverable digestion (9.4) µg/L
Manganese	4–1887	SR=0.0298(X)+0.6 X=0.9417(C)+0.13 S=0.0324(X)+0.88	SR=0.0268(X)+8.1 X=0.9725(C)+0.07 S=0.0557(X)+0.76
Molybdenum	17–1830	SR=0.0153(X)+0.91 X=0.9682(C)+0.1 S=0.0618(X)+1.6	SR=0.0400(X)+0.82 X=0.9707(C)−2.3 S=0.0811(X)+3.8
Nickel	17–47170	SR=0.0371(X)+2.2 X=0.9508(C)+0.4 S=0.0604(X)+4.4	SR=0.0529(X)+2.1 X=0.9869(C)+1.5 S=0.0526(X)+5.5
Potassium	347–14151	SR=0.0425(X)+3.6 X=0.8669(C)−36.4 S=0.0934(X)+77.8	SR=0.0393(X)+2.2 X=0.9355(C)−183.1 S=0.0481(X)+177.2
Selenium	69–1415	SR=0.0099(X)+144.2 X=0.9363(C)−2.5 S=0.0855(X)+17.8	SR=0.0329(X)+60.9 X=0.9737(C)−1.0 S=0.1523(X)+7.8
Silicon	189–9434	SR=0.0284(X)+9.3 X=0.5742(C)−35.6 S=0.4160(X)+37.8	SR=0.0443(X)+6.6 X=0.9737(C)−60.8 S=0.3288(X)+46.0
Silver	8–189	SR=0.1987(X)+8.4 X=0.4466(C)+5.07 S=0.5055(X)−3.05	SR=0.2133(X)+22.6 X=0.3987(C)+8.25 S=0.5478(X)−3.93
Sodium	35–47170	SR=0.2086(X)−1.74 X=0.9581(C)+39.6 S=0.2097(X)+33.0	SR=0.1836(X)−0.27 X=1.0526(C)+26.7 S=0.1473(X)+27.4
Thallium	79–1434	SR=0.0280(X)+105.8 X=0.9020(C)−7.3 S=0.1004(X)+18.3	SR=0.0884(X)+50.5 X=0.9238(C)+5.5 S=0.2156(X)+5.7
Vanadium	13–4698	SR=0.0364(X)+11.5 X=0.9615(C)−2.0 S=0.0618(X)+1.7	SR=0.0106(X)+48.0 X=0.9551(C)+0.4 S=0.0927(X)+1.6
Zinc	7–7076	SR=0.0220(X)+0.7 X=0.9356(C)−0.30 S=0.0914(X)+3.75 SR=0.0130(X)+10.7	SR=0.0472(X)+0.5 X=0.9500(C)+1.82 S=0.0597(X)+6.50 SR=0.0153(X)+7.78

AAAAAX=Mean Recovery, µg/L
AAAAAC=True Value for the Concentration, µg/L
AAAAAS=Multi-laboratory Standard Deviation, µg/L
SR=Single-analyst Standard Deviation, µg/L

[49 FR 43431, Oct. 26, 1984; 50 FR 695, 696, Jan. 4, 1985, as amended at 51 FR 23703, June 30, 1986; 55 FR 33440, Aug. 15, 1990]

APPENDIX D TO PART 136—PRECISION AND RECOVERY STATEMENTS FOR METHODS FOR MEASURING METALS

Twenty-eight selected methods from ''Methods for Chemical Analysis of Water and Wastes,'' EPA–600/4–79–020 (1979) have been subjected to interlaboratory method validation studies. The following precision and recovery statements are presented in this appendix and incorporated into part 136:

Method 202.1

For Aluminum, Method 202.1 (Atomic Absorption, Direct Aspiration) replace the Precision and Accuracy Section with the following:

Precision and Accuracy

An interlaboratory study on metal analyses by this method was conducted by the Quality Assurance Branch (QAB) of the Environmental Monitoring Systems Laboratory—Cincinnati (EMSL–CI). Synthetic concentrates containing various levels of this element were added to reagent water and a natural water or effluent of the analyst's choice. The digestion procedure was not specified. Results for the reagent water are given below. Results for other water types and study details are found in ''USEPA Method Study 7, Analyses for Trace Methods in water by Atomic Absorption Spectroscopy (Direction Aspiration) and Colorimetry'', National Technical Information Service, 5285 Port Royal Road, Springfield, VA 22161, Order No. PB86–208709/AS, Winter, J.A. and Britton, P.W., June, 1986.

For a concentration range of 500–1200 µg/L

X=0.979(C)+6.16

S=0.066(X)+125
SR=0.086(X)+40.5

Where:

C=True Value for the Concentration, µg/L
X=Mean Recovery, µg/L
S=Multi-laboratory Standard Deviation, µg/L
SR=Single-analyst Standard Deviation, µg/L

Method 206.4

For Arsenic, Method 206.4 (Spectrophotometric-SDDC) add the following to the Precision and Accuracy Section:

Precision and Accuracy

An interlaboratory study on metal analyses by this method was conducted by the Quality Assurance Branch (QAB) of the Environmental Monitoring Systems Laboratory—Cincinnati (EMSL–CI). Synthetic concentrates containing various levels of this element were added to reagent water and a natural water or effluent of the analyst's choice. Results for the reagent water are given below. Results for other water types and study details are found in ''USEPA Method Study 7, Analyses for Trace Methods in Water by Atomic Absorption Spectroscopy (Direct Aspiration) and Colorimetry'', National Technical Information Service, 5285 Port Royal Road, Springfield, VA 22161, Order No. PB86–208709/AS, Winter, J.A. and Britton, P.W., June, 1986.

For a concentration range of 20–292 µg/L

X=0.850(C)−0.25
S=0.198(X)+5.93
SR=0.122(X)+3.10

Where:

C=True Value for the Concentration, µg/L
X=Mean Recovery, µg/L
S=Multi-laboratory Standard Deviation, µg/L
SR=Single-analyst Standard Deviation, µg/L

Method 213.1

For Cadmium, Method 213.1 (Atomic Absorption, Direct Aspiration) replace the Precision and Accuracy Section with the following:

Precision and Accuracy

An interlaboratory study on metal analyses by this method was conducted by the Quality Assurance Branch (QAB) of the Environmental Monitoring Systems Laboratory—Cincinnati (EMSL–CI). Synthetic concentrates containing various levels of this element were added to reagent water and a natural water or effluent of the analyst's choice. The digestion procedure was not specified. Results for the reagent water are given below. Results for other water types and study details are found in ''USEPA Method Study 7, Analyses for Trace Methods in Water by Atomic Absorption Spectroscopy (Direct Aspiration) and Colorimetry'', National Technical Information Service, 5285 Port Royal Road, Springfield, VA 22161, Order No. PB86–208709/AS, Winter, J.A. and Britton, P.W., June, 1986.

For a concentration range of 14–78 µg/L

X=0.919(C)+2.97
S=0.108(X)+5.08
SR=0.120(X)+0.89

Where:

C=True Value for the Concentration, µg/L
X=Mean Recovery, µg/L
S=Multi-laboratory Standard Deviation, µg/L
SR=Single-analyst Standard Deviation, µg/L

Method 218.1

For Chromium, Method 218.1 (Atomic Absorption, Direct Aspiration) replace the Precision and Accuracy Section with the following:

Precision and Accuracy

An interlaboratory study on metal analyses by this method was conducted by the Quality Assurance Branch (QAB) of the Environmental Monitoring Systems Laboratory—Cincinnati (EMSL–CI). Synthetic concentrates containing various levels of this element were added to reagent water and a natural water or effluent of the analyst's choice. The digestion procedure was not specified. Results for the reagent water are given below. Results for other water types and study details are found in ''USEPA Method Study 7, Analyses for Trace Methods in Water by Atomic Absorption Spectroscopy (Direct Aspiration) and Colorimetry'', National Technical Information Service, 5285 Port Royal Road, Springfield, VA 22161, Order No. PB86–208709/AS, Winter, J.A. and Britton, P.W., June 1986.

For a concentration range of 74–407 µg/L

X=0.976(C)+3.94
S=0.131(X)+4.26
SR=0.052(X)+3.01

Where:

C=True Value for the Concentration, µg/L
X=Mean Recovery, µg/L
S=Multi-laboratory Standard Deviation, µg/L
SR=Single-analyst Standard Deviation, µg/L

Method 220.1

For Copper, Method 220.1 (Atomic Absorption, Direct Aspiration) replace the Precision and Accuracy Section with the following:

Precision and Accuracy

An interlaboratory study on metal analyses by this method was conducted by the

Quality Assurance Branch (QAB) of the Environmental Monitoring Systems Laboratory—Cincinnati (EMSL–CI). Synthetic concentrates containing various levels of this element were added to reagent water and a natural water or effluent of the analyst's choice. The digestion procedure was not specified. Results for the reagent water are given below. Results for other water types and study details are found in ''USEPA Method Study 7, Analyses for Trace Methods in Water by Atomic Absorption Spectroscopy (Direct Aspiration) and Colorimetry'', National Technical Information Service, 5285 Port Royal Road, Springfield, VA 22161, Order No. PB86–208709/AS, Winter, J.A. and Britton, P.W., June, 1986.

For concentration range 60–332 µg/L

X=0.963(C)+3.49

S=0.047(X)+12.3

SR=0.042(X)+4.60

Where:

C=True Value for the Concentration, µg/L

X=Mean Recovery, µg/L

S=Multi-laboratory Standard Deviation, µg/L

SR=Single-analyst Standard Deviation, µg/L

Method 236.1

For Iron, Method 236.1 (Atomic Absorption, Direct Aspiration) replace the Precision and Accuracy Section with the following:

Precision and Accuracy

An interlaboratory study on metal analyses by this method was conducted by the Quality Assurance Branch (QAB) of the Environmental Monitoring Systems Laboratory—Cincinnati (EMSL–CI). Synthetic concentrates containing various levels of this element were added to reagent water and a natural water or effluent of the analyst's choice. The digestion procedure was not specified. Results for the reagent water are given below. Results for other water types and study details are found in ''USEPA Method Study 7, Analyses for Trade Methods in Water by Atomic Absorption Spectroscopy (Direct Aspiration) and Colorimetry'', National Technical Information Service, 5285 Port Royal Road, Springfield, VA 22161, Order No. PB86–208709/AS, Winter, J.A. and Britton, P.W., June, 1986.

For concentration range 350–840 µg/L

X=0.999(C)−2.21

S=0.022(X)+41.0

SR=0.019(X)+21.2

Where:

C=True Value for the Concentration, µg/L

X=Mean Recovery, µg/L

S=Multi-Laboratory Standard Deviation, µg/L

SR=Single-analyst Standard Deviation, µg/L

Method 239.1

For Lead, Method 239.1 (Atomic Absorption, Direct Aspiration) replace Precision and Accuracy Section with the following:

Precision and Accuracy

An interlaboratory study on metal analyses by this method was conducted by the Quality Assurance Branch (QAB) of the Environmental Monitoring Systems Laboratory—Cincinnati (EMSL–CI). Synthetic concentrates containing various levels of this element were added to reagent water and a natural water or effluent of the analyst's choice. The digestion procedure was not specified. Results for the reagent water are given below. Results for other water types and study details are found in ''USEPA Method Study 7 Analyses for Trace Methods in Water by Atomic Absorption Spectroscopy (Direct Aspiration) and Colorimetry''; National Technical Information Service, 5285 Port Royal Road, Springfield, VA 22161, Order No. PB86–208709/AS, Winter, J.A. and Britton, P.W., June, 1986.

For concentration range of 84–367 µg/L

X=0.961(C)+13.8

S=0.028(C)+33.9

SR=0.011(X)+16.1

Where:

C=True Value for the Concentration, µg/L

X=Mean Recovery, µg/L

S=Multi-laboratory Standard Deviation, µg/L

SR=Single-analyst Standard Deviation, µg/L

Method 243.1

For Manganese, Method 243.1 (Atomic Absorption, Direct Aspiration) replace Precision and Accuracy Section with the following:

Precision and Accuracy

An interlaboratory study on metal analyses by this method was conducted by the Quality Assurance Branch (QAB) of the Environmental Monitoring Systems Laboratory—Cincinnati (EMSL–CI). Synthetic concentrates containing various levels of this element were added to reagent water and a natural water or effluent of the analyst's choice. The digestion procedure was not specified. Results for the reagent water are given below. Results for other water types and study details are found in ''USEPA Method Study 7, Analyses for Trace Methods in Water by Atomic Absorption Spectroscopy (Direct Aspiration) and Colorimetry'', National Technical Information Service, 5285 Port Royal Road, Springfield, VA 22161, Order No. PB86–208709/AS, Winter, J.A. and Britton, P.W., June, 1986.

For concentration range 84–469 µg/L

X=0.987(C)−1.27

S=0.042(X)+8.95
SR=0.023(X)+4.90

Where:

C=True Value for the Concentration, µg/L
X=Mean Recovery, µg/L
S=Multi-laboratory Standard Deviation, µg/L
SR=Single-analyst Standard Deviation, µg/L

Method 289.1

For Zinc, Method 289.1 (Atomic Absorption, Direct Aspiration) replace the Precision and Accuracy Section with the following:

Precision and Accuracy

An interlaboratory study on metal analyses by this method was conducted by the Quality Assurance Branch (QAB) of the Environmental Monitoring Systems Laboratory-Cincinnati (EMSL–CI). Synthetic concentrates containing various levels of this element were added to reagent water and a natural water or effluent of the analyst's choice. The digestion procedure was not specified. Results for the reagent water are given below. Results for other water types and study details are found in ''USEPA Method Study 7, Analyses for Trace Methods in Water by Atomic Absorption Spectroscopy (Direct Aspiration) and Colorimetry'', National Technical Information Service, 5285 Port Royal Road, Springfield, VA 22161, Order No. PB86–208709/AS, Winter, J. A. and Britton, P. W., June, 1986.

For concentration range 56–310 µg/L
X=0.999(C)+0.033
S=0.078(X)+10.8
SR=0.049(X)+1.10

Where:

C=True Value for the Concentration, µg/L
X=Mean Recovery, µg/L
S=Multi-laboratory Standard Deviation, µg/L
SR=Single-analyst Standard Deviation, µg/L

Method 202.2

For Aluminum, Method 202.2 (Atomic Absorption, Furnace Technique) replace the Precision and Accuracy Section statement with the following:

Precision and Accuracy

An interlaboratory study on metal analyses by this method was conducted by the Quality Assurance Branch (QAB) of the Environmental Monitoring Systems Laboratory-Cincinnati (EMSL–CI). Synthetic concentrates containing various levels of this element were added to reagent water, surface water, drinking water and three effluents. These samples were digested by the total digestion procedure, 4.1.3 in this manual. Results for the reagent water are given below. Results for other water types and study details are found in ''EPA Method Study 31, Trace Metals by Atomic Absorption (Furnace Techniques), ''National Technical Information Service, 5285 Port Royal Road, Springfield, VA 22161, Order No. PB 86–121 704/AS, by Copeland, F.R. and Maney, J.P., January 1986.

For a concentration range of 0.46 − 125 µg/L
X=1.1579(C) − 0.121
S=0.4286(X) − 0.124
SR=0.2908(X) − 0.082

Where:

C=True Value for the Concentration, µg/L
X=Mean Recovery, µg/L
S=Multi-laboratory Standard Deviation, µg/L
SR=Single-analyst Standard Deviation, µg/L

Method 204.2

For Antimony, Method 204.2 (Atomic Absorption, Furnace Technique) replace the Precision and Accuracy Section statement with the following:

Precision and Accuracy

An interlaboratory study on metal analyses by this method was conducted by the Quality Assurance Branch (QAB) of the Environmental Monitoring Systems Laboratory-Cincinnati (EMSL–CI). Synthetic concentrates containing various levels of this element were added to reagent water, surface water, drinking water and three effluents. These samples were digested by the total digestion procedure, 4.1.3 in this manual as modified by this method. Results for the reagent water are given below. Results for other water types and study details are found in ''EPA Method Study 31, Trace Metals by Atomic Absorption (Furnace Techniques),'' National Technical Information Service, 5285 Port Royal Road, Springfield, VA 22161, Order No. PB 86–121 704/AS, by Copeland, F.R. and Maney, J.P., January 1986.

For a concentration range of 10.50 − 240 µg/L
X=0.7219(C) − 0.986
S=0.3732(X)+0.854
SR=0.1874(X) − 0.461

Where:

C=True Value for the Concentration, µg/L
X=Mean Recovery, µg/L
S=Multi-laboratory Standard Deviation, µg/L
SR=Single-analyst Standard Deviation, µg/L

Method 206.2

For Arsenic, Method 206.2 (Atomic Absorption, Furnace Technique) add the following to the existing Precision and Accuracy statement:

Precision and Accuracy

An interlaboratory study on metal analyses by this method was conducted by the Quality Assurance Branch (QAB) of the Environmental Monitoring Systems Laboratory-Cincinnati (EMSL–CI). Synthetic concentrates containing various levels of this element were added to reagent water, surface water, drinking water and three effluents. Results for the reagent water are given below. Results for other water types and study details are found in ''EPA Method Study 31, Trace Metals by Atomic Absorption (Furnace Techniques),'' National Technical Information Service, 5285 Port Royal Road, Springfield, VA 22161, Order No. PB 86–121 704/AS, by Copeland, F.R. and Maney, J.P., January 1986.

For a concentration range of 9.78−237 µg/L

X=0.9652(C)+2.112
S=0.1411(X)+1.873
SR=0.0464(X)+2.109

Where:

C=True Value for the Concentration, µg/L
X=Mean Recovery, µg/L
S=Multi-laboratory Standard Deviation, µg/L
SR=Single-analyst Standard Deviation, µg/L

Method 208.2

For Barium, Method 208.2 (Atomic Absorption, Furnace Technique) add the following to the existing Precision and Accuracy information:

Precision and Accuracy

An interlaboratory study on metal analyses by this method was conducted by the Quality Assurance Branch (QAB) of the Environmental Monitoring Systems Laboratory—Cincinnati (EMSL–CI). Synthetic concentrates containing various levels of this element were added to reagent water, surface water, drinking water and three effluents. These samples were digested by the total digestion procedure, 4.1.3 in this manual. Results for the reagent water are given below. Results for other water types and study details are found in ''EPA Method Study 31, Trace Metals by Atomic Absorption (Furnace Techniques),'' National Technical Information Service, 5285 Port Royal Road, Springfield, VA 22161, Order No. PB 86–121 704/AS, by Copeland, F.R. and Maney, J.P., January 1986.

For a concentration range of 56.50–437 µg/L

X=0.8268(C)+59.459
S=0.2466(X)+6.436
SR=0.1393(X)−0.428

Where:

C=True Value for the Concentration, µg/L
X=Mean Recovery, µg/L
S=Multi-laboratory Standard Deviation, µg/L
SR=Single-analyst Standard Deviation, µg/L

Method 210.2

For Beryllium, Method 210.2 (Atomic Absorption, Furnace Technique) replace the existing Precision and Accuracy statement with the following:

Precision and Accuracy

An interlaboratory study on metal analyses by this method was conducted by the Quality Assurance Branch (QAB) of the Environmental Monitoring Systems Laboratory—Cincinnati (EMSL–CI). Synthetic concentrates containing various levels of this element were added to reagent water, surface water, drinking water and three effluents. These samples were digested by the total digestion procedure, 4.1.3 in this manual. Results for the reagent water are given below. Results for other water types and study details are found in ''EPA Method Study 31, Trace Metals by Atomic Absorption (Furnace Techniques),'' National Technical Information Service, 5285 Port Royal Road, Springfield, VA 22161, Order No. PB 86–121 704/AS, by Copeland, F.R. and Maney, J.P., January 1986.

For a concentration range of 0.45–11.4 µg/L

X=1.0682(C)−0.158
S=0.2167(X)+0.090
SR=0.1096(X)+0.061

Where:

C=True Value for the Concentration, µg/L
X=Mean Recovery, µg/L
S=Multi-laboratory Standard Deviation, µg/L
SR=Single-analyst Standard Deviation, µg/L

Method 213.2

For Cadmium, Method 213.2 (Atomic Absorption, Furnace Technique) add the following to the existing Precision and Accuracy information:

Precision and Accuracy

An interlaboratory study on metal analyses by this method was conducted by the Quality Assurance Branch (QAB) of the Environmental Monitoring System Laboratory—Cincinnati (EMSL–CI). Synthetic concentrates containing various levels of this element were added to reagent water, surface water, drinking water and three effluents. These samples were digested by the total digestion procedure, 4.1.3 in this manual. Results for the reagent water are given below. Results for other water types and study details are found in ''EPA Method Study 31,

Trace Metals by Atomic Absorption (Furnace Techniques),'' National Technical Information Service, 5285 Port Royal Road, Springfield, VA 22161, Order No. PB 86–121 704/AS, by Copeland, F.R. and Maney, J.P., January 1986.

For a concentration range of 0.43–12.5 µg/L

X=0.9826(C)+0.171
S=0.2300(X)+0.045
SR=0.1031(X)+0.116

Where:

C=True Value for the Concentration, µg/L
X=Mean Recovery, µg/L
S=Multi-laboratory Standard Deviation, µg/L
SR=Single-analyst Standard Devision, µg/L

Method 218.2

For Chromium, Method 218.2 (Atomic Absorption, Furnace Technique) add the following to the existing Precision and Accuracy Section:

Precision and Accuracy

An interlaboratory study on metal analyses by this method was conducted by the Quality Assurance Branch (QAB) of the Environmental Monitoring Systems Laboratory—Cincinnati (EMSL–CI). Synthetic concentrates containing various levels of this element were added to reagent water, surface water, drinking water and three effluents. These samples were digested by the total digestion procedure, 4.1.3 in this manual. Results for the reagent water are given below. Results for other water types and study details are found in ''EPA Method Study 31, Trace Metals by Atomic Absorption (Furnace Techniques),'' National Technical Information Service, 5285 Port Royal Road, Springfield, VA 22161, Order No. PB 86–121 704/AS, by Copeland, F.R. and Maney, J.P., January 1986.

For a concentration range of 9.87–246 µg/L

X=0.9120(C)+0.234
S=0.1684(X)+0.852
SR=0.1469(X)+0.315

Where:

C=True Value for the Concentration, µg/L
X=Mean Recovery, µg/L
S=Multi-laboratory Standard Deviation, µg/L
SR=Single-analyst Standard Devision, µg/L

Method 219.2

For Cobalt, Method 219.2 (Atomic Absorption, Furnace Technique), replace the Precision and Accuracy Section statement with the following:

Precision and Accuracy

An interlaboratory study on metal analyses by this method was conducted by the Quality Assurance Branch (QAB) of the Environmental Monitoring Systems Laboratory—Cincinnati (EMSL–CI). Synthetic concentrates containing various levels of this element were added to reagent water, surface water, drinking water and three effluents. These samples were digested by the total digestion procedure, 4.1.3 in this manual. Results for the reagent water are given below. Results for other water types and study details are found in ''EPA Method Study 31, Trace Metals by Atomic Absorption (Furnace Techniques),'' National Technical Information Service, 5285 Port Royal Road, Springfield, VA 22161 Order No. PB 86–121 704/AS, by Copeland, F.R. and Maney, J.P., January 1986.

For a concentration range of 21.10−461 µg/L

X=0.8875(C)+0.859
S=0.2481(X)−2.541
SR=0.0969(X)+0.134

Where:

C=True Value for the Concentration, µg/L
X=Mean Recovery, µg/L
S=Multi-laboratory Standard Deviation, µg/L
SR=Single-analyst Standard Deviation, µg/L

Method 220.2

For Copper, Method 220.2 (Atomic Absorption, Furnace Technique) replace the Precision and Accuracy Section statement with the following:

Precision and Accuracy

An interlaboratory study on metal analyses by this method was conducted by the Quality Assurance Branch (QAB) of the Environmental Monitoring Systems Laboratory—Cincinnati (EMSL–CI). Synthetic concentrates containing various levels of this element were added to reagent water, surface water, drinking water and three effluents. These samples were digested by the total digestion procedure, 4.1.3 in this manual. Results for the reagent water are given below. Results for other water types and study details are found in ''EPA Method Study 31, Trace Metals by Atomic Absorption (Furnace Techniques),'' National Technical Information Service, 5285 Port Royal Road, Springfield, VA 22161 Order No. PB 86–121 704/AS, by Copeland, F.R. and Maney, J.P., January 1986.

For a concentration range of 0.30−245 µg/L

X=0.9253(C)+0.010
S=0.2735(X)−0.058
SR=0.2197(X)−0.050

Where:

C=True Value for the Concentration, µg/L
X=Mean Recovery, µg/L
S=Multi-laboratory Standard Deviation, µg/L
SR=Single-analyst Standard Deviation, µg/L

Method 236.2

For Iron, Method 236.2 (Atomic Absorption, Furnace Technique) replace the Precision and Accuracy Section statement with the following:

Precision and Accuracy

An interlaboratory study on metal analyses by this method was conducted by the Quality Assurance Branch (QAB) of the Environmental Monitoring Systems Laboratory—Cincinnati (EMSL–CI). Synthetic concentrates containing various levels of this element were added to reagent water, surface water, drinking water and three effluents. These samples were digested by the total digestion procedure, 4.1.3 in this manual. Results for the reagent water are given below. Results for other water types and study details are found in ''EPA Method Study 31, Trace Metals by Atomic Absorption (Furnace Techniques),'' National Technical Information Service, 5285 Port Royal Road, Springfield, VA 22161 Order No. PB 86–121 704/AS, by Copeland, F.R. and Maney, J.P., January 1986.

For a concentration range of 0.37 − 455 µg/L

$X=1.4494(C)-0.229$
$S=0.3611(X)-0.079$
$SR=0.3715(X)-0.161$

Where:

C=True Value for the Concentration, µg/L
X=Mean Recovery, µg/L
S=Multi-laboratory Standard Deviation, µg/L
SR=Single-analyst Standard Deviation, µg/L

Method 239.2

For Lead, Method 239.2 (Atomic Absorption, Furnace Technique) add the following to the existing Precisions and Accuracy Section:

Precision and Accuracy

An interlaboratory study on metal analyses by this method was conducted by the Quality Assurance Branch (QAB) of the Environmental Monitoring Systems Laboratory—Cincinnati (EMSL–CI). Synthetic concentrates containing various levels of this element were added to reagent water, surface water, drinking water and three effluents. These samples were digested by the total digestion procedure, 4.1.3 in this manual. Results for the reagent water are given below. Results for other water types and study details are found in ''EPA Method Study 31, Trace Metals by Atomic Absorption (Furnace Techniques),'' National Technical Information Service, 5285 Port Royal Road, Springfield, VA 22161 Order No. PB 86–121 704/AS, by Copeland, F.R. and Maney, J.P., January 1986.

For a concentration range of 10.40 − 254 µg/L

$X=0.9430(C)-0.504$
$S=0.2224(X)+0.507$
$SR=0.1931(X)-0.378$

Where:

C=True Value for the Concentration, µg/L
X=Mean Recovery, µg/L
S=Multi-laboratory Standard Deviation, µg/L
SR=Single-analyst Standard Deviation, µg/L

Method 243.2

For Manganese, Method 243.2 (Atomic Absorption, Furnace Technique) replace the Precision and Accuracy Section statement with the following:

Precision and Accuracy

An interlaboratory study on metal analyses by this method was conducted by the Quality Assurance Branch (QAB) of the Environmental Monitoring Systems Laboratory—Cincinnati (EMSL—CI). Synthetic concentrates containing various levels of this element were added to reagent water, surface water, drinking water and three effluents. These samples were digested by the total digestion procedure, 4.1.3 in this manual. Results for the reagent water are given below. Results for other water types and study details are found in ''EPA Method Study 31, Trace Metals by Atomic Absorption (Furnace Techniques),'' National Technical Information Service, 5285 Port Royal Road, Springfield, VA 22161. Order No. PB 86–121 704/AS, by Copeland, F.R. and Maney, J.P., January 1986.

For a concentration range of 0.42–666 µg/L

$X=1.0480(C)+1.404$
$S=0.2001(X)+1.042$
$SR=0.1333(X)+0.680$

Where:

C=True Value for the Concentration, µg/L
X=Mean Recovery, µg/L
S=Multi-laboratory Standard Deviation, µg/L
SR=Single-analyst Standard Deviation, µg/L

Method 249.2

For Nickel, Method 249.2 (Atomic Absorption, Furnace Technique) replace the Precision and Accuracy Section statement with the following:

Precision and Accuracy

An interlaboratory study on metal analyses by this method was conducted by the Quality Assurance Branch (QAB) of the Environmental Monitoring Systems Laboratory—Cincinnati (EMSL—CI). Synthetic concentrates containing various levels of this element were added to reagent water, surface water, drinking water and three effluents.

These samples were digested by the total digestion procedure, 4.1.3 in this manual. Results for the reagent water are given below. Results for other water types and study details are found in ''EPA Method Study 31, Trace Metals by Atomic Absorption (Furnace Techniques),'' National Technical Information Service, 5285 Port Royal Road, Springfield, VA 22161. Order No. PB 86–121 704/AS, by Copeland, F.R. and Maney, J.P., January 1986.

For a concentration range of 26.20–482 µg/L

X=0.8812(C)+2.426
S=0.2475(X)+1.896
SR=0.1935(X)+1.315

Where:

C=True Value for the Concentration, µg/L
X=Mean Recovery, µg/L
S=Multi-laboratory Standard Deviation, µg/L
SR=Single-analyst Standard Deviation, µg/L

Method 270.2

For Selenium, Method 270.2 (Atomic Absorption, Furnace Technique) add the following to the existing Precision and Accuracy Section:

Precision and Accuracy

An interlaboratory study on metal analyses by this method was conducted by the Quality Assurance Branch (QAB) of the Environmental Monitoring Systems Laboratory—Cincinnati (EMSL—CI). Synthetic concentrates containing various levels of this element were added to reagent water, surface water, drinking water and three effluents. Results for the reagent water are given below. Results for other water types and study details are found in ''EPA Method Study 31, Trace Metals by Atomic Absorption (Furnace Techniques),'' National Technical Information Service, 5285 Port Royal Road, Springfield, VA 22161. Order No. PB 86–121 704/AS, by Copeland, F.R. and Maney, J.P., January 1986.

For a concentration range of 10.00–246 µg/L

X=0.9564(C)+0.476
S=0.1584(X)+0.878
SR=0.0772(X)+0.547

Where:

C=True Value for the Concentration, µg/L
X=Mean Recovery, µg/L
S=Multi-laboratory Standard Deviation, µg/L
SR=Single-analyst Standard Deviation, µg/L

Method 272.2

For Silver, Method 272.2 (Atomic Absorption, Furnace Technique) add the following to the existing Precision and Accuracy Section:

Precision and Accuracy

An interlaboratory study on metal analyses by this method was conducted by the Quality Assurance Branch (QAB) of the Environmental Monitoring Systems Laboratory—Cincinnati (EMSL—CI). Synthetic concentrates containing various levels of this element were added to reagent water, surface water, drinking water and three effluents. These samples were digested by the total digestion procedure, 4.1.3 in this manual. Results for the reagent water are given below. Results for other water types and study details are found in ''EPA Method Study 31, Trace Metals by Atomic Absorption (Furnace Techniques),'' National Technical Information Service, 5285 Port Royal Road, Springfield, VA 22161. Order No. PB 86–121 704/AS, by Copeland, F.R. and Maney, J.P., January 1986.

For a concentration range of 0.45–56.5 µg/L

X=0.9470(C)+0.181
S=0.1805(X)+0.153
SR=0.1417(X)+0.039

Where:

C=True Value for the Concentration, µg/L
X=Mean Recovery, µg/L
S=Multi-laboratory Standard Deviation, µg/L
SR=Single-analyst Standard Deviation, µg/L

Method 279.2

For Thalliu, Method 279.2 (Atomic Absorption, Furnace Technique) replace the Precision and Accuracy Section statement with the following:

Precision and Accuracy

An interlaboratory study on metal analyses by this method was conducted by the Quality Assurance Branch (QAB) of the Environmental Monitoring Systems Laboratory–Cincinnati (EMSL–CI). Synthetic concentrates containing various levels of this element were added to reagent water, surface water, drinking water and three effluents. These samples were digested by the total digestion procedure, 4.1.3 in this manual. Results for the reagent water are given below. Results for other water types and study details are found in ''EPA Method Study 31, Trace Metals by Atomic Absorption (Furnace Techniques),'' National Technical Information Service, 5285 Port Royal Road, Springfield, VA 22161 Order No. PB 86–121 704/AS, by Copeland, F.R. and Maney, J.P., January 1986.

For a concentration range of 10.00–252 µg/L.

X=0.8781(C)−0.715
S=0.1112(X)+0.669
SR=0.1005(X)+0.241

Where:

C=True Value for the Concentration, µg/L

X=Mean Recovery, µg/L
S=Multi-laboratory Standard Deviation, µg/L
SR=Single-analyst Standard Deviation, µg/L

Method 286.2

For Vanadium, Method 286.2 (Atomic Absorption, Furnace Technique) replace the Precision and Accuracy Section statement with the following:

Precision and Accuracy

An interlaboratory study on metal analyses by this method was conducted by the Quality Assurance Branch (QAB) of the Environmental Monitoring Systems Laboratory–Cincinnati (EMSL–CI). Synthetic concentrates containing various levels of this element were added to reagent water, surface water, drinking water and three effluents. These samples were digested by the total digestion procedure, 4.1.3 in this manual. Results for the reagent water are given below. Results for other water types and study details are found in ''EPA Method Study 31, Trace Metals by Atomic Absorption (Furnace Techniques),'' National Technical Information Service, 5285 Port Royal Road, Springfield, VA 22161 Order No. PB 86–121 704/AS, by Copeland, F.R. and Maney, J.P., January 1986.

For a concentration range of 1.36–982 µg/L.
X=0.8486(C)+0.252
S=0.3323(X)−0.428
SR=0.1195(X)−0.121

Where:

C=True Value for the Concentration, µg/L
X=Mean Recovery, µg/L
S=Multi-laboratory Standard Deviation, µg/L
SR=Single-analyst Standard Deviation, µg/L

Method 289.2

For Zinc, Method 289.2 (Atomic Absorption, Furnace Technique) replace the Precision and Accuracy Section statement with the following:

Precision and Accuracy

An interlaboratory study on metal analyses by this method was conducted by the Quality Assurance Branch (QAB) of the Environmental Monitoring Systems Laboratory–Cincinnati (EMSL–CI). Synthetic concentrates containing various levels of this element were added to reagent water, surface water, drinking water and three effluents. These samples were digested by the total digestion procedure, 4.1.3 in this manual. Results for the reagent water are given below. Results for other water types and study details are found in ''EPA Method Study 31, Trace Metals by Atomic Absorption (Furnace Techniques),'' National Technical Information Service, 5285 Port Royal Road, Springfield, VA 22161 Order No. PB 86–121 704/AS, by Copeland, F.R. and Maney, J.P., January 1986.

For a concentration range of 0.51–189 µg/L.
X=1.6710(C)+1.485
S=0.6740(X)−0.342
SR=0.3895(X)−0.384

Where:

C=True Value for the Concentration, µg/L
X=Mean Recovery, µg/L
S=Multi-laboratory Standard Deviation, µg/L
SR=Single-analyst Standard Deviation, µg/L

[55 FR 33442, Aug. 15, 1990]

PART 140—MARINE SANITATION DEVICE STANDARD

Sec.
140.1 Definitions.
140.2 Scope of standard.
140.3 Standard.
140.4 Complete prohibition.
140.5 Analytical procedures.

AUTHORITY: Sec. 312, as added Oct. 18, 1972, Pub. L. 92–500, sec. 2, 86 Stat. 871. Interpret or apply sec. 312(b)(1), 33 U.S.C. 1322 (b)(1).

SOURCE: 41 FR 4453, Jan. 29, 1976, unless otherwise noted.

§ 140.1 Definitions.

For the purpose of these standards the following definitions shall apply:

(a) *Sewage* means human body wastes and the wastes from toilets and other receptacles intended to receive or retain body wastes;

(b) *Discharge* includes, but is not limited to, any spilling, leaking, pumping, pouring, emitting, emptying, or dumping;

(c) *Marine sanitation device* includes any equipment for installation onboard a vessel and which is designed to receive, retain, treat, or discharge sewage and any process to treat such sewage;

(d) *Vessel* includes every description of watercraft or other artificial contrivance used, or capable of being used, as a means of transportation on waters of the United States;

(e) *New vessel* refers to any vessel on which construction was initiated on or after January 30, 1975;

(f) *Existing vessel* refers to any vessel on which construction was initiated before January 30, 1975;

(g) *Fecal coliform bacteria* are those organisms associated with the intestines of warm-blooded animals that are commonly used to indicate the presence of fecal material and the potential presence of organisms capable of causing human disease.

§ 140.2 Scope of standard.

The standard adopted herein applies only to vessels on which a marine sanitation device has been installed. The standard does not require the installation of a marine sanitation device on any vessel that is not so equipped. The standard applies to vessels owned and operated by the United States unless the Secretary of Defense finds that compliance would not be in the interest of national security.

§ 140.3 Standard.

(a) (1) In freshwater lakes, freshwater reservoirs or other freshwater impoundments whose inlets or outlets are such as to prevent the ingress or egress by vessel traffic subject to this regulation, or in rivers not capable of navigation by interstate vessel traffic subject to this regulation, marine sanitation devices certified by the U.S. Coast Guard (see 33 CFR part 159, published in 40 FR 4622, January 30, 1975), installed on all vessels shall be designed and operated to prevent the overboard discharge of sewage, treated or untreated, or of any waste derived from sewage. This shall not be construed to prohibit the carriage of Coast Guard-certified flow-through treatment devices which have been secured so as to prevent such discharges.

(2) In all other waters, Coast Guard-certified marine sanitation devices installed on all vessels shall be designed and operated to either retain, dispose of, or discharge sewage. If the device has a discharge, subject to paragraph (d) of this section, the effluent shall not have a fecal coliform bacterial count of greater than 1,000 per 100 milliliters nor visible floating solids. Waters where a Coast Guard-certified marine sanitation device permitting discharge is allowed include coastal waters and estuaries, the Great Lakes and inter-connected waterways, fresh-water lakes and impoundments accessible through locks, and other flowing waters that are navigable interstate by vessels subject to this regulation.

(b) This standard shall become effective on January 30, 1977 for new vessels and on January 30, 1980 for existing vessels (or, in the case of vessels owned and operated by the Department of Defense, two years and five years, for new and existing vessels, respectively, after promulgation of implementing regulations by the Secretary of Defense under section 312(d) of the Act).

(c) Any vessel which is equipped as of the date of promulgation of this regulation with a Coast Guard-certified flow-through marine sanitation device meeting the requirements of paragraph (a)(2) of this section, shall not be required to comply with the provisions designed to prevent the overboard discharge of sewage, treated or untreated, in paragraph (a)(1) of this section, for the operable life of that device.

(d) After January 30, 1980, subject to paragraphs (e) and (f) of this section, marine sanitation devices on all vessels on waters that are not subject to a prohibition of the overboard discharge of sewage, treated or untreated, as specified in paragraph (a)(1) of this section, shall be designed and operated to either retain, dispose of, or discharge sewage, and shall be certified by the U.S. Coast Guard. If the device has a discharge, the effluent shall not have a fecal coliform bacterial count of greater than 200 per 100 milliliters, nor suspended solids greater than 150 mg/1.

(e) Any existing vessel on waters not subject to a prohibition of the overboard discharge of sewage in paragraph (a)(1) of this section, and which is equipped with a certified device on or before January 30, 1978, shall not be required to comply with paragraph (d) of this section, for the operable life of that device.

(f) Any new vessel on waters not subject to the prohibition of the overboard discharge of sewage in paragraph (a)(1) of this section, and on which construction is initiated before January 31, 1980, which is equipped with a marine sanitation device before January 31, 1980, certified under paragraph (a)(2) of this section, shall not be required to comply with paragraph (d) of this section, for the operable life of that device.

(g) The degrees of treatment described in paragraphs (a) and (d) of this section are ''appropriate standards'' for purposes of Coast Guard and Department of Defense certification pursuant to section 312(g)(2) of the Act.

[41 FR 4453, Jan. 29, 1976, as amended at 60 FR 33932, June 29, 1995]

§140.4 Complete prohibition.

(a) A State may completely prohibit the discharge from all vessels of any sewage, whether treated or not, into some or all of the waters within such State by making a written application to the Administrator, Environmental Protection Agency, and by receiving the Administrator's affirmative determination pursuant to section 312(f)(3) of the Act. Upon receipt of an application under section 312(f)(3) of the Act, the Administrator will determine within 90 days whether adequate facilities for the safe and sanitary removal and treatment of sewage from all vessels using such waters are reasonably available. Applications made by States pursuant to section 312(f)(3) of the Act shall include:

(1) A certification that the protection and enhancement of the waters described in the petition require greater environmental protection than the applicable Federal standard;

(2) A map showing the location of commercial and recreational pump-out facilities;

(3) A description of the location of pump-out facilities within waters designated for no discharge;

(4) The general schedule of operating hours of the pump-out facilities;

(5) The draught requirements on vessels that may be excluded because of insufficient water depth adjacent to the facility;

(6) Information indicating that treatment of wastes from such pump-out facilities is in conformance with Federal law; and

(7) Information on vessel population and vessel usage of the subject waters.

(b) A State may make a written application to the Administrator, Environmental Protection Agency, under section 312(f)(4) of the Act, for the issuance of a regulation completely prohibiting discharge from a vessel of any sewage, whether treated or not, into particular waters of the United States or specified portions thereof, which waters are located within the boundaries of such State. Such application shall specify with particularly the waters, or portions thereof, for which a complete prohibition is desired. The application shall include identification of water recreational areas, drinking water intakes, aquatic sanctuaries, identifiable fish-spawning and nursery areas, and areas of intensive boating activities. If, on the basis of the State's application and any other information available to him, the Administrator is unable to make a finding that the waters listed in the application require a complete prohibition of any discharge in the waters or portions thereof covered by the application, he shall state the reasons why he cannot make such a finding, and shall deny the application. If the Administrator makes a finding that the waters listed in the application require a complete prohibition of any discharge in all or any part of the waters or portions thereof covered by the State's application, he shall publish notice of such findings together with a notice of proposed rule making, and then shall proceed in accordance with 5 U.S.C. 553. If the Administrator's finding is that applicable water quality standards require a complete prohibition covering a more restricted or more expanded area than that applied for by the State, he shall state the reasons why his finding differs in scope from that requested in the State's application.

(1) For the following waters the discharge from a vessel of any sewage (whether treated or not) is completely prohibited:

(i) Boundary Waters Canoe Area, formerly designated as the Superior, Little Indian Sioux, and Caribou Roadless Areas, in the Superior National Forest, Minnesota, as described in 16 U.S.C. 577–577d1.

(ii) Two portions of the Hudson River in New York State, the first is bounded by an east-west line through the most northern confluence of the Mohawk River which will be designated by the Troy-Waterford Bridge (126th Street Bridge) on the south and Lock 2 on the north, and the second of which is bounded on the north by the southern

end of Houghtaling Island and on the south by a line between the Village of Roseton on the western shore and Low Point on the eastern shore in the vicinity of Chelsea, as described in Items 2 and 3 of 6 NYCRR Part 858.4.

[41 FR 4453, Jan. 29, 1976, as amended at 42 FR 43837, Aug. 31, 1977; 60 FR 63945, Dec. 13, 1995]

§ 140.5 Analytical procedures.

In determining the composition and quality of effluent discharge from marine sanitation devices, the procedures contained in 40 CFR part 136, ''Guidelines Establishing Test Procedures for the Analysis of Pollutants,'' or subsequent revisions or amendments thereto, shall be employed.

PART 141—NATIONAL PRIMARY DRINKING WATER REGULATIONS

Subpart A—General

Sec.
141.1 Applicability.
141.2 Definitions.
141.3 Coverage.
141.4 Variances and exemptions.
141.5 Siting requirements.
141.6 Effective dates.

Subpart B—Maximum Contaminant Levels

141.11 Maximum contaminant levels for inorganic chemicals.
141.12 Maximum contaminant levels for organic chemicals.
141.13 Maximum contaminant levels for turbidity.
141.15 Maximum contaminant levels for radium-226, radium-228, and gross alpha particle radioactivity in community water systems.
141.16 Maximum contaminant levels for beta particle and photon radioactivity from man-made radionuclides in community water systems.

Subpart C—Monitoring and Analytical Requirements

141.21 Coliform sampling.
141.22 Turbidity sampling and analytical requirements.
141.23 Inorganic chemical sampling and analytical requirements.
141.24 Organic chemicals other than total trihalomethanes, sampling and analytical requirements.
141.25 Analytical methods for radioactivity.
141.26 Monitoring frequency for radioactivity in community water systems.
141.27 Alternate analytical techniques.
141.28 Certified laboratories.
141.29 Monitoring of consecutive public water systems.
141.30 Total trihalomethanes sampling, analytical and other requirements.

Subpart D—Reporting, Public Notification and Recordkeeping

141.31 Reporting requirements.
141.32 Public notification.
141.33 Record maintenance.
141.34 [Reserved]
141.35 Reporting and public notification for certain unregulated contaminants.

Subpart E—Special Regulations, Including Monitoring Regulations and Prohibition on Lead Use

141.40 Special monitoring for inorganic and organic contaminants.
141.41 Special monitoring for sodium.
141.42 Special monitoring for corrosivity characteristics.
141.43 Prohibition on use of lead pipes, solder, and flux.

Subpart F—Maximum Contaminant Level Goals

141.50 Maximum contaminant level goals for organic contaminants.
141.51 Maximum contaminant level goals for inorganic contaminants.
141.52 Maximum contaminant level goals for microbiological contaminants.

Subpart G—National Revised Primary Drinking Water Regulations: Maximum Contaminant Levels

141.60 Effective dates.
141.61 Maximum contaminant levels for organic contaminants.
141.62 Maximum contaminant levels for inorganic contaminants.
141.63 Maximum contaminant levels (MCLs) for microbiological contaminants.

Subpart H—Filtration and Disinfection

141.70 General requirements.
141.71 Criteria for avoiding filtration.
141.72 Disinfection.
141.73 Filtration.
141.74 Analytical and monitoring requirements.
141.75 Reporting and recordkeeping requirements.

Subpart I—Control of Lead and Copper

141.80 General requirements.

141.81 Applicability of corrosion control treatment steps to small, medium-size and large water systems.
141.82 Description of corrosion control treatment requirements.
141.83 Source water treatment requirements.
141.84 Lead service line replacement requirements.
141.85 Public education and supplemental monitoring requirements.
141.86 Monitoring requirements for lead and copper in tap water.
141.87 Monitoring requirements for water quality parameters.
141.88 Monitoring requirements for lead and copper in source water.
141.89 Analytical methods.
141.90 Reporting requirements.
141.91 Recordkeeping requirements.

Subpart J—Use of Non-Centralized Treatment Devices

141.100 Criteria and procedures for public water systems using point-of-entry devices.
141.101 Use of other non-centralized treatment devices.

Subpart K—Treatment Techniques

141.110 General requirements.
141.111 Treatment techniques for acrylamide and epichlorohydrin.

Subpart M—Information Collection Requirements (ICR) for Public Water Systems

141.140 Definitions specific to subpart M.
141.141 General requirements, applicability, and schedule for information collection.
141.142 Disinfection byproduct and related monitoring.
141.143 Microbial monitoring.
141.144 Disinfection byproduct precursor removal studies.

AUTHORITY: 42 U.S.C. 300f, 300g–1, 300g–2, 300g–3, 300g–4, 300g–5, 300g–6, 300j–4, and 300j–9.

SOURCE: 40 FR 59570, Dec. 24, 1975, unless otherwise noted.

NOTE: For community water systems serving 75,000 or more persons, monitoring must begin 1 year following promulation and the effective date of the MCL is 2 years following promulgation. For community water systems serving 10,000 to 75,000 persons, monitoring must begin within 3 years from the date of promulgation and the effective date of the MCL is 4 years from the date of promulgation. Effective immediately, systems that plan to make significant modifications to their treatment processes for the purpose of complying with the TTHM MCL are required to seek and obtain State approval of their treatment modification plans. This note affects §§141.2, 141.6, 141.12, 141.24 and 141.30. For additional information see 44 FR 68641, Nov. 29, 1979.

Subpart A—General

§141.1 Applicability.

This part establishes primary drinking water regulations pursuant to section 1412 of the Public Health Service Act, as amended by the Safe Drinking Water Act (Pub. L. 93–523); and related regulations applicable to public water systems.

§141.2 Definitions.

As used in this part, the term:

Act means the Public Health Service Act, as amended by the Safe Drinking Water Act, Public Law 93–523.

Action level, is the concentration of lead or copper in water specified in §141.80(c) which determines, in some cases, the treatment requirements contained in subpart I of this part that a water system is required to complete.

Best available technology or *BAT* means the best technology, treatment techniques, or other means which the Administrator finds, after examination for efficacy under field conditions and not solely under laboratory conditions, are available (taking cost into consideration). For the purposes of setting MCLs for synthetic organic chemicals, any BAT must be at least as effective as granular activated carbon.

Coagulation means a process using coagulant chemicals and mixing by which colloidal and suspended materials are destabilized and agglomerated into flocs.

Community water system means a public water system which serves at least 15 service connections used by year-round residents or regularly serves at least 25 year-round residents.

Compliance cycle means the nine-year calendar year cycle during which public water systems must monitor. Each compliance cycle consists of three three-year compliance periods. The first calendar year cycle begins January 1, 1993 and ends December 31, 2001; the second begins January 1, 2002 and ends December 31, 2010; the third begins

January 1, 2011 and ends December 31, 2019.

Compliance period means a three-year calendar year period within a compliance cycle. Each compliance cycle has three three-year compliance periods. Within the first compliance cycle, the first compliance period runs from January 1, 1993 to December 31, 1995; the second from January 1, 1996 to December 31, 1998; the third from January 1, 1999 to December 31, 2001.

Confluent growth means a continuous bacterial growth covering the entire filtration area of a membrane filter, or a portion thereof, in which bacterial colonies are not discrete.

Contaminant means any physical, chemical, biological, or radiological substance or matter in water.

Conventional filtration treatment means a series of processes including coagulation, flocculation, sedimentation, and filtration resulting in substantial particulate removal.

Corrosion inhibitor means a substance capable of reducing the corrosivity of water toward metal plumbing materials, especially lead and copper, by forming a protective film on the interior surface of those materials.

CT or *CTcalc* is the product of ''residual disinfectant concentration'' (C) in mg/1 determined before or at the first customer, and the corresponding ''disinfectant contact time'' (T) in minutes, i.e., ''C'' x ''T''. If a public water system applies disinfectants at more than one point prior to the first customer, it must determine the CT of each disinfectant sequence before or at the first customer to determine the total percent inactivation or ''total inactivation ratio.'' In determining the total inactivation ratio, the public water system must determine the residual disinfectant concentration of each disinfection sequence and corresponding contact time before any subsequent disinfection application point(s). ''$CT_{99.9}$'' is the CT value required for 99.9 percent (3–log) inactivation of *Giardia lamblia* cysts. $CT_{99.9}$ for a variety of disinfectants and conditions appear in tables 1.1–1.6, 2.1, and 3.1 of § 141.74(b)(3).

$$\frac{CTcalc}{CT_{99.9}}$$

is the inactivation ratio. The sum of the inactivation ratios, or total inactivation ratio shown as

$$\Sigma \frac{(CTcalc)}{(CT_{99.9})}$$

is calculated by adding together the inactivation ratio for each disinfection sequence. A total inactivation ratio equal to or greater than 1.0 is assumed to provide a 3-log inactivation of *Giardia lamblia* cysts.

Diatomaceous earth filtration means a process resulting in substantial particulate removal in which (1) a precoat cake of diatomaceous earth filter media is deposited on a support membrance (septum), and (2) while the water is filtered by passing through the cake on the septum, additional filter media known as body feed is continuously added to the feed water to maintain the permeability of the filter cake.

Direct filtration means a series of processes including coagulation and filtration but excluding sedimentation resulting in substantial particulate removal.

Disinfectant means any oxidant, including but not limited to chlorine, chlorine dioxide, chloramines, and ozone added to water in any part of the treatment or distribution process, that is intended to kill or inactivate pathogenic microorganisms.

Disinfectant contact time (''T'' in CT calculations) means the time in minutes that it takes for water to move from the point of disinfectant application or the previous point of disinfectant residual measurement to a point before or at the point where residual disinfectant concentration (''C'') is measured. Where only one ''C'' is measured, ''T'' is the time in minutes that it takes for water to move from the point of disinfectant application to a point before or at where residual disinfectant concentration (''C'') is measured. Where more than one ''C'' is measured, ''T'' is (a) for the first measurement of ''C'', the time in minutes that it takes for water to move from

the first or only point of disinfectant application to a point before or at the point where the first ''C'' is measured and (b) for subsequent measurements of ''C'', the time in minutes that it takes for water to move from the previous ''C'' measurement point to the ''C'' measurement point for which the particular ''T'' is being calculated. Disinfectant contact time in pipelines must be calculated based on ''plug flow'' by dividing the internal volume of the pipe by the maximum hourly flow rate through that pipe. Disinfectant contact time within mixing basins and storage reservoirs must be determined by tracer studies or an equivalent demonstration.

Disinfection means a process which inactivates pathogenic organisms in water by chemical oxidants or equivalent agents.

Domestic or other non-distribution system plumbing problem means a coliform contamination problem in a public water system with more than one service connection that is limited to the specific service connection from which the coliform-positive sample was taken.

Dose equivalent means the product of the absorbed dose from ionizing radiation and such factors as account for differences in biological effectiveness due to the type of radiation and its distribution in the body as specified by the International Commission on Radiological Units and Measurements (ICRU).

Effective corrosion inhibitor residual, for the purpose of subpart I of this part only, means a concentration sufficient to form a passivating film on the interior walls of a pipe.

Filtration means a process for removing particulate matter from water by passage through porous media.

First draw sample means a one-liter sample of tap water, collected in accordance with § 141.86(b)(2), that has been standing in plumbing pipes at least 6 hours and is collected without flushing the tap.

Flocculation means a process to enhance agglomeration or collection of smaller floc particles into larger, more easily settleable particles through gentle stirring by hydraulic or mechanical means.

Ground water under the direct influence of surface water means any water beneath the surface of the ground with (1) significant occurrence of insects or other macroorganisms, algae, or large-diameter pathogens such as *Giardia lamblia*, or (2) significant and relatively rapid shifts in water characteristics such as turbidity, temperature, conductivity, or pH which closely correlate to climatological or surface water conditions. Direct influence must be determined for individual sources in accordance with criteria established by the State. The State determination of direct influence may be based on site-specific measurements of water quality and/or documentation of well construction characteristics and geology with field evaluation.

Gross alpha particle activity means the total radioactivity due to alpha particle emission as inferred from measurements on a dry sample.

Gross beta particle activity means the total radioactivity due to beta particle emission as inferred from measurements on a dry sample.

Halogen means one of the chemical elements chlorine, bromine or iodine.

Initial compliance period means the first full three-year compliance period which begins at least 18 months after promulgation, except for contaminants listed at § 141.61(a) (19)–(21), (c) (19)–(33), and § 141.62(b) (11)–(15), initial compliance period means the first full three-year compliance period after promulgation for systems with 150 or more service connections (January 1993–December 1995), and first full three-year compliance period after the effective date of the regulation (January 1996–December 1998) for systems having fewer than 150 service connections.

Large water system, for the purpose of subpart I of this part only, means a water system that serves more than 50,000 persons.

Lead service line means a service line made of lead which connects the water main to the building inlet and any lead pigtail, gooseneck or other fitting which is connected to such lead line.

Legionella means a genus of bacteria, some species of which have caused a type of pneumonia called Legionnaires Disease.

Man-made beta particle and photon emitters means all radionuclides emitting beta particles and/or photons listed in Maximum Permissible Body Burdens and Maximum Permissible Concentration of Radionuclides in Air or Water for Occupational Exposure, NBS Handbook 69, except the daughter products of thorium–232, uranium–235 and uranium–238.

Maximum contaminant level means the maximum permissable level of a contaminant in water which is delivered to any user of a public water system.

Maximum contaminant level goal or *MCLG* means the maximum level of a contaminant in drinking water at which no known or anticipated adverse effect on the health of persons would occur, and which allows an adequate margin of safety. Maximum contaminant level goals are nonenforceable health goals.

Maximum Total Trihalomethane Potential (MTP) means the maximum concentration of total trihalomethanes produced in a given water containing a disinfectant residual after 7 days at a temperature of 25° C or above.

Medium-size water system, for the purpose of subpart I of this part only, means a water system that serves greater than 3,300 and less than or equal to 50,000 persons.

Near the first service connection means at one of the 20 percent of all service connections in the entire system that are nearest the water supply treatment facility, as measured by water transport time within the distribution system.

Non-community water system means a public water system that is not a community water system.

Non-transient non-community water system or *NTNCWS* means a public water system that is not a community water system and that regularly serves at least 25 of the same persons over 6 months per year.

Optimal corrosion control treatment, for the purpose of subpart I of this part only, means the corrosion control treatment that minimizes the lead and copper concentrations at users' taps while insuring that the treatment does not cause the water system to violate any national primary drinking water regulations.

Performance evaluation sample means a reference sample provided to a laboratory for the purpose of demonstrating that the laboratory can successfully analyze the sample within limits of performance specified by the Agency. The true value of the concentration of the reference material is unknown to the laboratory at the time of the analysis.

Person means an individual; corporation; company; association; partnership; municipality; or State, Federal, or tribal agency.

Picocurie (pCi) means the quantity of radioactive material producing 2.22 nuclear transformations per minute.

Point of disinfectant application is the point where the disinfectant is applied and water downstream of that point is not subject to recontamination by surface water runoff.

Point-of-entry treatment device is a treatment device applied to the drinking water entering a house or building for the purpose of reducing contaminants in the drinking water distributed throughout the house or building.

Point-of-use treatment device is a treatment device applied to a single tap used for the purpose of reducing contaminants in drinking water at that one tap.

Public water system or PWS means a system for the provision to the public of piped water for human consumption, if such system has at least fifteen service connections or regularly serves an average of at least twenty-five individuals daily at least 60 days out of the year. Such term includes (1) any collection, treatment, storage, and distribution facilities under control of the operator of such system and used primarily in connection with such system, and (2) any collection or pretreatment storage facilities not under such control which are used primarily in connection with such system. A public water system is either a ''community water system'' or a ''noncommunity water system.''

Rem means the unit of dose equivalent from ionizing radiation to the total body or any internal organ or organ system. A ''millirem (mrem)'' is 1/1000 of a rem.

Repeat compliance period means any subsequent compliance period after the initial compliance period.

Residual disinfectant concentration (''C'' in CT calculations) means the concentration of disinfectant measured in mg/l in a representative sample of water.

Sanitary survey means an onsite review of the water source, facilities, equipment, operation and maintenance of a public water system for the purpose of evaluating the adequacy of such source, facilities, equipment, operation and maintenance for producing and distributing safe drinking water.

Sedimentation means a process for removal of solids before filtration by gravity or separation.

Service line sample means a one-liter sample of water collected in accordance with §141.86(b)(3), that has been standing for at least 6 hours in a service line.

Single family structure, for the purpose of subpart I of this part only, means a building constructed as a single-family residence that is currently used as either a residence or a place of business.

Slow sand filtration means a process involving passage of raw water through a bed of sand at low velocity (generally less than 0.4 m/h) resulting in substantial particulate removal by physical and biological mechanisms.

Small water system, for the purpose of subpart I of this part only, means a water system that serves 3,300 persons or fewer.

Standard sample means the aliquot of finished drinking water that is examined for the presence of coliform bacteria.

State means the agency of the State or Tribal government which has jurisdiction over public water systems. During any period when a State or Tribal government does not have primary enforcement responsibility pursuant to section 1413 of the Act, the term ''State'' means the Regional Administrator, U.S. Environmental Protection Agency.

Supplier of water means any person who owns or operates a public water system.

Surface water means all water which is open to the atmosphere and subject to surface runoff.

System with a single service connection means a system which supplies drinking water to consumers via a single service line.

Too numerous to count means that the total number of bacterial colonies exceeds 200 on a 47-mm diameter membrane filter used for coliform detection.

Total trihalomethanes (TTHM) means the sum of the concentration in milligrams per liter of the trihalomethane compounds (trichloromethane [chloroform], dibromochloromethane, bromodichloromethane and tribromomethane [bromoform]), rounded to two significant figures.

Transient non-community water system or *TWS* means a non-community water system that does not regularly serve at least 25 of the same persons over six months per year.

Trihalomethane (THM) means one of the family of organic compounds, named as derivatives of methane, wherein three of the four hydrogen atoms in methane are each substituted by a halogen atom in the molecular structure.

Virus means a virus of fecal origin which is infectious to humans by waterborne transmission.

Waterborne disease outbreak means the significant occurrence of acute infectious illness, epidemiologically associated with the ingestion of water from a public water system which is deficient in treatment, as determined by the appropriate local or State agency.

[40 FR 59570, Dec. 24, 1975, as amended at 41 FR 28403, July 9, 1976; 44 FR 68641, Nov. 29, 1979; 51 FR 11410, Apr. 2, 1986; 52 FR 20674, June 2, 1987; 52 FR 25712, July 8, 1987; 53 FR 37410, Sept. 26, 1988; 54 FR 27526, 27562, June 29, 1989; 56 FR 3578, Jan. 30, 1991; 56 FR 26547, June 7, 1991; 57 FR 31838, July 17, 1992; 59 FR 34322, July 1, 1994; 61 FR 24368, May 14, 1996]

EFFECTIVE DATE NOTE: At 61 FR 24368, May 14, 1996, §141.2 was amended by adding ''or PWS'' to the definition for ''Public water system'', effective June 18, 1996 and will expire on Dec. 31, 2000.

§141.3 Coverage.

This part shall apply to each public water system, unless the public water system meets all of the following conditions:

(a) Consists only of distribution and storage facilities (and does not have

any collection and treatment facilities);

(b) Obtains all of its water from, but is not owned or operated by, a public water system to which such regulations apply:

(c) Does not sell water to any person; and

(d) Is not a carrier which conveys passengers in interstate commerce.

§ 141.4 Variances and exemptions.

(a) Variances or exemptions from certain provisions of these regulations may be granted pursuant to sections 1415 and 1416 of the Act by the entity with primary enforcement responsibility, except that variances or exemptions from the MCL for total coliforms and variances from any of the treatment technique requirements of subpart H of this part may not be granted.

(b) EPA has stayed the effective date of this section relating to the total coliform MCL of § 141.63(a) for systems that demonstrate to the State that the violation of the total coliform MCL is due to a persistent growth of total coliforms in the distribution system rather than fecal or pathogenic contamination, a treatment lapse or deficiency, or a problem in the operation or maintenance of the distribution system.

[54 FR 27562, June 29, 1989, as amended at 56 FR 1557, Jan. 15, 1991]

§ 141.5 Siting requirements.

Before a person may enter into a financial commitment for or initiate construction of a new public water system or increase the capacity of an existing public water system, he shall notify the State and, to the extent practicable, avoid locating part or all of the new or expanded facility at a site which:

(a) Is subject to a significant risk from earthquakes, floods, fires or other disasters which could cause a breakdown of the public water system or a portion thereof; or

(b) Except for intake structures, is within the floodplain of a 100–year flood or is lower than any recorded high tide where appropriate records exist. The U.S. Environmental Protection Agency will not seek to override land use decisions affecting public water systems siting which are made at the State or local government levels.

§ 141.6 Effective dates.

(a) Except as provided in paragraphs (a) through (i) of this section, and in § 141.80(a)(2), the regulations set forth in this part shall take effect on June 24, 1977.

(b) The regulations for total trihalomethanes set forth in § 141.12(c) shall take effect 2 years after the date of promulgation of these regulations for community water systems serving 75,000 or more individuals, and 4 years after the date of promulgation for communities serving 10,000 to 74,999 individuals.

(c) The regulations set forth in §§ 141.11 (a), (d) and (e); 141.14(a)(1); 141.14(b)(1)(i); 141.14(b)(2)(i); 141.14(d); 141.21 (a), (c) and (i); 141.22 (a) and (e); 141.23 (a)(3) and (a)(4); 141.23(f); 141.24(a)(3); 141.24 (e) and (f); 141.25(e); 141.27(a); 141.28 (a) and (b); 141.31 (a), (d) and (e); 141.32(b)(3); and 141.32(d) shall take effect immediately upon promulgation.

(d) The regulations set forth in § 141.41 shall take effect 18 months from the date of promulgation. Suppliers must complete the first round of sampling and reporting within 12 months following the effective date.

(e) The regulations set forth in § 141.42 shall take effect 18 months from the date of promulgation. All requirements in § 141.42 must be completed within 12 months following the effective date.

(f) The regulations set forth in § 141.11(c) and § 141.23(g) are effective May 2, 1986. Section 141.23(g)(4) is effective October 2, 1987.

(g) The regulations contained in § 141.6, paragraph (c) of the table in 141.12, and 141.62(b)(1) are effective July 1, 1991. The regulations contained in §§ 141.11(b), 141.23, 141.24, 142.57(b), 143.4(b)(12) and (b)(13), are effective July 30, 1992. The regulations contained in the revisions to §§ 141.32(e) (16), (25) through (27) and (46); 141.61(c)(16); and 141.62(b)(3) are effective January 1, 1993. The effective date of regulations contained in § 141.61(c) (2), (3), and (4) is postponed.

(h) Regulations for the analytic methods listed at § 141.23(k)(4) for

measuring antimony, beryllium, cyanide, nickel, and thallium are effective August 17, 1992. Regulations for the analytic methods listed at §141.24(f)(16) for dichloromethane, 1,2,4-trichlorobenzene, and 1,1,2-trichloroethane are effective August 17, 1992. Regulations for the analytic methods listed at §141.24(h)(12) for measuring dalapon, dinoseb, diquat, endothall, endrin, glyphosate, oxamyl, picloram, simazine, benzo(a)pyrene, di(2-ethylhexyl)adipate, di(2-ethylhexyl)phthalate, hexachlorobenzene, hexachlorocyclopentadiene, and 2,3,7,8-TCDD are effective August 17, 1992. The revision to §141.12(a) promulgated on July 17, 1992 is effective on August 17, 1992.

(i) Regulations for information collection requirements listed in subpart M are effective August 14, 1996, and shall remain effective until December 31, 2000.

[44 FR 68641, Nov. 29, 1979, as amended at 45 FR 57342, Aug. 27, 1980; 47 FR 10998, Mar. 12, 1982; 51 FR 11410, Apr. 2, 1986; 56 FR 30274, July 1, 1991; 57 FR 22178, May 27, 1992; 57 FR 31838, July 17, 1992; 59 FR 34322, July 1, 1994; 61 FR 24368, May 14, 1996]

EFFECTIVE DATE NOTE: At 61 FR 24368, May 14, 1996, §141.6 is amended in paragraph (a) by revising the reference ''(a) through (h)'' to read ''(a) through (i)'' and by adding paragraph (i), effective June 18, 1996 and will expire on Dec. 31, 2000.

Subpart B—Maximum Contaminant Levels

§141.11 Maximum contaminant levels for inorganic chemicals.

(a) The maximum contaminant level for arsenic applies only to community water systems. Compliance with the MCL for arsenic is calculated pursuant to §141.23.

(b) The maximum contaminant level for arsenic is 0.05 milligrams per liter.

(c) [Reserved]

(d) At the discretion of the State, nitrate levels not to exceed 20 mg/l may be allowed in a non-community water system if the supplier of water demonstrates to the satisfaction of the State that:

(1) Such water will not be available to children under 6 months of age; and

(2) There will be continuous posting of the fact that nitrate levels exceed 10 mg/l and the potential health effects of exposure; and

(3) Local and State public health authorities will be notified annually of nitrate levels that exceed 10 mg/l; and

(4) No adverse health effects shall result.

[40 FR 59570, Dec. 24, 1975, as amended at 45 FR 57342, Aug. 27, 1980; 47 FR 10998, Mar. 12, 1982; 51 FR 11410, Apr. 2, 1986; 56 FR 3578, Jan. 30, 1991; 56 FR 26548, June 7, 1991; 56 FR 30274, July 1, 1991; 56 FR 32113, July 15, 1991; 60 FR 33932, June 29, 1995]

§141.12 Maximum contaminant levels for organic chemicals.

The following are the maximum contaminant levels for organic chemicals. The maximum contaminant levels for organic chemicals in paragraph (a) of this section apply to all community water systems. Compliance with the maximum contaminant level in paragraph (a) of this section is calculated pursuant to §141.24. The maximum contaminant level for total trihalomethanes in paragraph (c) of this section applies only to community water systems which serve a population of 10,000 or more individuals and which add a disinfectant (oxidant) to the water in any part of the drinking water treatment process. Compliance with the maximum contaminant level for total trihalomethanes is calculated pursuant to §141.30.

	Level, milligrams per liter
(a) [Reserved]	
(b) [Reserved]	
(c) Total trihalomethanes (the sum of the concentrations of bromodichloromethane, dibromochloromethane, tribromomethane (bromoform) and trichloromethane (chloroform))	0.10

[56 FR 3578, Jan. 30, 1991, as amended at 57 FR 31838, July 17, 1992]

§141.13 Maximum contaminant levels for turbidity.

The maximum contaminant levels for turbidity are applicable to both community water systems and non-community water systems using surface water sources in whole or in part. The maximum contaminant levels for

turbidity in drinking water, measured at a representative entry point(s) to the distribution system, are:

EDITORIAL NOTE: At 54 FR 27527, June 29, 1988, § 141.13 was amended by adding introductory text, effective December 31, 1990. This section already contains an introductory text.

The requirements in this section apply to unfiltered systems until December 30, 1991, unless the State has determined prior to that date, in writing pursuant to § 1412(b)(7)(C)(iii), that filtration is required. The requirements in this section apply to filtered systems until June 29, 1993. The requirements in this section apply to unfiltered systems that the State has determined, in writing pursuant to § 1412(b)(7)(C)(iii), must install filtration, until June 29, 1993, or until filtration is installed, whichever is later.

(a) One turbidity unit (TU), as determined by a monthly average pursuant to § 141.22, except that five or fewer turbidity units may be allowed if the supplier of water can demonstrate to the State that the higher turbidity does not do any of the following:

(1) Interfere with disinfection;

(2) Prevent maintenance of an effective disinfectant agent throughout the distribution system; or

(3) Interfere with microbiological determinations.

(b) Five turbidity units based on an average for two consecutive days pursuant to § 141.22.

[40 FR 59570, Dec. 24, 1975]

§ 141.15 Maximum contaminant levels for radium-226, radium-228, and gross alpha particle radioactivity in community water systems.

The following are the maximum contaminant levels for radium-226, radium-228, and gross alpha particle radioactivity:

(a) Combined radium-226 and radium-228—5 pCi/1.

(b) Gross alpha particle activity (including radium-226 but excluding radon and uranium)—15 pCi/1.

[41 FR 28404, July 9, 1976]

§ 141.16 Maximum contaminant levels for beta particle and photon radioactivity from man-made radionuclides in community water systems.

(a) The average annual concentration of beta particle and photon radioactivity from man-made radionuclides in drinking water shall not produce an annual dose equivalent to the total body or any internal organ greater than 4 millirem/year.

(b) Except for the radionuclides listed in Table A, the concentration of man-made radionuclides causing 4 mrem total body or organ dose equivalents shall be calculated on the basis of a 2 liter per day drinking water intake using the 168 hour data listed in *''Maximum Permissible Body Burdens and Maximum Permissible Concentration of Radionuclides in Air or Water for Occupational Exposure,''* NBS Handbook 69 as amended August 1963, U.S. Department of Commerce. If two or more radionuclides are present, the sum of their annual dose equivalent to the total body or to any organ shall not exceed 4 millirem/year.

TABLE A—AVERAGE ANNUAL CONCENTRATIONS ASSUMED TO PRODUCE A TOTAL BODY OR ORGAN DOSE OF 4 MREM/YR

Radionuclide	Critical organ	pCi per liter
Tritium	Total body	20,000
Strontium–90	Bone marrow	8

[41 FR 28404, July 9, 1976]

Subpart C—Monitoring and Analytical Requirements

§ 141.21 Coliform sampling.

(a) *Routine monitoring.* (1) Public water systems must collect total coliform samples at sites which are representative of water throughout the distribution system according to a written sample siting plan. These plans are subject to State review and revision.

(2) The monitoring frequency for total coliforms for community water systems is based on the population served by the system, as follows:

TOTAL COLIFORM MONITORING FREQUENCY FOR COMMUNITY WATER SYSTEMS

Population served	Minimum number of samples per month
25 to 1,000 [1]	1
1,001 to 2,500	2
2,501 to 3,300	3
3,301 to 4,100	4
4,101 to 4,900	5
4,901 to 5,800	6
5,801 to 6,700	7
6,701 to 7,600	8
7,601 to 8,500	9
8,501 to 12,900	10
12,901 to 17,200	15
17,201 to 21,500	20
21,501 to 25,000	25
25,001 to 33,000	30
33,001 to 41,000	40
41,001 to 50,000	50
50,001 to 59,000	60
59,001 to 70,000	70
70,001 to 83,000	80
83,001 to 96,000	90
96,001 to 130,000	100
130,001 to 220,000	120
220,001 to 320,000	150
320,001 to 450,000	180
450,001 to 600,000	210
600,001 to 780,000	240
780,001 to 970,000	270
970,001 to 1,230,000	300
1,230,001 to 1,520,000	330
1,520,001 to 1,850,000	360
1,850,001 to 2,270,000	390
2,270,001 to 3,020,000	420
3,020,001 to 3,960,000	450
3,960,001 or more	480

[1] Includes public water systems which have at least 15 service connections, but serve fewer than 25 persons.

If a community water system serving 25 to 1,000 persons has no history of total coliform contamination in its current configuration and a sanitary survey conducted in the past five years shows that the system is supplied solely by a protected groundwater source and is free of sanitary defects, the State may reduce the monitoring frequency specified above, except that in no case may the State reduce the monitoring frequency to less than one sample per quarter. The State must approve the reduced monitoring frequency in writing.

(3) The monitoring frequency for total coliforms for non-community water systems is as follows:

(i) A non-community water system using only ground water (except ground water under the direct influence of surface water, as defined in § 141.2) and serving 1,000 persons or fewer must monitor each calendar quarter that the system provides water to the public, except that the State may reduce this monitoring frequency, in writing, if a sanitary survey shows that the system is free of sanitary defects. Beginning June 29, 1994, the State cannot reduce the monitoring frequency for a non-community water system using only ground water (except ground water under the direct influence of surface water, as defined in § 141.2) and serving 1,000 persons or fewer to less than once/year.

(ii) A non-community water system using only ground water (except ground water under the direct influence of surface water, as defined in § 141.2) and serving more than 1,000 persons during any month must monitor at the same frequency as a like-sized community water system, as specified in paragraph (a)(2) of this section, except the State may reduce this monitoring frequency, in writing, for any month the system serves 1,000 persons or fewer. The State cannot reduce the monitoring frequency to less than once/year. For systems using ground water under the direct influence of surface water, paragraph (a)(3)(iv) of this section applies.

(iii) A non-community water system using surface water, in total or in part, must monitor at the same frequency as a like-sized community water system, as specified in paragraph (a)(2) of this section, regardless of the number of persons it serves.

(iv) A non-community water system using ground water under the direct influence of surface water, as defined in § 141.2, must monitor at the same frequency as a like-sized community water system, as specified in paragraph (a)(2) of this section. The system must begin monitoring at this frequency beginning six months after the State determines that the ground water is under the direct influence of surface water.

(4) The public water system must collect samples at regular time intervals throughout the month, except that a system which uses only ground water (except ground water under the direct influence of surface water, as defined in § 141.2), and serves 4,900 persons or fewer, may collect all required samples

on a single day if they are taken from different sites.

(5) A public water system that uses surface water or ground water under the direct influence of surface water, as defined in § 141.2, and does not practice filtration in compliance with Subpart H must collect at least one sample near the first service connection each day the turbidity level of the source water, measured as specified in § 141.74(b)(2), exceeds 1 NTU. This sample must be analyzed for the presence of total coliforms. When one or more turbidity measurements in any day exceed 1 NTU, the system must collect this coliform sample within 24 hours of the first exceedance, unless the State determines that the system, for logistical reasons outside the system's control, cannot have the sample analyzed within 30 hours of collection. Sample results from this coliform monitoring must be included in determining compliance with the MCL for total coliforms in § 141.63.

(6) Special purpose samples, such as those taken to determine whether disinfection practices are sufficient following pipe placement, replacement, or repair, shall not be used to determine compliance with the MCL for total coliforms in § 141.63. Repeat samples taken pursuant to paragraph (b) of this section are not considered special purpose samples, and must be used to determine compliance with the MCL for total coliforms in § 141.63.

(b) *Repeat monitoring.* (1) If a routine sample is total coliform-positive, the public water system must collect a set of repeat samples within 24 hours of being notified of the positive result. A system which collects more than one routine sample/month must collect no fewer than three repeat samples for each total coliform-positive sample found. A system which collects one routine sample/month or fewer must collect no fewer than four repeat samples for each total coliform-positive sample found. The State may extend the 24-hour limit on a case-by-case basis if the system has a logistical problem in collecting the repeat samples within 24 hours that is beyond its control. In the case of an extension, the State must specify how much time the system has to collect the repeat samples.

(2) The system must collect at least one repeat sample from the sampling tap where the original total coliform-positive sample was taken, and at least one repeat sample at a tap within five service connections upstream and at least one repeat sample at a tap within five service connections downstream of the original sampling site. If a total coliform-positive sample is at the end of the distribution system, or one away from the end of the distribution system, the State may waive the requirement to collect at least one repeat sample upstream or downstream of the original sampling site.

(3) The system must collect all repeat samples on the same day, except that the State may allow a system with a single service connection to collect the required set of repeat samples over a four-day period or to collect a larger volume repeat sample(s) in one or more sample containers of any size, as long as the total volume collected is at least 400 ml (300 ml for systems which collect more than one routine sample/month).

(4) If one or more repeat samples in the set is total coliform-positive, the public water system must collect an additional set of repeat samples in the manner specified in paragraphs (b) (1)–(3) of this section. The additional samples must be collected within 24 hours of being notified of the positive result, unless the State extends the limit as provided in paragraph (b)(1) of this section. The system must repeat this process until either total coliforms are not detected in one complete set of repeat samples or the system determines that the MCL for total coliforms in § 141.63 has been exceeded and notifies the State.

(5) If a system collecting fewer than five routine samples/month has one or more total coliform-positive samples and the State does not invalidate the sample(s) under paragraph (c) of this section, it must collect at least five routine samples during the next month the system provides water to the public, except that the State may waive this requirement if the conditions of paragraph (b)(5) (i) or (ii) of this section are met. The State cannot waive

the requirement for a system to collect repeat samples in paragraphs (b) (1)–(4) of this section.

(i) The State may waive the requirement to collect five routine samples the next month the system provides water to the public if the State, or an agent approved by the State, performs a site visit before the end of the next month the system provides water to the public. Although a sanitary survey need not be performed, the site visit must be sufficiently detailed to allow the State to determine whether additional monitoring and/or any corrective action is needed. The State cannot approve an employee of the system to perform this site visit, even if the employee is an agent approved by the State to perform sanitary surveys.

(ii) The State may waive the requirement to collect five routine samples the next month the system provides water to the public if the State has determined why the sample was total coliform-positive and establishes that the system has corrected the problem or will correct the problem before the end of the next month the system serves water to the public. In this case, the State must document this decision to waive the following month's additional monitoring requirement in writing, have it approved and signed by the supervisor of the State official who recommends such a decision, and make this document available to the EPA and public. The written documentation must describe the specific cause of the total coliform-positive sample and what action the system has taken and/or will take to correct this problem. The State cannot waive the requirement to collect five routine samples the next month the system provides water to the public solely on the grounds that all repeat samples are total coliform-negative. Under this paragraph, a system must still take at least one routine sample before the end of the next month it serves water to the public and use it to determine compliance with the MCL for total coliforms in § 141.63, unless the State has determined that the system has corrected the contamination problem before the system took the set of repeat samples required in paragraphs (b) (1)–(4) of this section, and all repeat samples were total coliform-negative.

(6) After a system collects a routine sample and before it learns the results of the analysis of that sample, if it collects another routine sample(s) from within five adjacent service connections of the initial sample, and the initial sample, after analysis, is found to contain total coliforms, then the system may count the subsequent sample(s) as a repeat sample instead of as a routine sample.

(7) Results of all routine and repeat samples not invalidated by the State must be included in determining compliance with the MCL for total coliforms in § 141.63.

(c) *Invalidation of total coliform samples.* A total coliform-positive sample invalidated under this paragraph (c) does not count towards meeting the minimum monitoring requirements of this section. (1) The State may invalidate a total coliform-positive sample only if the conditions of paragraph (c)(1) (i), (ii), or (iii) of this section are met.

(i) The laboratory establishes that improper sample analysis caused the total coliform-positive result.

(ii) The State, on the basis of the results of repeat samples collected as required by paragraphs (b) (1) through (4) of this section, determines that the total coliform-positive sample resulted from a domestic or other non-distribution system plumbing problem. The State cannot invalidate a sample on the basis of repeat sample results unless all repeat sample(s) collected at the same tap as the original total coliform-positive sample are also total coliform-positive, and all repeat samples collected within five service connections of the original tap are total coliform-negative (e.g., a State cannot invalidate a total coliform-positive sample on the basis of repeat samples if all the repeat samples are total coliform-negative, or if the public water system has only one service connection).

(iii) The State has substantial grounds to believe that a total coliform-positive result is due to a circumstance or condition which does not reflect water quality in the distribution system. In this case, the system

must still collect all repeat samples required under paragraphs (b) (1)–(4) of this section, and use them to determine compliance with the MCL for total coliforms in § 141.63. To invalidate a total coliform-positive sample under this paragraph, the decision with the rationale for the decision must be documented in writing, and approved and signed by the supervisor of the State official who recommended the decision. The State must make this document available to EPA and the public. The written documentation must state the specific cause of the total coliform-positive sample, and what action the system has taken, or will take, to correct this problem. The State may not invalidate a total coliform-positive sample solely on the grounds that all repeat samples are total coliform-negative.

(2) A laboratory must invalidate a total coliform sample (unless total coliforms are detected) if the sample produces a turbid culture in the absence of gas production using an analytical method where gas formation is examined (e.g., the Multiple-Tube Fermentation Technique), produces a turbid culture in the absence of an acid reaction in the Presence-Absence (P–A) Coliform Test, or exhibits confluent growth or produces colonies too numerous to count with an analytical method using a membrane filter (e.g., Membrane Filter Technique). If a laboratory invalidates a sample because of such interference, the system must collect another sample from the same location as the original sample within 24 hours of being notified of the interference problem, and have it analyzed for the presence of total coliforms. The system must continue to re-sample within 24 hours and have the samples analyzed until it obtains a valid result. The State may waive the 24-hour time limit on a case-by-case basis.

(d) *Sanitary surveys.* (1)(i) Public water systems which do not collect five or more routine samples/month must undergo an initial sanitary survey by June 29, 1994, for community public water systems and June 29, 1999, for non-community water systems. Thereafter, systems must undergo another sanitary survey every five years, except that non-community water systems using only protected and disinfected ground water, as defined by the State, must undergo subsequent sanitary surveys at least every ten years after the initial sanitary survey. The State must review the results of each sanitary survey to determine whether the existing monitoring frequency is adequate and what additional measures, if any, the system needs to undertake to improve drinking water quality.

(ii) In conducting a sanitary survey of a system using ground water in a State having an EPA-approved wellhead protection program under section 1428 of the Safe Drinking Water Act, information on sources of contamination within the delineated wellhead protection area that was collected in the course of developing and implementing the program should be considered instead of collecting new information, if the information was collected since the last time the system was subject to a sanitary survey.

(2) Sanitary surveys must be performed by the State or an agent approved by the State. The system is responsible for ensuring the survey takes place.

(e) *Fecal coliforms/Escherichia coli (E. coli) testing.* (1) If any routine or repeat sample is total coliform-positive, the system must analyze that total coliform-positive culture medium to determine if fecal coliforms are present, except that the system may test for *E. coli* in lieu of fecal coliforms. If fecal coliforms or *E. coli* are present, the system must notify the State by the end of the day when the system is notified of the test result, unless the system is notified of the result after the State office is closed, in which case the system must notify the State before the end of the next business day.

(2) The State has the discretion to allow a public water system, on a case-by-case basis, to forgo fecal coliform or *E. coli* testing on a total coliform-positive sample if that system assumes that the total coliform-positive sample is fecal coliform-positive or *E. coli*-positive. Accordingly, the system must notify the State as specified in paragraph (e)(1) of this section and the provisions of § 141.63(b) apply.

(f) *Analytical methodology.* (1) The standard sample volume required for total coliform analysis, regardless of analytical method used, is 100 ml.

(2) Public water systems need only determine the presence or absence of total coliforms; a determination of total coliform density is not required.

(3) Public water systems must conduct total coliform analyses in accordance with one of the analytical methods in the following table. These methods are contained in the 18th edition of Standard Methods for the Examination of Water and Wastewater, 1992, American Public Health Association, 1015 Fifteenth Street NW., Washington, DC 20005. A description of the Colisure Test may be obtained from the Millipore Corporation, Technical Services Department, 80 Ashby Road, Bedford, MA 01730. The toll-free phone number is (800) 645–5476.

Organism	Methodology	Citation
Total Coliforms [1].	Total Coliform Fermentation Technique [2,3,4].	9221A, B.
	Total Coliform Membrane Filter Technique.	9222A, B, C.
	Presence-Absence (P–A) Coliform Test [4,5].	9221D.
	ONPG–MUG Test [6]	9223.
	Colisure Test [7].	

[1] The time from sample collection to initiation of analysis may not exceed 30 hours. Systems are encouraged but not required to hold samples below 10°C during transit.

[2] Lactose broth, as commercially available, may be used in lieu of laurel tryptose broth, if the system conducts at least 25 parallel tests between this medium and lauryl tryptose broth using the water normally tested, and this comparison demonstrates that the false-positive rate and false-negative rate for total coliforms, using lactose broth, is less than 10 percent.

[3] If inverted tubes are used to detect gas production, the media should cover these tubes at least one-half to two-thirds after the sample is added.

[4] No requirement exists to run the completed phase on 10 percent of all total coliform-positive confirmed tubes.

[5] Six-times formulation strength may be used if the medium is filter-sterilized rather than autoclaved.

[6] The ONPG–MUG Test is also known as the Autoanalysis Colilert System.

[7] The Colisure Test must be incubated for 28 hours before examining the results. If an examination of the results at 28 hours is not convenient, then results may be examined at any time between 28 hours and 48 hours.

(4) [Reserved]

(5) Public water systems must conduct fecal coliform analysis in accordance with the following procedure. When the MTF Technique or Presence-Absence (PA) Coliform Test is used to test for total coliforms, shake the lactose-positive presumptive tube or P–A vigorously and transfer the growth with a sterile 3-mm loop or sterile applicator stick into brilliant green lactose bile broth and EC medium to determine the presence of total and fecal coliforms, respectively. For EPA-approved analytical methods which use a membrane filter, transfer the total coliform-positive culture by one of the following methods: remove the membrane containing the total coliform colonies from the substrate with a sterile forceps and carefully curl and insert the membrane into a tube of EC medium (the laboratory may first remove a small portion of selected colonies for verification), swab the entire membrane filter surface with a sterile cotton swab and transfer the inoculum to EC medium (do not leave the cotton swab in the EC medium), or inoculate individual total coliform-positive colonies into EC Medium. Gently shake the inoculated tubes of EC medium to insure adequate mixing and incubate in a waterbath at 44.5 ± 0.2 °C for 24 ± 2 hours. Gas production of any amount in the inner fermentation tube of the EC medium indicates a positive fecal coliform test. The preparation of EC medium is described in the 18th edition of *Standard Methods for the Examination of Water and Wastewater,* 1992, Method 9221E—p. 9–52, paragraph 1a. Public water systems need only determine the presence or absence of fecal coliforms; a determination of fecal coliform density is not required.

(6) Public water systems must conduct analysis of *Escherichia coli* in accordance with one of the following analytical methods:

(i) EC medium supplemented with 50 µg/ml of 4-methylumbelliferyl-beta-D-glucuronide (MUG) (final concentration). EC medium is described in the 18th edition of *Standard Methods for the Examination of Water and Wastewater,* 1992, Method 9221E—p. 9–52, paragraph 1a. MUG may be added to EC medium before autoclaving. EC medium supplemented with 50 µg/ml of MUG is commercially available. At least 10 ml of EC medium supplemented with MUG must be used. The inner inverted fermentation tube may be omitted. The procedure for transferring a total coliform-positive culture to EC medium supplemented with MUG shall be as specified in paragraph (f)(5) of this section for transferring a total coliform-positive culture to EC medium. Observe fluorescence with an ultraviolet

light (366 nm) in the dark after incubating tube at 44.5 ± 0.2 °C for 24 ± 2 hours; or

(ii) Nutrient agar supplemented with 100 µg/ml 4-methylumbelliferyl-beta-D-glucuronide (MUG) (final concentration). Nutrient Agar is described in the 18th edition of *Standard Methods for the Examination of Water and Wastewater,* 1992, p. 9–47 to 9–48. This test is used to determine if a total coliform-positive sample, as determined by the Membrane Filter Technique or any other method in which a membrane filter is used, contains *E. coli.* Transfer the membrane filter containing a total coliform colony(ies) to nutrient agar supplemented with 100 µg/ml (final concentration) of MUG. After incubating the agar plate at 35 °C for 4 hours, observe the colony(ies) under ultraviolet light (366 nm) in the dark for fluorescence. If fluorescence is visible, *E. coli* are present.

(iii) Minimal Medium ONPG–MUG (MMO–MUG) Test, as set forth in the article ''National Field Evaluation of a Defined Substrate Method for the Simultaneous Detection of Total Coliforms and *Escherichia coli* from Drinking Water: Comparison with Presence-Absence Techniques'' (Edberg et al.), Applied and Environmental Microbiology, Volume 55, pp. 1003–1008, April 1989. (Note: The Autoanalysis Colilert System is an MMO–MUG test). If the MMO–MUG test is total coliform-positive after a 24-hour incubation, test the medium for fluorescence with a 366-nm ultraviolet light (preferably with a 6-watt lamp) in the dark. If fluorescence is observed, the sample is *E. coli*-positive. If fluorescence is questionable (cannot be definitively read) after 24 hours incubation, incubate the culture for an additional four hours (but not to exceed 28 hours total), and again test the medium for fluorescence. The MMO–MUG Test with hepes buffer in lieu of phosphate buffer is the only approved formulation for the detection of *E. coli.*

(iv) The Colisure Test. A description of the Colisure Test may be obtained from the Millipore Corporation, Technical Services Department, 80 Ashby Road, Bedford, MA 01730.

(7) As an option to paragraph (f)(6)(iii) of this section, a system with a total coliform-positive, MUG-negative, MMO–MUG test may further analyze the culture for the presence of *E. coli* by transferring a 0.1 ml, 28-hour MMO–MUG culture to EC Medium + MUG with a pipet. The formulation and incubation conditions of EC Medium + MUG, and observation of the results are described in paragraph (f)(6)(i) of this section.

(8) The following materials are incorporated by reference in this section with the approval of the Director of the Federal Register in accordance with 5 U.S.C. 552(a) and 1 CFR part 51. Copies of the analytical methods cited in *Standard Methods for the Examination of Water and Wastewater* may be obtained from the American Public Health Association *et al.*; 1015 Fifteenth Street, NW.; Washington, DC 20005. Copies of the methods set forth in *Microbiological Methods for Monitoring the Environment, Water and Wastes* may be obtained from ORD Publications, U.S. EPA, 26 W. Martin Luther King Drive, Cincinnati, Ohio 45268. Copies of the MMO–MUG Test as set forth in the article ''National Field Evaluation of a Defined Substrate Method for the Simultaneous Enumeration of Total Coliforms and *Escherichia coli* from Drinking Water: Comparison with the Standard Multiple Tube Fermentation Method'' (Edberg *et al.*) may be obtained from the American Water Works Association Research Foundation, 6666 West Quincy Avenue, Denver, CO 80235. A description of the Colisure Test may be obtained from the Millipore Corp., Technical Services Department, 80 Ashby Road, Bedford, MA 01730. Copies may be inspected at EPA's Drinking Water Docket; 401 M Street, SW.; Washington, DC 20460, or at the Office of the Federal Register; 800 North Capitol Street, NW., suite 700, Washington, DC.

(g) *Response to violation.* (1) A public water system which has exceeded the MCL for total coliforms in § 141.63 must report the violation to the State no later than the end of the next business day after it learns of the violation, and notify the public in accordance with § 141.32.

(2) A public water system which has failed to comply with a coliform monitoring requirement, including the sanitary survey requirement, must report

the monitoring violation to the State within ten days after the system discovers the violation, and notify the public in accordance with §141.32.

[54 FR 27562, June 29, 1989, as amended at 54 FR 30001, July 17, 1989; 55 FR 25064, June 19, 1990; 56 FR 642, Jan. 8, 1991; 57 FR 1852, Jan. 15, 1992; 57 FR 24747, June 10, 1992; 59 FR 62466, Dec. 5, 1994; 60 FR 34085, June 29, 1995]

§141.22 Turbidity sampling and analytical requirements.

The requirements in this section apply to unfiltered systems until December 30, 1991, unless the State has determined prior to that date, in writing pursuant to section 1412(b)(7)(iii), that filtration is required. The requirements in this section apply to filtered systems until June 29, 1993. The requirements in this section apply to unfiltered systems that the State has determined, in writing pursuant to section 1412(b)(7)(C)(iii), must install filtration, until June 29, 1993, or until filtration is installed, whichever is later.

(a) Samples shall be taken by suppliers of water for both community and non-community water systems at a representative entry point(s) to the water distribution system at least once per day, for the purposes of making turbidity measurements to determine compliance with §141.13. If the State determines that a reduced sampling frequency in a non-community will not pose a risk to public health, it can reduce the required sampling frequency. The option of reducing the turbidity frequency shall be permitted only in those public water systems that practice disinfection and which maintain an active residual disinfectant in the distribution system, and in those cases where the State has indicated in writing that no unreasonable risk to health existed under the circumstances of this option. Turbidity measurements shall be made as directed in §141.74(a)(1).

(b) If the result of a turbidity analysis indicates that the maximum allowable limit has been exceeded, the sampling and measurement shall be confirmed by resampling as soon as practicable and preferably within one hour. If the repeat sample confirms that the maximum allowable limit has been exceeded, the supplier of water shall report to the State within 48 hours. The repeat sample shall be the sample used for the purpose of calculating the monthly average. If the monthly average of the daily samples exceeds the maximum allowable limit, or if the average of two samples taken on consecutive days exceeds 5 TU, the supplier of water shall report to the State and notify the public as directed in §§141.31 and 141.32.

(c) Sampling for non-community water systems shall begin within two years after the effective date of this part.

(d) The requirements of this §141.22 shall apply only to public water systems which use water obtained in whole or in part from surface sources.

(e) The State has the authority to determine compliance or initiate enforcement action based upon analytical results or other information compiled by their sanctioned representatives and agencies.

[40 FR 59570, Dec. 24, 1975, as amended at 45 FR 57344, Aug. 27, 1980; 47 FR 8998, Mar. 3, 1982; 47 FR 10998, Mar. 12, 1982; 54 FR 27527, June 29, 1989; 59 FR 62466, Dec. 5, 1994]

§141.23 Inorganic chemical sampling and analytical requirements.

Community water systems shall conduct monitoring to determine compliance with the maximum contaminant levels specified in §141.62 in accordance with this section. Non-transient, non-community water systems shall conduct monitoring to determine compliance with the maximum contaminant levels specified in §141.62 in accordance with this section. Transient, non-community water systems shall conduct monitoring to determine compliance with the nitrate and nitrite maximum contaminant levels in §§141.11 and 141.62 (as appropriate) in accordance with this section.

(a) Monitoring shall be conducted as follows:

(1) Groundwater systems shall take a minimum of one sample at every entry point to the distribution system which is representative of each well after treatment (hereafter called a sampling point) beginning in the initial compliance period. The system shall take each sample at the same sampling point unless conditions make another

sampling point more representative of each source or treatment plant.

(2) Surface water systems shall take a minimum of one sample at every entry point to the distribution system after any application of treatment or in the distribution system at a point which is representative of each source after treatment (hereafter called a sampling point) beginning in the initial compliance period. The system shall take each sample at the same sampling point unless conditions make another sampling point more representative of each source or treatment plant.

NOTE: For purposes of this paragraph, surface water systems include systems with a combination of surface and ground sources.

(3) If a system draws water from more than one source and the sources are combined before distribution, the system must sample at an entry point to the distribution system during periods of normal operating conditions (i.e., when water is representative of all sources being used).

(4) The State may reduce the total number of samples which must be analyzed by allowing the use of compositing. Composite samples from a maximum of five samples are allowed, provided that the detection limit of the method used for analysis is less than one-fifth of the MCL. Compositing of samples must be done in the laboratory.

(i) If the concentration in the composite sample is greater than or equal to one-fifth of the MCL of any inorganic chemical, then a follow-up sample must be taken within 14 days at each sampling point included in the composite. These samples must be analyzed for the contaminants which exceeded one-fifth of the MCL in the composite sample. Detection limits for each analytical method and MCLs for each inorganic contaminant are the following:

DETECTION LIMITS FOR INORGANIC CONTAMINANTS

Contaminant	MCL (mg/l)	Methodology	Detection limit (mg/l)
Antimony	0.006	Atomic Absorption; Furnace	0.003
		Atomic Absorption; Platform	0.0008 [5]
		ICP-Mass Spectrometry	0.0004
		Hydride-Atomic Absorption	0.001
Asbestos	7 MFL [1]	Transmission Electron Microscopy	0.01 MFL
Barium	2	Atomic Absorption; furnace technique	0.002
		Atomic Absorption; direct aspiration	0.1
		Inductively Coupled Plasma	0.002 (0.001)
Beryllium	0.004	Atomic Absorption; Furnace	0.0002
		Atomic Absorption; Platform	0.00002 [5]
		Inductively Coupled Plasma [2]	0.0003
		ICP-Mass Spectrometry	0.0003
Cadmium	0.005	Atomic Absorption; furnace technique	0.0001
		Inductively Coupled Plasma	0.001
Chromium	0.1	Atomic Absorption; furnace technique	0.001
		Inductively Coupled Plasma	0.007 (0.001)
Cyanide	0.2	Distillation, Spectrophotometric [3]	0.02
		Distillation, Automated, Spectrophotometric [3]	0.005
		Distillation, Selective Electrode [3]	0.05
		Distillation, Amenable, Spectrophotometric [4]	0.02
Mercury	0.002	Manual Cold Vapor Technique	0.0002
		Automated Cold Vapor Technique	0.0002
Nickel	xl	Atomic Absorption; Furnace	0.001
		Atomic Absorption; Platform	0.0006 [5]
		Inductively Coupled Plasma [2]	0.005
		ICP-Mass Spectrometry	0.0005
Nitrate	10 (as N)	Manual Cadmium Reduction	0.01
		Automated Hydrazine Reduction	0.01
		Automated Cadmium Reduction	0.05
		Ion Selective Electrode	1
		Ion Chromatography	0.01
Nitrite	1 (as N)	Spectrophotometric	0.01
		Automated Cadmium Reduction	0.05
		Manual Cadmium Reduction	0.01
		Ion Chromatography	0.004
Selenium	0.05	Atomic Absorption; furnace	0.002
		Atomic Absorption; gaseous hydride	0.002
Thallium	0.002	Atomic Absorption; Furnace	0.001

DETECTION LIMITS FOR INORGANIC CONTAMINANTS—Continued

Contaminant	MCL (mg/l)	Methodology	Detection limit (mg/l)
		Atomic Absorption; Platform	0.0007[5]
		ICP-Mass Spectrometry	0.0003

[1] MFL = million fibers per liter >10 μm.
[2] Using a 2X preconcentration step as noted in Method 200.7. Lower MDLs may be achieved when using a 4X preconcentration.
[3] Screening method for total cyanides.
[4] Measures "free" cyanides.
[5] Lower MDLs are reported using stabilized temperature graphite furnace atomic absorption.

(ii) If the population served by the system is >3,300 persons, then compositing may only be permitted by the State at sampling points within a single system. In systems serving ≤3,300 persons, the State may permit compositing among different systems provided the 5-sample limit is maintained.

(iii) If duplicates of the original sample taken from each sampling point used in the composite are available, the system may use these instead of resampling. The duplicates must be analyzed and the results reported to the State within 14 days of collection.

(5) The frequency of monitoring for asbestos shall be in accordance with paragraph (b) of this section: the frequency of monitoring for antimony, barium, beryllium, cadmium, chromium, cyanide, fluoride, mercury, nickel, selenium and thallium shall be in accordance with paragraph (c) of this section; the frequency of monitoring for nitrate shall be in accordance with paragraph (d) of this section; and the frequency of monitoring for nitrite shall be in accordance with paragraph (e) of this section.

(b) The frequency of monitoring conducted to determine compliance with the maximum contaminant level for asbestos specified in § 141.62(b) shall be conducted as follows:

(1) Each community and non-transient, non-community water system is required to monitor for asbestos during the first three-year compliance period of each nine-year compliance cycle beginning in the compliance period starting January 1, 1993.

(2) If the system believes it is not vulnerable to either asbestos contamination in its source water or due to corrosion of asbestos-cement pipe, or both, it may apply to the State for a waiver of the monitoring requirement in paragraph (b)(1) of this section. If the State grants the waiver, the system is not required to monitor.

(3) The State may grant a waiver based on a consideration of the following factors:

(i) Potential asbestos contamination of the water source, and

(ii) The use of asbestos-cement pipe for finished water distribution and the corrosive nature of the water.

(4) A waiver remains in effect until the completion of the three-year compliance period. Systems not receiving a waiver must monitor in accordance with the provisions of paragraph (b)(1) of this section.

(5) A system vulnerable to asbestos contamination due solely to corrosion of asbestos-cement pipe shall take one sample at a tap served by asbestos-cement pipe and under conditions where asbestos contamination is most likely to occur.

(6) A system vulnerable to asbestos contamination due solely to source water shall monitor in accordance with the provision of paragraph (a) of this section.

(7) A system vulnerable to asbestos contamination due both to its source water supply and corrosion of asbestos-cement pipe shall take one sample at a tap served by asbestos-cement pipe and under conditions where asbestos contamination is most likely to occur.

(8) A system which exceeds the maximum contaminant levels as determined in § 141.23(i) of this section shall monitor quarterly beginning in the next quarter after the violation occurred.

(9) The State may decrease the quarterly monitoring requirement to the frequency specified in paragraph (b)(1) of this section provided the State has determined that the system is reliably

and consistently below the maximum contaminant level. In no case can a State make this determination unless a groundwater system takes a minimum of two quarterly samples and a surface (or combined surface/ground) water system takes a minimum of four quarterly samples.

(10) If monitoring data collected after January 1, 1990 are generally consistent with the requirements of § 141.23(b), then the State may allow systems to use that data to satisfy the monitoring requirement for the initial compliance period beginning January 1, 1993.

(c) The frequency of monitoring conducted to determine compliance with the maximum contaminant levels in § 141.62 for antimony, barium, beryllium, cadmium, chromium, cyanide, fluoride, mercury, nickel, selenium and thallium shall be as follows:

(1) Groundwater systems shall take one sample at each sampling point during each compliance period. Surface water systems (or combined surface/ground) shall take one sample annually at each sampling point.

(2) The system may apply to the State for a waiver from the monitoring frequencies specified in paragraph (c)(1) of this section. States may grant a public water system a waiver for monitoring of cyanide, provided that the State determines that the system is not vulnerable due to lack of any industrial source of cyanide.

(3) A condition of the waiver shall require that a system shall take a minimum of one sample while the waiver is effective. The term during which the waiver is effective shall not exceed one compliance cycle (i.e., nine years).

(4) The State may grant a waiver provided surface water systems have monitored annually for at least three years and groundwater systems have conducted a minimum of three rounds of monitoring. (At least one sample shall have been taken since January 1, 1990). Both surface and groundwater systems shall demonstrate that all previous analytical results were less than the maximum contaminant level. Systems that use a new water source are not eligible for a waiver until three rounds of monitoring from the new source have been completed.

(5) In determining the appropriate reduced monitoring frequency, the State shall consider:

(i) Reported concentrations from all previous monitoring;

(ii) The degree of variation in reported concentrations; and

(iii) Other factors which may affect contaminant concentrations such as changes in groundwater pumping rates, changes in the system's configuration, changes in the system's operating procedures, or changes in stream flows or characteristics.

(6) A decision by the State to grant a waiver shall be made in writing and shall set forth the basis for the determination. The determination may be initiated by the State or upon an application by the public water system. The public water system shall specify the basis for its request. The State shall review and, where appropriate, revise its determination of the appropriate monitoring frequency when the system submits new monitoring data or when other data relevant to the system's appropriate monitoring frequency become available.

(7) Systems which exceed the maximum contaminant levels as calculated in § 141.23(i) of this section shall monitor quarterly beginning in the next quarter after the violation occurred.

(8) The State may decrease the quarterly monitoring requirement to the frequencies specified in paragraphs (c)(1) and (c)(2) of this section provided it has determined that the system is reliably and consistently below the maximum contaminant level. In no case can a State make this determination unless a groundwater system takes a minimum of two quarterly samples and a surface water system takes a minimum of four quarterly samples.

(d) All public water systems (community; non-transient, non-community; and transient, non-community systems) shall monitor to determine compliance with the maximum contaminant level for nitrate in § 141.62.

(1) Community and non-transient, non-community water systems served by groundwater systems shall monitor annually beginning January 1, 1993; systems served by surface water shall

monitor quarterly beginning January 1, 1993.

(2) For community and non-transient, non-community water systems, the repeat monitoring frequency for groundwater systems shall be quarterly for at least one year following any one sample in which the concentration is ≥50 percent of the MCL. The State may allow a groundwater system to reduce the sampling frequency to annually after four consecutive quarterly samples are reliably and consistently less than the MCL.

(3) For community and non-transient, non-community water systems, the State may allow a surface water system to reduce the sampling frequency to annually if all analytical results from four consecutive quarters are <50 percent of the MCL. A surface water system shall return to quarterly monitoring if any one sample is ≥50 percent of the MCL.

(4) Each transient non-community water system shall monitor annually beginning January 1, 1993.

(5) After the initial round of quarterly sampling is completed, each community and non-transient non-community system which is monitoring annually shall take subsequent samples during the quarter(s) which previously resulted in the highest analytical result.

(e) All public water systems (community; non-transient, non-community; and transient, non-community systems) shall monitor to determine compliance with the maximum contaminant level for nitrite in § 141.62(b).

(1) All public water systems shall take one sample at each sampling point in the compliance period beginning January 1, 1993 and ending December 31, 1995.

(2) After the initial sample, systems where an analytical result for nitrite is <50 percent of the MCL shall monitor at the frequency specified by the State.

(3) For community, non-transient, non-community, and transient non-community water systems, the repeat monitoring frequency for any water system shall be quarterly for at least one year following any one sample in which the concentration is ≥50 percent of the MCL. The State may allow a system to reduce the sampling frequency to annually after determining the system is reliably and consistently less than the MCL.

(4) Systems which are monitoring annually shall take each subsequent sample during the quarter(s) which previously resulted in the highest analytical result.

(f) Confirmation samples:

(1) Where the results of sampling for asbestos, antimony, barium, beryllium, cadmium, chromium, cyanide, fluoride, mercury, nickel, selenium or thallium indicate an exceedance of the maximum contaminant level, the State may require that one additional sample be collected as soon as possible after the initial sample was taken (but not to exceed two weeks) at the same sampling point.

(2) Where nitrate or nitrite sampling results indicate an exceedance of the maximum contaminant level, the system shall take a confirmation sample within 24 hours of the system's receipt of notification of the analytical results of the first sample. Systems unable to comply with the 24-hour sampling requirement must immediately notify the consumers served by the area served by the public water system in accordance with § 141.32. Systems exercising this option must take and analyze a confirmation sample within two weeks of notification of the analytical results of the first sample.

(3) If a State-required confirmation sample is taken for any contaminant, then the results of the initial and confirmation sample shall be averaged. The resulting average shall be used to determine the system's compliance in accordance with paragraph (i) of this section. States have the discretion to delete results of obvious sampling errors.

(g) The State may require more frequent monitoring than specified in paragraphs (b), (c), (d) and (e) of this section or may require confirmation samples for positive and negative results at its discretion.

(h) Systems may apply to the State to conduct more frequent monitoring than the minimum monitoring frequencies specified in this section.

(i) Compliance with §§ 141.11 or 141.62(b) (as appropriate) shall be determined based on the analytical result(s) obtained at each sampling point.

(1) For systems which are conducting monitoring at a frequency greater than annual, compliance with the maximum contaminant levels for antimony, asbestos, barium, beryllium, cadmium, chromium, cyanide, fluoride, mercury, nickel, selenium or thallium is determined by a running annual average at any sampling point. If the average at any sampling point is greater than the MCL, then the system is out of compliance. If any one sample would cause the annual average to be exceeded, then the system is out of compliance immediately. Any sample below the method detection limit shall be calculated at zero for the purpose of determining the annual average.

(2) For systems which are monitoring annually, or less frequently, the system is out of compliance with the maximum contaminant levels for asbestos, antimony, barium, beryllium, cadmium, chromium, cyanide, fluoride, mercury, nickel, selenium or thallium if the level of a contaminant at any sampling point is greater than the MCL. If a confirmation sample is required by the State, the determination of compliance will be based on the average of the two samples.

(3) Compliance with the maximum contaminant levels for nitrate and nitrate is determined based on one sample if the levels of these contaminants are below the MCLs. If the levels of nitrate and/or nitrite exceed the MCLs in the initial sample, a confirmation sample is required in accordance with paragraph (f)(2) of this section, and compliance shall be determined based on the average of the initial and confirmation samples.

(4) If a public water system has a distribution system separable from other parts of the distribution system with no interconnections, the State may allow the system to give public notice to only the area served by that portion of the system which is out of compliance.

(j) Each public water system shall monitor at the time designated by the State during each compliance period.

(k) Inorganic analysis:

(1) Analysis for the following contaminants shall be conducted in accordance with the methods in the following table, or their equivalent as determined by EPA. Criteria for analyzing arsenic, barium, beryllium, cadmium, calcium, chromium, copper, lead, nickel, selenium, sodium, and thallium with digestion or directly without digestion, and other analytical test procedures are contained in *Technical Notes on Drinking Water Methods*, EPA–600/R–94–173, October 1994. This document also contains approved analytical test methods which remain available for compliance monitoring until July 1, 1996. These methods will not be available for use after July 1, 1996. This document is available from the National Technical Information Service, NTIS PB95–104766, U.S. Department of Commerce, 5285 Port Royal Road, Springfield, Virginia 22161. The toll-free number is 800–553–6847.

Contaminant	Methodology	EPA	ASTM [3]	SM [4]	Other
Antimony	ICP-Mass Spectrometry	[2] 200.8			
	Hydride-Atomic Absorption		D–3697–92		
	Atomic Absorption; Platform	[2] 200.9			
	Atomic Absorption; Furnace			3113B.	
Arsenic	Inductively Coupled Plasma	[2] 200.7		3120B.	
	ICP-Mass Spectrometry	[2] 200.8			
	Atomic Absorption; Platform	[2] 200.9			
	Atomic Absorption; Furnace		D–2972–93C	3113B.	
	Hydride Atomic Absorption		D–2972–93B	3114B.	
Asbestos	Transmission Electron Microscopy	[9] 100.1			
	Transmission Electron Microscopy	[10] 100.2			
Barium	Inductively Coupled Plasma	[2] 200.7		3120B.	
	ICP-Mass Spectrometry	[2] 200.8			
	Atomic Absorption; Direct			3111D.	
	Atomic Absorption; Furnace			3113B.	
Beryllium	Inductively Coupled Plasma	[2] 200.7		3120B.	
	ICP-Mass Spectrometry	[2] 200.8			
	Atomic Absorption; Platform	[2] 200.9			
	Atomic Absorption; Furnace		D–3645–93B	3113B.	
Cadmium	Inductively Coupled Plasma	[2] 200.7			
	ICP-Mass Spectrometry	[2] 200.8			
	Atomic Absorption; Platform	[2] 200.9			
	Atomic Absorption; Furnace			3113B.	
Chromium	Inductively Coupled Plasma	[2] 200.7		3120B.	
	ICP-Mass Spectrometry	[2] 200.8			
	Atomic Absorption; Platform	[2] 200.9			
	Atomic Absorption; Furnace			3113B.	
Cyanide	Manual Distillation followed by			4500–CN–C.	
	Spectrophotometric, Amenable		D2036–91B	4500CN–G.	
	Spectrophotometric Manual		D2036–91A	4500–CN–E	[5] I–3300–85
	Semi-automated	[6] 335.4			
	Selective Electrode			4500CN–F.	
Fluoride	Ion Chromatography	[6] 300.0	D4327–91	4110B.	
	Manual Distill.; Color. SPADNS			4500F–B,D.	
	Manual Electrode		D1179–93B	4500F–C.	
	Automated Electrode				[11] 380–75WE
	Automated Alizarin			4500F–E	[11] 129–71W
Mercury	Manual, Cold Vapor	[2] 245.1	D3223–91	3112B.	
	Automated, Cold Vapor	[1] 245.2			
	ICP-Mass Spectrometry	[2] 200.8			
Nickel	Inductively Coupled Plasma	[2] 200.7		3120B.	
	ICP-Mass Spectrometry	[2] 200.8			
	Atomic Absorption; Platform	[2] 200.9			
	Atomic Absorption; Direct			3111B.	
	Atomic Absorption; Furnace			3113B.	
Nitrate	Ion Chromatography	[6] 300.0	D4327–91	4110B	[8] B–1011
	Automated Cadmium Reduction	[6] 353.2	D3867–90A	4500–NO_3–F.	
	Ion Selective Electrode			4500–NO_3–D	[7] 601

Contaminant	Methodology	EPA	ASTM [3]	SM [4]	Other
	Manual Cadmium Reduction		D3867–90B	4500–NO_3–E.	
Nitrite	Ion Chromatography	[6] 300.0	D4327–91	4110B	[8] B–1011
	Automated Cadmium Reduction	[6] 353.2	D3867–90A	4500–NO_3–F.	
	Manual Cadmium Reduction		D3867–90B	4500–NO_3–E.	
	Spectrophotometric			4500–NO_2–B.	
Selenium	Hydride-Atomic Absorption		D3859–93A	3114B.	
	ICP-Mass Spectrometry	[2] 200.8			
	Atomic Absorption; Platform	[2] 200.9			
	Atomic Absorption; Furnace		D3859–93B	3113B.	
Thallium	ICP-Mass Spectrometry	[2] 200.8			
	Atomic Absorption; Platform	[2] 200.9			
Lead	Atomic absorption; furnace		D3559–90D	3113B.	
	ICP-Mass spectrometry	[2] 200.8			
	Atomic absorption; platform	[2] 200.9			
Copper	Atomic absorption; furnace		D1688–90C	3113B.	
	Atomic absorption; direct aspiration		D1688–90A	3111B.	
	ICP	[2] 200.7		3120B.	
	ICP-Mass spectrometry	[2] 200.8			
	Atomic absorption; platform	[2] 200.9			
pH	Electrometric	[1] 150.1	D1293–84	4500–H^+–B.	
		[1] 150.2			
Conductivity	Conductance		D1125–91A	2510B.	
Calcium	EDTA titrimetric		D511–93A	3500–Ca–D.	
	Atomic absorption; direct aspiration		D511–93B	3111B.	
	Inductively-coupled plasma	[2] 200.7		3120B.	
Alkalinity	Titrimetric		D1067–92B	2320B.	
	Electrometric titration				[5] I–1030–85
Orthophosphate [12]	Colorimetric, automated, ascorbic acid	[6] 365.1		4500–P–F.	
	Colorimetric, ascorbic acid, single reagent		D515–88A	4500–P–E.	
	Colorimetric, phosphomolybdate;				[5] I–1601–85
	automated-segmented flow;				[5] I–2601–90
	automated discrete				[5] I–2598–85
	Ion Chromatography	[6] 300.0	D4327–91	4110.	
Silica	Colorimetric, molybdate blue;				[5] I–1700–85
	automated-segmented flow				[5] I–2700–85
	Colorimetric		D859–88		
	Molybdosilicate			4500–Si–D.	
	Heteropoly blue			4500–Si–E.	
	Automated method for molybdate-reactive silica			4500–Si–F.	
	Inductively-coupled plasma	[2] 200.7		3120B.	
Temperature	Thermometric			2550.	
Sodium	Inductively-coupled plasma	[2] 200.7			
	Atomic Absorption; direct aspiration			3111B.	

FOOTNOTES:

[1] Methods 150.1, 150.2 and 245.2 are available from US EPA, EMSL, Cincinnati, OH 45268. The identical methods were formerly in ''Methods for Chemical Analysis of Water and Wastes'', EPA–600/4–79–020, March 1983, which is available at NTIS, PB84–128677.

[2] ''Methods for the Determination of Metals in Environmental Samples—Supplement I'', EPA–600/R–94–111, May 1994. Available at NTIS, PB 94–184942.

[3] The procedures shall be done in accordance with the *Annual Book of ASTM Standards,* 1994, Vols. 11.01 and 11.02, American Society for Testing and Materials. This incorporation by reference was approved by the Director of the Federal Register in accordance with 5 U.S.C. 552(a) and 1 CFR Part 51. Copies may be obtained from the American Society for Testing and Materials, 1916 Race Street, Philadelphia, PA 19103. Copies may be inspected at EPA's Drinking Water Docket, 401 M Street, SW., Washington, DC 20460; or at the Office of the Federal Register, 800 North Capitol Street, NW., Suite 700, Washington, DC.

[4] The procedures shall be done in accordance with the 18th edition of *Standard Methods for the Examination of Water and Wastewater,* 1992, American Public Health Association. This incorporation by reference was approved by the Director of the Federal Register in accordance with 5 U.S.C. 552(a) and 1 CFR Part 51. Copies may be obtained from the American Public Health Association, 1015 Fifteenth Street NW, Washington, DC 20005. Copies may be inspected at EPA's Drinking Water Docket, 401 M Street, SW., Washington, DC 20460; or at the Office of the Federal Register, 800 North Capitol Street, NW., Suite 700, Washington, DC.

[5] Available from Books and Open-File Reports Section, U.S. Geological Survey, Federal Center, Box 25425, Denver, CO 80225–0425.

[6] ''Methods for the Determination of Inorganic Substances in Environmental Samples'', EPA–600/R–93–100, August 1993. Available at NTIS, PB94–121811.

[7] The procedure shall be done in accordance with the Technical Bulletin 601 ''Standard Method of Test for Nitrate in Drinking Water'', July 1994, PN 221890–001, Analytical Technology, Inc. This incorporation by reference was approved by the Director of the Federal Register in accordance with 5 U.S.C. 552(a) and 1 CFR Part 51. Copies may be obtained from ATI Orion, 529 Main Street, Boston, MA 02129. Copies may be inspected at EPA's Drinking Water Docket, 401 M Street, SW., Washington, DC 20460; or at the Office of the Federal Register, 800 North Capitol Street, NW., Suite 700, Washington, DC.

[8] Method B–1011, ''Waters Test Method for Determination of Nitrite/Nitrate in Water Using Single Column Ion Chromatography'', Millipore Corporation, Waters Chromatography Division, 34 Maple Street, Milford, MA 01757.

[9] Method 100.1, ''Analytical Method For Determination of Asbestos Fibers in Water'', EPA–600/4–83–043, EPA, September 1983. Available at NTIS, PB83–260471.

[10] Method 100.2, ''Determination Of Asbestos Structure Over 10-µm In Length In Drinking Water'', EPA–600/R–94–134, June 1994. Available at NTIS, PB94–201902.

[11] The procedures shall be done in accordance with the Industrial Method No. 129–71W, ''Fluoride in Water and Wastewater'', December 1972, and Method No. 380–75WE, ''Fluoride in Water and Wastewater'', February 1976, Technicon Industrial Systems. This incorporation by reference was approved by the Director of the Federal Register in accordance with 5 U.S.C. 552(a) and 1 CFR Part 51. Copies may be obtained from the Technicon Industrial Systems, Tarrytown, NY 10591. Copies may be inspected at EPA's Drinking Water Docket, 401 M Street, SW., Washington, DC 20460; or at the Office of Federal Register, 800 Capitol Street, NW., Suite 700, Washington, DC.

[12] Unfiltered, no digestion or hydrolysis.

(2) Sample collection for antimony, asbestos, barium, beryllium, cadmium, chromium, cyanide, fluoride, mercury, nickel, nitrate, nitrite, selenium, and thallium under this section shall be conducted using the sample preservation, container, and maximum holding time procedures specified in the table below:

Contaminant	Preservative [1]	Container [2]	Time [3]
Antimony	Conc HNO_3 to pH <2	P or G	6 months.
Asbestos	Cool, 4°C	P or G	
Barium	Conc HNO_3 to pH <2	P or G	6 months.
Beryllium	Conc HNO_3 to pH <2	P or G	6 months.
Cadmium	Conc HNO_3 to pH <2	P or G	6 months.
Chromium	Conc HNO_3 to pH <2	P or G	6 months.
Cyanide	Cool, 4°C, NaOH to pH>12 [3]	P or G	14 days
Fluoride	None	P or G	1 month.
Mercury	Conc HNO_3 to pH <2	P or G	28 days.
Nickel	Conc HNO_3 to pH <2	P or G	6 months.
Nitrate			
Chlorinated	Cool, 4°C	P or G	28 days.
Non-chlorinated	Conc H_2SO_4 to pH <2	P or G	14 days.
Nitrite	Cool, 4°C	P or G	48 hours.
Selenium	Conc HNO_3 to pH <2	P or G	6 months.
Thallium	Conc HNO_3 to pH <2	P or G	6 months.

[1] P=plastic, hard or soft; G=glass, hard or soft.
[2] In all cases, samples should be analyzed as soon after collection as possible.
[3] See method(s) for the information for preservation.

(3) Analysis under this section shall only be conducted by laboratories that have been certified by EPA or the State. Laboratories may conduct sample analysis under provisional certification until January 1, 1996. To receive certification to conduct analyses for antimony, asbestos, barium, beryllium, cadmium, chromium, cyanide, fluoride, mercury, nickel, nitrate, nitrite and selenium and thallium, the laboratory must:

(i) Analyze Performance Evaluation samples which include those substances provided by EPA Environmental Monitoring Systems Laboratory or equivalent samples provided by the State.

(ii) Achieve quantitative results on the analyses that are within the following acceptance limits:

Contaminant	Acceptance limit
Antimony	±30 at ≥0.006 mg/1
Asbestos	2 standard deviations based on study statistics.
Barium	±15% at ≥0.15 mg/1
Beryllium	±15% at ≥0.001 mg/1
Cadmium	±20% at ≥0.002 mg/1
Chromium	±15% at ≥0.01 mg/1
Cyanide	±25% at ≥0.1 mg/1
Fluoride	±10% at ≥1 to 10 mg/1
Mercury	±30% at ≥0.0005 mg/1
Nickel	±15% at ≥0.01 mg/1
Nitrate	±10% at ≥0.4 mg/1
Nitrite	±15% at ≥0.4 mg/1
Selenium	±20% at ≥0.01 mg/1
Thallium	±30% at ≥0.002 mg/1

(l) Analyses for the purpose of determining compliance with § 141.11 shall be conducted using the requirements specified in paragraphs (l) through (q) of this section.

(1) Analyses for all community water systems utilizing surface water sources shall be completed by June 24, 1978. These analyses shall be repeated at yearly intervals.

(2) Analyses for all community water systems utilizing only ground water sources shall be completed by June 24, 1979. These analyses shall be repeated at three-year intervals.

(3) For non-community water systems, whether supplied by surface or ground sources, analyses for nitrate shall be completed by December 24, 1980. These analyses shall be repeated at intervals determined by the State.

(4) The State has the authority to determine compliance or initiate enforcement action based upon analytical results and other information compiled by their sanctioned representatives and agencies.

(m) If the result of an analysis made under paragraph (l) of this section indicates that the level of any contaminant listed in § 141.11 exceeds the maximum contaminant level, the supplier of the water shall report to the State within 7 days and initiate three additional analyses at the same sampling point within one month.

(n) When the average of four analyses made pursuant to paragraph (m) of this section, rounded to the same number of significant figures as the maximum contaminant level for the substance in question, exceeds the maximum contaminant level, the supplier of water shall notify the State pursuant to § 141.31 and give notice to the public pursuant to § 141.32. Monitoring after public notification shall be at a frequency designated by the State and shall continue until the maximum contaminant level has not been exceeded in two successive samples or until a monitoring schedule as a condition to a variance, exemption or enforcement action shall become effective.

(o) The provisions of paragraphs (m) and (n) of this section notwithstanding, compliance with the maximum contaminant level for nitrate shall be determined on the basis of the mean of two analyses. When a level exceeding the maximum contaminant level for nitrate is found, a second analysis shall be initiated within 24 hours, and if the mean of the two analyses exceeds the maximum contaminant level, the supplier of water shall report his findings to the State pursuant to § 141.31 and shall notify the public pursuant to § 141.32.

(p) For the initial analyses required by paragraph (l) (1), (2) or (3) of this section, data for surface waters acquired within one year prior to the effective date and data for ground waters acquired within 3 years prior to the effective date of this part may be substituted at the discretion of the State.

(q) [Reserved]

[56 FR 3579, Jan. 30, 1991, as amended at 56 FR 30274, July 1, 1991; 57 FR 31838, July 17, 1992; 59 FR 34322, July 1, 1994; 59 FR 62466, Dec. 5, 1994; 60 FR 33932, 34085, June 29, 1995]

§ 141.24 Organic chemicals other than total trihalomethanes, sampling and analytical requirements.

(a)–(d) [Reserved]

(e) Analyses for the contaminants in this section shall be conducted using the following EPA methods or their equivalent as approved by EPA. Methods 502.2, 505, 507, 508, 508A, 515.1 and 531.1 are in *Methods for the Determination of Organic Compounds in Drinking Water,* EPA–600/4–88–039, December 1988, Revised, July 1991. Methods 506, 547, 550, 550.1 and 551 are in *Methods for the Determination of Organic Compounds in Drinking Water—Supplement I,* EPA–600–4–90–020, July 1990. Methods 515.2, 524.2, 548.1, 549.1, 552.1 and 555 are in *Methods for the Determination of Organic Compounds in Drinking Water—Supplement II,* EPA–600/R–92–129, August 1992. Method 1613 is titled ''Tetra-through Octa-Chlorinated Dioxins and Furans by Isotope-Dilution HRGC/HRMS'', EPA–821–B–94–005, October 1994. These documents are available from the National Technical Information Service, NTIS PB91–231480, PB91–146027, PB92–207703 and PB95–104774, U.S. Department of Commerce, 5285 Port Royal Road, Springfield, Virginia 22161. The toll-free number is 800–553–6847. Method 6651 shall be followed in accordance with the 18th edition of *Standard Methods for the Examination of Water and Wastewater,* 1992, American Public Health Association. This incorporation by reference was approved by the Director of the Federal Register in accordance with 5 U.S.C. 552(a) and 1 CFR part 51. Copies may be obtained from the American Public Health Association, 1015 Fifteenth Street NW., Washington, DC 20005. Copies may be inspected at EPA's Drinking Water Docket, 401 M Street, SW., Washington, DC 20460; or at the Office of the Federal Register, 800 North Capitol Street, NW., Suite 700, Washington, DC. Method 6610 shall be followed in accordance with the *Supplement to the 18th edition of Standard Methods for the Examination of Water and Wastewater,* 1994, American Public Health Association. This incorporation by reference was approved by the Director of the Federal Register in accordance with 5 U.S.C. 552(a) and 1 CFR part 51. Copies may be obtained from the American Public Health Association, 1015 Fifteenth Street NW., Washington, DC 20005. Copies may be inspected at EPA's Drinking Water Docket, 401 M Street, SW., Washington, DC 20460; or at the Office of the Federal Register, 800 North Capitol Street, NW., Suite 700, Washington, DC. Other analytical test procedures are contained in *Technical Notes on Drinking Water Methods,* EPA–600/R–94–173, October 1994, NTIS PB95–104766. This document also contains approved analytical methods which remain available for compliance monitoring until July 1, 1996. These methods will not be available for use after July 1, 1996. EPA Methods 504.1, 508.1 and 525.2 are available from US EPA EMSL, Cincinnati, OH 45268. The phone number is 513–569–7586.

Contaminant	Method
Benzene	502.2, 524.2.
Carbon tetrachloride	502.2, 524.2, 551.
Chlorobenzene	502.2, 524.2.
1,2-Dichlorobenzene	502.2, 524.2.
1,4-Dichlorobenzene	502.2, 524.2.
1,2-Dichloroethane	502.2, 524.2.
cis-Dichloroethylene	502.2, 524.2.
trans-Dichloroethylene	502.2, 524.2.
Dichloromethane	502.2, 524.2.
1,2-Dichloropropane	502.2, 524.2.
Ethylbenzene	502.2, 524.2.
Styrene	502.2, 524.2.
Tetrachloroethylene	502.2, 524.2, 551.
1,1,1-Trichloroethane	502.2, 524.2, 551.
Trichloroethylene	502.2, 524.2, 551.
Toluene	502.2, 524.2.
1,2,4-Trichlorobenzene	502.2, 524.2.
1,1-Dichloroethylene	502.2, 524.2.
1,1,2-Trichloroethane	502.2, 524.2.
Vinyl chloride	502.2, 524.2.
Xylenes (total)	502.2, 524.2.
2,3,7,8-TCDD (dioxin)	1613.
2,4-D	515.2, 555, 515.1.
2,4,5-TP (Silvex)	515.2, 555, 515.1.
Alachlor	505 [1], 507, 525.2, 508.1.
Atrazine	505 [1], 507, 525.2, 508.1.
Benzo(a)pyrene	525.2, 550, 550.1.
Carbofuran	531.1, 6610.
Chlordane	505, 508, 525.2, 508.1.
Dalapon	552.1, 515.1.
Di(2-ethylhexyl) adipate	506, 525.2.
Di(2-ethylhexyl) phthalate	506, 525.2.
Dibromochloropropane (DBCP).	504.1, 551.
Dinoseb	515.2, 555, 515.1.
Diquat	549.1.
Endothall	548.1.
Endrin	505, 508, 525.2, 508.1.
Ethylene dibromide (EDB)	504.1, 551.
Glyphosate	547, 6651.
Heptachlor	505, 508, 525.2, 508.1.
Heptachlor Epoxide	505, 508, 525.2, 508.1.
Hexachlorobenzene	505, 508, 525.2, 508.1.
Hexachlorocyclopentadiene	505, 525.2, 508, 508.1.
Lindane	505, 508, 525.2, 508.1.
Methoxychlor	505, 508, 525.2, 508.1.
Oxamyl	531.1, 6610.

Contaminant	Method
PCBs[2] (as decachlorobiphenyl).	508A.
(as Aroclors)	505, 508.
Pentachlorophenol	515.2, 525.2, 555, 515.1.
Picloram	515.2, 555, 515.1.
Simazine	505[1], 507, 525.2, 508.1.
Toxaphene	505, 508, 525.2.
Total Trihalomethanes	502.2, 524.2, 551.

[1] A nitrogen-phosphorous detector should be substituted for the electron capture detector in Method 505 (or another approved method should be used) to determine alachlor, atrazine and simazine, if lower detection limits are required.

[2] PCBs are qualitatively identified as Aroclors and measured for compliance purposes as decachlorobiphenyl.

(f) Beginning with the initial compliance period, analysis of the contaminants listed in § 141.61(a) (1) through (21) for the purpose of determining compliance with the maximum contaminant level shall be conducted as follows:

(1) Groundwater systems shall take a minimum of one sample at every entry point to the distribution system which is representative of each well after treatment (hereafter called a sampling point). Each sample must be taken at the same sampling point unless conditions make another sampling point more representative of each source, treatment plant, or within the distribution system.

(2) Surface water systems (or combined surface/ground) shall take a minimum of one sample at points in the distribution system that are representative of each source or at each entry point to the distribution system after treatment (hereafter called a sampling point). Each sample must be taken at the same sampling point unless conditions make another sampling point more representative of each source, treatment plant, or within the distribution system.

(3) If the system draws water from more than one source and the sources are combined before distribution, the system must sample at an entry point to the distribution system during periods of normal operating conditions (i.e., when water representative of all sources is being used).

(4) Each community and non-transient non-community water system shall take four consecutive quarterly samples for each contaminant listed in § 141.61(a) (2) through (21) during each compliance period, beginning in the initial compliance period.

(5) If the initial monitoring for contaminants listed in § 141.61(a) (1) through (8) and the monitoring for the contaminants listed in § 141.61(a) (9) through (21) as allowed in paragraph (f)(18) has been completed by December 31, 1992, and the system did not detect any contaminant listed in § 141.61(a) (1) through (21), then each ground and surface water system shall take one sample annually beginning with the initial compliance period.

(6) After a minimum of three years of annual sampling, the State may allow groundwater systems with no previous detection of any contaiminant listed in § 141.61(a) to take one sample during each compliance period.

(7) Each community and non-transient non–community ground water system which does not detect a contaminant listed in § 141.61(a) (1) through (21) may apply to the State for a waiver from the requirements of paragraphs (f)(5) and (f)(6) of this section after completing the initial monitoring. (For purposes of this section, detection is defined as ≥0.0005 mg/l.) A waiver shall be effective for no more than six years (two compliance periods). States may also issue waivers to small systems for the initial round of monitoring for 1,2,4-trichlorobenzene.

(8) A State may grant a waiver after evaluating the following factor(s):

(i) Knowledge of previous use (including transport, storage, or disposal) of the contaminant within the watershed or zone of influence of the system. If a determination by the State reveals no previous use of the contaminant within the watershed or zone of influence, a waiver may be granted.

(ii) If previous use of the contaminant is unknown or it has been used previously, then the following factors shall be used to determine whether a waiver is granted.

(A) Previous analytical results.

(B) The proximity of the system to a potential point or non-point source of contamination. Point sources include spills and leaks of chemicals at or near a water treatment facility or at manufacturing, distribution, or storage facilities, or from hazardous and municipal waste landfills and other waste handling or treatment facilities.

(C) The environmental persistence and transport of the contaminants.

(D) The number of persons served by the public water system and the proximity of a smaller system to a larger system.

(E) How well the water source is protected against contamination, such as whether it is a surface or groundwater system. Groundwater systems must consider factors such as depth of the well, the type of soil, and wellhead protection. Surface water systems must consider watershed protection.

(9) As a condition of the waiver a groundwater system must take one sample at each sampling point during the time the waiver is effective (i.e., one sample during two compliance periods or six years) and update its vulnerability assessment considering the factors listed in paragraph (f)(8) of this section. Based on this vulnerability assessment the State must reconfirm that the system is non-vulnerable. If the State does not make this reconfirmation within three years of the initial determination, then the waiver is invalidated and the system is required to sample annually as specified in paragraph (5) of this section.

(10) Each community and non-transient non–community surface water system which does not detect a contaminant listed in §141.61(a) (1) through (21) may apply to the State for a waiver from the requirements of (f)(5) of this section after completing the initial monitoring. Composite samples from a maximum of five sampling points are allowed, provided that the detection limit of the method used for analysis is less than one-fifth of the MCL. Systems meeting this criterion must be determined by the State to be non-vulnerable based on a vulnerability assessment during each compliance period. Each system receiving a waiver shall sample at the frequency specified by the State (if any).

(11) If a contaminant listed in §141.61(a) (2) through (21) is detected at a level exceeding 0.0005 mg/l in any sample, then:

(i) The system must monitor quarterly at each sampling point which resulted in a detection.

(ii) The State may decrease the quarterly monitoring requirement specified in paragraph (f)(11)(i) of this section provided it has determined that the system is reliably and consistently below the maximum contaminant level. In no case shall the State make this determination unless a groundwater system takes a minimum of two quarterly samples and a surface water system takes a minimum of four quarterly samples.

(iii) If the State determines that the system is reliably and consistently below the MCL, the State may allow the system to monitor annually. Systems which monitor annually must monitor during the quarter(s) which previously yielded the highest analytical result.

(iv) Systems which have three consecutive annual samples with no detection of a contaminant may apply to the State for a waiver as specified in paragraph (f)(7) of this section.

(v) Groundwater systems which have detected one or more of the following two-carbon organic compounds: trichloroethylene, tetrachloroethylene, 1,2-dichloroethane, 1,1,1-trichloroethane, cis-1,2-dichloroethylene, trans-1,2-dichloroethylene, or 1,1-dichloroethylene shall monitor quarterly for vinyl chloride. A vinyl chloride sample shall be taken at each sampling point at which one or more of the two-carbon organic compounds was detected. If the results of the first analysis do not detect vinyl chloride, the State may reduce the quarterly monitoring frequency of vinyl chloride monitoring to one sample during each compliance period. Surface water systems are required to monitor for vinyl chloride as specified by the State.

(12) Systems which violate the requirements of §141.61(a) (1) through (21), as determined by paragraph (f)(15) of this section, must monitor quarterly. After a minimum of four consecutive quarterly samples which show the system is in compliance as specified in paragraph (f)(15) of this section the system and the State determines that the system is reliably and consistently below the maximum contaminant level, the system may monitor at the frequency and times specified in paragraph (f)(11)(iii) of this section.

(13) The State may require a confirmation sample for positive or negative results. If a confirmation sample is required by the State, the result must be averaged with the first sampling result and the average is used for the compliance determination as specified by paragraph (f)(15). States have discretion to delete results of obvious sampling errors from this calculation.

(14) The State may reduce the total number of samples a system must analyze by allowing the use of compositing. Composite samples from a maximum of five sampling points are allowed, provided that the detection limit of the method used for analysis is less than one-fifth of the MCL. Compositing of samples must be done in the laboratory and analyzed within 14 days of sample collection.

(i) If the concentration in the composite sample is greater than or equal to 0.0005 mg/l for any contaminant listed in § 141.61(a), then a follow-up sample must be taken within 14 days at each sampling point included in the composite, and be analyzed for that contaminant.

(ii) If duplicates of the original sample taken from each sampling point used in the composite are available, the system may use these instead of resampling. The duplicate must be analyzed and the results reported to the State within 14 days of collection.

(iii) If the population served by the system is > 3,300 persons, then compositing may only be permitted by the State at sampling points within a single system. In systems serving ≤ 3,300 persons, the State may permit compositing among different systems provided the 5-sample limit is maintained.

(iv) Compositing samples prior to GC analysis.

(A) Add 5 ml or equal larger amounts of each sample (up to 5 samples are allowed) to a 25 ml glass syringe. Special precautions must be made to maintain zero headspace in the syringe.

(B) The samples must be cooled at 4°C during this step to minimize volatilization losses.

(C) Mix well and draw out a 5-ml aliquot for analysis.

(D) Follow sample introduction, purging, and desorption steps described in the method.

(E) If less than five samples are used for compositing, a proportionately small syringe may be used.

(v) Compositing samples prior to GC/MS analysis.

(A) Inject 5-ml or equal larger amounts of each aqueous sample (up to 5 samples are allowed) into a 25-ml purging device using the sample introduction technique described in the method.

(B) The total volume of the sample in the purging device must be 25 ml.

(C) Purge and desorb as described in the method.

(15) Compliance with § 141.61(a) (1) through (21) shall be determined based on the analytical results obtained at each sampling point.

(i) For systems which are conducting monitoring at a frequency greater than annual, compliance is determined by a running annual average of all samples taken at each sampling point. If the annual average of any sampling point is greater than the MCL, then the system is out of compliance. If the initial sample or a subsequent sample would cause the annual average to be exceeded, then the system is out of compliance immediately.

(ii) If monitoring is conducted annually, or less frequently, the system is out of compliance if the level of a contaminant at any sampling point is greater than the MCL. If a confirmation sample is required by the State, the determination of compliance will be based on the average of two samples.

(iii) If a public water system has a distribution system separable from other parts of the distribution system with no interconnections, the State may allow the system to give public notice to only that area served by that portion of the system which is out of compliance.

(16) [Reserved]

(17) Analysis under this section shall only be conducted by laboratories that are certified by EPA or the State according to the following conditions (laboratories may conduct sample analysis under provisional certification until January 1, 1996):

(i) To receive certification to conduct analyses for the contaminants in § 141.61(a) (2) through (21) the laboratory must:

(A) Analyze Performance Evaluation samples which include these substances provided by EPA Environmental Monitoring Systems Laboratory or equivalent samples provided by the State.

(B) Achieve the quantitative acceptance limits under paragraphs (f)(17)(i)(C) and (D) of this section for at least 80 percent of the regulated organic chemicals listed in § 141.61(a) (2) through (21).

(C) Achieve quantitative results on the analyses performed under paragraph (f)(17)(i)(A) of this section that are within ±20% of the actual amount of the substances in the Performance Evaluation sample when the actual amount is greater than or equal to 0.010 mg/l.

(D) Achieve quantitative results on the analyses performed under paragraph (f)(17)(i)(A) of this section that are within ±40 percent of the actual amount of the substances in the Performance Evaluation sample when the actual amount is less than 0.010 mg/l.

(E) Achieve a method detection limit of 0.0005 mg/l, according to the procedures in appendix B of part 136.

(ii) To receive certification for vinyl chloride, the laboratory must:

(A) Analyze Performance Evaluation samples provided by EPA Environmental Monitoring Systems Laboratory or equivalent samples provided by the State.

(B) Achieve quantitative results on the analyses performed under paragraph (f)(17)(ii)(A) of this section that are within ±40 percent of the actual amount of vinyl chloride in the Performance Evaluation sample.

(C) Achieve a method detection limit of 0.0005 mg/l, according to the procedures in appendix B of part 136.

(D) Obtain certification for the contaminants listed in § 141.61(a)(2) through (21).

(18) States may allow the use of monitoring data collected after January 1, 1988, required under section 1445 of the Act for purposes of initial monitoring compliance. If the data are generally consistent with the other requirements of this section, the State may use these data (i.e., a single sample rather than four quarterly samples) to satisfy the initial monitoring requirement of paragraph (f)(4) of this section. Systems which use grandfathered samples and did not detect any contaminant listed § 141.61(a)(2) through (21) shall begin monitoring annually in accordance with paragraph (f)(5) of this section beginning with the initial compliance period.

(19) States may increase required monitoring where necessary to detect variations within the system.

(20) Each certified laboratory must determine the method detection limit (MDL), as defined in appendix B to part 136, at which it is capable of detecting VOCs. The acceptable MDL is 0.0005 mg/l. This concentration is the detection concentration for purposes of this section.

(21) Each public water system shall monitor at the time designated by the State within each compliance period.

(g) [Reserved]

(h) Analysis of the contaminants listed in § 141.61(c) for the purposes of determining compliance with the maximum contaminant level shall be conducted as follows:[7]

(1) Groundwater systems shall take a minimum of one sample at every entry point to the distribution system which is representative of each well after treatment (hereafter called a sampling point). Each sample must be taken at the same sampling point unless conditions make another sampling point more representative of each source or treatment plant.

(2) Surface water systems shall take a minimum of one sample at points in the distribution system that are representative of each source or at each entry point to the distribution system after treatment (hereafter called a sampling point). Each sample must be taken at the same sampling point unless conditions make another sampling point more representative of each source or treatment plant.

NOTE: For purposes of this paragraph, surface water systems include systems

[7] Monitoring for the contaminants aldicarb, aldicarb sulfoxide, and aldicarb sulfone shall be conducted in accordance with § 141.40.

with a combination of surface and ground sources.

(3) If the system draws water from more than one source and the sources are combined before distribution, the system must sample at an entry point to the distribution system during periods of normal operating conditions (i.e., when water representative of all sources is being used).

(4) Monitoring frequency:

(i) Each community and non-transient non-community water system shall take four consecutive quarterly samples for each contaminant listed in § 141.61(c) during each compliance period beginning with the initial compliance period.

(ii) Systems serving more than 3,300 persons which do not detect a contaminant in the initial compliance period may reduce the sampling frequency to a minimum of two quarterly samples in one year during each repeat compliance period.

(iii) Systems serving less than or equal to 3,300 persons which do not detect a contaminant in the initial compliance period may reduce the sampling frequency to a minimum of one sample during each repeat compliance period.

(5) Each community and non-transient water system may apply to the State for a waiver from the requirement of paragraph (h)(4) of this section. A system must reapply for a waiver for each compliance period.

(6) A State may grant a waiver after evaluating the following factor(s): Knowledge of previous use (including transport, storage, or disposal) of the contaminant within the watershed or zone of influence of the system. If a determination by the State reveals no previous use of the contaminant within the watershed or zone of influence, a waiver may be granted. If previous use of the contaminant is unknown or it has been used previously, then the following factors shall be used to determine whether a waiver is granted.

(i) Previous analytical results.

(ii) The proximity of the system to a potential point or non-point source of contamination. Point sources include spills and leaks of chemicals at or near a water treatment facility or at manufacturing, distribution, or storage facilities, or from hazardous and municipal waste landfills and other waste handling or treatment facilities. Non-point sources include the use of pesticides to control insect and weed pests on agricultural areas, forest lands, home and gardens, and other land application uses.

(iii) The environmental persistence and transport of the pesticide or PCBs.

(iv) How well the water source is protected against contamination due to such factors as depth of the well and the type of soil and the integrity of the well casing.

(v) Elevated nitrate levels at the water supply source.

(vi) Use of PCBs in equipment used in the production, storage, or distribution of water (i.e., PCBs used in pumps, transformers, etc.).

(7) If an organic contaminant listed in § 141.61(c) is detected (as defined by paragraph (h)(18) of this section) in any sample, then:

(i) Each system must monitor quarterly at each sampling point which resulted in a detection.

(ii) The State may decrease the quarterly monitoring requirement specified in paragraph (h)(7)(i) of this section provided it has determined that the system is reliably and consistently below the maximum contaminant level. In no case shall the State make this determination unless a groundwater system takes a minimum of two quarterly samples and a surface water system takes a minimum of four quarterly samples.

(iii) After the State determines the system is reliably and consistently below the maximum contaminant level the State may allow the system to monitor annually. Systems which monitor annually must monitor during the quarter that previously yielded the highest analytical result.

(iv) Systems which have 3 consecutive annual samples with no detection of a contaminant may apply to the State for a waiver as specified in paragraph (h)(6) of this section.

(v) If monitoring results in detection of one or more of certain related contaminants (aldicarb, aldicarb sulfone,

aldicarb sulfoxide and heptachlor, heptachlor epoxide), then subsequent monitoring shall analyze for all related contaminants.

(8) Systems which violate the requirements of § 141.61(c) as determined by paragraph (h)(11) of this section must monitor quarterly. After a minimum of four quarterly samples show the system is in compliance and the State determines the system is reliably and consistently below the MCL, as specified in paragraph (h)(11) of this section, the system shall monitor at the frequency specified in paragraph (h)(7)(iii) of this section.

(9) The State may require a confirmation sample for positive or negative results. If a confirmation sample is required by the State, the result must be averaged with the first sampling result and the average used for the compliance determination as specified by paragraph (h)(11) of this section. States have discretion to delete results of obvious sampling errors from this calculation.

(10) The State may reduce the total number of samples a system must analyze by allowing the use of compositing. Composite samples from a maximum of five sampling points are allowed, provided that the detection limit of the method used for analysis is less than one-fifth of the MCL. Compositing of samples must be done in the laboratory and analyzed within 14 days of sample collection.

(i) If the concentration in the composite sample detects one or more contaminants listed in § 141.61(c), then a follow-up sample must be taken within 14 days at each sampling point included in the composite, and be analyzed for that contaminant.

(ii) If duplicates of the original sample taken from each sampling point used in the composite are available, the system may use these duplicates instead of resampling. The duplicate must be analyzed and the results reported to the State within 14 days of collection.

(iii) If the population served by the system is >3,300 persons, then compositing may only be permitted by the State at sampling points within a single system. In systems serving ≤ 3,300 persons, the State may permit compositing among different systems provided the 5-sample limit is maintained.

(11) Compliance with § 141.61(c) shall be determined based on the analytical results obtained at each sampling point.

(i) For systems which are conducting monitoring at a frequency greater than annual, compliance is determined by a running annual average of all samples taken at each sampling point. If the annual average of any sampling point is greater than the MCL, then the system is out of compliance. If the initial sample or a subsequent sample would cause the annual average to be exceeded, then the system is out of compliance immediately. Any samples below the detection limit shall be calculated as zero for purposes of determining the annual average.

(ii) If monitoring is conducted annually, or less frequently, the system is out of compliance if the level of a contaminant at any sampling point is greater than the MCL. If a confirmation sample is required by the State, the determination of compliance will be based on the average of two samples.

(iii) If a public water system has a distribution system separable from other parts of the distribution system with no interconnections, the State may allow the system to give public notice to only that portion of the system which is out of compliance.

(12) [Reserved]

(13) Analysis for PCBs shall be conducted as follows using the methods in paragraph (e) of this section:

(i) Each system which monitors for PCBs shall analyze each sample using either Method 505 or Method 508.

(ii) If PCBs (as one of seven Aroclors) are detected (as designated in this paragraph) in any sample analyzed using Method 505 or 508, the system shall reanalyze the sample using Method 508A to quantitate PCBs (as decachlorobiphenyl).

Aroclor	Detection limit (mg/l)
1016	0.00008
1221	0.02
1232	0.0005
1242	0.0003
1248	0.0001
1254	0.0001

Aroclor	Detection limit (mg/l)
1260	0.0002

(iii) Compliance with the PCB MCL shall be determined based upon the quantitative results of analyses using Method 508A.

(14) If monitoring data collected after January 1, 1990, are generally consistent with the requirements of § 141.24(h), then the State may allow systems to use that data to satisfy the monitoring requirement for the initial compliance period beginning January 1, 1993.

(15) The State may increase the required monitoring frequency, where necessary, to detect variations within the system (e.g., fluctuations in concentration due to seasonal use, changes in water source).

(16) The State has the authority to determine compliance or initiate enforcement action based upon analytical results and other information compiled by their sanctioned representatives and agencies.

(17) Each public water system shall monitor at the time designated by the State within each compliance period.

(18) Detection as used in this paragraph shall be defined as greater than or equal to the following concentrations for each contaminant.

Contaminant	Detection limit (mg/l)
Alachlor	.0002
Aldicarb	.0005
Aldicarb sulfoxide	.0005
Aldicarb sulfone	.0008
Atrazine	.0001
Benzo[a]pyrene	.00002
Carbofuran	.0009
Chlordane	.0002
Dalapon	.001
1,2–Dibromo–3–chloropropane (DBCP)	.00002
Di (2-ethylhexyl) adipate	.0006
Di (2-ethylhexyl) phthalate	.0006
Dinoseb	.0002
Diquat	.0004
2,4-D	.0001
Endothall	.009
Endrin	.00001
Ethylene dibromide (EDB)	.00001
Glyphosate	.006
Heptachlor	.00004
Heptachlor epoxide	.00002
Hexachlorobenzene	.0001
Hexachlorocyclopentadiene	.0001
Lindane	.00002
Methoxychlor	.0001
Oxamyl	.002
Picloram	.0001
Polychlorinated biphenyls (PCBs) (as decachlorobiphenyl)	.0001
Pentachlorophenol	.00004
Simazine	.00007
Toxaphene	.001
2,3,7,8-TCDD (Dioxin)	.000000005
2,4,5-TP (Silvex)	.0002

(19) Anaylsis under this section shall only be conducted by laboratories that have received certification by EPA or the State and have met the following conditions:

(i) To receive certification to conduct analyses for the contaminants in § 141.61(c) the laboratory must:

(A) Analyze Performance Evaluation samples which include those substances provided by EPA Environmental Monitoring and Support Laboratory or equivalent samples provided by the State.

(B) Achieve quantitative results on the analyses that are within the following acceptance limits:

Contaminant	Acceptance limits (percent)
DBCP	±40
EDB	±40.
Alachlor	±45.
Atrazine	±45.
Benzo[a]pyrene	2 standard deviations.
Carbofuran	±45.
Chlordane	±45.
Dalapon	2 standard deviations.
Di(2-ethylhexyl)adipate	2 standard deviations.
Di(2-ethylhexyl)phthalate	2 standard deviations.
Dinoseb	2 standard deviations.
Diquat	2 standard deviations.
Endothall	2 standard deviations.
Endrin	±30.
Glyphosate	2 standard deviations.
Heptachlor	±45.
Heptachlor epoxide	±45.
Hexachlorobenzene	2 standard deviations.
Hexachloro- cyclopentadiene	2 standard deviations.
Lindane	±45.
Methoxychlor	±45.
Oxamyl	2 standard deviations.
PCBs (as Decachlorobiphenyl)	0–200.
Picloram	2 standard deviations.
Simazine	2 standard deviations.
Toxaphene	±45.
Aldicarb	2 standard deviations.
Aldicarb sulfoxide	2 standard deviations.
Aldicarb sulfone	2 standard deviations.
Pentachlorophenol	±50.
2,3,7,8-TCDD (Dioxin)	2 standard deviations.
2,4-D	±50.
2,4,5-TP (Silvex)	±50.

(ii) [Reserved]

(Approved by the Office of Management and Budget under control number 2040–0090)

[40 FR 59570, Dec. 24, 1975, as amended at 44 FR 68641, Nov. 29, 1979; 45 FR 57345, Aug. 27, 1980; 47 FR 10998, Mar. 12, 1982; 52 FR 25712, July 8, 1987; 53 FR 5147, Feb. 19, 1988; 53 FR 25110, July 1, 1988; 56 FR 3583, Jan. 30, 1991; 56 FR 30277, July 1, 1991; 57 FR 22178, May 27, 1992; 57 FR 31841, July 17, 1992; 59 FR 34323, July 1, 1994; 59 FR 62468, Dec. 5, 1994; 60 FR 34085, June 29, 1995]

§141.25 Analytical methods for radioactivity.

(a) Analysis for the following contaminants shall be conducted to determine compliance with §§141.15 and 141.16 (radioactivity) in accordance with the methods in the following Table, or their equivalent determined by EPA in accordance with §141.27.

Contaminant	Methodology	Reference (method or page number)								
		EPA[1]	EPA[2]	EPA[3]	EPA[4]	SM[5]	ASTM[6]	USGS[7]	DOE[8]	Other
Naturally occurring:										
Gross alpha[11] and beta	Evaporation	900.0	p 1	00–01	p 1	302, 7110 B		R–1120–76		
Gross alpha[11]	Co-precipitation	.....		00–02		7110 C				
Radium 226	Radon emanation,	903.1	p 16	Ra-04	p 19	7500-Ra C	D 3454–91	R–1141–76	Ra-05	N.Y.[9]
	Radio chemical	903.0	p 13	Ra-03		304, 305, ...	D 2460-90	R-1140-76		
						7500-Ra B				
Radium 228	Radio chemical	904.0	p 24	Ra-05	p 19	304, 7500-Ra D		R–1142–76		N.Y.[9] N.J.[10]
Uranium[12]	Radio chemical	908.0				7500-U B				
	Fluorometric	908.1				7500-U C (17th Ed.) ...	D2907-91	R-1180-76 R-1181-76	U-04	
	Alpha spectrometry.	.....		00–07	p33	7500-U C (18th or 19th Ed.).	D 3972–90	R-1182-76	U-02	
	Laser Phosphorimetry.	.....				.....	D 5174–91			
Man-made:										
Radioactive cesium	Radio chemical	901.0	p 4			7500-Cs B	D 2459–72	R-1111-76		
	Gamma ray spectrometry.	901.1			p 92	7120 (19th Ed.)	D 3649–91	R-1110-76	4.5.2.3	
Radioactive iodine	Radio chemical	902.0	p 6			7500-I B	D3649-91			
			p 9			7500-I C				
						7500-I D				
	Gamma ray spectrometry.	901.1			p 92	7120 (19th Ed.)	D 4785–88		4.5.2.3	
Radioactive Strontium 89, 90.	Radio chemical	905.0	p 29	Sr-04	p. 65	303, 7500-Sr B		R-1160-76	Sr-01 Sr-02	
Tritium	Liquid scintillation	906.0	p 34	H–02	p. 87	306, 7500–3H B	D 4107–91	R-1171-76		
Gamma emitters	Gamma ray	901.1			p92	7120 (19th Ed.)	D 3649–91	R-1110-76	4.5.2.3	
	Spectrometry	902.0				7500-Cs B	D 4785–88			
	.....	901.0				7500-I B				

The procedures shall be done in accordance with the documents listed below. The incorporation by reference of documents 1 through 10 was approved by the Director of the Federal Register in accordance with 5 U.S.C. 552(a) and 1 CFR part 51. Copies of the documents may be obtained from the sources listed below. Information regarding obtaining these documents can be obtained from the Safe Drinking Water Hotline at 800–426–4791. Documents may be inspected at EPA's Drinking Water Docket, 401 M Street, SW., Washington, DC 20460 (Telephone: 202–260–3027); or at the Office of Federal Register, 800 North Capitol Street, NW., Suite 700, Washington, DC.

[1] "Prescribed Procedures for Measurement of Radioactivity in Drinking Water", EPA 600/4–80–032 , August 1980. Available at U.S. Department of Commerce, National Technical Information Service (NTIS), 5285 Port Royal Road, Springfield, VA 22161 (Telephone 800–553–6847), PB 80–224744.

[2] "Interim Radiochemical Methodology for Drinking Water", EPA 600/4–75–008(revised), March 1976. Available at NTIS, ibid. PB 253258.

[3] "Radiochemistry Procedures Manual", EPA 520/5–84–006, December 1987. Available at NTIS, ibid. PB 84–215581.

[4] "Radiochemical Analytical Procedures for Analysis of Environmental Samples", March 1979. Available at NTIS, ibid. EMSL LV 053917.

[5] "Standard Methods for the Examination of Water and Wastewater", 13th, 17th, 18th, 19th Editions, 1971, 1989, 1992, 1995. Available at American Public Health Association, 1015 Fifteenth Street N.W., Washington, D.C. 20005. All methods are in the 17th, 18th and 19th editions except 7500-U C Fluorometric Uranium was discontinued after the 17th Edition, 7120 Gamma Emitters is only in the 19th Edition, and 302, 303, 304, 305 and 306 are only in the 13th Edition.

[6] *Annual Book of ASTM Standards,* Vol. 11.02, 1994. Available at American Society for Testing and Materials, 100 Barr Harbor Drive, West Conshohocken, PA 19428.

[7] "Methods for Determination of Radioactive Substances in Water and Fluvial Sediments", Chapter A5 in Book 5 of *Techniques of Water-Resources Investigations of the United States Geological Survey,* 1977. Available at U.S. Geological Survey (USGS) Information Services, Box 25286, Federal Center, Denver, CO 80225–0425.

[8] "EML Procedures Manual", 27th Edition, Volume 1, 1990. Available at the Environmental Measurements Laboratory, U.S. Department of Energy (DOE), 376 Hudson Street, New York, NY 10014–3621.

[9] "Determination of Ra-226 and Ra-228 (Ra-02)", January 1980, Revised June 1982. Available at Radiological Sciences Institute Center for Laboratories and Research, New York State Department of Health, Empire State Plaza, Albany, NY 12201.

[10] "Determination of Radium 228 in Drinking Water", August 1980. Available at State of New Jersey, Department of Environmental Protection, Division of Environmental Quality, Bureau of Radiation and Inorganic Analytical Services, 9 Ewing Street, Trenton, NJ 08625.

[11] Natural uranium and thorium-230 are approved as gross alpha calibration standards for gross alpha with co-precipitation and evaporation methods; americium-241 is approved with co-precipitation methods.

[12] If uranium (U) is determined by mass, a 0.67 pCi/µg of uranium conversion factor must be used. This conservative factor is based on the 1:1 activity ratio of U–234 to U–238 that is characteristic of naturally occurring uranium.

(b) When the identification and measurement of radionuclides other than those listed in paragraph (a) of this section is required, the following references are to be used, except in cases where alternative methods have been approved in accordance with § 141.27.

(1) *Procedures for Radiochemical Analysis of Nuclear Reactor Aqueous Solutions,* H. L. Krieger and S. Gold, EPA–R4–73–014. USEPA, Cincinnati, Ohio, May 1973.

(2) *HASL Procedure Manual,* Edited by John H. Harley. HASL 300, ERDA Health and Safety Laboratory, New York, NY., 1973.

(c) For the purpose of monitoring radioactivity concentrations in drinking water, the required sensitivity of the radioanalysis is defined in terms of a detection limit. The detection limit shall be that concentration which can be counted with a precision of plus or minus 100 percent at the 95 percent confidence level (1.96σ where σ is the standard deviation of the net counting rate of the sample).

(1) To determine compliance with § 141.15(a) the detection limit shall not exceed 1 pCi/1. To determine compliance with § 141.15(b) the detection limit shall not exceed 3 pCi/1.

(2) To determine compliance with § 141.16 the detection limits shall not exceed the concentrations listed in Table B.

TABLE B—DETECTION LIMITS FOR MAN-MADE BETA PARTICLE AND PHOTON EMITTERS

Radionuclide	Detection limit
Tritium	1,000 pCi/1.
Strontium–89	10 pCi/1.
Strontium–90	2 pCi/1.
Iodine–131	1 pCi/1.
Cesium–134	10 pCi/1.
Gross beta	4 pCi/1.
Other radionuclides	1/10 of the applicable limit.

(d) To judge compliance with the maximum contaminant levels listed in §§ 141.15 and 141.16, averages of data shall be used and shall be rounded to the same number of significant figures as the maximum contaminant level for the substance in question.

(e) The State has the authority to determine compliance or initiate enforcement action based upon analytical results or other information compiled by their sanctioned representatives and agencies.

[41 FR 28404, July 9, 1976, as amended at 45 FR 57345, Aug. 27, 1980; 62 FR 10173, Mar. 5, 1997]

§ 141.26 Monitoring frequency for radioactivity in community water systems.

(a) Monitoring requirements for gross alpha particle activity, radium–226 and radium–228.

(1) Initial sampling to determine compliance with § 141.15 shall begin within two years of the effective date of these regulations and the analysis shall be completed within three years of the effective date of these regulations. Compliance shall be based on the analysis of an annual composite of four consecutive quarterly samples or the average of the analyses of four samples obtained at quarterly intervals.

(i) A gross alpha particle activity measurement may be substituted for the required radium–226 and radium–228 analysis *Provided,* That the measured gross alpha particle activity does not exceed 5 pCi/1 at a confidence level of 95 percent (1.65σ where σ is the standard deviation of the net counting rate of the sample). In localities where radium–228 may be present in drinking water, it is recommended that the State require radium–226 and/or radium–228 analyses when the gross alpha particle activity exceeds 2 pCi/1.

(ii) When the gross alpha particle activity exceeds 5 pCi/1, the same or an equivalent sample shall be analyzed for radium–226. If the concentration of radium–226 exceeds 3 pCi/1 the same or an equivalent sample shall be analyzed for radium–228.

(2) For the initial analysis required by paragraph (a)(1) of this section, data acquired within one year prior to the effective date of this part may be substituted at the discretion of the State.

(3) Suppliers of water shall monitor at least once every four years following the procedure required by paragraph (a)(1) of this section. At the discretion of the State, when an annual record taken in conformance with paragraph (a)(1) of this section has established that the average annual concentration is less than half the maximum contaminant levels established by § 141.15,

analysis of a single sample may be substituted for the quarterly sampling procedure required by paragraph (a)(1) of this section.

(i) More frequent monitoring shall be conducted when ordered by the State in the vicinity of mining or other operations which may contribute alpha particle radioactivity to either surface or ground water sources of drinking water.

(ii) A supplier of water shall monitor in conformance with paragraph (a)(1) of this section within one year of the introduction of a new water source for a community water system. More frequent monitoring shall be conducted when ordered by the State in the event of possible contamination or when changes in the distribution system or treatment processing occur which may increase the concentration of radioactivity in finished water.

(iii) A community water system using two or more sources having different concentrations of radioactivity shall monitor source water, in addition to water from a free-flowing tap, when ordered by the State.

(iv) Monitoring for compliance with § 141.15 after the initial period need not include radium–228 *except when* required by the State, *Provided,* That the average annual concentration of radium–228 has been assayed at least once using the quarterly sampling procedure required by paragraph (a)(1) of this section.

(v) Suppliers of water shall conduct annual monitoring of any community water system in which the radium–226 concentration exceeds 3 pCi/1, when ordered by the State.

(4) If the average annual maximum contaminant level for gross alpha particle activity or total radium as set forth in § 141.15 is exceeded, the supplier of a community water system shall give notice to the State pursuant to § 141.31 and notify the public as required by § 141.32. Monitoring at quarterly intervals shall be continued until the annual average concentration no longer exceeds the maximum contaminant level or until a monitoring schedule as a condition to a variance, exemption or enforcement action shall become effective.

(b) Monitoring requirements for man-made radioactivity in community water systems.

(1) Within two years of the effective date of this part, systems using surface water sources and serving more than 100,000 persons and such other community water systems as are designated by the State shall be monitored for compliance with § 141.16 by analysis of a composite of four consecutive quarterly samples or analysis of four quarterly samples. Compliance with § 141.16 may be assumed without further analysis if the average annual concentration of gross beta particle activity is less than 50 pCi/1 and if the average annual concentrations of tritium and strontium–90 are less than those listed in table A, *Provided,* That if both radionuclides are present the sum of their annual dose equivalents to bone marrow shall not exceed 4 millirem/year.

(i) If the gross beta particle activity exceeds 50 pCi/1, an analysis of the sample must be performed to identify the major radioactive constituents present and the appropriate organ and total body doses shall be calculated to determine compliance with § 141.16.

(ii) Suppliers of water shall conduct additional monitoring, as ordered by the State, to determine the concentration of man-made radioactivity in principal watersheds designated by the State.

(iii) At the discretion of the State, suppliers of water utilizing only ground waters may be required to monitor for man-made radioactivity.

(2) For the initial analysis required by paragraph (b)(1) of this section data acquired within one year prior to the effective date of this part may be substituted at the discretion of the State.

(3) After the initial analysis required by paragraph (b)(1) of this section suppliers of water shall monitor at least every four years following the procedure given in paragraph (b)(1) of this section.

(4) Within two years of the effective date of these regulations the supplier of any community water system designated by the State as utilizing waters contaminated by effluents from nuclear facilities shall initiate quarterly monitoring for gross beta particle and iodine–131 radioactivity and annual

monitoring for strontium–90 and tritium.

(i) Quarterly monitoring for gross beta particle activity shall be based on the analysis of monthly samples or the analysis of a composite of three monthly samples. The former is recommended. If the gross beta particle activity in a sample exceeds 15 pCi/1, the same or an equivalent sample shall be analyzed for strontium–89 and cesium–134. If the gross beta particle activity exceeds 50 pCi/1, an analysis of the sample must be performed to identify the major radioactive constituents present and the appropriate organ and total body doses shall be calculated to determine compliance with § 141.16.

(ii) For iodine–131, a composite of five consecutive daily samples shall be analyzed once each quarter. As ordered by the State, more frequent monitoring shall be conducted when iodine–131 is identified in the finished water.

(iii) Annual monitoring for strontium–90 and tritium shall be conducted by means of the analysis of a composite of four consecutive quarterly samples or analysis of four quarterly samples. The latter procedure is recommended.

(iv) The State may allow the substitution of environmental surveillance data taken in conjunction with a nuclear facility for direct monitoring of manmade radioactivity by the supplier of water where the State determines such data is applicable to a particular community water system.

(5) If the average annual maximum contaminant level for man-made radioactivity set forth in § 141.16 is exceeded, the operator of a community water system shall give notice to the State pursuant to § 141.31 and to the public as required by § 141.32. Monitoring at monthly intervals shall be continued until the concentration no longer exceeds the maximum contaminant level or until a monitoring schedule as a condition to a variance, exemption or enforcement action shall become effective.

[41 FR 28404, July 9, 1976]

§ 141.27 Alternate analytical techniques.

(a) With the written permission of the State, concurred in by the Administrator of the U.S. EPA, an alternate analytical technique may be employed. An alternate technique shall be accepted only if it is substantially equivalent to the prescribed test in both precision and accuracy as it relates to the determination of compliance with any MCL. The use of the alternate analytical technique shall not decrease the frequency of monitoring required by this part.

[45 FR 57345, Aug. 27, 1980]

§ 141.28 Certified laboratories.

(a) For the purpose of determining compliance with §§ 141.21 through 141.27, 141.41 and 141.42, samples may be considered only if they have been analyzed by a laboratory certified by the State except that measurements for turbidity, free chlorine residual, temperature and pH may be performed by any person acceptable to the State.

(b) Nothing in this part shall be construed to preclude the State or any duly designated representative of the State from taking samples or from using the results from such samples to determine compliance by a supplier of water with the applicable requirements of this part.

[45 FR 57345, Aug. 27, 1980; 47 FR 10999, Mar. 12, 1982, as amended at 59 FR 34323, July 1, 1994]

§ 141.29 Monitoring of consecutive public water systems.

When a public water system supplies water to one or more other public water systems, the State may modify the monitoring requirements imposed by this part to the extent that the interconnection of the systems justifies treating them as a single system for monitoring purposes. Any modified monitoring shall be conducted pursuant to a schedule specified by the State and concurred in by the Administrator of the U.S. Environmental Protection Agency.

§ 141.30 Total trihalomethanes sampling, analytical and other requirements.

(a) Community water system which serve a population of 10,000 or more individuals and which add a disinfectant (oxidant) to the water in any part of the drinking water treatment process

shall analyze for total trihalomethanes in accordance with this section. For systems serving 75,000 or more individuals, sampling and analyses shall begin not later than 1 year after the date of promulgation of this regulation. For systems serving 10,000 to 74,999 individuals, sampling and analyses shall begin not later than 3 years after the date of promulgation of this regulation. For the purpose of this section, the minimum number of samples required to be taken by the system shall be based on the number of treatment plants used by the system, except that multiple wells drawing raw water from a single aquifer may, with the State approval, be considered one treatment plant for determining the minimum number of samples. All samples taken within an established frequency shall be collected within a 24-hour period.

(b)(1) For all community water systems utilizing surface water sources in whole or in part, and for all community water systems utilizing only ground water sources that have not been determined by the State to qualify for the monitoring requirements of paragraph (c) of this section, analyses for total trihalomethanes shall be performed at quarterly intervals on at least four water samples for each treatment plant used by the system. At least 25 percent of the samples shall be taken at locations within the distribution system reflecting the maximum residence time of the water in the system. The remaining 75 percent shall be taken at representative locations in the distribution system, taking into account number of persons served, different sources of water and different treatment methods employed. The results of all analyses per quarter shall be arithmetically averaged and reported to the State within 30 days of the system's receipt of such results. Results shall also be reported to EPA until such monitoring requirements have been adopted by the State. All samples collected shall be used in the computation of the average, unless the analytical results are invalidated for technical reasons. Sampling and analyses shall be conducted in accordance with the methods listed in paragraph (e) of this section.

(2) Upon the written request of a community water system, the monitoring frequency required by paragraph (b)(1) of this section may be reduced by the State to a minimum of one sample analyzed for TTHMs per quarter taken at a point in the distribution system reflecting the maximum residence time of the water in the system, upon a written determination by the State that the data from at least 1 year of monitoring in accordance with paragraph (b)(1) of this section and local conditions demonstrate that total trihalomethane concentrations will be consistently below the maximum contaminant level.

(3) If at any time during which the reduced monitoring frequency prescribed under this paragraph applies, the results from any analysis exceed 0.10 mg/l of TTHMs and such results are confirmed by at least one check sample taken promptly after such results are received, or if the system makes any significant change to its source of water or treatment program, the system shall immediately begin monitoring in accordance with the requirements of paragraph (b)(1) of this section, which monitoring shall continue for at least 1 year before the frequency may be reduced again. At the option of the State, a system's monitoring frequency may and should be increased above the minimum in those cases where it is necessary to detect variations of TTHM levels within the distribution system.

(c)(1) Upon written request to the State, a community water system utilizing only ground water sources may seek to have the monitoring frequency required by paragraph (b)(1) of this section reduced to a minimum of one sample for maximum TTHM potential per year for each treatment plant used by the system taken at a point in the distribution system reflecting maximum residence time of the water in the system. The system shall submit the results of at least one sample for maximum TTHM potential using the procedure specified in paragraph (g) of this section. A sample must be analyzed from each treatment plant used by the system and be taken at a point in the distribution system reflecting the maximum residence time of the water in

the system. The system's monitoring frequency may only be reduced upon a written determination by the State that, based upon the data submitted by the system, the system has a maximum TTHM potential of less than 0.10 mg/l and that, based upon an assessment of the local conditions of the system, the system is not likely to approach or exceed the maximum contaminant level for total TTHMs. The results of all analyses shall be reported to the State within 30 days of the system's receipt of such results. Results shall also be reported to EPA until such monitoring requirements have been adopted by the State. All samples collected shall be used for determining whether the system must comply with the monitoring requirements of paragraph (b) of this section, unless the analytical results are invalidated for technical reasons. Sampling and analyses shall be conducted in accordance with the methods listed in paragraph (e) of this section.

(2) If at any time during which the reduced monitoring frequency prescribed under paragraph (c)(1) of this section applies, the results from any analysis taken by the system for maximum TTHM potential are equal to or greater than 0.10 mg/l, and such results are confirmed by at least one check sample taken promptly after such results are received, the system shall immediately begin monitoring in accordance with the requirements of paragraph (b) of this section and such monitoring shall continue for at least one year before the frequency may be reduced again. In the event of any significant change to the system's raw water or treatment program, the system shall immediately analyze an additional sample for maximum TTHM potential taken at a point in the distribution system reflecting maximum residence time of the water in the system for the purpose of determining whether the system must comply with the monitoring requirements of paragraph (b) of this section. At the option of the State, monitoring frequencies may and should be increased above the minimum in those cases where this is necessary to detect variation of TTHM levels within the distribution system.

(d) Compliance with § 141.12(c) shall be determined based on a running annual average of quarterly samples collected by the system as prescribed in paragraph (b) (1) or (2) of this section. If the average of samples covering any 12 month period exceeds the Maximum Contaminant Level, the supplier of water shall report to the State pursuant to § 141.31 and notify the public pursuant to § 141.32. Monitoring after public notification shall be at a frequency designated by the State and shall continue until a monitoring schedule as a condition to a variance, exemption or enforcement action shall become effective.

(e) Sampling and analyses made pursuant to this section shall be conducted by one of the total trihalomethane methods as directed in § 141.24(e), and the *Technical Notes on Drinking Water Methods*, EPA–600/R–94–173, October 1994, which is available from NTIS, PB–104766. Samples for TTHM shall be dechlorinated upon collection to prevent further production of trihalomethanes, according to the procedures described in the methods, except acidification is not required if only THMs or TTHMs are to be determined. Samples for maximum TTHM potential should not be dechlorinated or acidified, and should be held for seven days at 25°C (or above) prior to analysis.

(f) Before a community water system makes any significant modifications to its existing treatment process for the purposes of achieving compliance with § 141.12(c), such system must submit and obtain State approval of a detailed plan setting forth its proposed modification and those safeguards that it will implement to ensure that the bacteriological quality of the drinking water served by such system will not be adversely affected by such modification. Each system shall comply with the provisions set forth in the State-approved plan. At a minimum, a State approved plan shall require the system modifying its disinfection practice to:

(1) Evaluate the water system for sanitary defects and evaluate the source water for biological quality;

(2) Evaluate its existing treatment practices and consider improvements

that will minimize disinfectant demand and optimize finished water quality throughout the distribution system;

(3) Provide baseline water quality survey data of the distribution system. Such data should include the results from monitoring for coliform and fecal coliform bacteria, fecal streptococci, standard plate counts at 35° C and 20° C, phosphate, ammonia nitrogen and total organic carbon. Virus studies should be required where source waters are heavily contaminated with sewage effluent;

(4) Conduct additional monitoring to assure continued maintenance of optimal biological quality in finished water, for example, when chloramines are introduced as disinfectants or when pre-chlorination is being discontinued. Additional monitoring should also be required by the State for chlorate, chlorite and chlorine dioxide when chlorine dioxide is used. Standard plate count analyses should also be required by the State as appropriate before and after any modifications;

(5) Consider inclusion in the plan of provisions to maintain an active disinfectant residual throughout the distribution system at all times during and after the modification.

(g) The water sample for determination of maximum total trihalomethane potential is taken from a point in the distribution system that reflects maximum residence time. Procedures for sample collection and handling are given in the methods. No reducing agent is added to ''quench'' the chemical reaction producing THMs at the time of sample collection. The intent is to permit the level of THM precursors to be depleted and the concentration of THMs to be maximized for the supply being tested. Four experimental parameters affecting maximum THM production are pH, temperature, reaction time and the presence of a disinfectant residual. These parameters are dealt with as follows: Measure the disinfectant residual at the selected sampling point. Proceed only if a measurable disinfectant residual is present. Collect triplicate 40 ml water samples at the pH prevailing at the time of sampling, and prepare a method blank according to the methods. Seal and store these samples together for seven days at 25°C or above. After this time period, open one of the sample containers and check for disinfectant residual. Absence of a disinfectant residual invalidates the sample for further analysis. Once a disinfectant residual has been demonstrated, open another of the sealed samples and determine total THM concentration using an approved analytical method.

[44 FR 68641, Nov. 29, 1979, as amended at 45 FR 15545, 15547, Mar. 11, 1980; 58 FR 41345, Aug. 3, 1993; 59 FR 62469, Dec. 5, 1994; 60 FR 34085, June 29, 1995]

Subpart D—Reporting, Public Notification and Recordkeeping

§141.31 Reporting requirements.

(a) Except where a shorter period is specified in this part, the supplier of water shall report to the State the results of any test measurement or analysis required by this part within (1) The first ten days following the month in which the result is received, or (2) the first ten days following the end of the required monitoring period as stipulated by the State, whichever of these is shortest.

(b) Except where a different reporting period is specified in this part, the supplier of water must report to the State within 48 hours the failure to comply with any national primary drinking water regulation (including failure to comply with monitoring requirements) set forth in this part.

(c) The supplier of water is not required to report analytical results to the State in cases where a State laboratory performs the analysis and reports the results to the State office which would normally receive such notification from the supplier.

(d) The water supply system, within ten days of completion of each public notification required pursuant to §141.32, shall submit to the State a representative copy of each type of notice distributed, published, posted, and/or made available to the persons served by the system and/or to the media.

(e) The water supply system shall submit to the State within the time stated in the request copies of any records required to be maintained under §141.33 hereof or copies of any

documents then in existence which the State or the Administrator is entitled to inspect pursuant to the authority of section 1445 of the Safe Drinking Water Act or the equivalent provisions of State law.

[40 FR 59570, Dec. 24, 1975, as amended at 45 FR 57345, Aug. 27, 1980]

§ 141.32 Public notification.

The requirements in this section are effective April 28, 1989. The requirements of § 141.36 apply until April 28, 1989.

(a) *Maximum contaminant level (MCL), treatment technique, and variance and exemption schedule violations.* The owner or operator of a public water system which fails to comply with an applicable MCL or treatment technique established by this part or which fails to comply with the requirements of any schedule prescribed pursuant to a variance or exemption, shall notify persons served by the system as follows:

(1) Except as provided in paragraph (a)(3) of this section, the owner or operator of a public water system must give notice:

(i) By publication in a daily newspaper of general circulation in the area served by the system as soon as possible, but in no case later than 14 days after the violation or failure. If the area served by a public water system is not served by a daily newspaper of general circulation, notice shall instead be given by publication in a weekly newspaper of general circulation serving the area; and

(ii) By mail delivery (by direct mail or with the water bill), or by hand delivery, not later than 45 days after the violation or failure. The State may waive mail or hand delivery if it determines that the owner or operator of the public water system in violation has corrected the violation or failure within the 45-day period. The State must make the waiver in writing and within the 45-day period; and

(iii) For violations of the MCLs of contaminants that may pose an acute risk to human health, by furnishing a copy of the notice to the radio and television stations serving the area served by the public water system as soon as possible but in no case later than 72 hours after the violation. The following violations are acute violations:

(A) Any violations specified by the State as posing an acute risk to human health.

(B) Violation of the MCL for nitrate or nitrite as defined in § 141.62 and determined according to § 141.23(i)(3).

(C) Violation of the MCL for total coliforms, when fecal coliforms or *E. coli* are present in the water distribution system, as specified in § 141.63(b).

(D) Occurrence of a waterborne disease outbreak, as defined in § 141.2, in an unfiltered system subject to the requirements of subpart H of this part, after December 30, 1991 (see § 141.71(b)(4)).

(2) Except as provided in paragraph (a)(3) of this section, following the initial notice given under paragraph (a)(1) of this section, the owner or operator of the public water system must give notice at least once every three months by mail delivery (by direct mail or with the water bill) or by hand delivery, for as long as the violation or failure exists.

(3)(i) In lieu of the requirements of paragraphs (a) (1) and (2) of this section, the owner or operator of a community water system in an area that is not served by a daily or weekly newspaper of general circulation must give notice by hand delivery or by continuous posting in conspicuous places within the area served by the system. Notice by hand delivery or posting must begin as soon as possible, but no later than 72 hours after the violation or failure for acute violations (as defined in paragraph (a)(1)(iii) of this section), or 14 days after the violation or failure (for any other violation). Posting must continue for as long as the violation or failure exists. Notice by hand delivery must be repeated at least every three months for as long as the violation or failure exists.

(ii) In lieu of the requirements of paragraphs (a) (1) and (2) of this section, the owner or operator of a noncommunity water system may give notice by hand delivery or by continuous posting in conspicuous places within the area served by the system. Notice by hand delivery or posting must begin as soon as possible, but no later than 72 hours after the violation or failure for

acute violations (as defined in paragraph (a)(1)(iii) of this section), or 14 days after the violation or failure (for any other violation). Posting must continue for as long as the violation or failure exists. Notice by hand delivery must be repeated at least every three months for as long as the violation or failure exists.

(b) *Other violations, variances, exemptions.* The owner or operator of a public water system which fails to perform monitoring required by section 1445(a) of the Act (including monitoring required by the National Primary Drinking Water Regulations (NPDWRs) of this part), fails to comply with a testing procedure established by this part, is subject to a variance granted under section 1415(a)(1)(A) or 1415(a)(2) of the Act, or is subject to an exemption under section 1416 of the Act, shall notify persons served by the system as follows:

(1) Except as provided in paragraph (b)(3) or (b)(4) of this section, the owner or operator of a public water system must give notice within three months of the violation or granting of a variance or exemption by publication in a daily newspaper of general circulation in the area served by the system. If the area served by a public water system is not served by a daily newspaper of general circulation, notice shall instead be given by publication in a weekly newspaper of general circulation serving the area.

(2) Except as provided in paragraph (b)(3) or (b)(4) of this section, following the initial notice given under paragraph (b)(1) of this section, the owner or operator of the public water system must give notice at least once every three months by mail delivery (by direct mail or with the water bill) or by hand delivery, for as long as the violation exists. Repeat notice of the existence of a variance or exemption must be given every three months for as long as the variance or exemption remains in effect.

(3)(i) In lieu of the requirements of paragraphs (b)(1) and (b)(2) of this section, the owner or operator of a community water system in an area that is not served by a daily or weekly newspaper of general circulation must give notice, within three months of the violation or granting of the variance or exemption, by hand delivery or by continuous posting in conspicuous places with the area served by the system. Posting must continue for as long as the violation exists or a variance or exemption remains in effect. Notice by hand delivery must be repeated at least every three months for as long as the violation exists or a variance or exemption remains in effect.

(ii) In lieu of the requirements of paragraphs (b)(1) and (b)(2) of this section, the owner or operator of a noncommunity water system may give notice, within three months of the violation or the granting of the variance or exemption, by hand delivery or by continuous posting in conspicuous places within the area served by the system. Posting must continue for as long as the violation exists, or a variance or exemption remains in effect. Notice by hand delivery must be repeated at least every three months for as long as the violation exists or a variance or exemption remains in effect.

(4) In lieu of the requirements of paragraphs (b)(1), (b)(2), and (b)(3) of this section, the owner or operator of a public water system, at the discretion of the State, may provide less frequent notice for minor monitoring violations as defined by the State, if EPA has approved the State's application for a program revision under § 142.16. Notice of such violations must be given no less frequently than annually.

(c) *Notice to new billing units.* The owner or operator of a community water system must give a copy of the most recent public notice for any outstanding violation of any maximum contaminant level, or any treatment technique requirement, or any variance or exemption schedule to all new billing units or new hookups prior to or at the time service begins.

(d) *General content of public notice.* Each notice required by this section must provide a clear and readily understandable explanation of the violation, any potential adverse health effects, the population at risk, the steps that the public water system is taking to correct such violation, the necessity for seeking alternative water supplies, if any, and any preventive measures the consumer should take until the

violation is corrected. Each notice shall be conspicuous and shall not contain unduly technical language, unduly small print, or similar problems that frustrate the purpose of the notice. Each notice shall include the telephone number of the owner, operator, or designee of the public water system as a source of additional information concerning the notice. Where appropriate, the notice shall be multi-lingual.

(e) *Mandatory health effects language.* When providing the information on potential adverse health effects required by paragraph (d) of this section in notices of violations of maximum contaminant levels or treatment technique requirements, or notices of the granting or the continued existence of exemptions or variances, or notices of failure to comply with a variance or exemption schedule, the owner or operator of a public water system shall include the language specified below for each contaminant. (If language for a particular contaminant is not specified below at the time notice is required, this paragraph does not apply.)

(1) *Trichloroethylene.* The United States Environmental Protection Agency (EPA) sets drinking water standards and has determined that trichloroethylene is a health concern at certain levels of exposure. This chemical is a common metal cleaning and dry cleaning fluid. It generally gets into drinking water by improper waste disposal. This chemical has been shown to cause cancer in laboratory animals such as rats and mice when the animals are exposed at high levels over their lifetimes. Chemicals that cause cancer in laboratory animals also may increase the risk of cancer in humans who are exposed at lower levels over long periods of time. EPA has set forth the enforceable drinking water standard for trichloroethylene at 0.005 parts per million (ppm) to reduce the risk of cancer or other adverse health effects which have been observed in laboratory animals. Drinking water which meets this standard is associated with little to none of this risk and should be considered safe.

(2) *Carbon tetrachloride.* The United States Environmental Protection Agency (EPA) sets drinking water standards and has determined that carbon tetrachloride is a health concern at certain levels of exposure. This chemical was once a popular household cleaning fluid. It generally gets into drinking water by improper waste disposal. This chemical has been shown to cause cancer in laboratory animals such as rats and mice when the animals are exposed at high levels over their lifetimes. Chemicals that cause cancer in laboratory animals also may increase the risk of cancer in humans who are exposed at lower levels over long periods of of time. EPA has set the enforceable drinking water standard for carbon tetrachloride at 0.005 parts per million (ppm) to reduce the risk of cancer or other adverse health effects which have been observed in laboratory animals. Drinking water which meets this standard is associated with little to none of this risk and should be considered safe.

(3) *1,2-Dichloroethane.* The United States Environmental Protection Agency (EPA) sets drinking water standards and has determined that 1,2-dichloroethane is a health concern at certain levels of exposure. This chemical is used as a cleaning fluid for fats, oils, waxes, and resins. It generally gets into drinking water from improper waste disposal. This chemical has been shown to cause cancer in laboratory animals such as rats and mice when the animals are exposed at high levels over their lifetimes. Chemicals that cause cancer in laboratory animals also may increase the risk of cancer in humans who are exposed at lower levels over long periods of time. EPA has set the enforceable drinking water standard for 1,2-dichloroethane at 0.005 parts per million (ppm) to reduce the risk of cancer or other adverse health effects which have been observed in laboratory animals. Drinking water which meets this standard is associated with little to none of this risk and should be considered safe.

(4) *Vinyl chloride.* The United States Environmental Protection Agency (EPA) sets drinking water standards and has determined that vinyl chloride is a health concern at certain levels of exposure. This chemical is used in industry and is found in drinking water as a result of the breakdown of related solvents. The solvents are used as

cleaners and degreasers of metals and generally get into drinking water by improper waste disposal. This chemical has been associated with significantly increased risks of cancer among certain industrial workers who were exposed to relatively large amounts of this chemical during their working careers. This chemical has also been shown to cause cancer in laboratory animals when the animals are exposed at high levels over their lifetimes. Chemicals that cause increased risk of cancer among exposed industrial workers and in laboratory animals also may increase the risk of cancer in humans who are exposed at lower levels over long periods of time. EPA has set the enforceable drinking water standard for vinyl chloride at 0.002 part per million (ppm) to reduce the risk of cancer or other adverse health effects which have been observed in humans and laboratory animals. Drinking water which meets this standard is associated with little to none of this risk and should be considered safe.

(5) *Benzene.* The United States Environmental Protection Agency (EPA) sets drinking water standards and has determined that benzene is a health concern at certain levels of exposure. This chemical is used as a solvent and degreaser of metals. It is also a major component of gasoline. Drinking water contamination generally results from leaking undergound gasoline and petroleum tanks or improper waste disposal. This chemical has been associated with significantly increased risks of leukemia among certain industrial workers who were exposed to relatively large amounts of this chemical during their working careers. This chemical has also been shown to cause cancer in laboratory animals when the animals are exposed at high levels over their lifetimes. Chemicals that cause increased risk of cancer among exposed industrial workers and in laboratory animals also may increase the risk of cancer in humans who are exposed at lower levels over long periods of time. EPA has set the enforceable drinking water standard for benzene at 0.005 parts per million (ppm) to reduce the risk of cancer or other adverse health effects which have been observed in humans and laboratory animals. Drinking water which meets this standard is associated with little to none of this risk and should be considered safe.

(6) *1,1-Dichloroethylene.* The United States Environmental Protection Agency (EPA) sets drinking water standards and has determined that 1,1-dichloroethylene is a health concern at certain levels of exposure. This chemical is used in industry and is found in drinking water as a result of the breakdown of related solvents. The solvents are used as cleaners and degreasers of metals and generally get into drinking water by improper waste disposal. This chemical has been shown to cause liver and kidney damage in laboratory animals such as rats and mice when the animals are exposed at high levels over their lifetimes. Chemicals which cause adverse effects in laboratory animals also may cause adverse health effects in humans who are exposed at lower levels over long periods of time. EPA has set the enforceable drinking water standard for 1,1-dichloroethylene at 0.007 parts per million (ppm) to reduce the risk of these adverse health effects which have been observed in laboratory animals. Drinking water which meets this standard is associated with little to none of this risk and should be considered safe.

(7) *Para-dichlorobenzene.* The United States Environmental Protection Agency (EPA) sets drinking water standards and has determined that para-dichlorobenzene is a health concern at certain levels of exposure. This chemical is a component of deodorizers, moth balls, and pesticides. It generally gets into drinking water by improper waste disposal. This chemical has been shown to cause liver and kidney damage in laboratory animals such as rats and mice when the animals are exposed to high levels over their lifetimes. Chemicals which cause adverse effects in laboratory animals also may cause adverse health effects in humans who are exposed at lower levels over long periods of time. EPA has set the enforceable drinking water standard for para-dichlorobenzene at 0.075 parts per million (ppm) to reduce the risk of these adverse health effects which have been observed in laboratory animals. Drinking water which meets this standard is associated with little to

none of this risk and should be considered safe.

(8) *1,1,1-Trichloroethane.* The United States Environmental Protection Agency (EPA) sets drinking water standards and has determined that the 1,1,1-trichloroethane is a health concern at certain levels of exposure. This chemical is used as a cleaner and degreaser of metals. It generally gets into drinking water by improper waste disposal. This chemical has been shown to damage the liver, nervous system, and circulatory system of laboratory animals such as rats and mice when the animals are exposed at high levels over their lifetimes. Some industrial workers who were exposed to relatively large amounts of this chemical during their working careers also suffered damage to the liver, nervous system, and circulatory system. Chemicals which cause adverse effects among exposed industrial workers and in laboratory animals also may cause adverse health effects in humans who are exposed at lower levels over long periods of time. EPA has set the enforceable drinking water standard for 1,1,1-trichloroethane at 0.2 parts per million (ppm) to protect against the risk of these adverse health effects which have been observed in humans and laboratory animals. Drinking water which meets this standard is associated with little to none of this risk and should be considered safe.

(9) *Fluoride.*

[NOTE: EPA is not specifying language that must be included in a public notice for a violation of the fluoride maximum contaminant level in this section because § 143.5 of this part includes the necessary information. See paragraph (f) of this section.]

(10) *Microbiological contaminants* (for use when there is a violation of the treatment technique requirements for filtration and disinfection in subpart H of this part). The United States Environmental Protection Agency (EPA) sets drinking water standards and has determined that the presence of microbiological contaminants are a health concern at certain levels of exposure. If water is inadequately treated, microbiological contaminants in that water may cause disease. Disease symptoms may include diarrhea, cramps, nausea, and possibly jaundice, and any associated headaches and fatigue. These symptoms, however, are not just associated with disease-causing organisms in drinking water, but also may be caused by a number of factors other than your drinking water. EPA has set enforceable requirements for treating drinking water to reduce the risk of these adverse health effects. Treatment such as filtering and disinfecting the water removes or destroys microbiological contaminants. Drinking water which is treated to meet EPA requirements is associated with little to none of this risk and should be considered safe.

(11) *Total coliforms* (To be used when there is a violation of § 141.63(a), and not a violation of § 141.63(b)). The United States Environmental Protection Agency (EPA) sets drinking water standards and has determined that the presence of total coliforms is a possible health concern. Total coliforms are common in the environment and are generally not harmful themselves. The presence of these bacteria in drinking water, however, generally is a result of a problem with water treatment or the pipes which distribute the water, and indicates that the water may be contaminated with organisms that can cause disease. Disease symptoms may include diarrhea, cramps, nausea, and possibly jaundice, and any associated headaches and fatigue. These symptoms, however, are not just associated with disease-causing organisms in drinking water, but also may be caused by a number of factors other than your drinking water. EPA has set an enforceable drinking water standard for total coliforms to reduce the risk of these adverse health effects. Under this standard, no more than 5.0 percent of the samples collected during a month can contain these bacteria, except that systems collecting fewer than 40 samples/month that have one total coliform-positive sample per month are not violating the standard. Drinking water which meets this standard is usually not associated with a health risk from disease-causing bacteria and should be considered safe.

(12) *Fecal Coliforms/E. coli* (To be used when there is a violation of § 141.63(b) or both § 141.63 (a) and (b)). The United States Environmental Protection

Agency (EPA) sets drinking water standards and has determined that the presence of fecal coliforms or *E. coli* is a serious health concern. Fecal coliforms and *E. coli* are generally not harmful themselves, but their presence in drinking water is serious because they usually are associated with sewage or animal wastes. The presence of these bacteria in drinking water is generally a result of a problem with water treatment or the pipes which distribute the water, and indicates that the water may be contaminated with organisms that can cause disease. Disease symptoms may include diarrhea, cramps, nausea, and possibly jaundice, and associated headaches and fatigue. These symptoms, however, are not just associated with disease-causing organisms in drinking water, but also may be caused by a number of factors other than your drinking water. EPA has set an enforceable drinking water standard for fecal coliforms and *E. coli* to reduce the risk of these adverse health effects. Under this standard all drinking water samples must be free of these bacteria. Drinking water which meets this standard is associated with little or none of this risk and should be considered safe. State and local health authorities recommend that consumers take the following precautions: [To be inserted by the public water system, according to instructions from State or local authorities].

(13) *Lead.* The United States Environmental Protection Agency (EPA) sets drinking water standards and has determined that lead is a health concern at certain exposure levels. Materials that contain lead have frequently been used in the construction of water supply distribution systems, and plumbing systems in private homes and other buildings. The most commonly found materials include service lines, pipes, brass and bronze fixtures, and solders and fluxes. Lead in these materials can contaminate drinking water as a result of the corrosion that takes place when water comes into contact with those materials. Lead can cause a variety of adverse health effects in humans. At relatively low levels of exposure, these effects may include interference with red blood cell chemistry, delays in normal physical and mental development in babies and young children, slight deficits in the attention span, hearing, and learning abilities of children, and slight increases in the blood pressure of some adults. EPA's national primary drinking water regulation requires all public water systems to optimize corrosion control to minimize lead contamination resulting from the corrosion of plumbing materials. Public water systems serving 50,000 people or fewer that have lead concentrations below 15 parts per billion (ppb) in more than 90% of tap water samples (the EPA ''action level'') have optimized their corrosion control treatment. Any water system that exceeds the action level must also monitor their source water to determine whether treatment to remove lead in source water is needed. Any water system that continues to exceed the action level after installation of corrosion control and/or source water treatment must eventually replace all lead service lines contributing in excess of 15 (ppb) of lead to drinking water. Any water system that exceeds the action level must also undertake a public education program to inform consumers of ways they can reduce their exposure to potentially high levels of lead in drinking water.

(14) *Copper.* The United States Environmental Protection Agency (EPA) sets drinking water standards and has determined that copper is a health concern at certain exposure levels. Copper, a reddish-brown metal, is often used to plumb residential and commercial structures that are connected to water distribution systems. Copper contaminating drinking water as a corrosion byproduct occurs as the result of the corrosion of copper pipes that remain in contact with water for a prolonged period of time. Copper is an essential nutrient, but at high doses it has been shown to cause stomach and intestinal distress, liver and kidney damage, and anemia. Persons with Wilson's disease may be at a higher risk of health effects due to copper than the general public. EPA's national primary drinking water regulation requires all public water systems to install optimal corrosion control to minimize copper contamination resulting from the corrosion of plumbing materials. Public water systems serving 50,000 people or

fewer that have copper concentrations below 1.3 parts per million (ppm) in more than 90% of tap water samples (the EPA ''action level'') are not required to install or improve their treatment. Any water system that exceeds the action level must also monitor their source water to determine whether treatment to remove copper in source water is needed.

(15) *Asbestos.* The United States Environmental Protection Agency (EPA) sets drinking water standards and has determined that asbestos fibers greater than 10 micrometers in length are a health concern at certain levels of exposure. Asbestos is a naturally occurring mineral. Most asbestos fibers in drinking water are less than 10 micrometers in length and occur in drinking water from natural sources and from corroded asbestos-cement pipes in the distribution system. The major uses of asbestos were in the production of cements, floor tiles, paper products, paint, and caulking; in transportation-related applications; and in the production of textiles and plastics. Asbestos was once a popular insulating and fire retardent material. Inhalation studies have shown that various forms of asbestos have produced lung tumors in laboratory animals. The available information on the risk of developing gastrointestinal tract cancer associated with the ingestion of asbestos from drinking water is limited. Ingestion of intermediate-range chrysotile asbestos fibers greater than 10 micrometers in length is associated with causing benign tumors in male rats. Chemicals that cause cancer in laboratory animals also may increase the risk of cancer in humans who are exposed over long periods of time. EPA has set the drinking water standard for asbestos at 7 million long fibers per liter to reduce the potential risk of cancer or other adverse health effects which have been observed in laboratory animals. Drinking water which meets the EPA standard is associated with little to none of this risk and should be considered safe with respect to asbestos.

(16) *Barium.* The United States Environmental Protection Agency (EPA) sets drinking water standards and has determined that barium is a health concern at certain levels of exposure. This inorganic chemical occurs naturally in some aquifers that serve as sources of ground water. It is also used in oil and gas drilling muds, automotive paints, bricks, tiles and jet fuels. It generally gets into drinking water after dissolving from naturally occurring minerals in the ground. This chemical may damage the heart and cardiovascular system, and is associated with high blood pressure in laboratory animals such as rats exposed to high levels during their lifetimes. In humans, EPA believes that effects from barium on blood pressure should not occur below 2 parts per million (ppm) in drinking water. EPA has set the drinking water standard for barium at 2 parts per million (ppm) to protect against the risk of these adverse health effects. Drinking water that meets the EPA standard is associated with little to none of this risk and is considered safe with respect to barium.

(17) *Cadmium.* The United States Environmental Protection Agency (EPA) sets drinking water standards and has determined that cadmium is a health concern at certain levels of exposure. Food and the smoking of tobacco are common sources of general exposure. This inorganic metal is a contaminant in the metals used to galvanize pipe. It generally gets into water by corrosion of galvanized pipes or by improper waste disposal. This chemical has been shown to damage the kidney in animals such as rats and mice when the animals are exposed at high levels over their lifetimes. Some industrial workers who were exposed to relatively large amounts of this chemical during working careers also suffered damage to the kidney. EPA has set the drinking water standard for cadmium at 0.005 parts per million (ppm) to protect against the risk of these adverse health effects. Drinking water that meets the EPA standard is associated with little to none of this risk and is considered safe with respect to cadmium.

(18) *Chromium.* The United States Environmental Protection Agency (EPA) sets drinking water standards and has determined that chromium is a health concern at certain levels of exposure. This inorganic metal occurs naturally in the ground and is often used in the electroplating of metals. It generally

gets into water from runoff from old mining operations and improper waste disposal from plating operations. This chemical has been shown to damage the kidney, nervous system, and the circulatory system of laboratory animals such as rats and mice when the animals are exposed at high levels. Some humans who were exposed to high levels of this chemical suffered liver and kidney damage, dermatitis and respiratory problems. EPA has set the drinking water standard for chromium at 0.1 parts per million (ppm) to protect against the risk of these adverse health effects. Drinking water that meets the EPA standard is associated with little to none of this risk and is considered safe with respect to chromium.

(19) *Mercury*. The United States Environmental Protection Agency (EPA) sets drinking water standards and has determined that mercury is a health concern at certain levels of exposure. This inorganic metal is used in electrical equipment and some water pumps. It usually gets into water as a result of improper waste disposal. This chemical has been shown to damage the kidney of laboratory animals such as rats when the animals are exposed at high levels over their lifetimes. EPA has set the drinking water standard for mercury at 0.002 parts per million (ppm) to protect against the risk of these adverse health effects. Drinking water that meets the EPA standard is associated with little to none of this risk and is considered safe with respect to mercury.

(20) *Nitrate*. The United States Environmental Protection Agency (EPA) sets drinking water standards and has determined that nitrate poses an acute health concern at certain levels of exposure. Nitrate is used in fertilizer and is found in sewage and wastes from human and/or farm animals and generally gets into drinking water from those activities. Excessive levels of nitrate in drinking water have caused serious illness and sometimes death in infants under six months of age. The serious illness in infants is caused because nitrate is converted to nitrite in the body. Nitrite interferes with the oxygen carrying capacity of the child's blood. This is an acute disease in that symptoms can develop rapidly in infants. In most cases, health deteriorates over a period of days. Symptoms include shortness of breath and blueness of the skin. Clearly, expert medical advice should be sought immediately if these symptoms occur. The purpose of this notice is to encourage parents and other responsible parties to provide infants with an alternate source of drinking water. Local and State health authorities are the best source for information concerning alternate sources of drinking water for infants. EPA has set the drinking water standard at 10 parts per million (ppm) for nitrate to protect against the risk of these adverse effects. EPA has also set a drinking water standard for nitrite at 1 ppm. To allow for the fact that the toxicity of nitrate and nitrite are additive, EPA has also established a standard for the sum of nitrate and nitrite at 10 ppm. Drinking water that meets the EPA standard is associated with little to none of this risk and is considered safe with respect to nitrate.

(21) *Nitrite*. The United States Environmental Protection Agency (EPA) sets drinking water standards and has determined that nitrite poses an acute health concern at certain levels of exposure. This inorganic chemical is used in fertilizers and is found in sewage and wastes from humans and/or farm animals and generally gets into drinking water as a result of those activities. While excessive levels of nitrite in drinking water have not been observed, other sources of nitrite have caused serious illness and sometimes death in infants under six months of age. The serious illness in infants is caused because nitrite interferes with the oxygen carrying capacity of the child's blood. This is an acute disease in that symptoms can develop rapidly. However, in most cases, health deteriorates over a period of days. Symptoms include shortness of breath and blueness of the skin. Clearly, expert medical advice should be sought immediately if these symptoms occur. The purpose of this notice is to encourage parents and other responsible parties to provide infants with an alternate source of drinking water. Local and State health authorities are the best source for information concerning alternate sources of

drinking water for infants. EPA has set the drinking water standard at 1 part per million (ppm) for nitrite to protect against the risk of these adverse effects. EPA has also set a drinking water standard for nitrate (converted to nitrite in humans) at 10 ppm and for the sum of nitrate and nitrite at 10 ppm. Drinking water that meets the EPA standard is associated with little to none of this risk and is considered safe with respect to nitrite.

(22) *Selenium.* The United States Environmental Protection Agency (EPA) sets drinking water standards and has determined that selenium is a health concern at certain high levels of exposure. Selenium is also an essential nutrient at low levels of exposure. This inorganic chemical is found naturally in food and soils and is used in electronics, photocopy operations, the manufacture of glass, chemicals, drugs, and as a fungicide and a feed additive. In humans, exposure to high levels of selenium over a long period of time has resulted in a number of adverse health effects, including a loss of feeling and control in the arms and legs. EPA has set the drinking water standard for selenium at 0.05 parts per million (ppm) to protect against the risk of these adverse health effects. Drinking water that meets the EPA standard is associated with little to none of this risk and is considered safe with respect to selenium.

(23) *Acrylamide.* The United States Environmental Protection Agency (EPA) sets drinking water standards and has determined that acrylamide is a health concern at certain levels of exposure. Polymers made from acrylamide are sometimes used to treat water supplies to remove particulate contaminants. Acrylamide has been shown to cause cancer in laboratory animals such as rats and mice when the animals are exposed at high levels over their lifetimes. Chemicals that cause cancer in laboratory animals also may increase the risk of cancer in humans who are exposed over long periods of time. Sufficiently large doses of acrylamide are known to cause neurological injury. EPA has set the drinking water standard for acrylamide using a treatment technique to reduce the risk of cancer or other adverse health effects which have been observed in laboratory animals. This treatment technique limits the amount of acrylamide in the polymer and the amount of the polymer which may be added to drinking water to remove particulates. Drinking water systems which comply with this treatment technique have little to no risk and are considered safe with respect to acrylamide.

(24) *Alachlor.* The United States Environmental Protection Agency (EPA) sets drinking water standards and has determined that alachlor is a health concern at certain levels of exposure. This organic chemical is a widely used pesticide. When soil and climatic conditions are favorable, alachlor may get into drinking water by runoff into surface water or by leaching into ground water. This chemical has been shown to cause cancer in laboratory animals such as rats and mice when the animals are exposed at high levels over their lifetimes. Chemicals that cause cancer in laboratory animals also may increase the risk of cancer in humans who are exposed over long periods of time. EPA has set the drinking water standard for alachlor at 0.002 parts per million (ppm) to reduce the risk of cancer or other adverse health effects which have been observed in laboratory animals. Drinking water that meets this standard is associated with little to none of this risk and is considered safe with respect to alachlor.

(25) *Aldicarb.* The United States Environmental Protection Agency (EPA) sets drinking water standards and has determined that aldicarb is a health concern at certain levels of exposure. Aldicarb is a widely used pesticide. Under certain soil and climatic conditions (e.g., sandy soil and high rainfall), aldicarb may leach into ground water after normal agricultural applications to crops such as potatoes or peanuts or may enter drinking water supplies as a result of surface runoff. This chemical has been shown to damage the nervous system in laboratory animals such as rats and dogs exposed to high levels. EPA has set the drinking water standard for aldicarb at 0.003 parts per million (ppm) to protect against the risk of adverse health effects. Drinking water that meets the

EPA standard is associated with little to none of this risk and is considered safe with respect to aldicarb.

(26) *Aldicarb sulfoxide.* The United States Environmental Protection Agency (EPA) sets drinking water standards and has determined that aldicarb sulfoxide is a health concern at certain levels of exposure. Aldicarb is a widely used pesticide. Aldicarb sulfoxide in ground water is primarily a breakdown product of aldicarb. Under certain soil and climatic conditions (e.g., sandy soil and high rainfall), aldicarb sulfoxide may leach into ground water after normal agricultural applications to crops such as potatoes or peanuts or may enter drinking water supplies as a result of surface runoff. This chemical has been shown to damage the nervous system in laboratory animals such as rats and dogs exposed to high levels. EPA has set the drinking water standard for aldicarb sulfoxide at 0.004 parts per million (ppm) to protect against the risk of adverse health effects. Drinking water that meets the EPA standard is associated with little to none of this risk and is considered safe with respect to aldicarb sulfoxide.

(27) *Aldicarb sulfone.* The United States Environmental Protection Agency (EPA) sets drinking water standards and has determined that aldicarb sulfone is a health concern at certain levels of exposure. Aldicarb is a widely used pesticide. Aldicarb sulfone is formed from the breakdown of aldicarb and is considered for registration as a pesticide under the name aldoxycarb. Under certain soil and climatic conditions (e.g., sandy soil and high rainfall), aldicarb sulfone may leach into ground water after normal agricultural applications to crops such as potatoes or peanuts or may enter drinking water supplies as a result of surface runoff. This chemical has been shown to damage the nervous system in laboratory animals such as rats and dogs exposed to high levels. EPA has set the drinking water standard for aldicarb sulfone at 0.002 parts per million (ppm) to protect against the risk of adverse health effects. Drinking water that meets the EPA standard is associated with little to none of this risk and is considered safe with respect to aldicarb sulfone.

(28) *Atrazine.* The United States Environmental Protection Agency (EPA) sets drinking water standards and has determined that atrazine is a health concern at certain levels of exposure. This organic chemical is a herbicide. When soil and climatic conditions are favorable, atrazine may get into drinking water by runoff into surface water or by leaching into ground water. This chemical has been shown to affect offspring of rats and the heart of dogs. EPA has set the drinking water standard for atrazine at 0.003 parts per million (ppm) to protect against the risk of these adverse health effects. Drinking water that meets the EPA standard is associated with little to none of this risk and is considered safe with respect to atrazine.

(29) *Carbofuran.* The United States Environmental Protection Agency (EPA) sets drinking water standards and has determined that carbofuran is a health concern at certain levels of exposure. This organic chemical is a pesticide. When soil and climatic conditions are favorable, carbofuran may get into drinking water by runoff into surface water or by leaching into ground water. This chemical has been shown to damage the nervous and reproductive systems of laboratory animals such as rats and mice exposed at high levels over their lifetimes. Some humans who were exposed to relatively large amounts of this chemical during their working careers also suffered damage to the nervous system. Effects on the nervous system are generally rapidly reversible. EPA has set the drinking water standard for carbofuran at 0.04 parts per million (ppm) to protect against the risk of these adverse health effects. Drinking water that meets the EPA standard is associated with little to none of this risk and is considered safe with respect to carbofuran.

(30) *Chlordane.* The United States Environmental Protection Agency (EPA sets drinking water standards and has determined that chlordane is a health concern at certain levels of exposure. This organic chemical is a pesticide used to control termites. Chlordane is not very mobile in soils. It usually gets

into drinking water after application near water supply intakes or wells. This chemical has been shown to cause cancer in laboratory animals such as rats and mice when the animals are exposed at high levels over their lifetimes. Chemicals that cause cancer in laboratory animals also may increase the risk of cancer in humans who are exposed over long periods of time. EPA has set the drinking water standard for chlordane at 0.002 parts per million (ppm) to reduce the risk of cancer or other adverse health effects which have been observed in laboratory animals. Drinking water that meets the EPA standard is associated with little to none of this risk and is considered safe with respect to chlordane.

(31) *Dibromochloropropane (DBCP).* The United States Environmental Protection Agency (EPA) sets drinking water standards and has determined that DBCP is a health concern at certain levels of exposure. This organic chemical was once a popular pesticide. When soil and climatic conditions are favorable, dibromochloropropane may get into drinking water by runoff into surface water or by leaching into ground water. This chemical has been shown to cause cancer in laboratory animals such as rats and mice when the animals are exposed at high levels over their lifetimes. Chemicals that cause cancer in laboratory animals also may increase the risk of cancer in humans who are exposed over long periods of time. EPA has set the drinking water standard for DBCP at 0.0002 parts per million (ppm) to reduce the risk of cancer or other adverse health effects which have been observed in laboratory animals. Drinking water that meets the EPA standard is associated with little to none of this risk and is considered safe with respect to DBCP.

(32) *o-Dichlorobenzene.* The United States Environmental Protection Agency (EPA) sets drinking water standards and has determined that o-dichlorobenzene is a health concern at certain levels of exposure. This organic chemical is used as a solvent in the production of pesticides and dyes. It generally gets into water by improper waste disposal. This chemical has been shown to damage the liver, kidney and the blood cells of laboratory animals such as rats and mice exposed to high levels during their lifetimes. Some industrial workers who were exposed to relatively large amounts of this chemical during working careers also suffered damage to the liver, nervous system, and circulatory system. EPA has set the drinking water standard for o-dichlorobenzene at 0.6 parts per million (ppm) to protect against the risk of these adverse health effects. Drinking water that meets the EPA standard is associated with little to none of this risk and is considered safe with respect to o-dichlorobenzene.

(33) *cis-1,2-Dichloroethylene.* The United States Environmental Protection Agency (EPA) establishes drinking water standards and has determined that cis-1,2-dichloroethylene is a health concern at certain levels of exposure. This organic chemical is used as a solvent and intermediate in chemical production. It generally gets into water by improper waste disposal. This chemical has been shown to damage the liver, nervous system, and circulatory system of laboratory animals such as rats and mice when exposed at high levels over their lifetimes. Some humans who were exposed to relatively large amounts of this chemical also suffered damage to the nervous system. EPA has set the drinking water standard for cis-1,2-dichloroethylene at 0.07 parts per million (ppm) to protect against the risk of these adverse health effects. Drinking water that meets the EPA standard is associated with little to none of this risk and is considered safe with respect to cis-1,2-dichloroethylene.

(34) *trans-1,2-Dichloroethylene.* The United States Environmental Protection Agency (EPA) establishes drinking water standards and has determined that trans-1,2-dichloroethylene is a health concern at certain levels of exposure. This organic chemical is used as a solvent and intermediate in chemical production. It generally gets into water by improper waste disposal. This chemical has been shown to damage the liver, nervous system, and the circulatory system of laboratory animals such as rats and mice when exposed at high levels over their lifetimes. Some humans who were exposed to relatively large amounts of this chemical also

suffered damage to the nervous system. EPA has set drinking water standard for trans-1,2-dichloroethylene at 0.1 parts per million (ppm) to protect against the risk of these adverse health effects. Drinking water that meets the EPA standard is associated with little to none of this risk and is considered safe with respect to trans-1,2-dichloroethylene.

(35) *1,2-Dichloropropane.* The United States Environmental Protection Agency (EPA) sets drinking water standards and has determined that 1,2-dichloropropane is a health concern at certain levels of exposure. This organic chemical is used as a solvent and pesticide. When soil and climatic conditions are favorable, 1,2-dichloropropane may get into drinking water by runoff into surface water or by leaching into ground water. It may also get into drinking water through improper waste disposal. This chemical has been shown to cause cancer in laboratory animals such as rats and mice when the animals are exposed at high levels over their lifetimes. Chemicals that cause cancer in laboratory animals also may increase the risk of cancer in humans who are exposed over long periods of time. EPA has set the drinking water standard for 1,2-dichloropropane at 0.005 parts per million (ppm) to reduce the risk of cancer or other adverse health effects which have been observed in laboratory animals. Drinking water that meets the EPA standard is associated with little to none of this risk and is considered safe with respect to 1,2-dichloropropane.

(36) *2,4–D.* The United States Environmental Protection Agency (EPA) sets drinking water standards and has determined that 2,4–D is a health concern at certain levels of exposure. This organic chemical is used as a herbicide and to control algae in reservoirs. When soil and climatic conditions are favorable, 2,4–D may get into drinking water by runoff into surface water or by leaching into ground water. This chemical has been shown to damage the liver and kidney of laboratory animals such as rats exposed at high levels during their lifetimes. Some humans who were exposed to relatively large amounts of this chemical also suffered damage to the nervous system. EPA has set the drinking water standard for 2,4–D at 0.07 parts per million (ppm) to protect against the risk of these adverse health effects. Drinking water that meets the EPA standard is associated with little to none of this risk and is considered safe with respect to 2,4–D.

(37) *Epichlorohydrin.* The United States Environmental Protection Agency (EPA) sets drinking water standards and has determined that epichlorohydrin is a health concern at certain levels of exposure. Polymers made from epichlorohydrin are sometimes used in the treatment of water supplies as a flocculent to remove particulates. Epichlorohydrin generally gets into drinking water by improper use of these polymers. This chemical has been shown to cause cancer in laboratory animals such as rats and mice when the animals are exposed at high levels over their lifetimes. Chemicals that cause cancer in laboratory animals also may increase the risk of cancer in humans who are exposed over long periods of time. EPA has set the drinking water standard for epichlorohydrin using a treatment technique to reduce the risk of cancer or other adverse health effects which have been observed in laboratory animals. This treatment technique limits the amount of epichlorohydrin in the polymer and the amount of the polymer which may be added to drinking water as a flocculent to remove particulates. Drinking water systems which comply with this treatment technique have little to no risk and are considered safe with respect to epichlorohydrin.

(38) *Ethylbenzene.* The United States Environmental Protection Agency (EPA) sets drinking water standards and has determined ethylbenzene is a health concern at certain levels of exposure. This organic chemical is a major component of gasoline. It generally gets into water by improper waste disposal or leaking gasoline tanks. This chemical has been shown to damage the kidney, liver, and nervous system of laboratory animals such as rats exposed to high levels during their lifetimes. EPA has set the drinking water standard for ethylbenzene at 0.7 part per million (ppm) to protect

against the risk of these adverse health effects. Drinking water that meets the EPA standard is associated with little to none of this risk and is considered safe with respect to ethylbenzene.

(39) *Ethylene dibromide (EDB).* The United States Environmental Protection Agency (EPA) sets drinking water standards and has determined that EDB is a health concern at certain levels of exposure. This organic chemical was once a popular pesticide. When soil and climatic conditions are favorable, EDB may get into drinking water by runoff into surface water or by leaching into ground water. This chemical has been shown to cause cancer in laboratory animals such as rats and mice when the animals are exposed at high levels over their lifetimes. Chemicals that cause cancer in laboratory animals also may increase the risk of cancer in humans who are exposed over long periods of time. EPA has set the drinking water standard for EDB at 0.00005 part per million (ppm) to reduce the risk of cancer or other adverse health effects which have been observed in laboratory animals. Drinking water that meets this standard is associated with little to none of this risk and is considered safe with respect to EDB.

(40) *Heptachlor.* The United States Environmental Protection Agency (EPA) sets drinking water standards and has determined that heptachlor is a health concern at certain levels of exposure. This organic chemical was once a popular pesticide. When soil and climatic conditions are favorable, heptachlor may get into drinking water by runoff into surface water or by leaching into ground water. This chemical has been shown to cause cancer in laboratory animals such as rats and mice when the animals are exposed at high levels over their lifetimes. Chemicals that cause cancer in laboratory animals also may increase the risk of cancer in humans who are exposed over long periods of time. EPA has set the drinking water standards for heptachlor at 0.0004 part per million (ppm) to reduce the risk of cancer or other adverse health effects which have been observed in laboratory animals. Drinking water that meets this standard is associated with little to none of this risk and is considered safe with respect to heptachlor.

(41) *Heptachlor epoxide.* The United States Environmental Protection Agency (EPA) sets drinking water standards and has determined that heptachlor epoxide is a health concern at certain levels of exposure. This organic chemical was once a popular pesticide. When soil and climatic conditions are favorable, heptachlor expoxide may get into drinking water by runoff into surface water or by leaching into ground water. This chemical has been shown to cause cancer in laboratory animals such as rats and mice when the animals are exposed at high levels over their lifetimes. Chemicals that cause cancer in laboratory animals also may increase the risk of cancer in humans who are exposed over long periods of time. EPA has set the drinking water standards for heptachlor epoxide at 0.0002 part per million (ppm) to reduce the risk of cancer or other adverse health effects which have been observed in laboratory animals. Drinking water that meets this standard is associated with little to none of this risk and is considered safe with respect to heptachlor epoxide.

(42) *Lindane.* The United States Environmental Protection Agency (EPA) sets drinking water standards and has determined that lindane is a health concern at certain levels of exposure. This organic chemical is used as a pesticide. When soil and climatic conditions are favorable, lindane may get into drinking water by runoff into surface water or by leaching into ground water. This chemical has been shown to damage the liver, kidney, nervous system, and immune system of laboratory animals such as rats, mice and dogs exposed at high levels during their lifetimes. Some humans who were exposed to relatively large amounts of this chemical also suffered damage to the nervous system and circulatory system. EPA has established the drinking water standard for lindane at 0.0002 part per million (ppm) to protect against the risk of these adverse health effects. Drinking water that meets the EPA standard is associated with little to none of this risk and is considered safe with respect to lindane.

(43) *Methoxychlor.* The United States Environmental Protection Agency (EPA) sets drinking water standards and has determined that methoxychlor is a health concern at certain levels of exposure. This organic chemical is used as a pesticide. When soil and climatic conditions are favorable, methoxychlor may get into drinking water by runoff into surface water or by leaching into ground water. This chemical has been shown to damage the liver, kidney, nervous system, and reproductive system of laboratory animals such as rats exposed at high levels during their lifetimes. It has also been shown to produce growth retardation in rats. EPA has set the drinking water standard for methoxychlor at 0.04 part per million (ppm) to protect against the risk of these adverse health effects. Drinking water that meets the EPA standard is associated with little to none of this risk and is considered safe with respect to methoxychlor.

(44) *Monochlorobenzene.* The United States Environmental Protection Agency (EPA) sets drinking water standards and has determined that monochlorobenzene is a health concern at certain levels of exposure. This organic chemical is used as a solvent. It generally gets into water by improper waste disposal. This chemical has been shown to damage the liver, kidney and nervous system of laboratory animals such as rats and mice exposed to high levels during their lifetimes. EPA has set the drinking water standard for monochlorobenzene at 0.1 part per million (ppm) to protect against the risk of these adverse health effects. Drinking water that meets the EPA standard is associated with little to none of this risk and is considered safe with respect to monochlorobenzene.

(45) *Polychlorinated biphenyls (PCBs).* The United States Environmental Protection Agency (EPA) sets drinking water standards and has determined that polychlorinated biphenyls (PCBs) are a health concern at certain levels of exposure. These organic chemicals were once widely used in electrical transformers and other industrial equipment. They generally get into drinking water by improper waste disposal or leaking electrical industrial equipment. This chemical has been shown to cause cancer in laboratory animals such as rats and mice when the animals are exposed at high levels over their lifetimes. Chemicals that cause cancer in laboratory animals also may increase the risk of cancer in humans who are exposed over long periods of time. EPA has set the drinking water standard for PCBs at 0.0005 part per million (ppm) to reduce the risk of cancer or other adverse health effects which have been observed in laboratory animals. Drinking water that meets this standard is associated with little to none of this risk and is considered safe with respect to PCBs.

(46) *Pentachlorophenol.* The United States Environmental Protection Agency (EPA) sets drinking water standards and has determined that pentachlorophenol is a health concern at certain levels of exposure. This organic chemical is used as a wood preservative, herbicide, disinfectant, and defoliant. It generally gets into drinking water by runoff into surface water or leaching into ground water. This chemical has been shown to produce adverse reproductive effects and to damage the liver and kidneys of laboratory animals such as rats exposed to high levels during their lifetimes. Some humans who were exposed to relatively large amounts of this chemical also suffered damage to the liver and kidneys. This chemical has been shown to cause cancer in laboratory animals such as rats and mice when the animals are exposed to high levels over their lifetimes. Chemicals that cause cancer in laboratory animals also may increase the risk of cancer in humans who are exposed over long periods of time. EPA has set the drinking water standard for pentachlorophenol at 0.001 parts per million (ppm) to protect against the risk of cancer or other adverse health effects. Drinking water that meets the EPA standard is associated with little to none of this risk and is considered safe with respect to pentachlorophenol.

(47) *Styrene.* The United States Environmental Protection Agency (EPA) sets drinking water standards and has determined that styrene is a health concern at certain levels of exposure. This organic chemical is commonly used to make plastics and is sometimes

a component of resins used for drinking water treatment. Styrene may get into drinking water from improper waste disposal. This chemical has been shown to damage the liver and nervous system in laboratory animals when exposed at high levels during their lifetimes. EPA has set the drinking water standard for styrene at 0.1 part per million (ppm) to protect against the risk of these adverse health effects. Drinking water that meets the EPA standard is associated with little to none of this risk and is considered safe with respect to styrene.

(48) *Tetrachloroethylene.* The United States Environmental Protection Agency (EPA) sets drinking water standards and has determined that tetrachloroethylene is a health concern at certain levels of exposure. This organic chemical has been a popular solvent, particularly for dry cleaning. It generally gets into drinking water by improper waste disposal. This chemical has been shown to cause cancer in laboratory animals such as rats and mice when the animals are exposed at high levels over their lifetimes. Chemicals that cause cancer in laboratory animals also may increase the risk of cancer in humans who are exposed over long periods of time. EPA has set the drinking water standard for tetrachloroethylene at 0.005 part per million (ppm) to reduce the risk of cancer or other adverse health effects which have been observed in laboratory animals. Drinking water that meets this standard is associated with little to none of this risk and is considered safe with respect to tetrachloroethylene.

(49) *Toluene.* The United States Environmental Protection Agency (EPA) sets drinking water standards and has determined that toluene is a health concern at certain levels of exposure. This organic chemical is used as a solvent and in the manufacture of gasoline for airplanes. It generally gets into water by improper waste disposal or leaking underground storage tanks. This chemical has been shown to damage the kidney, nervous system, and circulatory system of laboratory animals such as rats and mice exposed to high levels during their lifetimes. Some industrial workers who were exposed to relatively large amounts of this chemical during working careers also suffered damage to the liver, kidney and nervous system. EPA has set the drinking water standard for toluene at 1 part per million (ppm) to protect against the risk of adverse health effects. Drinking water that meets the EPA standard is associated with little to none of this risk and is considered safe with respect to toluene.

(50) *Toxaphene.* The United States Environmental Protection Agency (EPA) sets drinking water standards and has determined that toxaphene is a health concern at certain levels of exposure. This organic chemical was once a pesticide widely used on cotton, corn, soybeans, pineapples and other crops. When soil and climatic conditions are favorable, toxaphene may get into drinking water by runoff into surface water or by leaching into ground water. This chemical has been shown to cause cancer in laboratory animals such as rats and mice when the animals are exposed at high levels over their lifetimes. Chemicals that cause cancer in laboratory animals also may increase the risk of cancer in humans who are exposed over long periods of time. EPA has set the drinking water standard for toxaphene at 0.003 part per million (ppm) to reduce the risk of cancer or other adverse health effects which have been observed in laboratory animals. Drinking water that meets this standard is associated with little to none of this risk and is considered safe with respect to toxaphene.

(51) *2,4,5–TP.* The United States Environmental Protection Agency (EPA) sets drinking water standards and has determined that 2,4,5–TP is a health concern at certain levels of exposure. This organic chemical is used as a herbicide. When soil and climatic conditions are favorable, 2,4,5–TP may get into drinking water by runoff into surface water or by leaching into ground water. This chemical has been shown to damage the liver and kidney of laboratory animals such as rats and dogs exposed to high levels during their lifetimes. Some industrial workers who were exposed to relatively large amounts of this chemical during working careers also suffered damage to the

nervous system. EPA has set the drinking water standard for 2,4,5–TP at 0.05 part per million (ppm) to protect against the risk of these adverse health effects. Drinking water that meets the EPA standard is associated with little to none of this risk and is considered safe with respect to 2,4,5–TP.

(52) *Xylenes.* The United States Environmental Protection Agency (EPA) sets drinking water standards and has determined that xylene is a health concern at certain levels of exposure. This organic chemical is used in the manufacture of gasoline for airplanes and as a solvent for pesticides, and as a cleaner and degreaser of metals. It usually gets into water by improper waste disposal. This chemical has been shown to damage the liver, kidney and nervous system of laboratory animals such as rats and dogs exposed to high levels during their lifetimes. Some humans who were exposed to relatively large amounts of this chemical also suffered damage to the nervous system. EPA has set the drinking water standard for xylene at 10 parts per million (ppm) to protect against the risk of these adverse health effects. Drinking water that meets the EPA standard is associated with little to none of this risk and is considered safe with respect to xylene.

(53) *Antimony*. The United States Environmental Protection Agency (EPA) sets drinking water standards and has determined that antimony is a health concern at certain levels of exposure. This inorganic chemical occurs naturally in soils, ground water and surface waters and is often used in the flame retardant industry. It is also used in ceramics, glass, batteries, fireworks and explosives. It may get into drinking water through natural weathering of rock, industrial production, municipal waste disposal or manufacturing processes. This chemical has been shown to decrease longevity, and altered blood levels of cholesterol and glucose in laboratory animals such as rats exposed to high levels during their lifetimes. EPA has set the drinking water standard for antimony at 0.006 parts per million (ppm) to protect against the risk of these adverse health effects. Drinking water which meets the EPA standard is associated with little to none of this risk and should be considered safe with respect to antimony.

(54) *Beryllium.* The United States Environmental Protection Agency (EPA) sets drinking water standards and has determined that beryllium is a health concern at certain levels of exposure. This inorganic metal occurs naturally in soils, ground water and surface waters and is often used in electrical equipment and electrical components. It generally gets into water from runoff from mining operations, discharge from processing plants and improper waste disposal. Beryllium compounds have been associated with damage to the bones and lungs and induction of cancer in laboratory animals such as rats and mice when the animals are exposed at high levels over their lifetimes. There is limited evidence to suggest that beryllium may pose a cancer risk via drinking water exposure. Therefore, EPA based the health assessment on noncancer effects with an extra uncertainty factor to account for possible carcinogenicity. Chemicals that cause cancer in laboratory animals also may increase the risk of cancer in humans who are exposed over long periods of time. EPA has set the drinking water standard for beryllium at 0.004 part per million (ppm) to protect against the risk of these adverse health effects. Drinking water which meets the EPA standard is associated with little to none of this risk and should be considered safe with respect to beryllium.

(55) *Cyanide.* The United States Environmental Protection Agency (EPA) sets drinking water standards and has determined that cyanide is a health concern at certain levels of exposure. This inorganic chemical is used in electroplating, steel processing, plastics, synthetic fabrics and fertilizer products. It usually gets into water as a result of improper waste disposal. This chemical has been shown to damage the spleen, brain and liver of humans fatally poisoned with cyanide. EPA has set the drinking water standard for cyanide at 0.2 parts per million (ppm) to protect against the risk of these adverse health effects. Drinking water which meets the EPA standard is associated with little to none of this risk

and should be considered safe with respect to cyanide.

(56) [Reserved]

(57) *Thallium.* The United States Environmental Protection Agency (EPA) sets drinking water standards and has determined that thallium is a health concern at certain high levels of exposure. This inorganic metal is found naturally in soils and is used in electronics, pharmaceuticals, and the manufacture of glass and alloys. This chemical has been shown to damage the kidney, liver, brain and intestines of laboratory animals when the animals are exposed at high levels over their lifetimes. EPA has set the drinking water standard for thallium at 0.002 parts per million (ppm) to protect against the risk of these adverse health effects. Drinking water which meets the EPA standard is associated with little to none of this risk and should be considered safe with respect to thallium.

(58) *Benzo[a]pyrene.* The United States Environmental Protection Agency (EPA) sets drinking water standards and has determined that benzo[a]pyrene is a health concern at certain levels of exposure. Cigarette smoke and charbroiled meats are common source of general exposure. The major source of benzo[a]pyrene in drinking water is the leaching from coal tar lining and sealants in water storage tanks. This chemical has been shown to cause cancer in animals such as rats and mice when the animals are exposed at high levels. EPA has set the drinking water standard for benzo[a]pyrene at 0.0002 parts per million (ppm) to protect against the risk of cancer. Drinking water which meets the EPA standard is associated with little to none of this risk and should be considered safe with respect to benzo[a]pyrene.

(59) *Dalapon.* The United States Environmental Protection Agency (EPA) sets drinking water standards and has determined that dalapon is a health concern at certain levels of exposure. This organic chemical is a widely used herbicide. It may get into drinking water after application to control grasses in crops, drainage ditches and along railroads. This chemical has been shown to cause damage to the kidney and liver in laboratory animals when the animals are exposed to high levels over their lifetimes. EPA has set the drinking water standard for dalapon at 0.2 parts per million (ppm) to protect against the risk of these adverse health effects. Drinking water which meets the EPA standard is associated with little to none of this risk and should be considered safe with respect to dalapon.

(60) *Dichloromethane.* The United States Environmental Protection Agency (EPA) sets drinking water standards and has determined that dichloromethane (methylene chloride) is a health concern at certain levels of exposure. This organic chemical is a widely used solvent. It is used in the manufacture of paint remover, as a metal degreaser and as an aerosol propellant. It generally gets into drinking water after improper discharge of waste disposal. This chemical has been shown to cause cancer in laboratory animals such as rats and mice when the animals are exposed at high levels over their lifetimes. Chemicals that cause cancer in laboratory animals also may increase the risk of cancer in humans who are exposed over long periods of time. EPA has set the drinking water standard for dichloromethane at 0.005 parts per million (ppm) to reduce the risk of cancer or other adverse health effects which have been observed in laboratory animals. Drinking water which meets this standard is associated with little to none of this risk and should be considered safe with respect to dichloromethane.

(61) *Di (2-ethylhexyl)adipate.* The United States Environmental Protection Agency (EPA) sets drinking water standards and has determined that di(2-ethylhexyl)adipate is a health concern at certain levels of exposure. Di(2-ethylhexyl)adipate is a widely used plasticizer in a variety of products, including synthetic rubber, food packaging materials and cosmetics. It may get into drinking water after improper waste disposal. This chemical has been shown to damage liver and testes in laboratory animals such as rats and mice exposed to high levels. EPA has set the drinking water standard for di(2-ethylhexyl)adipate at 0.4 parts per million (ppm) to protect against the

risk of adverse health effects. Drinking water which meets the EPA standards is associated with little to none of this risk and should be considered safe with respect to di(2-ethylhexyl)adipate.

(62) *Di(2-ethylhexyl)phthalate.* The United States Environmental Protection Agency (EPA) sets drinking water standards and has determined that di(2-ethylhexyl)phthalate is a health concern at certain levels of exposure. Di(2-ethylhexyl)phthalate is a widely used plasticizer, which is primarily used in the production of polyvinyl chloride (PVC) resins. It may get into drinking water after improper waste disposal. This chemical has been shown to cause cancer in laboratory animals such as rats and mice exposed to high levels over their lifetimes. EPA has set the drinking water standard for di(2-ethylhexyl)phthalate at 0.006 parts per million (ppm) to reduce the risk of cancer or other adverse health effects which have been observed in laboratory animals. Drinking water which meets the EPA standard is associated with little to none of this risk and should be considered safe with respect to di(2-ethylhexyl)phthalate.

(63) *Dinoseb.* The United States Environmental Protection Agency (EPA) sets drinking water standards and has determined that dinoseb is a health concern at certain levels of exposure. Dinoseb is a widely used pesticide and generally gets into drinking water after application on orchards, vineyards and other crops. This chemical has been shown to damage the thyroid and reproductive organs in laboratory animals such as rats exposed to high levels. EPA has set the drinking water standard for dinoseb at 0.007 parts per million (ppm) to protect against the risk of adverse health effects. Drinking water which meets the EPA standard is associated with little to none of this risk and should be considered safe with respect to dinoseb.

(64) *Diquat.* The United States Environmental Protection Agency (EPA) sets drinking water standards and has determined that diquat is a health concern at certain levels of exposure. This organic chemical is a herbicide used to control terrestrial and aquatic weeds. It may get into drinking water by runoff into surface water. This chemical has been shown to damage the liver, kidney and gastrointestinal tract and causes cataract formation in laboratory animals such as dogs and rats exposed at high levels over their lifetimes. EPA has set the drinking water standard for diquat at 0.02 parts per million (ppm) to protect against the risk of these adverse health effects. Drinking water which meets the EPA standard is associated with little to none of this risk and should be considered safe with respect to diquat.

(65) *Endothall.* The United States Environmental Protection Agency (EPA) has determined that endothall is a health concern at certain levels of exposure. This organic chemical is a herbicide used to control terrestrial and aquatic weeds. It may get into water by runoff into surface water. This chemical has been shown to damage the liver, kidney, gastrointestinal tract and reproductive system of laboratory animals such as rats and mice exposed at high levels over their lifetimes. EPA has set the drinking water standard for endothall at 0.1 parts per million (ppm) to protect against the risk of these adverse health effects. Drinking water which meets the EPA standard is associated with little to none of this risk and should be considered safe with respect to endothall.

(66) *Endrin.* The United States Environmental Protection Agency (EPA) sets drinking water standards and has determined that endrin is a health concern at certain levels of exposure. This organic chemical is a pesticide no longer registered for use in the United States. However, this chemical is persistent in treated soils and accumulates in sediments and aquatic and terrestrial biota. This chemical has been shown to cause damage to the liver, kidney and heart in laboratory animals such as rats and mice when the animals are exposed at high levels over their lifetimes. EPA has set the drinking water standard for endrin at 0.002 parts per million (ppm) to protect against the risk of these adverse health effects which have been observed in laboratory animals. Drinking water that meets the EPA standard is associated with little to none of this risk and should be considered safe with respect to endrin.

(67) *Glyphosate.* The United States Environmental Protection Agency (EPA) sets drinking water standards and has determined that glyphosate is a health concern at certain levels of exposure. This organic chemical is a herbicide used to control grasses and weeds. It may get into drinking water by runoff into surface water. This chemical has been shown to cause damage to the liver and kidneys in laboratory animals such as rats and mice when the animals are exposed at high levels over their lifetimes. EPA has set the drinking water standard for glyphosate at 0.7 parts per million (ppm) to protect against the risk of these adverse health effects. Drinking water which meets the EPA standard is associated with little to none of this risk and should be considered safe with respect to glyphosate.

(68) *Hexachlorobenzene.* The United States Environmental Protection Agency (EPA) sets drinking water standards and has determined that hexachlorobenzene is a health concern at certain levels of exposure. This organic chemical is produced as an impurity in the manufacture of certain solvents and pesticides. This chemical has been shown to cause cancer in laboratory animals such as rats and mice when the animals are exposed to high levels during their lifetimes. Chemicals that cause cancer in laboratory animals also may increase the risk of cancer in humans who are exposed over long periods of time. EPA has set the drinking water standard for hexachlorobenzene at 0.001 parts per million (ppm) to protect against the risk of cancer and other adverse health effects. Drinking water which meets the EPA standard is associated with little to none of this risk and should be considered safe with respect to hexachlorobenzene.

(69) *Hexachlorocyclopentadiene.* The United States Environmental Protection Agency (EPA) establishes drinking water standards and has determined that hexachlorocyclopentadiene is a health concern at certain levels of exposure. This organic chemical is used as an intermediate in the manufacture of pesticides and flame retardants. It may get into water by discharge from production facilities. This chemical has been shown to damage the kidney and the stomach of laboratory animals when exposed at high levels over their lifetimes. EPA has set the drinking water standard for hexachlorocyclopentadiene at 0.05 parts per million (ppm) to protect against the risk of these adverse health effects. Drinking water which meets the EPA standard is associated with little to none of this risk and should be considered safe with respect to hexachlorocyclopentadiene.

(70) *Oxamyl.* The United States Environmental Protection Agency (EPA) establishes drinking water standards and has determined that oxamyl is a health concern at certain levels of exposure. This organic chemical is used as a pesticide for the control of insects and other pests. It may get into drinking water by runoff into surface water or leaching into ground water. This chemical has been shown to damage the kidneys of laboratory animals such as rats when exposed at high levels over their lifetimes. EPA has set the drinking water standard for oxamyl at 0.2 parts per million (ppm) to protect against the risk of these adverse health effects. Drinking water which meets the EPA standard is associated with little to none of this risk and should be considered safe with respect to oxamyl.

(71) *Picloram.* The United States Environmental Protection Agency (EPA) sets drinking water standards and has determined that picloram is a health concern at certain levels of exposure. This organic chemical is used as a pesticide for broadleaf weed control. It may get into drinking water by runoff into surface water or leaching into ground water as a result of pesticide application and improper waste disposal. This chemical has been shown to cause damage to the kidneys and liver in laboratory animals such as rats when the animals are exposed at high levels over their lifetimes. EPA has set the drinking water standard for picloram at 0.5 parts per million (ppm) to protect against the risk of these adverse health effects. Drinking water which meets the EPA standard is associated with little to none of this risk and should be considered safe with respect to picloram.

(72) *Simazine.* The United States Environmental Protection Agency (EPA) sets drinking water standards and has determined that simazine is a health concern at certain levels of exposure. This organic chemical is a herbicide used to control annual grasses and broadleaf weeds. It may leach into ground water or runs off into surface water after application. This chemical may cause cancer in laboratory animals such as rats and mice exposed at high levels during their lifetimes. Chemicals that cause cancer in laboratory animals also may increase the risk of cancer in humans who are exposed over long periods of time. EPA has set the drinking water standard for simazine at 0.004 parts per million (ppm) to reduce the risk of cancer or other adverse health effects. Drinking water which meets the EPA standard is associated with little to none of this risk and should be considered safe with respect to simazine.

(73) *1,2,4-Trichlorobenzene.* The United States Environmental Protection Agency (EPA) sets drinking water standards and has determined that 1,2,4-trichlorobenzene is a health concern at certain levels of exposure. This organic chemical is used as a dye carrier and as a precursor in herbicide manufacture. It generally gets into drinking water by discharges from industrial activities. This chemical has been shown to cause damage to several organs, including the adrenal glands. EPA has set the drinking water standard for 1,2,4-trichlorobenzene at 0.07 parts per million (ppm) to protect against the risk of these adverse health effects. Drinking water which meets the EPA standard is associated with little to none of this risk and should be considered safe with respect to 1,2,4-trichlorobenzene.

(74) *1,1,2-Trichloroethane.* The United States Environmental Protection Agency (EPA) sets drinking water standards and has determined 1,1,2-trichloroethane is a health concern at certain levels of exposure. This organic chemical is an intermediate in the production of 1,1-dichloroethylene. It generally gets into water by industrial discharge of wastes. This chemical has been shown to damage the kidney and liver of laboratory animals such as rats exposed to high levels during their lifetimes. EPA has set the drinking water standard for 1,1,2-trichloroethane at 0.005 parts per million (ppm) to protect against the risk of these adverse health effects. Drinking water which meets the EPA standard is associated with little to none of this risk and should be considered safe with respect to 1,1,2-trichloroethane.

(75) *2,3,7,8–TCDD (Dioxin).* The United States Environmental Protection Agency (EPA) sets drinking water standards and has determined that dioxin is a health concern at certain levels of exposure. This organic chemical is an impurity in the production of some pesticides. It may get into drinking water by industrial discharge of wastes. This chemical has been shown to cause cancer in laboratory animals such as rats and mice when the animals are exposed at high levels over their lifetimes. Chemicals that cause cancer in laboratory animals also may increase the risk of cancer in humans who are exposed over long periods of time. EPA has set the drinking water standard for dioxin at 0.00000003 parts per million (ppm) to reduce the risk of cancer or other adverse health effects which have been observed in laboratory animals. Drinking water which meets this standard is associated with little to none of this risk and should be considered safe with respect to dioxin.

(f) *Public notices for fluoride.* Notice of violations of the maximum contaminant level for fluoride, notices of variances and exemptions from the maximum contaminant level for fluoride, and notices of failure to comply with variance and exemption schedules for the maximum contaminant level for fluoride shall consist of the public notice prescribed in § 143.5(b), plus a description of any steps which the system is taking to come into compliance.

(g) *Public notification by the State.* The State may give notice to the public required by this section on behalf of the owner or operator of the public water system if the State complies with the requirements of this section. However, the owner or operator of the public

water system remains legally responsible for ensuring that the requirements of this section are met.

[52 FR 41546, Oct. 28, 1987, as amended at 54 FR 15188, Apr. 17, 1989; 54 FR 27527, 27566, June 29, 1989; 55 FR 25064, June 19, 1990; 56 FR 3587, Jan. 30, 1991; 56 FR 26548, June 7, 1991; 56 FR 30279, July 1, 1991; 57 FR 31843, July 17, 1992; 59 FR 34323, July 1, 1994; 60 FR 33932, June 29, 1995]

§ 141.33 Record maintenance.

Any owner or operator of a public water system subject to the provisions of this part shall retain on its premises or at a convenient location near its premises the following records:

(a) Records of bacteriological analyses made pursuant to this part shall be kept for not less than 5 years. Records of chemical analyses made pursuant to this part shall be kept for not less than 10 years. Actual laboratory reports may be kept, or data may be transferred to tabular summaries, provided that the following information is included:

(1) The date, place, and time of sampling, and the name of the person who collected the sample;

(2) Identification of the sample as to whether it was a routine distribution system sample, check sample, raw or process water sample or other special purpose sample;

(3) Date of analysis;

(4) Laboratory and person responsible for performing analysis;

(5) The analytical technique/method used; and

(6) The results of the analysis.

(b) Records of action taken by the system to correct violations of primary drinking water regulations shall be kept for a period not less than 3 years after the last action taken with respect to the particular violation involved.

(c) Copies of any written reports, summaries or communications relating to sanitary surveys of the system conducted by the system itself, by a private consultant, or by any local, State or Federal agency, shall be kept for a period not less than 10 years after completion of the sanitary survey involved.

(d) Records concerning a variance or exemption granted to the system shall be kept for a period ending not less than 5 years following the expiration of such variance or exemption.

§ 141.34 [Reserved]

§ 141.35 Reporting and public notification for certain unregulated contaminants.

(a) The requirements of this section only apply to the contaminants listed in § 141.40.

(b) The owner or operator of a community water system or non-transient, non-community water system who is required to monitor under § 141.40 shall send a copy of the results of such monitoring within 30 days of receipt and any public notice under paragraph (d) of this section to the State.

(c) The State, or the community water system or non-transient, non-community water system if the State has not adopted regulations equivalent to § 141.40, shall furnish the following information to the Administrator for each sample analyzed under § 141.40:

(1) Results of all analytical methods, including negatives;

(2) Name and address of the system that supplied the sample;

(3) Contaminant(s);

(4) Analytical method(s) used;

(5) Date of sample;

(6) Date of analysis.

(d) The owner or operator shall notify persons served by the system of the availability of the results of sampling conducted under § 141.40 by including a notice in the first set of water bills issued by the system after the receipt of the results or written notice within three months. The notice shall identify a person and supply the telephone number to contact for information on the monitoring results. For surface water systems, public notification is required only after the first quarter's monitoring and must include a statement that additional monitoring will be conducted for three more quarters with the results available upon request.

[52 FR 25714, July 8, 1987; 53 FR 25110, July 1, 1988]

Subpart E—Special Regulations, Including Monitoring Regulations and Prohibition on Lead Use

§ 141.40 Special monitoring for inorganic and organic contaminants.

(a) All community and non-transient, non-community water systems shall monitor for the contaminants listed in paragraph (e) in this section by date specified in table 1:

TABLE 1—MONITORING SCHEDULE BY SYSTEM SIZE

Number of persons served	Monitoring to begin no later than—
Over 10,000	Jan. 1, 1988.
3,300 to 10,000	Jan. 1, 1989.
Less than 3,300	Jan. 1, 1991.

(b) Surface water systems shall sample at points in the distribution system representative of each water source or at entry points to the distribution system after any application of treatment. The minimum number of samples is one year of quarterly samples per water source.

(c) Ground water systems shall sample at points of entry to the distribution system representative of each well after any application of treatment. The minimum number of samples is one sample per entry point to the distribution system.

(d) The State may require confirmation samples for positive or negative results.

(e) Community water systems and non-transient, non-community water systems shall monitor for the following contaminants except as provided in paragraph (f) of this section:

(1) Chloroform
(2) Bromodichloromethane
(3) Chlorodibromomethane
(4) Bromoform
(5) Dibromomethane
(6) m-Dichlorobenzene
(7) [Reserved]
(8) 1,1-Dichloropropene
(9) 1,1-Dichloroethane
(10) 1,1,2,2-Tetrachloroethane
(11) 1,3-Dichloropropane
(12) Chloromethane
(13) Bromomethane
(14) 1,2,3-Trichloropropane
(15) 1,1,1,2-Tetrachloroethane
(16) Chloroethane
(17) 2,2-Dichloropropane
(18) o-Chlorotoluene
(19) p-Chlorotoluene
(20) Bromobenzene
(21) 1,3-Dichloropropene

(f) [Reserved]

(g) Analysis for the unregulated contaminants listed under paragraphs (e) and (j) of this section shall be conducted using EPA Methods 502.2 or 524.2, or their equivalent as determined by EPA, except analysis for bromodichloromethane, bromoform, chlorodibromomethane and chloroform under paragraph (e) of this section also may be conducted by EPA Method 551, and analysis for 1,2,3-trichloropropane also may be conducted by EPA Method 504.1. A source for the EPA methods is referenced at § 141.24(e).

(h) Analysis under this section shall only be conducted by laboratories certified under § 141.24(f)(17).

(i) Public water systems may use monitoring data collected any time after January 1, 1983 to meet the requirements for unregulated monitoring, provided that the monitoring program was consistent with the requirements of this section. In addition, the results of EPA's Ground Water Supply Survey may be used in a similar manner for systems supplied by a single well.

(j) Monitoring for the following compounds is required at the discretion of the State:

(1) 1,2,4-Trimethylbenzene;
(2) 1,2,3-Trichlorobenzene;
(3) n-Propylbenzene;
(4) n-Butylbenzene;
(5) Naphthalene;
(6) Hexachlorobutadiene;
(7) 1,3,5-Trimethylbenzene;
(8) p-Isopropyltoluene;
(9) Isopropylbenzene;
(10) Tert-butylbenzene;
(11) Sec-butylbenzene;
(12) Fluorotrichloromethane;
(13) Dichlorodifluoromethane;
(14) Bromochloromethane.

(k) Instead of performing the monitoring required by this section, a community water system or non-transient non-community water system serving fewer than 150 service connections may send a letter to the State stating that

the system is available for sampling. This letter must be sent to the State no later than January 1, 1991. The system shall not send such samples to the State, unless requested to do so by the State.

(l) All community and non-transient, non-community water systems shall repeat the monitoring required in § 141.40 no less frequently than every five years from the dates specified in § 141.40(a).

(m) States or public water systems may composite up to five samples when monitoring for substances in § 141.40 (e) and (j) of this section.

(n) Monitoring of the contaminants listed in § 141.40(n) (11) and (12) shall be conducted as follows:

(1) Each community and non-transient, non-community water system shall take four consecutive quarterly samples at each sampling point for each contaminant listed in paragraph (n)(11) of this section and report the results to the State. Monitoring must be completed by December 31, 1995.

(2) Each community and non-transient non-community water system shall take one sample at each sampling point for each contaminant listed in paragraph (n)(12) of this section and report the results to the States. Monitoring must be completed by December 31, 1995.

(3) Each community and non-transient non-community water system may apply to the State for a waiver from the requirements of paragraph (n)(1) and (2) of this section.

(4) The State may grant a waiver for the requirement of paragraph (n)(1) of this section based on the criteria specified in § 141.24(h)(6). The State may grant a waiver from the requirement of paragraph (n)(2) of this section if previous analytical results indicate contamination would not occur, provided this data was collected after January 1, 1990.

(5) Groundwater systems shall take a minimum of one sample at every entry point to the distribution system which is representative of each well after treatment (hereafter called a sampling point). Each sample must be taken at the same sampling point unless conditions make another sampling point more representative of each source or treatment plant.

(6) Surface water systems shall take a minimum of one sample at points in the distribution system that are representative of each source or at each entry point to the distribution system after treatment (hereafter called a sampling point). Each sample must be taken at the same sampling point unless conditions make another sampling point more representative of each source or treatment plant.

NOTE: For purposes of this paragraph, surface water systems include systems with a combination of surface and ground sources.

(7) If the system draws water from more than one source and the sources are combined before distribution, the system must sample at an entry point to the distribution system during periods of normal operating conditions (i.e., when water representative of all sources is being used).

(8) The State may require a confirmation sample for positive or negative results.

(9) The State may reduce the total number of samples a system must analyze by allowing the use of compositing. Composite samples from a maximum of five sampling points are allowed. Compositing of samples must be done in the laboratory and the composite sample must be analyzed within 14 days of collection. If the population served by the system is >3,300 persons, then compositing may only be permitted by the State at sampling points within a single system. In systems serving ≤3,300 persons, the State may permit compositing among different systems provided the 5-sample limit is maintained.

(10) Instead of performing the monitoring required by this section, a community water system or non-transient non-community water system serving fewer than 150 service connections may send a letter to the State stating that the system is available for sampling. This letter must be sent to the State by January 1, 1994. The system shall not send such samples to the State, unless requested to do so by the State.

(11) Systems shall monitor for the unregulated organic contaminants listed below, using the method(s) identified below and using the analytical test procedures contained in *Technical Notes on Drinking Water Methods*, EPA–600/R–94–173, October 1994, which is available at NTIS, PB95–104766. Method 6610 shall be followed in accordance with the *Standard Methods for the Examination of Water and Wastewater 18th Edition Supplement*, 1994, American Public Health Association. This incorporation by reference was approved by the Director of the Federal Register in accordance with 5 U.S.C. 552(a) and 1 CFR part 51. Copies may be obtained from the American Public Health Association, 1015 Fifteenth Street NW, Washington, DC 20005. Copies may be inspected at EPA's Drinking Water Docket, 401 M Street, SW., Washington, DC 20460; or at the Office of the Federal Register, 800 North Capitol Street, NW., Suite 700, Washington, DC. A source for EPA methods 505, 507, 508, 508.1, 515.2, 525.2 and 531.1 is referenced at §141.24(e).

Contaminants	Method
aldicarb	531.1, 6610.
aldicarb sulfone	531.1, 6610.
aldicarb sulfoxide	531.1, 6610.
aldrin	505, 508, 525.2, 508.1.
butachlor	507, 525.2.
carbaryl	531.1, 6610.
dicamba	515.2, 555, 515.1.
dieldrin	505, 508, 525.2, 508.1.
3-hydroxycarbofuran	531.1, 6610.
methomyl	531.1, 6610.
metolachlor	507, 525.2, 508.1.
metribuzin	507, 525.2, 508.1.
propachlor	508, 525.2, 508.1.

(12) Systems shall monitor for sulfate, an unregulated inorganic contaminant, by using the methods listed at §143.4(b).

[52 FR 25715, July 8, 1987; 53 FR 25110, July 1, 1988, as amended at 56 FR 3592, Jan. 30, 1991; 57 FR 31845, July 17, 1992; 59 FR 34323, July 1, 1994; 59 FR 62469, Dec. 5, 1994]

§141.41 Special monitoring for sodium.

(a) Suppliers of water for community public water systems shall collect and analyze one sample per plant at the entry point of the distribution system for the determination of sodium concentration levels; samples must be collected and analyzed annually for systems utilizing surface water sources in whole or in part, and at least every three years for systems utilizing solely ground water sources. The minimum number of samples required to be taken by the system shall be based on the number of treatment plants used by the system, except that multiple wells drawing raw water from a single aquifer may, with the State approval, be considered one treatment plant for determining the minimum number of samples. The supplier of water may be required by the State to collect and analyze water samples for sodium more frequently in locations where the sodium content is variable.

(b) The supplier of water shall report to EPA and/or the State the results of the analyses for sodium within the first 10 days of the month following the month in which the sample results were received or within the first 10 days following the end of the required monitoring period as stipulated by the State, whichever of these is first. If more than annual sampling is required the supplier shall report the average sodium concentration within 10 days of the month following the month in which the analytical results of the last sample used for the annual average was received. The supplier of water shall not be required to report the results to EPA where the State has adopted this regulation and results are reported to the State. The supplier shall report the results to EPA where the State has not adopted this regulation.

(c) The supplier of water shall notify appropriate local and State public health officials of the sodium levels by written notice by direct mail within three months. A copy of each notice required to be provided by this paragraph shall be sent to EPA and/or the State within 10 days of its issuance. The supplier of water is not required to notify appropriate local and State public health officials of the sodium levels where the State provides such notices in lieu of the supplier.

(d) Analyses for sodium shall be conducted as directed in §141.23(k)(1).

[45 FR 57345, Aug. 27, 1980, as amended at 59 FR 62470, Dec. 5, 1994]

§141.42 Special monitoring for corrosivity characteristics.

(a)–(c) [Reserved]

(d) Community water supply systems shall identify whether the following construction materials are present in their distribution system and report to the State:

Lead from piping, solder, caulking, interior lining of distribution mains, alloys and home plumbing.
Copper from piping and alloys, service lines, and home plumbing.
Galvanized piping, service lines, and home plumbing.
Ferrous piping materials such as cast iron and steel.
Asbestos cement pipe.

In addition, States may require identification and reporting of other materials of construction present in distribution systems that may contribute contaminants to the drinking water, such as:

Vinyl lined asbestos cement pipe.
Coal tar lined pipes and tanks.

[45 FR 57346, Aug. 27, 1980; 47 FR 10999, Mar. 12, 1982, as amended at 59 FR 62470, Dec. 5, 1994]

§ 141.43 Prohibition on use of lead pipes, solder, and flux.

(a) *In general*—(1) *Prohibition*. Any pipe, solder, or flux, which is used after June 19, 1986, in the installation or repair of—

(i) Any public water system, or

(ii) Any plumbing in a residential or nonresidential facility providing water for human consumption which is connected to a public water system shall be lead free as defined by paragraph (d) of this section. This paragraph (a)(1) shall not apply to leaded joints necessary for the repair of cast iron pipes.

(2) Each public water system shall identify and provide notice to persons that may be affected by lead contamination of their drinking water where such contamination results from either or both of the following:

(i) The lead content in the construction materials of the public water distribution system,

(ii) Corrosivity of the water supply sufficient to cause leaching of lead.

Notice shall be provided notwithstanding the absence of a violation of any national drinking water standard. The manner and form of notice are specified in § 141.34 of this part.

(b) *State enforcement*—(1) *Enforcement of prohibition*. The requirements of paragraph (a)(1) of this section shall be enforced in all States effective June 19, 1988. States shall enforce such requirements through State or local plumbing codes, or such other means of enforcement as the State may determine to be appropriate.

(2) *Enforcement of public notice requirements.* The requirements of paragraph (a)(2) of this section, shall apply in all States effective June 19, 1988.

(c) *Penalties.* If the Administrator determines that a State is not enforcing the requirements of paragraph (a) of this section, as required pursuant to paragraph (b) of this section, the Administrator may withhold up to 5 percent of Federal funds available to that State for State program grants under section 1443(a) of the Act.

(d) *Definition of lead free.* For purposes of this section, the term *lead free*

(1) When used with respect to solders and flux refers to solders and flux containing not more than 0.2 percent lead, and

(2) When used with respect to pipes and pipe fittings refers to pipes and pipe fittings containing not more than 8.0 percent lead.

[52 FR 20674, June 2, 1987]

Subpart F—Maximum Contaminant Level Goals

§ 141.50 Maximum contaminant level goals for organic contaminants.

(a) MCLGs are zero for the following contaminants:

(1) Benzene
(2) Vinyl chloride
(3) Carbon tetrachloride
(4) 1,2-dichloroethane
(5) Trichloroethylene
(6) Acrylamide
(7) Alachlor
(8) Chlordane
(9) Dibromochloropropane
(10) 1,2-Dichloropropane
(11) Epichlorohydrin
(12) Ethylene dibromide
(13) Heptachlor
(14) Heptachlor epoxide
(15) Pentachlorophenol
(16) Polychlorinated biphenyls (PCBs)
(17) Tetrachloroethylene

(18) Toxaphene
(19) Benzo[a]pyrene
(20) Dichloromethane (methylene chloride)
(21) Di(2-ethylhexyl)phthalate
(22) Hexachlorobenzene
(23) 2,3,7,8-TCDD (Dioxin)

(b) MCLGs for the following contaminants are as indicated:

Contaminant	MCLG in mg/l
(1) 1,1-Dichloroethylene	0.007
(2) 1,1,1-Trichloroethane	0.20
(3) para-Dichlorobenzene	0.075
(4) Aldicarb	0.001
(5) Aldicarb sulfoxide	0.001
(6) Aldicarb sulfone	0.001
(7) Atrazine	0.003
(8) Carbofuran	0.04
(9) o-Dichlorobenzene	0.6
(10) cis-1,2-Dichloroethylene	0.07
(11) trans-1,2-Dichloroethylene	0.1
(12) 2,4-D	0.07
(13) Ethylbenzene	0.7
(14) Lindane	0.0002
(15) Methoxychlor	0.04
(16) Monochlorobenzene	0.1
(17) Styrene	0.1
(18) Toluene	1
(19) 2,4,5-TP	0.05
(20) Xylenes (total)	10
(21) Dalapon	0.2
(22) Di(2-ethylhexyl)adipate	.4
(23) Dinoseb	.007
(24) Diquat	.02
(25) Endothall	.1
(26) Endrin	.002
(27) Glyphosate	.7
(28) Hexachlorocyclopentadiene	.05
(29) Oxamyl (Vydate)	.2
(30) Picloram	.5
(31) Simazine	.004
(32) 1,2,4-Trichlorobenzene	.07
(33) 1,1,2-Trichloroethane	.003

[50 FR 46901, Nov. 13, 1985, as amended at 52 FR 20674, June 2, 1987; 52 FR 25716, July 8, 1987; 56 FR 3592, Jan. 30, 1991; 56 FR 30280, July 1, 1991; 57 FR 31846, July 17, 1992]

§141.51 Maximum contaminant level goals for inorganic contaminants.

(a) [Reserved]

(b) MCLGs for the following contaminants are as indicated:

Contaminant	MCLG (mg/l)
Antimony	0.006
Asbestos	7 Million fibers/liter (longer than 10 μm).
Barium	2
Beryllium	.004
Cadmium	0.005
Chromium	0.1
Copper	1.3
Cyanide (as free Cyanide)	.2
Fluoride	4.0
Lead	zero
Mercury	0.002
Nitrate	10 (as Nitrogen).
Nitrite	1 (as Nitrogen).
Total Nitrate+Nitrite	10 (as Nitrogen).
Selenium	0.05
Thallium	.0005

[50 FR 47155, Nov. 14, 1985, as amended at 52 FR 20674, June 2, 1987; 56 FR 3593, Jan. 30, 1991; 56 FR 26548, June 7, 1991; 56 FR 30280, July 1, 1991; 57 FR 31846, July 17, 1992; 60 FR 33932, June 29, 1995]

§141.52 Maximum contaminant level goals for microbiological contaminants.

MCLGs for the following contaminants are as indicated:

Contaminant	MCLG
(1) *Giardia lamblia*	zero
(2) Viruses	zero
(3) *Legionella*	zero
(4) Total coliforms (including fecal coliforms and *Escherichia coli*).	zero.

[54 FR 27527, 27566, June 29, 1989; 55 FR 25064, June 19, 1990]

Subpart G—National Revised Primary Drinking Water Regulations: Maximum Contaminant Levels

§141.60 Effective dates.

(a) The effective dates for §141.61 are as follows:

(1) The effective date for paragraphs (a)(1) through (a)(8) of §141.61 is January 9, 1989.

(2) The effective date for paragraphs (a)(9) through (a)(18) and (c)(1) through (c)(18) of §141.61 is July 30, 1992.

(3) The effective date for paragraphs (a)(19) through (a)(21), (c)(19) through (c)(25), and (c)(27) through (c)(33) of §141.61 is January 17, 1994. The effective date of §141.61(c)(26) is August 17, 1992.

(b) The effective dates for §141.62 are as follows:

(1) The effective date of paragraph (b)(1) of §141.62 is October 2, 1987.

(2) The effective date for paragraphs (b)(2) and (b)(4) through (b)(10) of §141.62 is July 30, 1992.

(3) The effective date for paragraphs (b)(11) through (b)(15) of § 141.62 is January 17, 1994.

[56 FR 3593, Jan. 30, 1991, as amended at 57 FR 31846, July 17, 1992; 59 FR 34324, July 1, 1994]

§ 141.61 Maximum contaminant levels for organic contaminants.

(a) The following maximum contaminant levels for organic contaminants apply to community and non-transient, non-community water systems.

CAS No.	Contaminant	MCL (mg/l)
(1) 75–01–4	Vinyl chloride	0.002
(2) 71–43–2	Benzene	0.005
(3) 56–23–5	Carbon tetrachloride	0.005
(4) 107–06–2	1,2-Dichloroethane	0.005
(5) 79–01–6	Trichloroethylene	0.005
(6) 106–46–7	para-Dichlorobenzene	0.075
(7) 75–35–4	1,1-Dichloroethylene	0.007
(8) 71–55–6	1,1,1-Trichloroethane	0.2
(9) 156–59–2	cis-1,2-Dichloroethylene	0.07
(10) 78–87–5	1,2-Dichloropropane	0.005
(11) 100–41–4	Ethylbenzene	0.7
(12) 108–90–7	Monochlorobenzene	0.1
(13) 95–50–1	o-Dichlorobenzene	0.6
(14) 100–42–5	Styrene	0.1
(15) 127–18–4	Tetrachloroethylene	0.005
(16) 108–88–3	Toluene	1
(17) 156–60–5	trans-1,2-Dichloroethylene	0.1
(18) 1330–20–7	Xylenes (total)	10
(19) 75–09–2	Dichloromethane	0.005
(20) 120–82–1	1,2,4-Trichloro- benzene	.07
(21) 79–00–5	1,1,2-Trichloro- ethane	.005

(b) The Administrator, pursuant to section 1412 of the Act, hereby identifies as indicated in the Table below granular activated carbon (GAC), packed tower aeration (PTA), or oxidation (OX) as the best technology treatment technique, or other means available for achieving compliance with the maximum contaminant level for organic contaminants identified in paragraphs (a) and (c) of this section:

BAT FOR ORGANIC CONTAMINANTS LISTED IN § 141.61 (a) AND (c)

CAS No.	Contaminant	GAC	PTA	OX
15972–60–8	Alachlor	X		
116–06–3	Aldicarb	X		
1646–88–4	Aldicarb sulfone	X		
1646–87–3	Aldicarb sulfoxide	X		
1912–24–9	Atrazine	X		
71–43–2	Benzene	X	X	
50–32–8	Benzo[a]pyrene	X		
1563–66–2	Carbofuran	X		
56–23–5	Carbon tetrachloride	X	X	
57–74–9	Chlordane	X		
75–99–0	Dalapon	X		
94–75–7	2,4-D	X		
103–23–1	Di (2-ethylhexyl) adipate	X	X	
117–81–7	Di (2-ethylhexyl) phthalate	X		
96–12–8	Dibromochloropropane (DBCP)	X	X	
95–50–1	o-Dichlorobenzene	X	X	
106–46–7	para-Dichlorobenzene	X	X	
107–06–2	1,2-Dichloroethane	X	X	
75–35–4	1,1-Dichloroethylene	X	X	
156–59–2	cis-1,2-Dichloroethylene	X	X	
156–60–5	trans-1,2-Dichloroethylene	X	X	
75–09–2	Dichloromethane		X	
78–87–5	1,2-Dichloropropane	X	X	
88–85–7	Dinoseb	X		
85–00–7	Diquat	X		
145–73–3	Endothall	X		
72–20–8	Endrin	X		
100–41–4	Ethylbenzene	X	X	

BAT FOR ORGANIC CONTAMINANTS LISTED IN § 141.61 (a) AND (c)—Continued

CAS No.	Contaminant	GAC	PTA	OX
106–93–4	Ethylene Dibromide (EDB)	X	X	
1071–83–6	Gylphosate			X
76–44–8	Heptachlor	X		
1024–57–3	Heptachlor epoxide	X		
118–74–1	Hexachlorobenzene	X		
77–47–3	Hexachlorocyclopentadiene	X	X	
58–89–9	Lindane	X		
72–43–5	Methoxychlor	X		
108–90–7	Monochlorobenzene	X	X	
23135–22–0	Oxamyl (Vydate)	X		
87–86–5	Pentachlorophenol	X		
1918–02–1	Picloram	X		
1336–36–3	Polychlorinated biphenyls (PCB)	X		
122–34–9	Simazine	X		
100–42–5	Styrene	X	X	
1746–01–6	2,3,7,8-TCDD (Dioxin)	X		
127–18–4	Tetrachloroethylene	X	X	
108–88–3	Toluene	X	X	
8001–35–2	Toxaphene	X		
93–72–1	2,4,5-TP (Silvex)	X		
120–82–1	1,2,4-Trichlorobenzene	X	X	
71–55–6	1,1,1-Trichloroethane	X	X	
79–00–5	1,1,2-Trichloroethane	X	X	
79–01–6	Trichloroethylene	X	X	
75–01–4	Vinyl chloride		X	
1330–20–7	Xylene	X	X	

(c) The following maximum contaminant levels for synthetic organic contaminants apply to community water systems and non-transient, non-community water systems:

CAS No.	Contaminant	MCL (mg/l)
(1) 15972–60–8	Alachlor	0.002
(2) 116–06–3	Aldicarb	0.003
(3) 1646–87–3	Aldicarb sulfoxide	0.004
(4) 1646–87–4	Aldicarb sulfone	0.002
(5) 1912–24–9	Atrazine	0.003
(6) 1563–66–2	Carbofuran	0.04
(7) 57–74–9	Chlordane	0.002
(8) 96–12–8	Dibromochloropropane	0.0002
(9) 94–75–7	2,4-D	0.07
(10) 106–93–4	Ethylene dibromide	0.00005
(11) 76–44–8	Heptachlor	0.0004
(12) 1024–57–3	Heptachlor epoxide	0.0002
(13) 58–89–9	Lindane	0.0002
(14) 72–43–5	Methoxychlor	0.04
(15) 1336–36–3	Polychlorinated biphenyls	0.0005
(16) 87–86–5	Pentachlorophenol	0.001
(17) 8001–35–2	Toxaphene	0.003
(18) 93–72–1	2,4,5-TP	0.05
(19) 50–32–8	Benzo[a]pyrene	0.0002
(20) 75–99–0	Dalapon	0.2
(21) 103–23–1	Di(2-ethylhexyl) adipate	0.4
(22) 117–81–7	Di(2-ethylhexyl) phthalate	0.006
(23) 88–85–7	Dinoseb	0.007
(24) 85–00–7	Diquat	0.02
(25) 145–73–3	Endothall	0.1
(26) 72–20–8	Endrin	0.002
(27) 1071–53–6	Glyphosate	0.7
(28) 118–74–1	Hexacholorbenzene	0.001
(29) 77–47–4	Hexachlorocyclopentadiene	0.05
(30) 23135–22–0	Oxamyl (Vydate)	0.2
(31) 1918–02–1	Picloram	0.5
(32) 122–34–9	Simazine	0.004
(33) 1746–01–6	2,3,7,8-TCDD (Dioxin)	3×10^{-8}

[56 FR 3593, Jan. 30, 1991, as amended at 56 FR 30280, July 1, 1991; 57 FR 31846, July 17, 1992; 59 FR 34324, July 1, 1994]

§ 141.62 Maximum contaminant levels for inorganic contaminants.

(a) [Reserved]

(b) The maximum contaminant levels for inorganic contaminants specified in paragraphs (b) (2)—(6), (b)(10), and (b)(11)—(15) of this section apply to community water systems and non-transient, non-community water systems. The maximum contaminant level specified in paragraph (b)(1) of this section only applies to community water systems. The maximum contaminant levels specified in (b)(7), (b)(8), and (b)(9) of this section apply to community water systems; non-transient, non-community water systems; and transient non-community water systems.

Contaminant	MCL (mg/l)
(1) Fluoride	4.0
(2) Asbestos	7 Million Fibers/liter (longer than 10 μm).
(3) Barium	2
(4) Cadmium	0.005
(5) Chromium	0.1
(6) Mercury	0.002
(7) Nitrate	10 (as Nitrogen)
(8) Nitrite	1 (as Nitrogen)
(9) Total Nitrate and Nitrite	10 (as Nitrogen)
(10) Selenium	0.05
(11) Antimony	0.006
(12) Beryllium	0.004
(13) Cyanide (as free Cyanide).	0.2
(14) [Reserved]	
(15) Thallium	0.002

(c) The Administrator, pursuant to section 1412 of the Act, hereby identifies the following as the best technology, treatment technique, or other means available for achieving compliance with the maximum contaminant levels for inorganic contaminants identified in paragraph (b) of this section, except fluoride:

BAT FOR INORGANIC COMPOUNDS LISTED IN SECTION 141.62(B)

Chemical Name	BAT(s)
Antimony	2,7
Asbestos	2,3,8
Barium	5,6,7,9
Beryllium	1,2,5,6,7
Cadmium	2,5,6,7
Chromium	2,5,6[2],7
Cyanide	5,7,10
Mercury	2[1],4,6[1],7[1]

BAT FOR INORGANIC COMPOUNDS LISTED IN SECTION 141.62(B)—Continued

Chemical Name	BAT(s)
Nickel	5,6,7
Nitrate	5,7,9
Nitrite	5,7
Selenium	1,2[3],6,7,9
Thallium	1,5

[1] BAT only if influent Hg concentrations ≤10μg/1.
[2] BAT for Chromium III only.
[3] BAT for Selenium IV only.

Key to BATS in Table

1=Activated Alumina
2=Coagulation/Filtration
3=Direct and Diatomite Filtration
4=Granular Activated Carbon
5=Ion Exchange
6=Lime Softening
7=Reverse Osmosis
8=Corrosion Control
9=Electrodialysis
10=Chlorine
11=Ultraviolet

[56 FR 3594, Jan. 30, 1991, as amended at 56 FR 30280, July 1, 1991; 57 FR 31847, July 17, 1992; 59 FR 34325, July 1, 1994; 60 FR 33932, June 29, 1995]

§ 141.63 Maximum contaminant levels (MCLs) for microbiological contaminants.

(a) The MCL is based on the presence or absence of total coliforms in a sample, rather than coliform density.

(1) For a system which collects at least 40 samples per month, if no more than 5.0 percent of the samples collected during a month are total coliform-positive, the system is in compliance with the MCL for total coliforms.

(2) For a system which collects fewer than 40 samples/month, if no more than one sample collected during a month is total coliform-positive, the system is in compliance with the MCL for total coliforms.

(b) Any fecal coliform-positive repeat sample or *E. coli*-positive repeat sample, or any total coliform-positive repeat sample following a fecal coliform-positive or *E. coli*-positive routine sample constitutes a violation of the MCL for total coliforms. For purposes of the public notification requirements in § 141.32, this is a violation that may pose an acute risk to health.

(c) A public water system must determine compliance with the MCL for total coliforms in paragraphs (a) and (b) of this section for each month in

which it is required to monitor for total coliforms.

(d) The Administrator, pursuant to section 1412 of the Act, hereby identifies the following as the best technology, treatment techniques, or other means available for achieving compliance with the maximum contaminant level for total coliforms in paragraphs (a) and (b) of this section:

(1) Protection of wells from contamination by coliforms by appropriate placement and construction;

(2) Maintenance of a disinfectant residual throughout the distribution system;

(3) Proper maintenance of the distribution system including appropriate pipe replacement and repair procedures, main flushing programs, proper operation and maintenance of storage tanks and reservoirs, and continual maintenance of positive water pressure in all parts of the distribution system;

(4) Filtration and/or disinfection of surface water, as described in subpart H, or disinfection of ground water using strong oxidants such as chlorine, chlorine dioxide, or ozone; and

(5) For systems using ground water, compliance with the requirements of an EPA-approved State Wellhead Protection Program developed and implemented under section 1428 of the SDWA.

[54 FR 27566, June 29, 1989; 55 FR 25064, June 19, 1990]

Subpart H—Filtration and Disinfection

SOURCE: 54 FR 27527, June 29, 1989, unless otherwise noted.

§141.70 General requirements.

(a) The requirements of this subpart H constitute national primary drinking water regulations. These regulations establish criteria under which filtration is required as a treatment technique for public water systems supplied by a surface water source and public water systems supplied by a ground water source under the direct influence of surface water. In addition, these regulations establish treatment technique requirements in lieu of maximum contaminant levels for the following contaminants: *Giardia lamblia*, viruses, heterotrophic plate count bacteria, *Legionella*, and turbidity. Each public water system with a surface water source or a ground water source under the direct influence of surface water must provide treatment of that source water that complies with these treatment technique requirements. The treatment technique requirements consist of installing and properly operating water treatment processes which reliably achieve:

(1) At least 99.9 percent (3-log) removal and/or inactivation of *Giardia lamblia* cysts between a point where the raw water is not subject to recontamination by surface water runoff and a point downstream before or at the first customer; and

(2) At least 99.99 percent (4-log) removal and/or inactivation of viruses between a point where the raw water is not subject to recontamination by surface water runoff and a point downstream before or at the first customer.

(b) A public water system using a surface water source or a ground water source under the direct influence of surface water is considered to be in compliance with the requirements of paragraph (a) of this section if:

(1) It meets the requirements for avoiding filtration in §141.71 and the disinfection requirements in §141.72(a); or

(2) It meets the filtration requirements in §141.73 and the disinfection requirements in §141.72(b).

(c) Each public water system using a surface water source or a ground water source under the direct influence of surface water must be operated by qualified personnel who meet the requirements specified by the State.

§141.71 Criteria for avoiding filtration.

A public water system that uses a surface water source must meet all of the conditions of paragraphs (a) and (b) of this section, and is subject to paragraph (c) of this section, beginning December 30, 1991, unless the State has determined, in writing pursuant to §1412(b)(7)(C)(iii), that filtration is required. A public water system that uses a ground water source under the direct influence of surface water must

meet all of the conditions of paragraphs (a) and (b) of this section and is subject to paragraph (c) of this section, beginning 18 months after the State determines that it is under the direct influence of surface water, or December 30, 1991, whichever is later, unless the State has determined, in writing pursuant to § 1412(b)(7)(C)(iii), that filtration is required. If the State determines in writing pursuant to § 1412(b)(7)(C)(iii) before December 30, 1991, that filtration is required, the system must have installed filtration and meet the criteria for filtered systems specified in §§ 141.72(b) and 141.73 by June 29, 1993. Within 18 months of the failure of a system using surface water or a ground water source under the direct influence of surface water to meet any one of the requirements of paragraphs (a) and (b) of this section or after June 29, 1993, whichever is later, the system must have installed filtration and meet the criteria for filtered systems specified in §§ 141.72(b) and 141.73.

(a) *Source water quality conditions.* (1) The fecal coliform concentration must be equal to or less than 20/100 ml, or the total coliform concentration must be equal to or less than 100/100 ml (measured as specified in § 141.74 (a) (1) and (2) and (b)(1)), in representative samples of the source water immediately prior to the first or only point of disinfectant application in at least 90 percent of the measurements made for the 6 previous months that the system served water to the public on an ongoing basis. If a system measures both fecal and total coliforms, the fecal coliform criterion, but not the total coliform criterion, in this paragraph must be met.

(2) The turbidity level cannot exceed 5 NTU (measured as specified in § 141.74 (a)(4) and (b)(2)) in representative samples of the source water immediately prior to the first or only point of disinfectant application unless: (i) the State determines that any such event was caused by circumstances that were unusual and unpredictable; and (ii) as a result of any such event, there have not been more than two events in the past 12 months the system served water to the public, or more than five events in the past 120 months the system served water to the public, in which the turbidity level exceeded 5 NTU. An ''event'' is a series of consecutive days during which at least one turbidity measurement each day exceeds 5 NTU.

(b) *Site-specific conditions.* (1)(i) The public water system must meet the requirements of § 141.72(a)(1) at least 11 of the 12 previous months that the system served water to the public, on an ongoing basis, unless the system fails to meet the requirements during 2 of the 12 previous months that the system served water to the public, and the State determines that at least one of these failures was caused by circumstances that were unusual and unpredictable.

(ii) The public water system must meet the requirements of § 141.72(a)(2) at all times the system serves water to the public.

(iii) The public water system must meet the requirements of § 141.72(a)(3) at all times the system serves water to the public unless the State determines that any such failure was caused by circumstances that were unusual and unpredictable.

(iv) The public water system must meet the requirements of § 141.72(a)(4) on an ongoing basis unless the State determines that failure to meet these requirements was not caused by a deficiency in treatment of the source water.

(2) The public water system must maintain a watershed control program which minimizes the potential for contamination by *Giardia lamblia* cysts and viruses in the source water. The State must determine whether the watershed control program is adequate to meet this goal. The adequacy of a program to limit potential contamination by *Giardia lamblia* cysts and viruses must be based on: the comprehensiveness of the watershed review; the effectiveness of the system's program to monitor and control detrimental activities occurring in the watershed; and the extent to which the water system has maximized land ownership and/or controlled land use within the watershed. At a minimum, the watershed control program must:

(i) Characterize the watershed hydrology and land ownership;

(ii) Identify watershed characteristics and activities which may have an adverse effect on source water quality; and

(iii) Monitor the occurrence of activities which may have an adverse effect on source water quality.

The public water system must demonstrate through ownership and/or written agreements with landowners within the watershed that it can control all human activities which may have an adverse impact on the microbiological quality of the source water. The public water system must submit an annual report to the State that identifies any special concerns about the watershed and how they are being handled; describes activities in the watershed that affect water quality; and projects what adverse activities are expected to occur in the future and describes how the public water system expects to address them. For systems using a ground water source under the direct influence of surface water, an approved wellhead protection program developed under section 1428 of the Safe Drinking Water Act may be used, if the State deems it appropriate, to meet these requirements.

(3) The public water system must be subject to an annual on-site inspection to assess the watershed control program and disinfection treatment process. Either the State or a party approved by the State must conduct the on-site inspection. The inspection must be conducted by competent individuals such as sanitary and civil engineers, sanitarians, or technicians who have experience and knowledge about the operation and maintenance of a public water system, and who have a sound understanding of public health principles and waterborne diseases. A report of the on-site inspection summarizing all findings must be prepared every year. The on-site inspection must indicate to the State's satisfaction that the watershed control program and disinfection treatment process are adequately designed and maintained. The on-site inspection must include:

(i) A review of the effectiveness of the watershed control program;

(ii) A review of the physical condition of the source intake and how well it is protected;

(iii) A review of the system's equipment maintenance program to ensure there is low probability for failure of the disinfection process;

(iv) An inspection of the disinfection equipment for physical deterioration;

(v) A review of operating procedures;

(vi) A review of data records to ensure that all required tests are being conducted and recorded and disinfection is effectively practiced; and

(vii) Identification of any improvements which are needed in the equipment, system maintenance and operation, or data collection.

(4) The public water system must not have been identified as a source of a waterborne disease outbreak, or if it has been so identified, the system must have been modified sufficiently to prevent another such occurrence, as determined by the State.

(5) The public water system must comply with the maximum contaminant level (MCL) for total coliforms in § 141.63 at least 11 months of the 12 previous months that the system served water to the public, on an ongoing basis, unless the State determines that failure to meet this requirement was not caused by a deficiency in treatment of the source water.

(6) The public water system must comply with the requirements for trihalomethanes in §§ 141.12 and 141.30.

(c) *Treatment technique violations.* (1) A system that (i) fails to meet any one of the criteria in paragraphs (a) and (b) of this section and/or which the State has determined that filtration is required, in writing pursuant to § 1412(b)(7)(C)(iii), and (ii) fails to install filtration by the date specified in the introductory paragraph of this section is in violation of a treatment technique requirement.

(2) A system that has not installed filtration is in violation of a treatment technique requirement if:

(i) The turbidity level (measured as specified in § 141.74(a)(4) and (b)(2)) in a representative sample of the source water immediately prior to the first or only point of disinfection application exceeds 5 NTU; or

(ii) The system is identified as a source of a waterborne disease outbreak.

§ 141.72 Disinfection.

A public water system that uses a surface water source and does not provide filtration treatment must provide the disinfection treatment specified in paragraph (a) of this section beginning December 30, 1991, unless the State determines that filtration is required in writing pursuant to § 1412 (b)(7)(C)(iii). A public water system that uses a ground water source under the direct influence of surface water and does not provide filtration treatment must provide disinfection treatment specified in paragraph (a) of this section beginning December 30, 1991, or 18 months after the State determines that the ground water source is under the influence of surface water, whichever is later, unless the State has determined that filtration is required in writing pursuant to § 1412(b)(7)(C)(iii). If the State has determined that filtration is required, the system must comply with any interim disinfection requirements the State deems necessary before filtration is installed. A system that uses a surface water source that provides filtration treatment must provide the disinfection treatment specified in paragraph (b) of this section beginnng June 29, 1993, or beginning when filtration is installed, whichever is later. A system that uses a ground water source under the direct influence of surface water and provides filtration treatment must provide disinfection treatment as specified in paragraph (b) of this section by June 29, 1993, or beginning when filtration is installed, whichever is later. Failure to meet any requirement of this section after the applicable date specified in this introductory paragraph is a treatment technique violation.

(a) *Disinfection requirements for public water systems that do not provide filtration.* Each public water system that does not provide filtration treatment must provide disinfection treatment as follows:

(1) The disinfection treatment must be sufficient to ensure at least 99.9 percent (3-log) inactivation of *Giardia lamblia* cysts and 99.99 percent (4-log) inactivation of viruses, every day the system serves water to the public, except any one day each month. Each day a system serves water to the public, the public water system must calculate the CT value(s) from the system's treatment parameters, using the procedure specified in § 141.74(b)(3), and determine whether this value(s) is sufficient to achieve the specified inactivation rates for *Giardia lamblia* cysts and viruses. If a system uses a disinfectant other than chlorine, the system may demonstrate to the State, through the use of a State-approved protocol for on-site disinfection challenge studies or other information satisfactory to the State, that $CT_{99.9}$ values other than those specified in tables 2.1 and 3.1 in § 141.74(b)(3) or other operational parameters are adequate to demonstrate that the system is achieving minimum inactivation rates required by paragraph (a)(1) of this section.

(2) The disinfection system must have either (i) redundant components, including an auxiliary power supply with automatic start-up and alarm to ensure that disinfectant application is maintained continuously while water is being delivered to the distribution system, or (ii) automatic shut-off of delivery of water to the distribution system whenever there is less than 0.2 mg/l of residual disinfectant concentration in the water. If the State determines that automatic shut-off would cause unreasonable risk to health or interfere with fire protection, the system must comply with paragraph (a)(2)(i) of this section.

(3) The residual disinfectant concentration in the water entering the distribution system, measured as specified in § 141.74 (a)(5) and (b)(5), cannot be less than 0.2 mg/l for more than 4 hours.

(4)(i) The residual disinfectant concentration in the distribution system, measured as total chlorine, combined chlorine, or chlorine dioxide, as specified in § 141.74 (a)(5) and (b)(6), cannot be undetectable in more than 5 percent of the samples each month, for any two consecutive months that the system serves water to the public. Water in the distribution system with a heterotrophic bacteria concentration less than or equal to 500/ml, measured

as heterotrophic plate count (HPC) as specified in § 141.74(a)(3), is deemed to have a detectable disinfectant residual for purposes of determining compliance with this requirement. Thus, the value ''V'' in the following formula cannot exceed 5 percent in one month, for any two consecutive months.

$$V = \frac{c+d+e}{a+b} \times 100$$

where:

a=number of instances where the residual disinfectant concentration is measured;

b=number of instances where the residual disinfectant concentration is not measured but heterotrophic bacteria plate count (HPC) is measured;

c=number of instances where the residual disinfectant concentration is measured but not detected and no HPC is measured;

d=number of instances where the residual disinfectant concentration is measured but not detected and where the HPC is >500/ml; and

e=number of instances where the residual disinfectant concentration is not measured and HPC is >500/ml.

(ii) If the State determines, based on site-specific considerations, that a system has no means for having a sample transported and analyzed for HPC by a certified laboratory under the requisite time and temperature conditions specified by § 141.74(a)(3) and that the system is providing adequate disinfection in the distribution system, the requirements of paragraph (a)(4)(i) of this section do not apply to that system.

(b) *Disinfection requirements for public water systems which provide filtration.* Each public water system that provides filtration treatment must provide disinfection treatment as follows.

(1) The disinfection treatment must be sufficient to ensure that the total treatment processes of that system achieve at least 99.9 percent (3-log) inactivation and/or removal of *Giardia lamblia* cysts and at least 99.99 percent (4-log) inactivation and/or removal of viruses, as determined by the State.

(2) The residual disinfectant concentration in the water entering the distribution system, measured as specified in § 141.74 (a)(5) and (c)(2), cannot be less than 0.2 mg/l for more than 4 hours.

(3)(i) The residual disinfectant concentration in the distribution system, measured as total chlorine, combined chlorine, or chlorine dioxide, as specified in § 141.74 (a)(5) and (c)(3), cannot be undetectable in more than 5 percent of the samples each month, for any two consecutive months that the system serves water to the public. Water in the distribution system with a heterotrophic bacteria concentration less than or equal to 500/ml, measured as heterotrophic plate count (HPC) as specified in § 141.74(a)(3), is deemed to have a detectable disinfectant residual for purposes of determining compliance with this requirement. Thus, the value ''V'' in the following formula cannot exceed 5 percent in one month, for any two consecutive months.

$$V = \frac{c+d+e}{a+b} \times 100$$

where:

a=number of instances where the residual disinfectant concentration is measured;

b=number of instances where the residual disinfectant concentration is not measured but heterotrophic bacteria plate count (HPC) is measured;

c=number of instances where the residual disinfectant concentration is measured but not detected and no HPC is measured;

d=number of instances where no residual disinfectant concentration is detected and where the HPC is >500/ml; and

e=number of instances where the residual disinfectant concentration is not measured and HPC is >500/ml.

(ii) If the State determines, based on site-specific considerations, that a system has no means for having a sample transported and analyzed for HPC by a certified laboratory under the requisite time and temperature conditions specified in § 141.74(a)(3) and that the system is providing adequate disinfection in the distribution system, the requirements of paragraph (b)(3)(i) of this section do not apply.

§ 141.73 Filtration.

A public water system that uses a surface water source or a ground water source under the direct influence of surface water, and does not meet all of the criteria in § 141.71 (a) and (b) for

avoiding filtration, must provide treatment consisting of both disinfection, as specified in § 141.72(b), and filtration treatment which complies with the requirements of paragraph (a), (b), (c), (d), or (e) of this section by June 29, 1993, or within 18 months of the failure to meet any one of the criteria for avoiding filtration in § 141.71 (a) and (b), whichever is later. Failure to meet any requirement of this section after the date specified in this introductory paragraph is a treatment technique violation.

(a) *Conventional filtration treatment or direct filtration.* (1) For systems using conventional filtration or direct filtration, the turbidity level of representative samples of a system's filtered water must be less than or equal to 0.5 NTU in at least 95 percent of the measurements taken each month, measured as specified in § 141.74 (a)(4) and (c)(1), except that if the State determines that the system is capable of achieving at least 99.9 percent removal and/or inactivation of *Giardia lamblia* cysts at some turbidity level higher than 0.5 NTU in at least 95 percent of the measurements taken each month, the State may substitute this higher turbidity limit for that system. However, in no case may the State approve a turbidity limit that allows more than 1 NTU in more than 5 percent of the samples taken each month, measured as specified in § 141.74 (a)(4) and (c)(1).

(2) The turbidity level of representative samples of a system's filtered water must at no time exceed 5 NTU, measured as specified in § 141.74 (a)(4) and (c)(1).

(b) *Slow sand filtration.* (1) For systems using slow sand filtration, the turbidity level of representative samples of a system's filtered water must be less than or equal to 1 NTU in at least 95 percent of the measurements taken each month, measured as specified in § 141.74 (a)(4) and (c)(1), except that if the State determines there is no significant interference with disinfection at a higher turbidity level, the State may substitute this higher turbidity limit for that system.

(2) The turbidity level of representative samples of a system's filtered water must at no time exceed 5 NTU, measured as specified in § 141.74 (a)(4) and (c)(1).

(c) *Diatomaceous earth filtration.* (1) For systems using diatomaceous earth filtration, the turbidity level of representative samples of a system's filtered water must be less than or equal to 1 NTU in at least 95 percent of the measurements taken each month, measured as specified in § 141.74 (a)(4) and (c)(1).

(2) The turbidity level of representative samples of a system's filtered water must at no time exceed 5 NTU, measured as specified in § 141.74 (a)(4) and (c)(1).

(d) *Other filtration technologies.* A public water system may use a filtration technology not listed in paragraphs (a)–(c) of this section if it demonstrates to the State, using pilot plant studies or other means, that the alternative filtration technology, in combination with disinfection treatment that meets the requirements of § 141.72(b), consistently achieves 99.9 percent removal and/or inactivation of *Giardia lamblia* cysts and 99.99 percent removal and/or inactivation of viruses. For a system that makes this demonstration, the requirements of paragraph (b) of this section apply.

§ 141.74 Analytical and monitoring requirements.

(a) *Analytical requirements.* Only the analytical method(s) specified in this paragraph, or otherwise approved by EPA, may be used to demonstrate compliance with the requirements of §§ 141.71, 141.72, and 141.73. Measurements for pH, temperature, turbidity, and residual disinfectant concentrations must be conducted by a party approved by the State. Measurements for total coliforms, fecal coliforms, and HPC must be conducted by a laboratory certified by the State or EPA to do such analysis. Until laboratory certification criteria are developed for the analysis of HPC and fecal coliforms, any laboratory certified for total coliform analysis by EPA is deemed certified for HPC and fecal coliform analysis. The following procedures shall be performed in accordance with the publications listed in the following section. This incorporation by reference was approved by the Director of the

Federal Register in accordance with 5 U.S.C. 552(a) and 1 CFR part 51. Copies of the methods published in *Standard Methods for the Examination of Water and Wastewater* may be obtained from the American Public Health Association et al., 1015 Fifteenth Street, NW., Washington, DC 20005; copies of the Minimal Medium ONPG–MUG Method as set forth in the article ''National Field Evaluation of a Defined Substrate Method for the Simultaneous Enumeration of Total Coliforms and *Esherichia coli* from Drinking Water: Comparison with the Standard Multiple Tube Fermentation Method'' (Edberg et al.), Applied and Environmental Microbiology, Volume 54, pp. 1595–1601, June 1988 (as amended under Erratum, Applied and Environmental Microbiology, Volume 54, p. 3197, December, 1988), may be obtained from the American Water Works Association Research Foundation, 6666 West Quincy Avenue, Denver, Colorado, 80235; and copies of the Indigo Method as set forth in the article ''Determination of Ozone in Water by the Indigo Method'' (Bader and Hoigne), may be obtained from Ozone Science & Engineering, Pergamon Press Ltd., Fairview Park, Elmsford, New York 10523. Copies may be inspected at the U.S. Environmental Protection Agency, Room EB15, 401 M Street, SW., Washington, DC 20460 or at the Office of the Federal Register, 800 North Capitol Street, NW., suite 700, Washington, DC.

(1) Public water systems must conduct analysis of pH and temperature in accordance with one of the methods listed at § 141.23(k)(1). Public water systems must conduct analysis of total coliforms, fecal coliforms, heterotrophic bacteria, and turbidity in accordance with one of the following analytical methods and by using analytical test procedures contained in *Technical Notes on Drinking Water Methods,* EPA–600/R–94–173, October 1994, which is available at NTIS PB95–104766.

Organism	Methodology	Citation [1]
Total Coliforms [2]. ..	Total Coliform Fermentation Technique [3,4,5].	9221A, B, C
	Total Coliform Membrane Filter Technique.	9222A, B, C
	ONPG-Mug Test [6]	9223
Fecal Coliforms [2] ..	Fecal Coliform Procedure [7].	9221E
	Fecal Coliform Membrane Filter Procedure.	9222D
Heterotrophic bacteria [2].	Pour Plate method	9215B
Turbidity	Nephelometric Method.	2130B
	Nephelometric Method.	180.1 [8]
	Great Lakes Instruments.	Method 2 [9]

[1] Except where noted, all methods refer to the 18th edition of *Standard Methods for the Examination of Water and Wastewater,* 1992, American Public Health Association, 1015 Fifteenth Street NW, Washington, D.C. 20005.

[2] The time from sample collection to initiation of analysis may not exceed 8 hours. Systems are encouraged but not required to hold samples below 10°C during transit.

[3] Lactose broth, as commercially available, may be used in lieu of lauryl tryptose broth, if the system conducts at least 25 parallel tests between this medium and lauryl tryptose broth using the water normally tested, and this comparison demonstrates that the false positive rate and false negative rate for total coliforms, using lactose broth, is less than 10 percent.

[4] Media should cover inverted tubes at least one-half to two-thirds after the sample is added.

[5] No requirement exists to run the completed phase on 10 percent of all total coliform-positive confirmed tubes.

[6] The ONPG-MUG Test is also known as the Autoanalysis Colilert System.

[7] A–1 Broth may be held up to three months in a tightly closed screwcap tube at 4°C.

[8] ''Methods for the Determination of Inorganic Substances in Environmental Samples'', EPA–600/R–93–100, August 1993. Available at NTIS, PB94–121811.

[9] GLI Method 2, ''Turbidity'', November 2, 1992, Great Lakes Instruments, Inc., 8855 North 55th Street, Milwaukee, Wisconsin 53223.

(2) Public water systems must measure residual disinfectant concentrations with one of the analytical methods in the following table. The methods are contained in the 18th edition of *Standard Methods for the Examination of Water and Wastewater,* 1992. Other analytical test procedures are contained *in Technical Notes on Drinking Water Methods,* EPA–600/R–94–173, October 1994, which is available at NTIS PB95–104766. If approved by the State, residual disinfectant concentrations for free chlorine and combined chlorine also may be measured by using DPD colorimetric test kits. Free and total chlorine residuals may be measured continuously by adapting a specified chlorine residual method for use with a continuous monitoring instrument provided the chemistry, accuracy, and precision remain same. Instruments used for continuous monitoring must be calibrated with a grab sample measurement at least every five days, or with a protocol approved by the State.

Residual	Methodology	Methods
Free Chlorine.	Amperometric Titration	4500–Cl D
	DPD Ferrous Titrimetric.	4500–Cl F
	DPD Colorimetric	4500–Cl G
	Syringaldazine (FACTS).	4500–Cl H
Total Chlorine.	Amperometric Titration	4500–Cl D
	Amperometric Titration (low level measurement).	4500–Cl E
	DPD Ferrous Titrimetric.	4500–Cl F
	DPD Colorimetric	4500–Cl G
	Iodometric Electrode	4500–Cl I
Chlorine Dioxide.	Amperometric Titration	4500–ClO_2 C
	DPD Method	4500–ClO_2 D
	Amperometric Titration	4500–ClO_2 E
Ozone	Indigo Method	4500–O_3 B

(b) *Monitoring requirements for systems that do not provide filtration.* A public water system that uses a surface water source and does not provide filtration treatment must begin monitoring, as specified in this paragraph (b), beginning December 31, 1990, unless the State has determined that filtration is required in writing pursuant to §1412(b)(7)(C)(iii), in which case the State may specify alternative monitoring requirements, as appropriate, until filtration is in place. A public water system that uses a ground water source under the direct influence of surface water and does not provide filtration treatment must begin monitoring as specified in this paragraph (b) beginning December 31, 1990, or 6 months after the State determines that the ground water source is under the direct influence of surface water, whichever is later, unless the State has determined that filtration is required in writing pursuant to §1412(b)(7)(C)(iii), in which case the State may specify alternative monitoring requirements, as appropriate, until filtration is in place.

(1) Fecal coliform or total coliform density measurements as required by §141.71(a)(1) must be performed on representative source water samples immediately prior to the first or only point of disinfectant application. The system must sample for fecal or total coliforms at the following minimum frequency each week the system serves water to the public:

System size (persons served)	Samples/week[1]
≤500	1
501 to 3,300	2
3,301 to 10,000	3
10,001 to 25,000	4
>25,000	5

[1] Must be taken on separate days.

Also, one fecal or total coliform density measurement must be made every day the system serves water to the public and the turbidity of the source water exceeds 1 NTU (these samples count towards the weekly coliform sampling requirement) unless the State determines that the system, for logistical reasons outside the system's control, cannot have the sample analyzed within 30 hours of collection.

(2) Turbidity measurements as required by §141.71(a)(2) must be performed on representative grab samples of source water immediately prior to the first or only point of disinfectant application every four hours (or more frequently) that the system serves water to the public. A public water system may substitute continuous turbidity monitoring for grab sample monitoring if it validates the continuous measurement for accuracy on a regular basis using a protocol approved by the State.

(3) The total inactivation ratio for each day that the system is in operation must be determined based on the $CT_{99.9}$ values in tables 1.1–1.6, 2.1, and 3.1 of this section, as appropriate. The parameters necessary to determine the total inactivation ratio must be monitored as follows:

(i) The temperature of the disinfected water must be measured at least once per day at each residual disinfectant concentration sampling point.

(ii) If the system uses chlorine, the pH of the disinfected water must be measured at least once per day at each chlorine residual disinfectant concentration sampling point.

(iii) The disinfectant contact time(s) (''T'') must be determined for each day during peak hourly flow.

(iv) The residual disinfectant concentration(s) (''C'') of the water before or at the first customer must be measured each day during peak hourly flow.

(v) If a system uses a disinfectant other than chlorine, the system may

demonstrate to the State, through the use of a State-approved protocol for on-site disinfection challenge studies or other information satisfactory to the State, that $CT_{99.9}$ values other than those specified in tables 2.1 and 3.1 in this section other operational parameters are adequate to demonstrate that the system is achieving the minimum inactivation rates required by § 141.72(a)(1).

TABLE 1.1—CT VALUES ($CT_{99.9}$) FOR 99.9 PERCENT INACTIVATION OF GIARDIA LAMBLIA CYSTS BY FREE CHLORINE AT 0.5 °C OR LOWER [1]

Residual (mg/l)	pH						
	≤6.0	6.5	7.0	7.5	8.0	8.5	≤9.0
≤0.4	137	163	195	237	277	329	390
0.6	141	168	200	239	286	342	407
0.8	145	172	205	246	295	354	422
1.0	148	176	210	253	304	365	437
1.2	152	180	215	259	313	376	451
1.4	155	184	221	266	321	387	464
1.6	157	189	226	273	329	397	477
1.8	162	193	231	279	338	407	489
2.0	165	197	236	286	346	417	500
2.2	169	201	242	297	353	426	511
2.4	172	205	247	298	361	435	522
2.6	175	209	252	304	368	444	533
2.8	178	213	257	310	375	452	543
3.0	181	217	261	316	382	460	552

[1] These CT values achieve greater than a 99.99 percent inactivation of viruses. CT values between the indicated pH values may be determined by linear interpolation. CT values between the indicated temperatures of different tables may be determined by linear interpolation. If no interpolation is used, use the $CT_{99.9}$ value at the lower temperature and at the higher pH.

TABLE 1.2—CT VALUES ($CT_{99.9}$) for 99.9 PERCENT INACTIVATION OF GIARDIA LAMBLIA CYSTS BY FREE CHLORINE AT 5.0 °C[1]

Free residual (mg/l)	pH						
	≦6.0	6.5	7.0	7.5	8.0	8.5	≦9.0
≦0.4 ...	97	117	139	166	198	236	279
0.6 ..	100	120	143	171	204	244	291
0.8 ..	103	122	146	175	210	252	301
1.0 ..	105	125	149	179	216	260	312
1.2 ..	107	127	152	183	221	267	320
1.4 ..	109	130	155	187	227	274	329
1.6 ..	111	132	158	192	232	281	337
1.8 ..	114	135	162	196	238	287	345
2.0 ..	116	138	165	200	243	294	353
2.2 ..	118	140	169	204	248	300	361
2.4 ..	120	143	172	209	253	306	368
2.6 ..	122	146	175	213	258	312	375
2.8 ..	124	148	178	217	263	318	382
3.0 ..	126	151	182	221	268	324	389

[1] These CT values achieve greater than a 99.99 percent inactivation of viruses. CT values between the indicated pH values may be determined by linear interpolation. CT values between the indicated temperatures of different tables may be determined by linear interpolation. If no interpolation is used, use the $CT_{99.9}$ value at the lower temperature, and at the higher pH.

TABLE 1.3—CT VALUES ($CT_{99.9}$) for 99.9 PERCENT INACTIVATION OF GIARDIA LAMBLIA CYSTS BY FREE CHLORINE AT 10.0 °C[1]

Free residual (mg/l)	pH						
	≦6.0	6.5	7.0	7.5	8.0	8.5	≦9.0
≦0.4 ...	73	88	104	125	149	177	209
0.6 ..	75	90	107	128	153	183	218
0.8 ..	78	92	110	131	158	189	226
1.0 ..	79	94	112	134	162	195	234
1.2 ..	80	95	114	137	166	200	240
1.4 ..	82	98	116	140	170	206	247
1.6 ..	83	99	119	144	174	211	253
1.8 ..	86	101	122	147	179	215	259
2.0 ..	87	104	124	150	182	221	265
2.2 ..	89	105	127	153	186	225	271
2.4 ..	90	107	129	157	190	230	276
2.6 ..	92	110	131	160	194	234	281
2.8 ..	93	111	134	163	197	239	287
3.0 ..	95	113	137	166	201	243	292

[1] These CT values achieve greater than a 99.99 percent inactivation of viruses. CT values between the indicated pH values may be determined by linear interpolation. CT values between the indicated temperatures of different tables may be determined by linear interpolation. If no interpolation is used, use the $CT_{99.9}$ value at the lower temperature, and at the higher pH.

TABLE 1.4—CT VALUES ($CT_{99.9}$) for 99.9 PERCENT INACTIVATION OF GIARDIA LAMBLIA CYSTS BY FREE CHLORINE AT 15.0 °C[1]

Free residual (mg/l)	pH						
	≦6.0	6.5	7.0	7.5	8.0	8.5	≦9.0
≦0.4 ...	49	59	70	83	99	118	140
0.6 ..	50	60	72	86	102	122	146
0.8 ..	52	61	73	88	105	126	151
1.0 ..	53	63	75	90	108	130	156
1.2 ..	54	64	76	92	111	134	160
1.4 ..	55	65	78	94	114	137	165
1.6 ..	56	66	79	96	116	141	169
1.8 ..	57	68	81	98	119	144	173
2.0 ..	58	69	83	100	122	147	177
2.2 ..	59	70	85	102	124	150	181
2.4 ..	60	72	86	105	127	153	184
2.6 ..	61	73	88	107	129	156	188
2.8 ..	62	74	89	109	132	159	191
3.0 ..	63	76	91	111	134	162	195

[1] These CT values achieve greater than a 99.99 percent inactivation of viruses. CT values between the indicated pH values may be determined by linear interpolation. CT values between the indicated temperatures of different tables may be determined by linear interpolation. If no interpolation is used, use the $CT_{99.9}$ value at the lower temperature, and at the higher pH.

TABLE 1.5—CT Values ($CT_{99.9}$) FOR 99.9 PERCENT INACTIVATION OF GIARDIA LAMBLIA CYSTS BY FREE CHLORINE AT 20 °C[1]

Free residual (mg/l)	pH						
	≤ 6.0	6.5	7.0	7.5	8.0	8.5	≤ 9.0
≤ 0.4 ...	36	44	52	62	74	89	105
0.6	38	45	54	64	77	92	109
0.8	39	46	55	66	79	95	113
1.0	39	47	56	67	81	98	117
1.2	40	48	57	69	83	100	120
1.4	41	49	58	70	85	103	123
1.6	42	50	59	72	87	105	126

TABLE 1.5—CT Values ($CT_{99.9}$) FOR 99.9 PERCENT INACTIVATION OF GIARDIA LAMBLIA CYSTS BY FREE CHLORINE AT 20 °C[1]—Continued

Free residual (mg/l)	pH						
	≤ 6.0	6.5	7.0	7.5	8.0	8.5	≤ 9.0
1.8	43	51	61	74	89	108	129
2.0	44	52	62	75	91	110	132
2.2	44	53	63	77	93	113	135
2.4	45	54	65	78	95	115	138
2.6	46	55	66	80	97	117	141
2.8	47	56	67	81	99	119	143
3.0	47	57	68	83	101	122	146

[1] These CT values achieve greater than a 99.99 percent inactivation of viruses. CT values between the indicated pH values may be determined by linear interpolation. CT values between the indicated temperatures of different tables may be determined by linear interpolation. If no interpolation is used, use the $CT_{99.9}$ value at the lower temperature, and at the higher pH.

TABLE 1.6—CT Values ($CT_{99.9}$) FOR 99.9 PERCENT INACTIVATION OF GIARDIA LAMBLIA CYSTS BY FREE CHLORINE AT 25 °C[1] AND HIGHER

Free residual (mg/l)	pH						
	≤ 6.0	6.5	7.0	7.5	8.0	8.5	≤ 9.0
≤ 0.4 ...	24	29	35	42	50	59	70
0.6	25	30	36	43	51	61	73
0.8	26	31	37	44	53	63	75
1.0	26	31	37	45	54	65	78
1.2	27	32	38	46	55	67	80
1.4	27	33	39	47	57	69	82
1.6	28	33	40	48	58	70	84
1.8	29	34	41	49	60	72	86
2.0	29	35	41	50	61	74	88
2.2	30	35	42	51	62	75	90
2.4	30	36	43	52	63	77	92
2.6	31	37	44	53	65	78	94
2.8	31	37	45	54	66	80	96
3.0	32	38	46	55	67	81	97

[1] These CT values achieve greater than a 99.99 percent inactivation of viruses. CT values between the indicated pH values may be determined by linear interpolation. CT values between the indicated temperatures of different tables may be determined by linear interpolation. If no interpolation is used, use the $CT_{99.9}$ value at the lower temperature, and at the higher pH.

TABLE 2.1—CT VALUES ($CT_{99.9}$) FOR 99.9 PERCENT INACTIVATION OF GIARDIA LAMBLIA CYSTS BY CHLORINE DIOXIDE AND OZONE[1]

	Temperature					
	< 1 °C	5 °C	10 °C	15 °C	20 °C	≥ 25 °C
Chlorine dioxide	63	26	23	19	15	11
Ozone	2.9	1.9	1.4	0.95	0.72	0.48

[1] These CT values achieve greater than 99.99 percent inactivation of viruses. CT values between the indicated temperatures may be determined by linear interpolation. If no interpolation is used, use the $CT_{99.9}$ value at the lower temperature for determining $CT_{99.9}$ values between indicated temperatures.

TABLE 3.1—CT VALUES ($CT_{99.9}$) FOR 99.9 PERCENT INACTIVATION OF GIARDIA LAMBLIA CYSTS BY CHLORAMINES[1]

Temperature					
< 1 °C	5 °C	10 °C	15 °C	20 °C	25 °C
3,800	2,200	1,850	1,500	1,100	750

[1] These values are for pH values of 6 to 9. These CT values may be assumed to achieve greater than 99.99 percent inactivation of viruses only if chlorine is added and mixed in the water prior to the addition of ammonia. If this condition is not met, the system must demonstrate, based on on-site studies or other information, as approved by the State, that the system is achieving at least 99.99 percent inactivation of viruses. CT values between the indicated temperatures may be determined by linear interpolation. If no interpolation is used, use the $CT_{99.9}$ value at the lower temperature for determining $CT_{99.9}$ values between indicated temperatures.

(4) The total inactivation ratio must be calculated as follows:

(i) If the system uses only one point of disinfectant application, the system may determine the total inactivation ratio based on either of the following two methods:

(A) One inactivation ratio ($CTcalc/CT_{99.9}$) is determined before or at the first customer during peak hourly flow and if the $CTcalc/CT_{99.9} \geq 1.0$, the 99.9 percent *Giardia lamblia* inactivation requirement has been achieved; or

(B) Successive $CTcalc/CT_{99.9}$ values, representing sequential inactivation ratios, are determined between the point of disinfectant application and a point before or at the first customer during peak hourly flow. Under this alternative, the following method must be used to calculate the total inactivation ratio:

(1) Determine $\frac{CTcalc}{CT_{99.9}}$ for each sequence.

(2) Add the $\frac{CTcalc}{CT_{99.9}}$ values together $\left(\Sigma \frac{(CTcalc)}{CT_{99.9}} \right)$

(3) If $\Sigma \left(\frac{CTcalc}{CT_{99.9}} \right) \geq 1.0$, the 99.9 percent *Giardia lamblia* inactivation requirement has been achieved.

(ii) If the system uses more than one point of disinfectant application before or at the first customer, the system must determine the CT value of each disinfection sequence immediately prior to the next point of disinfectant application during peak hourly flow. The $CTcalc/CT_{99.9}$ value of each sequence and

$$\sum \frac{CTcalc}{CT_{99.9}}$$

must be calculated using the method in paragraph (b)(4)(i)(B) of this section to determine if the system is in compliance with § 142.72(a).

(iii) Although not required, the total percent inactivation for a system with one or more points of residual disinfectant concentration monitoring may be calculated by solving the following equation:

$$\text{Percent inactivation} = 100 - \frac{100}{10^z}$$

$$\text{where } z = 3 \times \sum \left(\frac{CTcalc}{CT_{99.9}} \right)$$

(5) The residual disinfectant concentration of the water entering the distribution system must be monitored continuously, and the lowest value must be recorded each day, except that if there is a failure in the continuous monitoring equipment, grab sampling every 4 hours may be conducted in lieu of continuous monitoring, but for no more than 5 working days following the failure of the equipment, and systems serving 3,300 or fewer persons may take grab samples in lieu of providing continuous monitoring on an ongoing basis at the frequencies prescribed below:

System size by population	Samples/day[1]
<500	1
501 to 1,000	2
1,001 to 2,500	3
2,501 to 3,300	4

[1] The day's samples cannot be taken at the same time. The sampling intervals are subject to State review and approval.

If at any time the residual disinfectant concentration falls below 0.2 mg/l in a system using grab sampling in lieu of continuous monitoring, the system must take a grab sample every 4 hours until the residual concentration is equal to or greater than 0.2 mg/l.

(6)(i) The residual disinfectant concentration must be measured at least at the same points in the distribution system and at the same time as total coliforms are sampled, as specified in § 141.21, except that the State may allow a public water system which uses both a surface water source or a ground water source under direct influence of surface water, and a ground water source, to take disinfectant residual samples at points other than the total coliform sampling points if the State determines that such points are more representative of treated (disinfected) water quality within the distribution system. Heterotrophic bacteria, measured as heterotrophic plate count (HPC) as specified in paragraph (a)(3) of

this section, may be measured in lieu of residual disinfectant concentration.

(ii) If the State determines, based on site-specific considerations, that a system has no means for having a sample transported and analyzed for HPC by a certified laboratory under the requisite time and temperature conditions specified by paragraph (a)(3) of this section and that the system is providing adequate disinfection in the distribution system, the requirements of paragraph (b)(6)(i) of this section do not apply to that system.

(c) *Monitoring requirements for systems using filtration treatment.* A public water system that uses a surface water source or a ground water source under the influence of surface water and provides filtration treatment must monitor in accordance with this paragraph (c) beginning June 29, 1993, or when filtration is installed, whichever is later.

(1) Turbidity measurements as required by § 141.73 must be performed on representative samples of the system's filtered water every four hours (or more frequently) that the system serves water to the public. A public water system may substitute continuous turbidity monitoring for grab sample monitoring if it validates the continuous measurement for accuracy on a regular basis using a protocol approved by the State. For any systems using slow sand filtration or filtration treatment other than conventional treatment, direct filtration, or diatomaceous earth filtration, the State may reduce the sampling frequency to once per day if it determines that less frequent monitoring is sufficient to indicate effective filtration performance. For systems serving 500 or fewer persons, the State may reduce the turbidity sampling frequency to once per day, regardless of the type of filtration treatment used, if the State determines that less frequent monitoring is sufficient to indicate effective filtration performance.

(2) The residual disinfectant concentration of the water entering the distribution system must be monitored continuously, and the lowest value must be recorded each day, except that if there is a failure in the continuous monitoring equipment, grab sampling every 4 hours may be conducted in lieu of continuous monitoring, but for no more than 5 working days following the failure of the equipment, and systems serving 3,300 or fewer persons may take grab samples in lieu of providing continuous monitoring on an ongoing basis at the frequencies each day prescribed below:

System size by population	Samples/day [1]
±500	1
501 to 1,000	2
1,001 to 2,500	3
2,501 to 3,300	4

[1] The day's samples cannot be taken at the same time. The sampling intervals are subject to State review and approval.

If at any time the residual disinfectant concentration falls below 0.2 mg/l in a system using grab sampling in lieu of continuous monitoring, the system must take a grab sample every 4 hours until the residual disinfectant concentration is equal to or greater than 0.2 mg/l.

(3)(i) The residual disinfectant concentration must be measured at least at the same points in the distribution system and at the same time as total coliforms are sampled, as specified in § 141.21, except that the State may allow a public water system which uses both a surface water source or a ground water source under direct influence of surface water, and a ground water source to take disinfectant residual samples at points other than the total coliform sampling points if the State determines that such points are more representative of treated (disinfected) water quality within the distribution system. Heterotrophic bacteria, measured as heterotrophic plate count (HPC) as specified in paragraph (a)(3) of this section, may be measured in lieu of residual disinfectant concentration.

(ii) If the State determines, based on site-specific considerations, that a system has no means for having a sample transported and analyzed for HPC by a certified laboratory under the requisite time and temperature conditions specified by paragraph (a)(3) of this section and that the system is providing adequate disinfection in the distribution system, the requirements of paragraph

(c)(3)(i) of this section do not apply to that system.

[54 FR 27527, June 29, 1989, as amended at 59 FR 62470, Dec. 5, 1994; 60 FR 34086, June 29, 1995]

§141.75 Reporting and recordkeeping requirements.

(a) A public water system that uses a surface water source and does not provide filtration treatment must report monthly to the State the information specified in this paragraph (a) beginning December 31, 1990, unless the State has determined that filtration is required in writing pursuant to section 1412(b)(7)(C)(iii), in which case the State may specify alternative reporting requirements, as appropriate, until filtration is in place. A public water system that uses a ground water source under the direct influence of surface water and does not provide filtration treatment must report monthly to the State the information specified in this paragraph (a) beginning December 31, 1990, or 6 months after the State determines that the ground water source is under the direct influence of surface water, whichever is later, unless the State has determined that filtration is required in writing pursuant to §1412(b)(7)(C)(iii), in which case the State may specify alternative reporting requirements, as appropriate, until filtration is in place.

(1) Source water quality information must be reported to the State within 10 days after the end of each month the system serves water to the public. Information that must be reported includes:

(i) The cumulative number of months for which results are reported.

(ii) The number of fecal and/or total coliform samples, whichever are analyzed during the month (if a system monitors for both, only fecal coliforms must be reported), the dates of sample collection, and the dates when the turbidity level exceeded 1 NTU.

(iii) The number of samples during the month that had equal to or less than 20/100 ml fecal coliforms and/or equal to or less than 100/100 ml total coliforms, whichever are analyzed.

(iv) The cumulative number of fecal or total coliform samples, whichever are analyzed, during the previous six months the system served water to the public.

(v) The cumulative number of samples that had equal to or less than 20/100 ml fecal coliforms or equal to or less than 100/100 ml total coliforms, whichever are analyzed, during the previous six months the system served water to the public.

(vi) The percentage of samples that had equal to or less than 20/100 ml fecal coliforms or equal to or less than 100/100 ml total coliforms, whichever are analyzed, during the previous six months the system served water to the public.

(vii) The maximum turbidity level measured during the month, the date(s) of occurrence for any measurement(s) which exceeded 5 NTU, and the date(s) the occurrence(s) was reported to the State.

(viii) For the first 12 months of recordkeeping, the dates and cumulative number of events during which the turbidity exceeded 5 NTU, and after one year of recordkeeping for turbidity measurements, the dates and cumulative number of events during which the turbidity exceeded 5 NTU in the previous 12 months the system served water to the public.

(ix) For the first 120 months of recordkeeping, the dates and cumulative number of events during which the turbidity exceeded 5 NTU, and after 10 years of recordkeeping for turbidity measurements, the dates and cumulative number of events during which the turbidity exceeded 5 NTU in the previous 120 months the system served water to the public.

(2) Disinfection information specified in §141.74(b) must be reported to the State within 10 days after the end of each month the system serves water to the public. Information that must be reported includes:

(i) For each day, the lowest measurement of residual disinfectant concentration in mg/l in water entering the distribution system.

(ii) The date and duration of each period when the residual disinfectant concentration in water entering the distribution system fell below 0.2 mg/l and when the State was notified of the occurrence.

(iii) The daily residual disinfectant concentration(s) (in mg/l) and disinfectant contact time(s) (in minutes) used for calculating the CT value(s).

(iv) If chlorine is used, the daily measurement(s) of pH of disinfected water following each point of chlorine disinfection.

(v) The daily measurement(s) of water temperature in °C following each point of disinfection.

(vi) The daily CTcalc and CTcalc/$CT_{99.9}$ values for each disinfectant measurement or sequence and the sum of all CTcalc/$CT_{99.9}$ values ((CTcalc/$CT_{99.9}$)) before or at the first customer.

(vii) The daily determination of whether disinfection achieves adequate *Giardia* cyst and virus inactivation, i.e., whether (CTcalc/$CT_{99.9}$) is at least 1.0 or, where disinfectants other than chlorine are used, other indicator conditions that the State determines are appropriate, are met.

(viii) The following information on the samples taken in the distribution system in conjunction with total coliform monitoring pursuant to § 141.72:

(A) Number of instances where the residual disinfectant concentration is measured;

(B) Number of instances where the residual disinfectant concentration is not measured but heterotrophic bacteria plate count (HPC) is measured;

(C) Number of instances where the residual disinfectant concentration is measured but not detected and no HPC is measured;

(D) Number of instances where the residual disinfectant concentration is detected and where HPC is >500/ml;

(E) Number of instances where the residual disinfectant concentration is not measured and HPC is >500/ml;

(F) For the current and previous month the system served water to the public, the value of ''V'' in the following formula:

$$V = \frac{c+d+e}{a+b} \times 100$$

where

a=the value in paragraph (a)(2)(viii)(A) of this section,

b=the value in paragraph (a)(2)(viii)(B) of this section,

c=the value in paragraph (a)(2)(viii)(C) of this section,

d=the value in paragraph (a)(2)(viii)(D) of this section, and

e=the value in paragraph (a)(2)(viii)(E) of this section.

(G) If the State determines, based on site-specific considerations, that a system has no means for having a sample transported and analyzed for HPC by a certified laboratory under the requisite time and temperature conditions specified by § 141.74(a)(3) and that the system is providing adequate disinfection in the distribution system, the requirements of paragraph (a)(2)(viii) (A)–(F) of this section do not apply to that system.

(ix) A system need not report the data listed in paragraphs (a)(2) (i), and (iii)–(vi) of this section if all data listed in paragraphs (a)(2) (i)–(viii) of this section remain on file at the system, and the State determines that:

(A) The system has submitted to the State all the information required by paragraphs (a)(2) (i)–(viii) of this section for at least 12 months; and

(B) The State has determined that the system is not required to provide filtration treatment.

(3) No later than ten days after the end of each Federal fiscal year (September 30), each system must provide to the State a report which summarizes its compliance with all watershed control program requirements specified in § 141.71(b)(2).

(4) No later than ten days after the end of each Federal fiscal year (September 30), each system must provide to the State a report on the on-site inspection conducted during that year pursuant to § 141.71(b)(3), unless the on-site inspection was conducted by the State. If the inspection was conducted by the State, the State must provide a copy of its report to the public water system.

(5)(i) Each system, upon discovering that a waterborne disease outbreak potentially attributable to that water system has occurred, must report that occurrence to the State as soon as possible, but no later than by the end of the next business day.

(ii) If at any time the turbidity exceeds 5 NTU, the system must inform the State as soon as possible, but no

later than the end of the next business day.

(iii) If at any time the residual falls below 0.2 mg/l in the water entering the distribution system, the system must notify the State as soon as possible, but no later than by the end of the next business day. The system also must notify the State by the end of the next business day whether or not the residual was restored to at least 0.2 mg/l within 4 hours.

(b) A public water system that uses a surface water source or a ground water source under the direct influence of surface water and provides filtration treatment must report monthly to the State the information specified in this paragraph (b) beginning June 29, 1993, or when filtration is installed, whichever is later.

(1) Turbidity measurements as required by § 141.74(c)(1) must be reported within 10 days after the end of each month the system serves water to the public. Information that must be reported includes:

(i) The total number of filtered water turbidity measurements taken during the month.

(ii) The number and percentage of filtered water turbidity measurements taken during the month which are less than or equal to the turbidity limits specified in § 141.73 for the filtration technology being used.

(iii) The date and value of any turbidity measurements taken during the month which exceed 5 NTU.

(2) Disinfection information specified in § 141.74(c) must be reported to the State within 10 days after the end of each month the system serves water to the public. Information that must be reported includes:

(i) For each day, the lowest measurement of residual disinfectant concentration in mg/l in water entering the distribution system.

(ii) The date and duration of each period when the residual disinfectant concentration in water entering the distribution system fell below 0.2 mg/l and when the State was notified of the occurrence.

(iii) The following information on the samples taken in the distribution system in conjunction with total coliform monitoring pursuant to § 141.72:

(A) Number of instances where the residual disinfectant concentration is measured;

(B) Number of instances where the residual disinfectant concentration is not measured but heterotrophic bacteria plate count (HPC) is measured;

(C) Number of instances where the residual disinfectant concentration is measured but not detected and no HPC is measured;

(D) Number of instances where no residual disinfectant concentration is detected and where HPC is >500/ml;

(E) Number of instances where the residual disinfectant concentration is not measured and HPC is >500/ml;

(F) For the current and previous month the system serves water to the public, the value of ''V'' in the following formula:

$$V = \frac{c+d+e}{a+b} \times 100$$

where

a=the value in paragraph (b)(2)(iii)(A) of this section,

b=the value in paragraph (b)(2)(iii)(B) of this section,

c=the value in paragraph (b)(2)(iii)(C) of this section,

d=the value in paragraph (b)(2)(iii)(D) of this section, and

e=the value in paragraph (b)(2)(iii)(E) of this section.

(G) If the State determines, based on site-specific considerations, that a system has no means for having a sample transported and analyzed for HPC by a certified laboratory within the requisite time and temperature conditions specified by § 141.74(a)(3) and that the system is providing adequate disinfection in the distribution system, the requirements of paragraph (b)(2)(iii) (A)–(F) of this section do not apply.

(iv) A system need not report the data listed in paragraph (b)(2)(i) of this section if all data listed in paragraphs (b)(2) (i)–(iii) of this section remain on file at the system and the State determines that the system has submitted all the information required by paragraphs (b)(2) (i)–(iii) of this section for at least 12 months.

(3)(i) Each system, upon discovering that a waterborne disease outbreak potentially attributable to that water

system has occurred, must report that occurrence to the State as soon as possible, but no later than by the end of the next business day.

(ii) If at any time the turbidity exceeds 5 NTU, the system must inform the State as soon as possible, but no later than the end of the next business day.

(iii) If at any time the residual falls below 0.2 mg/l in the water entering the distribution system, the system must notify the State as soon as possible, but no later than by the end of the next business day. The system also must notify the State by the end of the next business day whether or not the residual was restored to at least 0.2 mg/l within 4 hours.

Subpart I—Control of Lead and Copper

SOURCE: 56 FR 26548, June 7, 1991, unless otherwise noted.

§ 141.80 General requirements.

(a) *Applicability and effective dates.* (1) The requirements of this subpart I constitute the national primary drinking water regulations for lead and copper. Unless otherwise indicated, each of the provisions of this subpart applies to community water systems and non-transient, non-community water systems (hereinafter referred to as ''water systems'' or ''systems'').

(2) The requirements set forth in §§ 141.86 to 141.91 shall take effect on July 7, 1991. The requirements set forth in §§ 141.80 to 141.85 shall take effect on December 7, 1992.

(b) *Scope.* These regulations establish a treatment technique that includes requirements for corrosion control treatment, source water treatment, lead service line replacement, and public education. These requirements are triggered, in some cases, by lead and copper action levels measured in samples collected at consumers' taps.

(c) *Lead and copper action levels.* (1) The lead action level is exceeded if the concentration of lead in more than 10 percent of tap water samples collected during any monitoring period conducted in accordance with § 141.86 is greater than 0.015 mg/L (i.e., if the ''90th percentile'' lead level is greater than 0.015 mg/L).

(2) The copper action level is exceeded if the concentration of copper in more than 10 percent of tap water samples collected during any monitoring period conducted in accordance with § 141.86 is greater than 1.3 mg/L (i.e., if the ''90th percentile'' copper level is greater than 1.3 mg/L).

(3) The 90th percentile lead and copper levels shall be computed as follows:

(i) The results of all lead or copper samples taken during a monitoring period shall be placed in ascending order from the sample with the lowest concentration to the sample with the highest concentration. Each sampling result shall be assigned a number, ascending by single integers beginning with the number 1 for the sample with the lowest contaminant level. The number assigned to the sample with the highest contaminant level shall be equal to the total number of samples taken.

(ii) The number of samples taken during the monitoring period shall be multiplied by 0.9.

(iii) The contaminant concentration in the numbered sample yielded by the calculation in paragraph (c)(3)(ii) is the 90th percentile contaminant level.

(iv) For water systems serving fewer than 100 people that collect 5 samples per monitoring period, the 90th percentile is computed by taking the average of the highest and second highest concentrations.

(d) *Corrosion control treatment requirements.* (1) All water systems shall install and operate optimal corrosion control treatment as defined in § 141.2.

(2) Any water system that complies with the applicable corrosion control treatment requirements specified by the State under §§ 141.81 and 141.82 shall be deemed in compliance with the treatment requirement contained in paragraph (d)(1) of this section.

(e) *Source water treatment requirements.* Any system exceeding the lead or copper action level shall implement all applicable source water treatment requirements specified by the State under § 141.83.

(f) *Lead service line replacement requirements.* Any system exceeding the lead action level after implementation

of applicable corrosion control and source water treatment requirements shall complete the lead service line replacement requirements contained in §141.84.

(g) *Public education requirements.* Any system exceeding the lead action level shall implement the public education requirements contained in §141.85.

(h) *Monitoring and analytical requirements.* Tap water monitoring for lead and copper, monitoring for water quality parameters, source water monitoring for lead and copper, and analyses of the monitoring results under this subpart shall be completed in compliance with §§141.86, 141.87, 141.88, and 141.89.

(i) *Reporting requirements.* Systems shall report to the State any information required by the treatment provisions of this subpart and §141.90.

(j) *Recordkeeping requirements.* Systems shall maintain records in accordance with §141.91.

(k) *Violation of national primary drinking water regulations.* Failure to comply with the applicable requirements of §§141.80–141.91, including requirements established by the State pursuant to these provisions, shall constitute a violation of the national primary drinking water regulations for lead and/or copper.

[56 FR 26548, June 7, 1991; 57 FR 28788, June 29, 1992]

§141.81 Applicability of corrosion control treatment steps to small, medium-size and large water systems.

(a) Systems shall complete the applicable corrosion control treatment requirements described in §141.82 by the deadlines established in this section.

(1) A large system (serving >50,000 persons) shall complete the corrosion control treatment steps specified in paragraph (d) of this section, unless it is deemed to have optimized corrosion control under paragraph (b)(2) or (b)(3) of this section.

(2) A small system (serving ≤3300 persons) and a medium-size system (serving >3,300 and ≤50,000 persons) shall complete the corrosion control treatment steps specified in paragraph (e) of this section, unless it is deemed to have optimized corrosion control under paragraph (b)(1), (b)(2), or (b)(3) of this section.

(b) A system is deemed to have optimized corrosion control and is not required to complete the applicable corrosion control treatment steps identified in this section if the system satisfies one of the following criteria:

(1) A small or medium-size water system is deemed to have optimized corrosion control if the system meets the lead and copper action levels during each of two consecutive six-month monitoring periods conducted in accordance with §141.86.

(2) Any water system may be deemed by the State to have optimized corrosion control treatment if the system demonstrates to the satisfaction of the State that it has conducted activities equivalent to the corrosion control steps applicable to such system under this section. If the State makes this determination, it shall provide the system with written notice explaining the basis for its decision and shall specify the water quality control parameters representing optimal corrosion control in accordance with §141.82(f). A system shall provide the State with the following information in order to support a determination under this paragraph:

(i) The results of all test samples collected for each of the water quality parameters in §141.82(c)(3).

(ii) A report explaining the test methods used by the water system to evaluate the corrosion control treatments listed in §141.82(c)(1), the results of all tests conducted, and the basis for the system's selection of optimal corrosion control treatment;

(iii) A report explaining how corrosion control has been installed and how it is being maintained to insure minimal lead and copper concentrations at consumers' taps; and

(iv) The results of tap water samples collected in accordance with §141.86 at least once every six months for one year after corrosion control has been installed.

(3) Any water system is deemed to have optimized corrosion control if it submits results of tap water monitoring conducted in accordance with §141.86 and source water monitoring conducted in accordance with §141.88 that demonstrates for two consecutive six-month monitoring periods that the difference between the 90th percentile

tap water lead level computed under § 141.80(c)(3), and the highest source water lead concentration, is less than the Practical Quantitation Level for lead specified in § 141.89(a)(1)(ii).

(c) Any small or medium-size water system that is required to complete the corrosion control steps due to its exceedance of the lead or copper action level may cease completing the treatment steps whenever the system meets both action levels during each of two consecutive monitoring periods conducted pursuant to § 141.86 and submits the results to the State. If any such water system thereafter exceeds the lead or copper action level during any monitoring period, the system (or the State, as the case may be) shall recommence completion of the applicable treatment steps, beginning with the first treatment step which was not previously completed in its entirety. The State may require a system to repeat treatment steps previously completed by the system where the State determines that this is necessary to implement properly the treatment requirements of this section. The State shall notify the system in writing of such a determination and explain the basis for its decision. The requirement for any small- or medium-size system to implement corrosion control treatment steps in accordance with paragraph (e) of this section (including systems deemed to have optimized corrosion control under paragraph (b)(1) of this section) is triggered whenever any small- or medium-size system exceeds the lead or copper action level.

(d) *Treatment steps and deadlines for large systems.* Except as provided in paragraph (b) (2) and (3) of this section, large systems shall complete the following corrosion control treatment steps (described in the referenced portions of §§ 141.82, 141.86, and 141.87) by the indicated dates.

(1) *Step 1:* The system shall conduct initial monitoring (§ 141.86(d)(1) and § 141.87(b)) during two consecutive six-month monitoring periods by January 1, 1993.

(2) *Step 2:* The system shall complete corrosion control studies (§ 141.82(c)) by July 1, 1994.

(3) *Step 3:* The State shall designate optimal corrosion control treatment (§ 141.82(d)) by January 1, 1995.

(4) *Step 4:* The system shall install optimal corrosion control treatment (§ 141.82(e)) by January 1, 1997.

(5) *Step 5:* The system shall complete follow-up sampling (§ 141.86(d)(2) and § 141.87(c)) by January 1, 1998.

(6) *Step 6:* The State shall review installation of treatment and designate optimal water quality control parameters (§ 141.82(f)) by July 1, 1998.

(7) *Step 7:* The system shall operate in compliance with the State-specified optimal water quality control parameters (§ 141.82(g)) and continue to conduct tap sampling (§ 141.86(d)(3) and § 141.87(d)).

(e) *Treatment Steps and deadlines for small and medium-size systems.* Except as provided in paragraph (b) of this section, small and medium-size systems shall complete the following corrosion control treatment steps (described in the referenced portions of §§ 141.82, 141.86 and 141.87) by the indicated time periods.

(1) *Step 1:* The system shall conduct initial tap sampling (§ 141.86(d)(1) and § 141.87(b)) until the system either exceeds the lead or copper action level or becomes eligible for reduced monitoring under § 141.86(d)(4). A system exceeding the lead or copper action level shall recommend optimal corrosion control treatment (§ 141.82(a)) within six months after it exceeds one of the action levels.

(2) *Step 2:* Within 12 months after a system exceeds the lead or copper action level, the State may require the system to perform corrosion control studies (§ 141.82(b)). If the State does not require the system to perform such studies, the State shall specify optimal corrosion control treatment (§ 141.82(d)) within the following timeframes:

(i) For medium-size systems, within 18 months after such system exceeds the lead or copper action level,

(ii) For small systems, within 24 months after such system exceeds the lead or copper action level.

(3) *Step 3:* If the State requires a system to perform corrosion control studies under step 2, the system shall complete the studies (§ 141.82(c)) within 18 months after the State requires that such studies be conducted.

(4) *Step 4:* If the system has performed corrosion control studies under step 2, the State shall designate optimal corrosion control treatment (§141.82(d)) within 6 months after completion of step 3.

(5) *Step 5:* The system shall install optimal corrosion control treatment (§141.82(e)) within 24 months after the State designates such treatment.

(6) *Step 6:* The system shall complete follow-up sampling (§141.86(d)(2) and §141.87(c)) within 36 months after the State designates optimal corrosion control treatment.

(7) *Step 7:* The State shall review the system's installation of treatment and designate optimal water quality control parameters (§141.82(f)) within 6 months after completion of step 6.

(8) *Step 8:* The system shall operate in compliance with the State-designated optimal water quality control parameters (§141.82(g)) and continue to conduct tap sampling (§141.86(d)(3) and §141.87(d)).

[56 FR 26548, June 7, 1991, as amended at 59 FR 33862, June 30, 1994]

§141.82 Description of corrosion control treatment requirements.

Each system shall complete the corrosion control treatment requirements described below which are applicable to such system under §141.81.

(a) *System recommendation regarding corrosion control treatment.* Based upon the results of lead and copper tap monitoring and water quality parameter monitoring, small and medium-size water systems exceeding the lead or copper action level shall recommend installation of one or more of the corrosion control treatments listed in paragraph (c)(1) of this section which the system believes constitutes optimal corrosion control for that system. The State may require the system to conduct additional water quality parameter monitoring in accordance with §141.87(b) to assist the State in reviewing the system's recommendation.

(b) *State decision to require studies of corrosion control treatment (applicable to small and medium-size systems).* The State may require any small or medium-size system that exceeds the lead or copper action level to perform corrosion control studies under paragraph (c) of this section to identify optimal corrosion control treatment for the system.

(c) *Performance of corrosion control studies.* (1) Any public water system performing corrosion control studies shall evaluate the effectiveness of each of the following treatments, and, if appropriate, combinations of the following treatments to identify the optimal corrosion control treatment for that system:

(i) Alkalinity and pH adjustment;

(ii) Calcium hardness adjustment; and

(iii) The addition of a phosphate or silicate based corrosion inhibitor at a concentration sufficient to maintain an effective residual concentration in all test tap samples.

(2) The water system shall evaluate each of the corrosion control treatments using either pipe rig/loop tests, metal coupon tests, partial-system tests, or analyses based on documented analogous treatments with other systems of similar size, water chemistry and distribution system configuration.

(3) The water system shall measure the following water quality parameters in any tests conducted under this paragraph before and after evaluating the corrosion control treatments listed above:

(i) Lead;

(ii) Copper;

(iii) pH;

(iv) Alkalinity;

(v) Calcium;

(vi) Conductivity;

(vii) Orthophosphate (when an inhibitor containing a phosphate compound is used);

(viii) Silicate (when an inhibitor containing a silicate compound is used);

(ix) Water temperature.

(4) The water system shall identify all chemical or physical constraints that limit or prohibit the use of a particular corrosion control treatment and document such constraints with at least one of the following:

(i) Data and documentation showing that a particular corrosion control treatment has adversely affected other water treatment processes when used by another water system with comparable water quality characteristics; and/or

(ii) Data and documentation demonstrating that the water system has previously attempted to evaluate a particular corrosion control treatment and has found that the treatment is ineffective or adversely affects other water quality treatment processes.

(5) The water system shall evaluate the effect of the chemicals used for corrosion control treatment on other water quality treatment processes.

(6) On the basis of an analysis of the data generated during each evaluation, the water system shall recommend to the State in writing the treatment option that the corrosion control studies indicate constitutes optimal corrosion control treatment for that system. The water system shall provide a rationale for its recommendation along with all supporting documentation specified in paragraphs (c) (1) through (5) of this section.

(d) *State designation of optimal corrosion control treatment.* (1) Based upon consideration of available information including, where applicable, studies performed under paragraph (c) of this section and a system's recommended treatment alternative, the State shall either approve the corrosion control treatment option recommended by the system, or designate alternative corrosion control treatment(s) from among those listed in paragraph (c)(1) of this section. When designating optimal treatment the State shall consider the effects that additional corrosion control treatment will have on water quality parameters and on other water quality treatment processes.

(2) The State shall notify the system of its decision on optimal corrosion control treatment in writing and explain the basis for this determination. If the State requests additional information to aid its review, the water system shall provide the information.

(e) *Installation of optimal corrosion control.* Each system shall properly install and operate throughout its distribution system the optimal corrosion control treatment designated by the State under paragraph (d) of this section.

(f) *State review of treatment and specification of optimal water quality control parameters.* The State shall evaluate the results of all lead and copper tap samples and water quality parameter samples submitted by the water system and determine whether the system has properly installed and operated the optimal corrosion control treatment designated by the State in paragraph (d) of this section. Upon reviewing the results of tap water and water quality parameter monitoring by the system, both before and after the system installs optimal corrosion control treatment, the State shall designate:

(1) A minimum value or a range of values for pH measured at each entry point to the distribution system;

(2) A minimum pH value, measured in all tap samples. Such value shall be equal to or greater than 7.0, unless the State determines that meeting a pH level of 7.0 is not technologically feasible or is not necessary for the system to optimize corrosion control;

(3) If a corrosion inhibitor is used, a minimum concentration or a range of concentrations for the inhibitor, measured at each entry point to the distribution system and in all tap samples, that the State determines is necessary to form a passivating film on the interior walls of the pipes of the distribution system;

(4) If alkalinity is adjusted as part of optimal corrosion control treatment, a minimum concentration or a range of concentrations for alkalinity, measured at each entry point to the distribution system and in all tap samples;

(5) If calcium carbonate stabilization is used as part of corrosion control, a minimum concentration or a range of concentrations for calcium, measured in all tap samples.

The values for the applicable water quality control parameters listed above shall be those that the State determines to reflect optimal corrosion control treatment for the system. The State may designate values for additional water quality control parameters determined by the State to reflect optimal corrosion control for the system. The State shall notify the system in writing of these determinations and explain the basis for its decisions.

(g) *Continued operation and monitoring.* All systems shall maintain water quality parameter values at or above

minimum values or within ranges designated by the State under paragraph (f) of this section in each sample collected under §141.87(d). If the water quality parameter value of any sample is below the minimum value or outside the range designated by the State, then the system is out of compliance with this paragraph. As specified in §141.87(d), the system may take a confirmation sample for any water quality parameter value no later than 3 days after the first sample. If a confirmation sample is taken, the result must be averaged with the first sampling result and the average must be used for any compliance determinations under this paragraph. States have discretion to delete results of obvious sampling errors from this calculation.

(h) *Modification of State treatment decisions.* Upon its own initiative or in response to a request by a water system or other interested party, a State may modify its determination of the optimal corrosion control treatment under paragraph (d) of this section or optimal water quality control parameters under paragraph (f) of this section. A request for modification by a system or other interested party shall be in writing, explain why the modification is appropriate, and provide supporting documentation. The State may modify its determination where it concludes that such change is necessary to ensure that the system continues to optimize corrosion control treatment. A revised determination shall be made in writing, set forth the new treatment requirements, explain the basis for the State's decision, and provide an implementation schedule for completing the treatment modifications.

(i) *Treatment decisions by EPA in lieu of the State.* Pursuant to the procedures in §142.19, the EPA Regional Administrator may review treatment determinations made by a State under paragraphs (d), (f), or (h) of this section and issue federal treatment determinations consistent with the requirements of those paragraphs where the Regional Administrator finds that:

(1) A State has failed to issue a treatment determination by the applicable deadlines contained in §141.81,

(2) A State has abused its discretion in a substantial number of cases or in cases affecting a substantial population, or

(3) The technical aspects of a State's determination would be indefensible in an expected Federal enforcement action taken against a system.

§141.83 Source water treatment requirements.

Systems shall complete the applicable source water monitoring and treatment requirements (described in the referenced portions of paragraph (b) of this section, and in §§141.86, and 141.88) by the following deadlines.

(a) *Deadlines for completing source water treatment steps*—(1) *Step 1:* A system exceeding the lead or copper action level shall complete lead and copper source water monitoring (§141.88(b)) and make a treatment recommendation to the State (§141.83(b)(1)) within 6 months after exceeding the lead or copper action level.

(2) *Step 2:* The State shall make a determination regarding source water treatment (§141.83(b)(2)) within 6 months after submission of monitoring results under step 1.

(3) *Step 3:* If the State requires installation of source water treatment, the system shall install the treatment (§141.83(b)(3)) within 24 months after completion of step 2.

(4) *Step 4:* The system shall complete follow-up tap water monitoring (§141.86(d)(2) and source water monitoring (§141.88(c)) within 36 months after completion of step 2.

(5) *Step 5:* The State shall review the system's installation and operation of source water treatment and specify maximum permissible source water levels (§141.83(b)(4)) within 6 months after completion of step 4.

(6) *Step 6:* The system shall operate in compliance with the State-specified maximum permissible lead and copper source water levels (§141.83(b)(4)) and continue source water monitoring (§141.88(d)).

(b) *Description of source water treatment requirements*—(1) *System treatment recommendation.* Any system which exceeds the lead or copper action level shall recommend in writing to the State the installation and operation of one of the source water treatments

listed in paragraph (b)(2) of this section. A system may recommend that no treatment be installed based upon a demonstration that source water treatment is not necessary to minimize lead and copper levels at users' taps.

(2) *State determination regarding source water treatment.* The State shall complete an evaluation of the results of all source water samples submitted by the water system to determine whether source water treatment is necessary to minimize lead or copper levels in water delivered to users' taps. If the State determines that treatment is needed, the State shall either require installation and operation of the source water treatment recommended by the system (if any) or require the installation and operation of another source water treatment from among the following: Ion exchange, reverse osmosis, lime softening or coagulation/filtration. If the State requests additional information to aid in its review, the water system shall provide the information by the date specified by the State in its request. The State shall notify the system in writing of its determination and set forth the basis for its decision.

(3) *Installation of source water treatment.* Each system shall properly install and operate the source water treatment designated by the State under paragraph (b)(2) of this section.

(4) *State review of source water treatment and specification of maximum permissible source water levels.* The State shall review the source water samples taken by the water system both before and after the system installs source water treatment, and determine whether the system has properly installed and operated the source water treatment designated by the State. Based upon its review, the State shall designate the maximum permissible lead and copper concentrations for finished water entering the distribution system. Such levels shall reflect the contaminant removal capability of the treatment properly operated and maintained. The State shall notify the system in writing and explain the basis for its decision.

(5) *Continued operation and maintenance.* Each water system shall maintain lead and copper levels below the maximum permissible concentrations designated by the State at each sampling point monitored in accordance with § 141.88. The system is out of compliance with this paragraph if the level of lead or copper at any sampling point is greater than the maximum permissible concentration designated by the State.

(6) *Modification of State treatment decisions.* Upon its own initiative or in response to a request by a water system or other interested party, a State may modify its determination of the source water treatment under paragraph (b)(2) of this section, or maximum permissible lead and copper concentrations for finished water entering the distribution system under paragraph (b)(4) of this section. A request for modification by a system or other interested party shall be in writing, explain why the modification is appropriate, and provide supporting documentation. The State may modify its determination where it concludes that such change is necessary to ensure that the system continues to minimize lead and copper concentrations in source water. A revised determination shall be made in writing, set forth the new treatment requirements, explain the basis for the State's decision, and provide an implementation schedule for completing the treatment modifications.

(7) *Treatment decisions by EPA in lieu of the State.* Pursuant to the procedures in § 142.19, the EPA Regional Administrator may review treatment determinations made by a State under paragraphs (b) (2), (4), or (6) of this section and issue Federal treatment determinations consistent with the requirements of those paragraphs where the Administrator finds that:

(i) A State has failed to issue a treatment determination by the applicable deadlines contained in § 141.83(a),

(ii) A state has abused its discretion in a substantial number of cases or in cases affecting a substantial population, or

(iii) The technical aspects of a State's determination would be indefensible in an expected Federal enforcement action taken against a system.

§141.84 Lead service line replacement requirements.

(a) Systems that fail to meet the lead action level in tap samples taken pursuant to §141.86(d)(2), after installing corrosion control and/or source water treatment (whichever sampling occurs later), shall replace lead service lines in accordance with the requirements of this section. If a system is in violation of §141.81 or §141.83 for failure to install source water or corrosion control treatment, the State may require the system to commence lead service line replacement under this section after the date by which the system was required to conduct monitoring under §141.86(d)(2) has passed.

(b) A system shall replace annually at least 7 percent of the initial number of lead service lines in its distribution system. The initial number of lead service lines is the number of lead lines in place at the time the replacement program begins. The system shall identify the initial number of lead service lines in its distribution system based upon a materials evaluation, including the evaluation required under §141.86(a). The first year of lead service line replacement shall begin on the date the action level was exceeded in tap sampling referenced in paragraph (a) of this section.

(c) A system is not required to replace an individual lead service line if the lead concentration in all service line samples from that line, taken pursuant to §141.86(b)(3), is less than or equal to 0.015 mg/L.

(d) A water system shall replace the entire service line (up to the building inlet) unless it demonstrates to the satisfaction of the State under paragraph (e) of this section that it controls less than the entire service line. In such cases, the system shall replace the portion of the line which the State determines is under the system's control. The system shall notify the user served by the line that the system will replace the portion of the service line under its control and shall offer to replace the building owner's portion of the line, but is not required to bear the cost of replacing the building owner's portion of the line. For buildings where only a portion of the lead service line is replaced, the water system shall inform the resident(s) that the system will collect a first flush tap water sample after partial replacement of the service line is completed if the resident(s) so desire. In cases where the resident(s) accept the offer, the system shall collect the sample and report the results to the resident(s) within 14 days following partial lead service line replacement.

(e) A water system is presumed to control the entire lead service line (up to the building inlet) unless the system demonstrates to the satisfaction of the State, in a letter submitted under §141.90(e)(4), that it does not have any of the following forms of control over the entire line (as defined by state statutes, municipal ordinances, public service contracts or other applicable legal authority): authority to set standards for construction, repair, or maintenance of the line, authority to replace, repair, or maintain the service line, or ownership of the service line. The State shall review the information supplied by the system and determine whether the system controls less than the entire service line and, in such cases, shall determine the extent of the system's control. The State's determination shall be in writing and explain the basis for its decision.

(f) The State shall require a system to replace lead service lines on a shorter schedule than that required by this section, taking into account the number of lead service lines in the system, where such a shorter replacement schedule is feasible. The State shall make this determination in writing and notify the system of its finding within 6 months after the system is triggered into lead service line replacement based on monitoring referenced in paragraph (a) of this section.

(g) Any system may cease replacing lead service lines whenever first draw samples collected pursuant to §141.86(b)(2) meet the lead action level during each of two consecutive monitoring periods and the system submits the results to the State. If first draw tap samples collected in any such system thereafter exceeds the lead action level, the system shall recommence replacing lead service lines pursuant to paragraph (b) of this section.

(h) To demonstrate compliance with paragraphs (a) through (d) of this section, a system shall report to the State the information specified in § 141.90(e).

[56 FR 26548, June 7, 1991; 57 FR 28788, June 29, 1992]

§ 141.85 Public education and supplemental monitoring requirements.

A water system that exceeds the lead action level based on tap water samples collected in accordance with § 141.86 shall deliver the public education materials contained in paragraphs (a) and (b) of this section in accordance with the requirements in paragraph (c) of this section.

(a) *Content of written materials.* A water system shall include the following text in all of the printed materials it distributes through its lead public education program. Any additional information presented by a system shall be consistent with the information below and be in plain English that can be understood by laypersons.

(1) *Introduction.* The United States Environmental Protection Agency (EPA) and [insert name of water supplier] are concerned about lead in your drinking water. Although most homes have very low levels of lead in their drinking water, some homes in the community have lead levels above the EPA action level of 15 parts per billion (ppb), or 0.015 milligrams of lead per liter of water (mg/L). Under Federal law we are required to have a program in place to minimize lead in your drinking water by [insert date when corrosion control will be completed for your system]. This program includes corrosion control treatment, source water treatment, and public education. We are also required to replace each lead service line that we control if the line contributes lead concentrations of more than 15 ppb after we have completed the comprehensive treatment program. If you have any questions about how we are carrying out the requirements of the lead regulation please give us a call at [insert water system's phone number]. This brochure explains the simple steps you can take to protect you and your family by reducing your exposure to lead in drinking water.

(2) *Health effects of lead.* Lead is a common metal found throughout the environment in lead-based paint, air, soil, household dust, food, certain types of pottery porcelain and pewter, and water. Lead can pose a significant risk to your health if too much of it enters your body. Lead builds up in the body over many years and can cause damage to the brain, red blood cells and kidneys. The greatest risk is to young children and pregnant women. Amounts of lead that won't hurt adults can slow down normal mental and physical development of growing bodies. In addition, a child at play often comes into contact with sources of lead contamination—like dirt and dust—that rarely affect an adult. It is important to wash children's hands and toys often, and to try to make sure they only put food in their mouths.

(3) *Lead in drinking water.* (i) Lead in drinking water, although rarely the sole cause of lead poisoning, can significantly increase a person's total lead exposure, particularly the exposure of infants who drink baby formulas and concentrated juices that are mixed with water. The EPA estimates that drinking water can make up 20 percent or more of a person's total exposure to lead.

(ii) Lead is unusual among drinking water contaminants in that it seldom occurs naturally in water supplies like rivers and lakes. Lead enters drinking water primarily as a result of the corrosion, or wearing away, of materials containing lead in the water distribution system and household plumbing. These materials include lead-based solder used to join copper pipe, brass and chrome plated brass faucets, and in some cases, pipes made of lead that connect your house to the water main (service lines). In 1986, Congress banned the use of lead solder containing greater than 0.2% lead, and restricted the lead content of faucets, pipes and other plumbing materials to 8.0%.

(iii) When water stands in lead pipes or plumbing systems containing lead for several hours or more, the lead may dissolve into your drinking water. This means the first water drawn from the tap in the morning, or later in the afternoon after returning from work or

school, can contain fairly high levels of lead.

(4) *Steps you can take in the home to reduce exposure to lead in drinking water.* (i) Despite our best efforts mentioned earlier to control water corrosivity and remove lead from the water supply, lead levels in some homes or buildings can be high. To find out whether you need to take action in your own home, have your drinking water tested to determine if it contains excessive concentrations of lead. Testing the water is essential because you cannot see, taste, or smell lead in drinking water. Some local laboratories that can provide this service are listed at the end of this booklet. For more information on having your water tested, please call [insert phone number of water system].

(ii) If a water test indicates that the drinking water drawn from a tap in your home contains lead above 15 ppb, then you should take the following precautions:

(A) Let the water run from the tap before using it for drinking or cooking any time the water in a faucet has gone unused for more than six hours. The longer water resides in your home's plumbing the more lead it may contain. Flushing the tap means running the cold water faucet until the water gets noticeably colder, usually about 15–30 seconds. If your house has a lead service line to the water main, you may have to flush the water for a longer time, perhaps one minute, before drinking. Although toilet flushing or showering flushes water through a portion of your home's plumbing system, you still need to flush the water in each faucet before using it for drinking or cooking. Flushing tap water is a simple and inexpensive measure you can take to protect your family's health. It usually uses less than one or two gallons of water and costs less than [insert a cost estimate based on flushing two times a day for 30 days] per month. To conserve water, fill a couple of bottles for drinking water after flushing the tap, and whenever possible use the first flush water to wash the dishes or water the plants. If you live in a high-rise building, letting the water flow before using it may not work to lessen your risk from lead. The plumbing systems have more, and sometimes larger pipes than smaller buildings. Ask your landlord for help in locating the source of the lead and for advice on reducing the lead level.

(B) Try not to cook with, or drink water from the hot water tap. Hot water can dissolve more lead more quickly than cold water. If you need hot water, draw water from the cold tap and heat it on the stove.

(C) Remove loose lead solder and debris from the plumbing materials installed in newly constructed homes, or homes in which the plumbing has recently been replaced, by removing the faucet strainers from all taps and running the water from 3 to 5 minutes. Thereafter, periodically remove the strainers and flush out any debris that has accumulated over time.

(D) If your copper pipes are joined with lead solder that has been installed illegally since it was banned in 1986, notify the plumber who did the work and request that he or she replace the lead solder with lead-free solder. Lead solder looks dull gray, and when scratched with a key looks shiny. In addition, notify your State [insert name of department responsible for enforcing the Safe Drinking Water Act in your State] about the violation.

(E) Determine whether or not the service line that connects your home or apartment to the water main is made of lead. The best way to determine if your service line is made of lead is by either hiring a licensed plumber to inspect the line or by contacting the plumbing contractor who installed the line. You can identify the plumbing contractor by checking the city's record of building permits which should be maintained in the files of the [insert name of department that issues building permits]. A licensed plumber can at the same time check to see if your homes's plumbing contains lead solder, lead pipes, or pipe fittings that contain lead. The public water system that delivers water to your home should also maintain records of the materials located in the distribution system. If the service line that connects your dwelling to the water main contributes more than 15 ppb to drinking water, after our comprehensive treatment program is in place, we are required to replace the line. If the line

is only partially controlled by the [insert name of the city, county, or water system that controls the line], we are required to provide you with information on how to replace your portion of the service line, and offer to replace that portion of the line at your expense and take a follow-up tap water sample within 14 days of the replacement. Acceptable replacement alternatives include copper, steel, iron, and plastic pipes.

(F) Have an electrician check your wiring. If grounding wires from the electrical system are attached to your pipes, corrosion may be greater. Check with a licensed electrician or your local electrical code to determine if your wiring can be grounded elsewhere. DO NOT attempt to change the wiring yourself because improper grounding can cause electrical shock and fire hazards.

(iii) The steps described above will reduce the lead concentrations in your drinking water. However, if a water test indicates that the drinking water coming from your tap contains lead concentrations in excess of 15 ppb after flushing, or after we have completed our actions to minimize lead levels, then you may want to take the following additional measures:

(A) Purchase or lease a home treatment device. Home treatment devices are limited in that each unit treats only the water that flows from the faucet to which it is connected, and all of the devices require periodic maintenance and replacement. Devices such as reverse osmosis systems or distillers can effectively remove lead from your drinking water. Some activated carbon filters *may* reduce lead levels at the tap, however all lead reduction claims should be investigated. Be sure to check the actual performance of a specific home treatment device before and after installing the unit.

(B) Purchase bottled water for drinking and cooking.

(iv) You can consult a variety of sources for additional information. Your family doctor or pediatrician can perform a blood test for lead and provide you with information about the health effects of lead. State and local government agencies that can be contacted include:

(A) [insert the name of city or county department of public utilities] at [insert phone number] can provide you with information about your community's water supply, and a list of local laboratories that have been certified by EPA for testing water quality;

(B) [insert the name of city or county department that issues building permits] at [insert phone number] can provide you with information about building permit records that should contain the names of plumbing contractors that plumbed your home; and

(C) [insert the name of the State Department of Public Health] at [insert phone number] or the [insert the name of the city or county health department] at [insert phone number] can provide you with information about the health effects of lead and how you can have your child's blood tested.

(v) The following is a list of some State approved laboratories in your area that you can call to have your water tested for lead. [Insert names and phone numbers of at least two laboratories].

(b) *Content of broadcast materials.* A water system shall include the following information in all public service announcements submitted under its lead public education program to television and radio stations for broadcasting:

(1) Why should everyone want to know the facts about lead and drinking water? Because unhealthy amounts of lead can enter drinking water through the plumbing in your home. That's why I urge you to do what I did. I had my water tested for [insert free or $ per sample]. You can contact the [insert the name of the city or water system] for information on testing and on simple ways to reduce your exposure to lead in drinking water.

(2) To have your water tested for lead, or to get more information about this public health concern, please call [insert the phone number of the city or water system].

(c) *Delivery of a public education program.* (1) In communities where a significant proportion of the population speaks a language other than English, public education materials shall be communicated in the appropriate language(s).

(2) A community water system that fails to meet the lead action level on the basis of tap water samples collected in accordance with §141.86 shall, within 60 days:

(i) Insert notices in each customer's water utility bill containing the information in paragraph (a) of this section, along with the following alert on the water bill itself in large print: ''SOME HOMES IN THIS COMMUNITY HAVE ELEVATED LEAD LEVELS IN THEIR DRINKING WATER. LEAD CAN POSE A SIGNIFICANT RISK TO YOUR HEALTH. PLEASE READ THE ENCLOSED NOTICE FOR FURTHER INFORMATION.''

(ii) Submit the information in paragraph (a) of this section to the editorial departments of the major daily and weekly newspapers circulated throughout the community.

(iii) Deliver pamphlets and/or brochures that contain the public education materials in paragraphs (a) (2) and (4) of this section to facilities and organizations, including the following:

(A) Public schools and/or local school boards;

(B) City or county health department;

(C) Women, Infants, and Children and/or Head Start Program(s) whenever available;

(D) Public and private hospitals and/or clinics;

(E) Pediatricians;

(F) Family planning clinics; and

(G) Local welfare agencies.

(iv) Submit the public service announcement in paragraph (b) of this section to at least five of the radio and television stations with the largest audiences that broadcast to the community served by the water system.

(3) A community water system shall repeat the tasks contained in paragraphs (c)(2) (i), (ii) and (iii) of this section every 12 months, and the tasks contained in paragraphs (c)(2)(iv) of this section every 6 months for as long as the system exceeds the lead action level.

(4) Within 60 days after it exceeds the lead action level, a non-transient non-community water system shall deliver the public education materials contained in paragraphs (a) (1), (2), and (4) of this section as follows:

(i) Post informational posters on lead in drinking water in a public place or common area in each of the buildings served by the system; and

(ii) Distribute informational pamphlets and/or brochures on lead in drinking water to each person served by the non-transient non-community water system.

(5) A non-transient non-community water system shall repeat the tasks contained in paragraph (c)(4) of this section at least once during each calendar year in which the system exceeds the lead action level.

(6) A water system may discontinue delivery of public education materials if the system has met the lead action level during the most recent six-month monitoring period conducted pursuant to §141.86. Such a system shall recommence public education in accordance with this section if it subsequently exceeds the lead action level during any monitoring period.

(d) *Supplemental monitoring and notification of results.* A water system that fails to meet the lead action level on the basis of tap samples collected in accordance with §141.86 shall offer to sample the tap water of any customer who requests it. The system is not required to pay for collecting or analyzing the sample, nor is the system required to collect and analyze the sample itself.

[56 FR 26548, June 7, 1991; 57 FR 28788, June 29, 1992]

§141.86 Monitoring requirements for lead and copper in tap water.

(a) *Sample site location.* (1) By the applicable date for commencement of monitoring under paragraph (d)(1) of this section, each water system shall complete a materials evaluation of its distribution system in order to identify a pool of targeted sampling sites that meets the requirements of this section, and which is sufficiently large to ensure that the water system can collect the number of lead and copper tap samples required in paragraph (c) of this section. All sites from which first draw samples are collected shall be selected from this pool of targeted sampling sites. Sampling sites may not include faucets that have point-of-use or point-

of-entry treatment devices designed to remove inorganic contaminants.

(2) A water system shall use the information on lead, copper, and galvanized steel that it is required to collect under § 141.42(d) of this part [special monitoring for corrosivity characteristics] when conducting a materials evaluation. When an evaluation of the information collected pursuant to § 141.42(d) is insufficient to locate the requisite number of lead and copper sampling sites that meet the targeting criteria in paragraph (a) of this section, the water system shall review the sources of information listed below in order to identify a sufficient number of sampling sites. In addition, the system shall seek to collect such information where possible in the course of its normal operations (e.g., checking service line materials when reading water meters or performing maintenance activities):

(i) All plumbing codes, permits, and records in the files of the building department(s) which indicate the plumbing materials that are installed within publicly and privately owned structures connected to the distribution system;

(ii) All inspections and records of the distribution system that indicate the material composition of the service connections that connect a structure to the distribution system; and

(iii) All existing water quality information, which includes the results of all prior analyses of the system or individual structures connected to the system, indicating locations that may be particularly susceptible to high lead or copper concentrations.

(3) The sampling sites selected for a community water system's sampling pool (''tier l sampling sites'') shall consist of single family structures that:

(i) Contain copper pipes with lead solder installed after 1982 or contain lead pipes; and/or

(ii) Are served by a lead service line. When multiple-family residences comprise at least 20 percent of the structures served by a water system, the system may include these types of structures in its sampling pool.

(4) Any community water system with insufficient tier 1 sampling sites shall complete its sampling pool with ''tier 2 sampling sites'', consisting of buildings, including multiple-family residences that:

(i) Contain copper pipes with lead solder installed after 1982 or contain lead pipes; and/or

(ii) Are served by a lead service line.

(5) Any community water system with insufficient tier 1 and tier 2 sampling sites shall complete its sampling pool with ''tier 3 sampling sites'', consisting of single family structures that contain copper pipes with lead solder installed before 1983.

(6) The sampling sites selected for a non-transient noncommunity water system (''tier l sampling sites'') shall consist of buildings that:

(i) Contain copper pipes with lead solder installed after 1982 or contain lead pipes; and/or

(ii) Are served by a lead service line.

(7) A non-transient non-community water system with insufficient tier 1 sites that meet the targeting criteria in paragraph (a)(6) of this section shall complete its sampling pool with sampling sites that contain copper pipes with lead solder installed before 1983.

(8) Any water system whose sampling pool does not consist exclusively of tier 1 sites shall demonstrate in a letter submitted to the State under § 141.90(a)(2) why a review of the information listed in paragraph (a)(2) of this section was inadequate to locate a sufficient number of tier l sites. Any community water system which includes tier 3 sampling sites in its sampling pool shall demonstrate in such a letter why it was unable to locate a sufficient number of tier l and tier 2 sampling sites.

(9) Any water system whose distribution system contains lead service lines shall draw 50 percent of the samples it collects during each monitoring period from sites that contain lead pipes, or copper pipes with lead solder, and 50 percent of the samples from sites served by a lead service line. A water system that cannot identify a sufficient number of sampling sites served by a lead service line shall demonstrate in a letter submitted to the State under § 141.90(a)(4) why the system was unable to locate a sufficient number of such sites. Such a water system shall collect first draw samples

from all of the sites identified as being served by such lines.

(b) *Sample collection methods.* (1) All tap samples for lead and copper collected in accordance with this subpart, with the exception of lead service line samples collected under § 141.84(c), shall be first draw samples.

(2) Each first draw tap sample for lead and copper shall be one liter in volume and have stood motionless in the plumbing system of each sampling site for at least six hours. First draw samples from residential housing shall be collected from the cold water kitchen tap or bathroom sink tap. First-draw samples from a nonresidential building shall be collected at an interior tap from which water is typically drawn for consumption. First draw samples may be collected by the system or the system may allow residents to collect first draw samples after instructing the residents of the sampling procedures specified in this paragraph. To avoid problems of residents handling nitric acid, acidification of first draw samples may be done up to 14 days after the sample is collected. If the sample is not acidified immediately after collection, then the sample must stand in the original container for at least 28 hours after acidification. If a system allows residents to perform sampling, the system may not challenge, based on alleged errors in sample collection, the accuracy of sampling results.

(3) Each service line sample shall be one liter in volume and have stood motionless in the lead service line for at least six hours. Lead service line samples shall be collected in one of the following three ways:

(i) At the tap after flushing the volume of water between the tap and the lead service line. The volume of water shall be calculated based on the interior diameter and length of the pipe between the tap and the lead service line;

(ii) Tapping directly into the lead service line; or

(iii) If the sampling site is a building constructed as a single-family residence, allowing the water to run until there is a significant change in temperature which would be indicative of water that has been standing in the lead service line.

(4) A water system shall collect each first draw tap sample from the same sampling site from which it collected a previous sample. If, for any reason, the water system cannot gain entry to a sampling site in order to collect a follow-up tap sample, the system may collect the follow-up tap sample from another sampling site in its sampling pool as long as the new site meets the same targeting criteria, and is within reasonable proximity of the original site.

(c) *Number of samples.* Water systems shall collect at least one sample during each monitoring period specified in paragraph (d) of this section from the number of sites listed in the first column below (''standard monitoring''). A system conducting reduced monitoring under paragraph (d)(4) of this section may collect one sample from the number of sites specified in the second column below during each monitoring period specified in paragraph (d)(4) of this section.

System size (No. people served)	No. of sites (standard monitoring)	No. of sites (reduced monitoring)
>100,000	100	50
10,001–100,000	60	30
3,301 to 10,000	40	20
501 to 3,300	20	10
101 to 500	10	5
≤100	5	5

(d) *Timing of monitoring*—(1) *Initial tap sampling.*

The first six-month monitoring period for small, medium-size and large systems shall begin on the following dates:

System size (No. people served)	First six-month monitoring period begins on
>50,000	January 1, 1992.
3,301 to 50,000	July 1, 1992.
≤3,300	July 1, 1993.

(i) All large systems shall monitor during two consecutive six-month periods.

(ii) All small and medium-size systems shall monitor during each six-month monitoring period until:

(A) The system exceeds the lead or copper action level and is therefore required to implement the corrosion control treatment requirements under § 141.81, in which case the system shall

continue monitoring in accordance with paragraph (d)(2) of this section, or

(B) The system meets the lead and copper action levels during two consecutive six-month monitoring periods, in which case the system may reduce monitoring in accordance with paragraph (d)(4) of this section.

(2) *Monitoring after installation of corrosion control and source water treatment.* (i) Any large system which installs optimal corrosion control treatment pursuant to § 141.81(d)(4) shall monitor during two consecutive six-month monitoring periods by the date specified in § 141.81(d)(5).

(ii) Any small or medium-size system which installs optimal corrosion control treatment pursuant to § 141.81(e)(5) shall monitor during two consecutive six-month monitoring periods by the date specified in § 141.81(e)(6).

(iii) Any system which installs source water treatment pursuant to § 141.83(a)(3) shall monitor during two consecutive six-month monitoring periods by the date specified in § 141.83(a)(4).

(3) *Monitoring after State specifies water quality parameter values for optimal corrosion control.* After the State specifies the values for water quality control parameters under § 141.82(f), the system shall monitor during each subsequent six-month monitoring period, with the first monitoring period to begin on the date the State specifies the optimal values under § 141.82(f).

(4) *Reduced monitoring.* (i) A small or medium-size water system that meets the lead and copper action levels during each of two consecutive six-month monitoring periods may reduce the number of samples in accordance with paragraph (c) of this section, and reduce the frequency of sampling to once per year.

(ii) Any water system that maintains the range of values for the water quality control parameters reflecting optimal corrosion control treatment specified by the State under § 141.82(f) during each of two consecutive six-month monitoring periods may request that the State allow the system to reduce the frequency of monitoring to once per year and to reduce the number of lead and copper samples in accordance with paragraph (c) of this section. The State shall review the information submitted by the water system and shall make its decision in writing, setting forth the basis for its determination. The State shall review, and where appropriate, revise its determination when the system submits new monitoring or treatment data, or when other data relevant to the number and frequency of tap sampling becomes available.

(iii) A small or medium-size water system that meets the lead and copper action levels during three consecutive years of monitoring may reduce the frequency of monitoring for lead and copper from annually to once every three years. Any water system that maintains the range of values for the water quality control parameters reflecting optimal corrosion control treatment specified by the State under § 141.82(f) during three consecutive years of monitoring may request that the State allow the system to reduce the frequency of monitoring from annually to once every three years. The State shall review the information submitted by the water system and shall make its decision in writing, setting forth the basis for its determination. The State shall review, and where appropriate, revise its determination when the system submits new monitoring or treatment data, or when other data relevant to the number and frequency of tap sampling becomes available.

(iv) A water system that reduces the number and frequency of sampling shall collect these samples from sites included in the pool of targeted sampling sites identified in paragraph (a) of this section. Systems sampling annually or less frequently shall conduct the lead and copper tap sampling during the months of June, July, August or September.

(v) A small- or medium-size water system subject to reduced monitoring that exceeds the lead or copper action level shall resume sampling in accordance with paragraph (d)(3) of this section and collect the number of samples specified for standard monitoring under paragraph (d) of this section. Such system shall also conduct water quality parameter monitoring in accordance with § 141.87 (b), (c) or (d) (as

appropriate) during the monitoring period in which it exceeded the action level. Any water system subject to the reduced monitoring frequency that fails to operate within the range of values for the water quality parameters specified by the State under §141.82(f) shall resume tap water sampling in accordance with paragraph (d)(3) of this section and collect the number of samples specified for standard monitoring under paragraph (c) of this section.

(e) *Additional monitoring by systems.* The results of any monitoring conducted in addition to the minimum requirements of this section shall be considered by the system and the State in making any determinations (i.e., calculating the 90th percentile lead or copper level) under this subpart.

[56 FR 26548, June 7, 1991; 56 FR 32113, July 15, 1991; 57 FR 28788, June 29, 1992]

§141.87 Monitoring requirements for water quality parameters.

All large water systems, and all small- and medium-size systems that exceed the lead or copper action level shall monitor water quality parameters in addition to lead and copper in accordance with this section. The requirements of this section are summarized in the table at the end of this section.

(a) *General requirements*—(1) *Sample collection methods.* (i) Tap samples shall be representative of water quality throughout the distribution system taking into account the number of persons served, the different sources of water, the different treatment methods employed by the system, and seasonal variability. Tap sampling under this section is not required to be conducted at taps targeted for lead and copper sampling under §141.86(a). [Note: Systems may find it convenient to conduct tap sampling for water quality parameters at sites used for coliform sampling under 40 CFR 141.21.]

(ii) Samples collected at the entry point(s) to the distribution system shall be from locations representative of each source after treatment. If a system draws water from more than one source and the sources are combined before distribution, the system must sample at an entry point to the distribution system during periods of normal operating conditions (i.e., when water is representative of all sources being used).

(2) *Number of samples.* (i) Systems shall collect two tap samples for applicable water quality parameters during each monitoring period specified under paragraphs (b) through (e) of this section from the following number of sites.

System size (No. people served)	No. of sites for water quality parameters
>100,000	25
10,001–100,000	10
3,301 to 10,000	3
501 to 3,300	2
101 to 500	1
≤100	1

(ii) Systems shall collect two samples for each applicable water quality parameter at each entry point to the distribution system during each monitoring period specified in paragraph (b) of this section. During each monitoring period specified in paragraphs (c)–(e) of this section, systems shall collect one sample for each applicable water quality parameter at each entry point to the distribution system.

(b) *Initial sampling* All large water systems shall measure the applicable water quality parameters as specified below at taps and at each entry point to the distribution system during each six-month monitoring period specified in §141.86(d)(1). All small and medium-size systems shall measure the applicable water quality parameters at the locations specified below during each six-month monitoring period specified in §141.86(d)(1) during which the system exceeds the lead or copper action level.

(1) At taps:

(i) pH;

(ii) Alkalinity;

(iii) Orthophosphate, when an inhibitor containing a phosphate compound is used;

(iv) Silica, when an inhibitor containing a silicate compound is used;

(v) Calcium;

(vi) Conductivity; and

(vii) Water temperature.

(2) At each entry point to the distribution system: all of the applicable parameters listed in paragraph (b)(1) of this section.

(c) *Monitoring after installation of corrosion control.* Any large system which installs optimal corrosion control treatment pursuant to §141.81(d)(4) shall measure the water quality parameters at the locations and frequencies specified below during each six-month monitoring period specified in §141.86(d)(2)(i). Any small or medium-size system which installs optimal corrosion control treatment shall conduct such monitoring during each six-month monitoring period specified in §141.86(d)(2)(ii) in which the system exceeds the lead or copper action level.

(1) At taps, two samples for:

(i) pH;

(ii) Alkalinity;

(iii) Orthophosphate, when an inhibitor containing a phosphate compound is used;

(iv) Silica, when an inhibitor containing a silicate compound is used;

(v) Calcium, when calcium carbonate stabilization is used as part of corrosion control.

(2) At each entry point to the distribution system, one sample every two weeks (bi-weekly) for:

(i) pH;

(ii) When alkalinity is adjusted as part of optimal corrosion control, a reading of the dosage rate of the chemical used to adjust alkalinity, and the alkalinity concentration; and

(iii) When a corrosion inhibitor is used as part of optimal corrosion control, a reading of the dosage rate of the inhibitor used, and the concentration of orthophosphate or silica (whichever is applicable).

(d) *Monitoring after State specifies water quality parameter values for optimal corrosion control.* After the State specifies the values for applicable water quality control parameters reflecting optimal corrosion control treatment under §141.82(f), all large systems shall measure the applicable water quality parameters in accordance with paragraph (c) of this section during each monitoring period specified in §141.86(d)(3). Any small or medium-size system shall conduct such monitoring during each monitoring period specified in §141.86(d)(3) in which the system exceeds the lead or copper action level. The system may take a confirmation sample for any water quality parameter value no later than 3 days after the first sample. If a confirmation sample is taken, the result must be averaged with the first sampling result and the average must be used for any compliance determinations under §141.82(g). States have discretion to delete results of obvious sampling errors from this calculation.

(e) *Reduced monitoring.* (1) Any water system that maintains the range of values for the water quality parameters reflecting optimal corrosion control treatment during each of two consecutive six-month monitoring periods under paragraph (d) of this section shall continue monitoring at the entry point(s) to the distribution system as specified in paragraph (c)(2) of this section. Such system may collect two tap samples for applicable water quality parameters from the following reduced number of sites during each six-month monitoring period.

System size (No. of people served)	Reduced No. of sites for water quality parameters
>100,000	10
10,001 to 100,000	7
3,301 to 10,000	3
501 to 3,300	2
101 to 500	1
≤100	1

(2) Any water system that maintains the range of values for the water quality parameters reflecting optimal corrosion control treatment specified by the State under §141.82(f) during three consecutive years of monitoring may reduce the frequency with which it collects the number of tap samples for applicable water quality parameters specified in this paragraph (e)(1) of this section from every six months to annually. Any water system that maintains the range of values for the water quality parameters reflecting optimal corrosion control treatment specified by the State under §141.82(f) during three consecutive years of annual monitoring under this paragraph may reduce the frequency with which it collects the number of tap samples for applicable water quality parameters specified in paragraph (e)(1) from annually to every three years.

(3) A water system that conducts sampling annually shall collect these

samples evenly throughout the year so as to reflect seasonal variability.

(4) Any water system subject to the reduced monitoring frequency that fails to operate within the range of values for the water quality parameters specified by the State in §141.82(f) shall resume tap water sampling in accordance with the number and frequency requirements in paragraph (d) of this section.

(f) *Additional monitoring by systems.* The results of any monitoring conducted in addition to the minimum requirements of this section shall be considered by the system and the State in making any determinations (i.e., determining concentrations of water quality parameters) under this section or §141.82.

SUMMARY OF MONITORING REQUIREMENTS FOR WATER QUALITY PARAMETERS [1]

Monitoring Period	Parameters [2]	Location	Frequency
Initial Monitoring	pH, alkalinity, orthophosphate or silica [3], calcium, conductivity, temperature.	Taps and at entry point(s) to distribution system.	Every 6 months
After Installation of Corrosion Control	pH, alkalinity, orthophosphate or silica [3], calcium [4].	Taps	Every 6 months
	pH, alkalinity dosage rate and concentration (if alkalinity adjusted as part of corrosion control), inhibitor dosage rate and inhibitor residual [5].	Entry point(s) to distribution system.	Biweekly
After State Specifies Parameter Values For Optimal Corrosion Control.	pH, alkalinity, orthophosphate or silica [3], calcium [4].	Taps	Every 6 months
	pH, alkalinity dosage rate and concentration (if alkalinity adjusted as part of corrosion control), inhibitor dosage rate and inhibitor residual [5].	Entry point(s) to distribution system.	Biweekly
Reduced Monitoring	pH, alkalinity, orthophosphate or silica [3], calcium [4].	Taps	Every 6 months at a reduced number of sites
	pH, alkalinity dosage rate and concentration (if alkalinity adjusted as part of corrosion control), inhibitor dosage rate and inhibitor residual [5].	Entry point(s) to distribution system.	Biweekly

[1] Table is for illustrative purposes; consult the text of this section for precise regulatory requirements.
[2] Small and medium-size systems have to monitor for water quality parameters only during monitoring periods in which the system exceeds the lead or copper action level.
[3] Orthophosphate must be measured only when an inhibitor containing a phosphate compound is used. Silica must be measured only when an inhibitor containing silicate compound is used.
[4] Calcium must be measured only when calcium carbonate stabilization is used as part of corrosion control.
[5] Inhibitor dosage rates and inhibitor residual concentrations (orthophosphate or silica) must be measured only when an inhibitor is used.

[56 FR 26548, June 7, 1991; 57 FR 28788, June 29, 1992, as amended at 59 FR 33862, June 30, 1994]

§141.88 Monitoring requirements for lead and copper in source water.

(a) *Sample location, collection methods, and number of samples.* (1) A water system that fails to meet the lead or copper action level on the basis of tap samples collected in accordance with §141.86 shall collect lead and copper source water samples in accordance with the requirements regarding sample location, number of samples, and collection methods specified in §141.23(a) (1)–(4) (inorganic chemical sampling). (Note: The timing of sampling for lead and copper shall be in accordance with paragraphs (b) and (c) of this section, and not dates specified in §141.23(a) (1) and (2)).

(2) Where the results of sampling indicate an exceedance of maximum permissible source water levels established under §141.83(b)(4), the State may require that one additional sample be collected as soon as possible after

the initial sample was taken (but not to exceed two weeks) at the same sampling point. If a State-required confirmation sample is taken for lead or copper, then the results of the initial and confirmation sample shall be averaged in determining compliance with the State-specified maximum permissible levels. Any sample value below the detection limit shall be considered to be zero. Any value above the detection limit but below the PQL shall either be considered as the measured value or be considered one-half the PQL.

(b) *Monitoring frequency after system exceeds tap water action level.* Any system which exceeds the lead or copper action level at the tap shall collect one source water sample from each entry point to the distribution system within six months after the exceedance.

(c) *Monitoring frequency after installation of source water treatment.* Any system which installs source water treatment pursuant to § 141.83(a)(3) shall collect an additional source water sample from each entry point to the distribution system during two consecutive six-month monitoring periods by the deadline specified in § 141.83(a)(4).

(d) *Monitoring frequency after State specifies maximum permissible source water levels or determines that source water treatment is not needed.* (1) A system shall monitor at the frequency specified below in cases where the State specifies maximum permissible source water levels under § 141.83(b)(4) or determines that the system is not required to install source water treatment under § 141.83(b)(2).

(i) A water system using only groundwater shall collect samples once during the three-year compliance period (as that term is defined in § 141.2) in effect when the applicable State determination under paragraph (d)(1) of this section is made. Such systems shall collect samples once during each subsequent compliance period.

(ii) A water system using surface water (or a combination of surface and groundwater) shall collect samples once during each year, the first annual monitoring period to begin on the date on which the applicable State determination is made under paragraph (d)(1) of this section.

(2) A system is not required to conduct source water sampling for lead and/or copper if the system meets the action level for the specific contaminant in tap water samples during the entire source water sampling period applicable to the system under paragraph (d)(1) (i) or (ii) of this section.

(e) *Reduced monitoring frequency.* (1) A water system using only groundwater which demonstrates that finished drinking water entering the distribution system has been maintained below the maximum permissible lead and/or copper concentrations specified by the State in § 141.83(b)(4) during at least three consecutive compliance periods under paragraph (d)(1) of this section may reduce the monitoring frequency for lead and/or copper to once during each nine-year compliance cycle (as that term is defined in § 141.2).

(2) A water system using surface water (or a combination of surface and ground waters) which demonstrates that finished drinking water entering the distribution system has been maintained below the maximum permissible lead and copper concentrations specified by the State in § 141.83(b)(4) for at least three consecutive years may reduce the monitoring frequency in paragraph (d)(1) of this section to once during each nine-year compliance cycle (as that term is defined in § 141.2).

(3) A water system that uses a new source of water is not eligible for reduced monitoring for lead and/or copper until concentrations in samples collected from the new source during three consecutive monitoring periods are below the maximum permissible lead and copper concentrations specified by the State in § 141.83(a)(5).

[56 FR 26548, June 7, 1991; 57 FR 28788 and 28789, June 29, 1992]

§ 141.89 Analytical methods.

(a) Analyses for lead, copper, pH, conductivity, calcium, alkalinity, orthophosphate, silica, and temperature shall be conducted with the methods in § 141.23(k)(1).

(1) Analyses under this section shall only be conducted by laboratories that have been certified by EPA or the State. To obtain certification to conduct analyses for lead and copper, laboratories must:

(i) Analyze performance evaluation samples which include lead and copper provided by EPA Environmental Monitoring and Support Laboratory or equivalent samples provided by the State; and

(ii) Achieve quantitative acceptance limits as follows:

(A) For lead: ±30 percent of the actual amount in the Performance Evaluation sample when the actual amount is greater than or equal to 0.005 mg/L. The Practical Quantitation Level, or PQL for lead is 0.005 mg/L.

(B) For Copper: ±10 percent of the actual amount in the Performance Evaluation sample when the actual amount is greater than or equal to 0.050 mg/L. The Practical Quantitation Level, or PQL for copper is 0.050 mg/L;

(iii) Achieve method detection limits according to the procedures in appendix B of part 136 of this title as follows:

(A) Lead: 0.001 mg/L (only if source water compositing is done under §141.23(a)(4)); and

(B) Copper: 0.001 mg/L or 0.020 mg/L when atomic absorption direct aspiration is used (only if source water compositing is done under §141.23(a)(4)).

(iv) Be currently certified by EPA or the State to perform analyses to the specifications described in paragraph (a)(2) of this section.

(2) States have the authority to allow the use of previously collected monitoring data for purposes of monitoring, if the data were collected and analyzed in accordance with the requirements of this subpart.

(3) All lead and copper levels measured between the PQL and MDL must be either reported as measured or they can be reported as one-half the PQL specified for lead and copper in paragraph (a)(1)(ii) of this section. All levels below the lead and copper MDLs must be reported as zero.

(4) All copper levels measured between the PQL and the MDL must be either reported as measured or they can be reported as one-half the PQL (0.025 mg/L). All levels below the copper MDL must be reported as zero.

(b) [Reserved]

[56 FR 26548, June 7, 1991, as amended at 57 FR 28789, June 29, 1992; 57 FR 31847, July 17, 1992; 59 FR 33863, June 30, 1994; 59 FR 62470, Dec. 5, 1994]

§141.90 Reporting requirements.

All water systems shall report all of the following information to the State in accordance with this section.

(a) *Reporting requirements for tap water monitoring for lead and copper and for water quality parameter monitoring.* (1) A water system shall report the information specified below for all tap water samples within the first 10 days following the end of each applicable monitoring period specified in §§141.86 and 141.87 and 141.88 (i.e., every six-months, annually, or every 3 years).

(i) The results of all tap samples for lead and copper including the location of each site and the criteria under §141.86(a) (3), (4), (5), (6), and/or (7) under which the site was selected for the system's sampling pool;

(ii) A certification that each first draw sample collected by the water system is one-liter in volume and, to the best of their knowledge, has stood motionless in the service line, or in the interior plumbing of a sampling site, for at least six hours;

(iii) Where residents collected samples, a certification that each tap sample collected by the residents was taken after the water system informed them of proper sampling procedures specified in §141.86(b)(2);

(iv) The 90th percentile lead and copper concentrations measured from among all lead and copper tap water samples collected during each monitoring period (calculated in accordance with §141.80(c)(3));

(v) With the exception of initial tap sampling conducted pursuant to §141.86(d)(1), the system shall designate any site which was not sampled during previous monitoring periods, and include an explanation of why sampling sites have changed;

(vi) The results of all tap samples for pH, and where applicable, alkalinity, calcium, conductivity, temperature, and orthophosphate or silica collected under §141.87 (b)–(e);

(vii) The results of all samples collected at the entry point(s) to the distribution system for applicable water quality parameters under §141.87 (b)–(e).

(2) By the applicable date in §141.86(d)(1) for commencement of monitoring, each community water system

which does not complete its targeted sampling pool with tier 1 sampling sites meeting the criteria in § 141.86(a)(3) shall send a letter to the State justifying its selection of tier 2 and/or tier 3 sampling sites under § 141.86 (a)(4) and/or (a)(5).

(3) By the applicable date in § 141.86(d)(1) for commencement of monitoring, each non-transient, non-community water system which does not complete its sampling pool with tier 1 sampling sites meeting the criteria in § 141.86(a)(6) shall send a letter to the State justifying its selection of sampling sites under § 141.86(a)(7).

(4) By the applicable date in § 141.86(d)(1) for commencement of monitoring, each water system with lead service lines that is not able to locate the number of sites served by such lines required under § 141.86(a)(9) shall send a letter to the State demonstrating why it was unable to locate a sufficient number of such sites based upon the information listed in § 141.86(a)(2).

(5) Each water system that requests that the State reduce the number and frequency of sampling shall provide the information required under § 141.86(d)(4).

(b) *Source water monitoring reporting requirements.* (1) A water system shall report the sampling results for all source water samples collected in accordance with § 141.88 within the first 10 days following the end of each source water monitoring period (i.e., annually, per compliance period, per compliance cycle) specified in § 141.88.

(2) With the exception of the first round of source water sampling conducted pursuant to § 141.88(b), the system shall specify any site which was not sampled during previous monitoring periods, and include an explanation of why the sampling point has changed.

(c) *Corrosion control treatment reporting requirements.* By the applicable dates under § 141.81, systems shall report the following information:

(1) For systems demonstrating that they have already optimized corrosion control, information required in § 141.81(b) (2) or (3).

(2) For systems required to optimize corrosion control, their recommendation regarding optimal corrosion control treatment under § 141.82(a).

(3) For systems required to evaluate the effectiveness of corrosion control treatments under §141.82(c), the information required by that paragraph.

(4) For systems required to install optimal corrosion control designated by the State under § 141.82(d), a letter certifying that the system has completed installing that treatment.

(d) *Source water treatment reporting requirements.* By the applicable dates in § 141.83, systems shall provide the following information to the State:

(1) If required under § 141.83(b)(1), their recommendation regarding source water treatment;

(2) For systems required to install source water treatment under § 141.83(b)(2), a letter certifying that the system has completed installing the treatment designated by the State within 24 months after the State designated the treatment.

(e) *Lead service line replacement reporting requirements.* Systems shall report the following information to the State to demonstrate compliance with the requirements of § 141.84:

(1) Within 12 months after a system exceeds the lead action level in sampling referred to in § 141.84(a), the system shall demonstrate in writing to the State that it has conducted a material evaluation, including the evaluation in § 141.86(a), to identify the initial number of lead service lines in its distribution system, and shall provide the State with the system's schedule for replacing annually at least 7 percent of the initial number of lead service lines in its distribution system.

(2) Within 12 months after a system exceeds the lead action level in sampling referred to in § 141.84(a), and every 12 months thereafter, the system shall demonstrate to the State in writing that the system has either:

(i) Replaced in the previous 12 months at least 7 percent of the initial lead service lines (or a greater number of lines specified by the State under § 141.84(f)) in its distribution system, or

(ii) Conducted sampling which demonstrates that the lead concentration in all service line samples from an individual line(s), taken pursuant to § 141.86(b)(3), is less than or equal to 0.015 mg/L. In such cases, the total number of lines replaced and/or which

meet the criteria in § 141.84(c) shall equal at least 7 percent of the initial number of lead lines identified under paragraph (a) of this section (or the percentage specified by the State under § 141.84(f)).

(3) The annual letter submitted to the State under paragraph (e)(2) of this section shall contain the following information:

(i) The number of lead service lines scheduled to be replaced during the previous year of the system's replacement schedule;

(ii) The number and location of each lead service line replaced during the previous year of the system's replacement schedule;

(iii) If measured, the water lead concentration and location of each lead service line sampled, the sampling method, and the date of sampling.

(4) As soon as practicable, but in no case later than three months after a system exceeds the lead action level in sampling referred to in § 141.84(a), any system seeking to rebut the presumption that it has control over the entire lead service line pursuant to § 141.84(d) shall submit a letter to the State describing the legal authority (e.g., state statutes, municipal ordinances, public service contracts or other applicable legal authority) which limits the system's control over the service lines and the extent of the system's control.

(f) *Public education program reporting requirements.* By December 31st of each year, any water system that is subject to the public education requirements in § 141.85 shall submit a letter to the State demonstrating that the system has delivered the public education materials that meet the content requirements in § 141.85 (a) and (b) and the delivery requirements in § 141.85(c). This information shall include a list of all the newspapers, radio stations, television stations, facilities and organizations to which the system delivered public education materials during the previous year. The water system shall submit the letter required by this paragraph annually for as long as it exceeds the lead action level.

(g) *Reporting of additional monitoring data.* Any system which collects sampling data in addition to that required by this subpart shall report the results to the State within the first ten days following the end of the applicable monitoring period under §§ 141.86, 141.87 and 141.88 during which the samples are collected.

[56 FR 26548, June 7, 1991; 57 FR 28789, June 29, 1992, as amended at 59 FR 33864, June 30, 1994]

§ 141.91 Recordkeeping requirements.

Any system subject to the requirements of this subpart shall retain on its premises original records of all sampling data and analyses, reports, surveys, letters, evaluations, schedules, State determinations, and any other information required by §§ 141.81 through 141.88. Each water system shall retain the records required by this section for no fewer than 12 years.

Subpart J—Use of Non-Centralized Treatment Devices

SOURCE: 52 FR 25716, July 8, 1987, unless otherwise noted.

§ 141.100 Criteria and procedures for public water systems using point-of-entry devices.

(a) Public water systems may use point-of-entry devices to comply with maximum contaminant levels only if they meet the requirements of this section.

(b) It is the responsibility of the public water system to operate and maintain the point-of-entry treatment system.

(c) The public water system must develop and obtain State approval for a monitoring plan before point-of-entry devices are installed for compliance. Under the plan approved by the State, point-of-entry devices must provide health protection equivalent to central water treatment. ''Equivalent'' means that the water would meet all national primary drinking water regulations and would be of acceptable quality similar to water distributed by a well-operated central treatment plant. In addition to the VOCs, monitoring must include physical measurements and observations such as total flow treated and mechanical condition of the treatment equipment.

(d) Effective technology must be properly applied under a plan ap-

proved by the State and the microbiological safety of the water must be maintained.

(1) The State must require adequate certification of performance, field testing, and, if not included in the certification process, a rigorous engineering design review of the point-of-entry devices.

(2) The design and application of the point-of-entry devices must consider the tendency for increase in heterotrophic bacteria concentrations in water treated with activated carbon. It may be necessary to use frequent backwashing, post-contactor disinfection, and Heterotrophic Plate Count monitoring to ensure that the microbiological safety of the water is not compromised.

(e) *All consumers shall be protected.* Every building connected to the system must have a point-of-entry device installed, maintained, and adequately monitored. The State must be assured that every building is subject to treatment and monitoring, and that the rights and responsibilities of the public water system customer convey with title upon sale of property.

[52 FR 25716, July 8, 1987; 53 FR 25111, July 1, 1988]

§ 141.101 Use of other non-centralized treatment devices.

Public water systems shall not use bottled water or point-of-use devices to achieve compliance with an MCL. Bottled water or point-of-use devices may be used on a temporary basis to avoid an unreasonable risk to health.

Subpart K—Treatment Techniques

SOURCE: 56 FR 3594, Jan. 30, 1991, unless otherwise noted.

§ 141.110 General requirements.

The requirements of subpart K of this part constitute national primary drinking water regulations. These regulations establish treatment techniques in lieu of maximum contaminant levels for specified contaminants.

§ 141.111 Treatment techniques for acrylamide and epichlorohydrin.

Each public water system must certify annually in writing to the State (using third party or manufacturer's certification) that when acrylamide and epichlorohydrin are used in drinking water systems, the combination (or product) of dose and monomer level does not exceed the levels specified as follows:

Acrylamide=0.05% dosed at 1 ppm (or equivalent)

Epichlorohydrin=0.01% dosed at 20 ppm (or equivalent)

Certifications can rely on manufacturers or third parties, as approved by the State.

Subpart M—Information Collection Requriements (ICR) for Public Water Systems

SOURCE: 61 FR 24368, May 14, 1996, unless otherwise noted.

EFFECTIVE DATE NOTE: At 61 FR 24368, May 14, 1996, subpart M consisting of §§ 141.140 through 141.144 were added, effective June 18, 1996 and will expire on Dec. 31, 2000.

§ 141.140 Definitions specific to subpart M.

The following definitions apply only to the requirements of subpart M of this part and are arranged alphabetically.

Distribution system means the components of a PWS that are under the control of that PWS located after the point where the finished water sample is taken and that provide distribution, storage, and/or booster disinfection of finished water.

Distribution System Equivalent (DSE) sample means a sample collected from the distribution system for the purpose of comparing it with the ''simulated distribution system (SDS) sample''. The DSE sample shall be selected using the following criteria:

(1) No additional disinfectant added between the treatment plant and the site where the DSE sample is collected;

(2) Approximate detention time of water is available; and

(3) There is no blending with finished water from other treatment plants.

Entry point to distribution system means a location following one or more finished water sample points but prior to the beginning of the distribution system.

Finished water means water that does not undergo further treatment by a treatment plant other than maintenance of a disinfection residual.

Haloacetic acids (five) (HAA5) means the sum of the concentration in micrograms per liter of the haloacetic acids mono-, di-, and trichloroacetic acid; mono-, and di-, bromoacetic acid, rounded to two significant figures.

Haloacetic acids (six) (HAA6) means the concentration in micrograms per liter of the haloacetic acids mono-, di-, and trichloroacetic acid; mono-, and di- bromoacetic acid; and bromochloroacetic acid, rounded to two significant figures.

Haloacetonitriles (HAN) means the concentration in micrograms per liter of the haloacetonitriles dichloro-, trichloro-, bromochloro-, and dibromoacetonitrile, rounded to two significant figures.

Haloketones (HK) means the concentration in micrograms per liter of the haloketones 1,1-dichloropropanone and 1,1,1- trichloropropanone, rounded to two significant figures.

Intake means the physical location at which the PWS takes water from a water resource. Thereafter, the water is under the control of that PWS.

Notice of applicability means a notice sent by EPA to a PWS that indicates that EPA believes that the PWS must comply with some or all requirements of subpart M. The PWS is required to reply to this notice by providing information specified in the notice (e.g., retail and wholesale population served, types of water sources used, volume of water treated) by the date provided in subpart M.

Process train means some number of unit processes connected in series starting from the treatment plant influent and ending with finished water. A particular unit process may be in more than one process train.

Purchased finished water means finished water purchased by one PWS from another PWS (the wholesaler). Purchased finished water includes both purchased finished water that is redisinfected and purchased finished water that is not.

Simulated distribution system (SDS) sample means a finished water sample incubated at the temperature and detention time of a ''DSE sample'' collected from the distribution system. Analytical results of the SDS sample will be compared with the DSE sample to determine how well the SDS sample predicts disinfection byproduct formation in the actual distribution system sample.

Total finished water means the flow (volume per unit of time) of finished water obtained from all treatment plants operated by a PWS and includes purchased finished water. This flow includes water entering the distribution system and water sold to another PWS.

Treatment plant means the PWS components that have as their exclusive source of water a shared treatment plant influent and that deliver finished water to a common point which is located prior to the point at which finished water enters a distribution system or is diverted for sale to another PWS. For these components of the PWS to be considered part of one treatment plant, the PWS must be able to collect one representative treatment plant influent sample, either at a single sample point or by a composite of multiple influent samples, and there must exist a single sampling point where a representative sample of finished water can be collected. For the purpose of subpart M, a treatment plant is considered to include any site where a disinfectant or oxidant is added to water prior to the water entering the distribution system. Facilities in which ground water is disinfected prior to entering a distribution system, and facilities in which purchased finished water has a disinfectant added prior to entering a distribution system, are considered treatment plants.

Treatment plant influent means water that represents the water quality challenge to a particular plant.

Treatment system means all treatment plants operated by one PWS.

Trihalomethanes (four) (THM4) means the sum of the concentration in micrograms per liter of the trihalomethanes chloroform, bromodichloromethane, dibromochloromethane, and bromoform, rounded to two significant figures.

Unit process means a component of a treatment process train which serves any treatment purpose such as mixing or sedimentation for which design and operating information is requested in § 141.142(a), Table 6c, of this subpart.

Water resource means a body of water before it passes through an intake structure. Examples of a water resource include a river, lake, or aquifer. For a PWS which purchases finished water, the water resource is the wholesale PWS which supplies the purchased finished water. Generally water resources are not under the direct control of a PWS.

Watershed control practice means protection of a water resource from microbiological contamination prior to the water entering an intake. These protective measures might include, but are not limited to, a watershed control program approved under § 141.71(b)(2) of this part, or land use restrictions.

§ 141.141 General requirements, applicability, and schedule for information collection.

(a) *General requirements.* (1) The purpose of subpart M is to collect specified information from certain PWSs for a limited period of time. Accordingly, subpart M is of limited duration and is effective for a defined period (see §§ 141.6(i) and 141.141(e) of this part). Since subpart M does not establish continuing obligations, a PWS that has completed all of its requirements at the required duration and frequency may discontinue its information collection efforts even if subpart M is still in effect.

(2) For the purpose of this subpart, a PWS shall make applicability determinations based on completion of data gathering, calculations, and treatment plant categorization specified in appendix A to paragraph (a) of this section.

(3) For the purpose of this subpart, a PWS that uses multiple wells drawing from the same aquifer and has no central treatment plant is considered to have one treatment plant for those wells and shall conduct required monitoring under this specification. A PWS with multiple wells in one or more aquifers that are treated in the same treatment plant is considered to have one treatment plant for those wells and shall conduct required monitoring under this specification.

(i) To the extent possible, the PWS should sample at the well with the largest flow and at the same well each month for the duration of required monitoring.

(ii) A PWS must report information from § 141.142(a) tables 6a through 6e of this subpart for each well that the PWS sampled.

(4) For the purpose of this subpart, a PWS shall treat ground water sources that have been classified by the State as under the direct influence of surface water by May 14, 1996, as surface water sources. A PWS shall treat ground water sources that either have not been classified by the State (as under the direct influence of surface water or not) or have been classified by the State as ground water, by May 14, 1996, as ground water sources.

APPENDIX A TO 40 CFR 141.141(a)

Purpose. The purpose of this appendix is to enable the PWS to assign proportional amounts of its retail and wholesale population served to specific treatment plants. The PWS shall then use these values to determine which specific requirements in subpart M that it must comply with and on what schedule.

Period of applicability determination. For the purpose of this appendix, a PWS shall make applicability determinations based on population calculated as annual averages based on PWS records of treatment system or treatment plant operation during calendar year 1995.

—If a natural disaster made a treatment system or treatment plant inoperable for one or more calendar months in 1995, the applicability determination will be based on those months in 1995 during which the treatment system or treatment plant was in operation, plus the calendar months from 1994 that are representative of those months of 1995 during which the treatment system or treatment plant was inoperable. The total time period shall be 12 months.

—If the treatment system or treatment plant was not in operation during one or more calendar months during 1995 due to a seasonal reduction in demand for finished water, the months that the treatment system or treatment plant was not in operation are to be included in the 12 months of applicability determination with zero flow indicating no operation.

—If the treatment system or treatment plant was not in operation for one or more

calendar months in 1995 due to construction and/or maintenance, the applicability determination will be based on those months in 1995 during which the treatment system or treatment plant was in operation, plus the calendar months from 1994 that correspond to those months of 1995 during which the treatment system or treatment plant was inoperable. The total time period shall be 12 months.

—Treatment systems or treatment plants whose total operational lifetime is fewer than 12 calendar months as of December 1995 are not required to comply with subpart M requirements.

—PWSs that purchase all their water from one or more other PWSs and do not further treat any of their water are not required to comply with subpart M requirements.

Applicability determination. To determine applicability, the PWS is required to collect certain operational data and perform specified mathematical operations. All operational data and calculated values will be expressed as either ''F'' (for flow) or ''P'' (for population), with a one or two character subscript. Table A–1 contains a more detailed explanation.

TABLE A–1.—: APPENDIX A SUBSCRIPT IDENTIFICATION PROTOCOL

General.

1. ''F'' indicates a flow value. The PWS must use million gallons per day (MGD) to express the flow throughout its calculations.
2. ''P'' indicates a population value, expressed as a number of people.

Subscripts.

1. ''P_R'' is retail population, ''F_W'' is wholesale flow, and ''F_N'' is purchased finished water that is not further treated.
2. Each ''F'' value (in Table A–2) or ''P'' value (in Table A–4) will have a two character designator.
   a. The first character in the subscript indicates the source type. Possible entries are ''S'' (for surface water or ground water under the direct influence of surface water), ''G'' (for ground water not under the direct influence of surface water), ''P'' (for finished water purchased from another PWS and further treated at the entrance to the distribution system, such as by redisinfection), and ''C'' (for combined, or the sum of all water treated by the PWS, including purchased water that is further treated at the entrance to the distribution system).
   b. The second character in the subscript indicates the specific identification of the treatment plant. This will be a number (e.g., 1, 2, 3, * * *, with # being a non-specific designator) and ''T'' (for a Total).

Data from operational records. The PWS shall determine the following information based on operational records.

—P_R=Retail population served by the PWS
=_____ (number of people)

—F_N=treated water bought from one or more other PWSs and not further treated at the entry point to the distribution system
=_____ (MGD)

—F_W= finished water sold to one or more other PWSs, regardless of whether buying PWSs further treat the finished water
=_____ (MGD)

—Flows from specific water resources to specific treatment plants. For each treatment plant operated by the PWS, the PWS must determine the flow from each water resource that provides water to the treatment plant. In the following table, the PWS must enter flow from each type of water resource into the appropriate block, using the subscript identification protocol in table A–1.

—$F_{S\#}$=surface water treated at treatment plant ''#''
=_____ (MGD) (enter into Table A–2)

—$F_{G\#}$=ground water treated at treatment plant ''#''
=_____ (MGD) (enter into Table A–2)

—$F_{P\#}$=treated water bought from one or more other PWSs and further treated at treatment plant ''#'' prior to the entry point to the distribution system
=_____ (MGD) (enter into Table A–2)

TABLE A–2.—TREATED FLOW VALUES

Water resources (by type source)	Sources of treated water (FLOW)			
	Treatment plants			
	#1	#2	#3	#4
Surface water (S)	(F_{S1})	(F_{S2})	(F_{S3})	(F_{S4})
Ground water (G)	(F_{G1})	(F_{G2})	(F_{G3})	(F_{G4})
Purchased finished water that is further treated (P)	(F_{P1})	(F_{P2})	(F_{P3})	(F_{P4})
Combined (C)	(F_{C1})	(F_{C2})	(F_{C3})	(F_{C4})

NOTE: The $F_{C\#}$ value is calculated by adding the $F_{S\#}$, $F_{G\#}$, and $F_{P\#}$ values in the column above.

—F_{CT}=finished water produced in all of the PWS's treatment plants (calculated by adding the combined flows from each treatment plant (Σ ($F_{C\#}$)).

=_____ (MGD)

Calculated values. The PWS must calculate the following values.

—Population equivalents. Divide the flow values in Table A–2 by the conversion factor K below (a PWS-specific per capita finished water usage rate) and enter in the corresponding box in Table A–3 below. For each treatment plant operated by the PWS, the PWS must determine the population served by each type of water resource that provides water to the treatment plant.

Conversion factor=K=(F_{CT}+F_N−F_W)/P_R=_____

For Table A–3, P=F/K, using F values from Table A–2 (e.g., P_{S1}=F_{S1}/K).

TABLE A–3: POPULATION SERVED VALUES

Water resources (by type source)	Population served by treated water (number of people)			
	Treatment plants			
	#1	#2	#3	#4
Surface water (S)	(P_{S1})	(P_{S2})	(P_{S3})	(P_{S4})
Ground water (G)	(P_{G1})	(P_{G2})	(P_{G3})	(P_{G4})
Purchased finished water that is further treated (P)	(P_{P1})	(P_{P2})	(P_{P3})	(P_{P4})
Combined (C)	(P_{C1})	(P_{C2})	(P_{C3})	(P_{C4})

Note: The $P_{C\#}$ value is calculated by adding the $P_{S\#}$, $P_{G\#}$, and $P_{P\#}$ values in the column above.

—P_{CT}=number of people served by finished water produced in all of the PWS's treatment plants (calculated by adding the combined populations served by each treatment plant (Σ ($P_{C\#}$)))

=_____ (people)

NOTE: A PWS that sells all its finished water and thus has no retail population must calculate the population served by the PWS by raising the PWS's average treated flow (in MGD) to the 0.95 power and multiplying the result by 7,700. As an equation, this would appear as:

PWS population served=7,700 (PWS's average treated flow in MGD)$^{0.95}$

The PWS may then calculate the population served by each of its treatment plants by multiplying the PWS population served times the average treated flow from the treatment plant divided by the average treated flow for the PWS. As an equation, this would appear as:

$$\text{Treatment plant population served} = \frac{\text{PWS population served} \times \text{treatment plant flow}}{\text{PWS average treated flow}}$$

Treatment plant categorization. A PWS must categorize its treatment plants to determine its specific compliance requirements by reviewing Table A–4 below.

TABLE A–4.—TREATMENT PLANT CATEGORIES

Treatment plant category	P_{CT}	$P_{C\#}$	$P_{S\#}$	$P_{G\#}$
A	≥100,000	≥100,000	≥1	NA.
B	≥100,000	≥100,000	Zero	NA.
C	≥100,000	$P_{C\#}$ is <100,000 and is largest $P_{C\#}$ in PWS	≥1	NA.
D	≥100,000	$P_{C\#}$ is <100,000 and is largest $P_{C\#}$ in PWS	Zero	NA.
E	≥100,000	<100,000 and is not largest $P_{C\#}$ in PWS	≥1	NA.
F	≥100,000	<100,000 and is not largest $P_{C\#}$ in PWS	Zero	NA.
G	50,000–99,999 and $P_{GT} \geq 50,000$	NA	NA	Largest $P_{G\#}$.

NA—not applicable.

(b) *Applicability*.

(1) Table 1 of this paragraph is a summary of treatment plant categorization under the provisions of appendix A to paragraph (a) of this section.

TABLE 1.—TREATMENT PLANT CATEGORIES

Treatment plant category	PWS combined population served	Treatment plant combined population served	Treatment plant surface water population served	Treatment plant ground water population served
A	≥100,000	≥100,000	≥1	NA.
B	≥100,000	≥100,000	zero	NA.
C	≥100,000	Plant serves <100,000 and is largest plant	≥1	NA.
D	≥100,000	Plant serves <100,000 and is largest plant	zero	<100,000.
E	≥100,000	Plant serves <100,000 and is not largest plant in PWS	≥1	NA.
F	≥100,000	Plant serves <100,000 and is not largest plant in PWS	zero	<100,000.
G	50,000–99,999 and ≥ 50,000 served by ground water.	NA	NA	Largest ground water plant.

NA–not applicable.

(2) Table 2 of this paragraph specifies applicability for requirements contained in §§ 141.142, 141.143, and 141.144 of this part, based on treatment plant categorization determined under the provisions of appendix A to paragraph (a) of this section.

TABLE 2—SUBPART M APPLICABILITY

Subpart M Requirements	Categories of treatment plants[1]						
	A	B	C	D	E	F	G
§ 141.142.—DBP and Related Monitoring							
Table 1a and 1b	X	X	X	X	X	X	.....
Table 2[2]	X	X	X	X	X	X	.....
Table 3[2]	X	X	X	X	X	X	.....
Table 4a and 4b[2]	X	X	X	X	X	X	.....
Table 5a and 5b[2]	X	X	X	X	X	X	.....
Table 6	X	X	X	X	X	X	.....
§ 141.143—Microbiological Monitoring							
Treatment plant influent monitoring	X	.....	X	.....	X	.....	.....
Finished water monitoring[3]	X	.....	X	.....	X	.....	.....
§ 141.144—Applicability Monitoring and Treatment Studies							
Treatment study applicability monitoring	X	X	X	X	.....	.....	X
Pilot-scale treatment studies[4]	X	X	.....	.....	.....	.....	.....
Bench- or pilot-scale treatment studies[4]	X	X	X	X	.....	.....	X

[1] As determined by Appendix A to paragraph (a) of this section.
[2] Table 2 required only for treatment plants using chloramines. Table 3 required only for treatment plants using hypochlorite solution. Table 4a and 4b required only for treatment plants using ozone. Table 5a and 5b required only for treatment plants using chlorine dio xide.
[3] Only required for a PWS that, during any of the first twelve months of monitoring at the treatment plant influent, detects 10 or more *Giardia* cysts, or 10 or more *Cryptosporidium* oocysts, or one or more total culturable viruses in one liter of water; or calculates a numerical value of the *Giardia* or *Cryptosporidium* concentration equal to or greater than 1000 per 100 liters or virus concentration equal to or greater than 100 per 100 liters; or detects no pathogens in the sample and calculates a numerical value of the detection limit for *Giardia* or *Cryptosporidium* concentration equal to or greater than 1000 per 100 liters or virus concentration equal to or greater than 100 per 100 liters.
[4] Pilot-scale treatment studies are required for treatment plants that serve a population of 500,000 or greater. Bench- or pilot-scale treatment studies are required for treatment plants that serve a population of fewer than 500,000.

(c) *Disinfection Byproduct and Related Monitoring.* A PWS must comply with the monitoring requirements in § 141.142 of this subpart for treatment plants in treatment plant categories A, B, C, D, and E listed in table 1 in paragraph (b)(1) of this section. The PWS shall monitor monthly for 18 consecutive months at each treatment plant, even if a treatment plant was not used for one or more calendar months. When the treatment plant is not operating, the PWS shall file the report required under § 141.142(c) of this subpart to indicate zero flow, and need only conduct treatment plant influent monitoring under the provisions of § 141.142 of this subpart. A PWS must comply with the monitoring requirements in § 141.142 of this subpart for treatment plants in treatment plant categories F listed in table 1 in paragraph (b)(1) of this section monthly for 18 consecutive months at each treatment plant, except if a treatment plant was not used for one or more calendar months. When the treatment plant is not operating, the PWS shall file the report required under § 141.142(c) of this subpart to indicate zero flow, and is not required to conduct treatment plant influent monitoring under the provisions of § 141.142 of this subpart.

(d) *Microbiological Monitoring.* A PWS must comply with the monitoring requirements in § 141.143 of this subpart for treatment plants in treatment plant categories A, C, and E listed in table 1 in paragraph (b)(1) of this section and table 3 of this paragraph. The

PWS shall conduct 18 consecutive months of microbiological monitoring at each treatment plant, even if it is not operated each calendar month.

TABLE 3.—MICROBIOLOGICAL MONITORING REQUIREMENTS FOR SUBPART M

Microbial sample	Treatment plant category	
	A, C and E	
	Treatment plant influent	Finished water [1]
Total culturable viruses	1/month [2]	1/month.
Total coliforms	1/month	1/month.
Fecal coliforms or *E. coli*	1/month	1/month.
Giardia	1/month	1/month.[3]
Cryptosporidium	1/month	1/month.[3]

[1] Only required for a PWS that, during any of the first twelve months of monitoring at the treatment plant influent, detects 10 or more *Giardia* cysts, or 10 or more *Cryptosporidium* oocysts, or one or more total culturable viruses in one liter of water; or calculates a numerical value of the *Giardia* or *Cryptosporidium* concentration equal to or greater than 1000 per 100 liters or virus concentration equal to or greater than 100 per 100 liters; or detects no pathogens in the sample and calculates a numerical value of the detection limit for *Giardia* or *Cryptosporidium* concentration equal to or greater than 1000 per 100 liters or virus concentration equal to or greater than 100 per 100 liters. The PWS shall collect one sample of finished water during each month that the treatment plant is operated at each such treatment plant beginning in the first calendar month after the PWS learns of such a result. A PWS shall continue finished water monitoring monthly until 18 months of treatment plant influent monitoring has been completed.

[2] A PWS may avoid virus monitoring if the PWS has monitored total coliforms, fecal coliforms, or *E. coli* in the source water for at least five days/week for any period of six consecutive months beginning after January 1, 1994, and 90% of all samples taken in that six-month period contained no greater than 100 total coliforms/100 ml, or 20 fecal coliforms/100 ml, or 20 *E. coli*/100 ml.

[3] A PWS may avoid the requirement for finished water monitoring of *Giardia* and *Cryptosporidium* if the PWS notifies EPA that it will comply with the alternative monitoring requirements in § 141.143(a)(2)(iii). The PWS must still conduct finished water monitoring for all other microorganisms, except that *Giardia* and *Cryptosporidium* monitoring in the finished water is not required.

(e) *Disinfection Byproduct Precursor Removal Studies (Treatment Studies).*

(1) A PWS shall comply with treatment study applicability monitoring in paragraph (e)(2) of this section at each treatment plant in treatment plant categories A, B, C, D, and G listed in table 1 in paragraph (b)(1) of this section. A PWS shall comply with the treatment study requirements in § 141.144 of this subpart at each such treatment plant, except for those treatment plants:

(i) Meeting the source water quality, disinfection practice, or disinfection byproduct precursor removal practice criteria in paragraph (e)(3) of this section, for which no treatment study is required; or

(ii) Meeting the common water resource criteria in paragraph (e)(4) of this section, for which several PWSs may conduct treatment studies jointly, in lieu of separately; or

(iii) Meeting the common water resource criteria in paragraph (e)(5) of this section, for which a PWS may contribute funds towards research, in lieu of conducting a treatment study; or

(iv) At which a previous treatment study that meets the criteria in paragraph (e)(6) of this section has already been conducted, for which a PWS may use the results of this previous treatment study, in lieu of conducting another treatment study; or

(v) Operated by the PWS that use the same water resource, as classified by the procedure in paragraph (e)(4) of this section. The PWS is not required to conduct more than one treatment study for those treatment plants. If both pilot-scale and bench-scale treatment studies would otherwise be required for treatment plants on the same water resource, the PWS shall conduct a pilot-scale study. A PWS with multiple water resources shall conduct treatment studies for each treatment plant that uses different water resources.

(2) Treatment study applicability monitoring.

(i) PWSs shall monitor total organic carbon (TOC) monthly for 12 months. Treatment plants using surface water shall monitor treatment plant influent. Treatment plants using ground water shall monitor finished water.

(ii) Treatment study applicability monitoring for THM4 and HAA5 is only required by a PWS that intends to qualify for avoiding a treatment study under the provisions of paragraph (e)(3)(i) of this section.

(iii) Total organic halides formed under the uniform formation conditions (UFCTOX) monitoring is only required by a PWS that intends to qualify for a joint treatment study under the provisions of paragraph (e)(4)(i)(A)(*2*) of this section or for the alternative to conducting a treatment study under the provisions of paragraph (e)(5) of this section.

(3) *Criteria under which no treatment study is required.* A PWS identified in paragraph (e)(1) of this section is not required to conduct a treatment study at any treatment plant that satisfies any criteria in paragraphs (e)(3) (i) through (iv) of this section, provided that the PWS has also complied with the requirements in paragraph (e)(7)(i) of this section and EPA has approved the PWS's request to avoid the treatment study.

(i) Treatment plants that use chlorine as both the primary and residual disinfectant and have, as an annual average of four quarterly averages, levels of less than 40 µg/l for THM4 and less than 30 µg/l for HAA5. Quarterly averages are the arithmetic average of the four distribution system samples collected under the requirements of § 141.142(a)(1) of this subpart.

(ii) Treatment plants using surface water that do not exceed a TOC annual average of 4.0 mg/l in the treatment plant influent, measured in accordance with §§ 141.141(f)(4) and 141.144(a) of this subpart and calculated by averaging the initial 12 monthly TOC samples.

(iii) Treatment plants using only ground water not under the direct influence of surface water that do not exceed a TOC annual average of 2.0 mg/l in the finished water, measured in accordance with §§ 141.141(f)(4) and 141.144(a) of this subpart and calculated by averaging the initial 12 monthly TOC samples.

(iv) Treatment plants that already use full scale membrane or GAC technology. For a treatment plant that already uses full-scale GAC or membrane technology capable of achieving precursor removal, a PWS shall conduct monitoring and submit full-scale plant data required for disinfection byproduct and related monitoring by § 141.142(a) of this subpart, ensuring that the GAC or membrane processes are included in the process train being monitored. For a treatment plant to be considered to have membrane technology to achieve precursor removal, the PWS shall have used nanofiltration or reverse osmosis membranes. GAC capable of removing precursors is defined as GAC with an empty bed contact time (EBCT) of 15 minutes or greater, with a time between carbon reactivation or replacement of no more than nine months. PWSs that operate treatment plants that use GAC with either an EBCT of less than 15 minutes or a replacement or reactivation frequency for GAC longer than nine months may submit a request to avoid treatment studies under the provisions of paragraph (e)(7)(i) of this section by including data demonstrating effective DBP precursor removal.

(4) *Criteria under which joint treatment studies are allowed.* (i) PWSs that use common water resources and have similar treatment trains may conduct joint treatment studies. A common water resource for all types of surface water resources requires the mean treatment plant influent TOC or UFCTOX of each of the cooperating treatment plants to be within 10% of the average of the mean treatment plant influent TOCs or UFCTOX of all the cooperating treatment plants. A common water resource for all types of ground water resources requires the mean treatment plant finished water TOC or UFCTOX of each of the cooperating treatment plants to be within 10% of the average of the mean treatment plant finished water TOCs or UFCTOX of all the cooperating treatment plants. The mean is calculated from the monthly TOC or UFCTOX monitoring data for the initial twelve months of monitoring under § 141.144(a) of this subpart. Similar treatment trains means that, for example, softening plants may not conduct joint studies with conventional treatment plants. In addition, the applicable requirements in paragraphs (e)(4)(i) (A) through (C) of this section shall be met for the water resource to be considered a common water resource. If otherwise eligible, a PWS may choose to either perform a joint treatment study with other eligible systems or contribute

funds to a cooperative research program, as described in paragraph (e)(5) of this section, as an alternative to conducting a treatment study.

(A) *River sources.* Treatment plants with river intakes are considered to have a common water resource if the PWS meets either criteria in paragraphs (e)(4)(i)(A) (*1*) or (*2*) of this section.

(*1*) The intakes are no more than 20 river miles apart and TOC at each treatment plant influent is within 10% of the mean TOC of all the treatment plant influents.

(*2*) The intakes are at least 20, but no more than 200, river miles apart and the PWS demonstrates that the mean water resource UFCTOX is within 10% of the mean UFCTOX of all the treatment plant influents, based on UFCTOX analytical results of the same 12 consecutive months for all cooperating treatment plants.

(B) *Lake/reservoir.* Treatment plants with lake or reservoir intakes are considered to have a common water resource if the same lake or reservoir serves all the cooperating treatment plants and TOC at each treatment plant influent is within 10% of the mean TOC of all the treatment plant influents.

(C) *Ground water not under the direct influence of surface water.* Treatment plants with intakes from a single aquifer are considered to have a common water resource if treatment plant finished water TOC at each treatment plant is within 10% of the mean finished water TOC of all the treatment plants.

(ii) PWSs that meet the requirements of paragraph (e)(4)(i) of this section shall conduct at least the number and type of joint studies noted in the following tables. Joint studies shall only be conducted among treatment plants in the same size category, i.e. a population served of either ≥500,000 or of <500,000. The maximum number of treatment plants with a population served ≥500,000 persons allowed to join together to conduct a study is three. The maximum number of treatment plants with a population served <500,000 persons allowed to join together to conduct a study is six.

JOINT STUDIES REQUIREMENT FOR TREATMENT PLANTS WITH A POPULATION SERVED OF <500,000

Number of plants	Minimum studies to be conducted
2	1 pilot (GAC or membrane).
3	1 pilot and 1 bench (GAC or membrane).
4	2 pilots (GAC and/or membrane).
5	2 pilots (GAC and/or membrane), 1 bench (GAC or membrane).
6	2 pilots and 2 bench (GAC and/or membrane).

JOINT STUDIES REQUIREMENT FOR TREATMENT PLANTS WITH A POPULATION SERVED OF ≥500,000

Number of plants	Minimum studies to be conducted
2	1 pilot (GAC or membrane), 2 bench (GAC and/or membrane).
3	2 pilots (GAC and/or membrane).

(5) *Criteria under which an alternative to conducting a treatment study is allowed.* In lieu of conducting the required treatment study, a PWS may apply to EPA to contribute funds to a cooperative research effort. The PWS shall submit an application to EPA Technical Support Division, ICR Precursor Removal Studies Coordinator, 26 W. Martin Luther King Drive, Cincinnati, OH 45268. The application shall show that the treatment plant for which the waiver of the treatment study is sought uses a common water resource, as described in paragraph (e)(4) of this section, that is being studied by another PWS or cooperative of PWSs operating treatment plants in the same size category. A PWS operating treatment plants serving a population of fewer than 500,000 may also contribute to this fund if there is a common water resource (as defined in paragraph (e)(4) of this section) treatment plant serving 500,000 or more conducting a treatment study. If EPA approves the application, the PWS shall contribute funds in the amount specified in paragraph (e)(5)(i) of this section to the Disinfection Byproducts/Microbial Research Fund, to be administered by the American Water Works Association Research Foundation (AWWARF) under the direction of an independent research council, for use in a dedicated cooperative research

program related to disinfectants, disinfection byproducts, and enhanced surface water treatment.

(i) The PWS shall contribute $300,000 for a treatment plant with a population served of 500,000 or more. The PWS shall contribute $100,000 for a treatment plant with a population served of fewer than 500,000.

(ii) The PWS shall send the contribution to the address specified in EPA's approval letter not later than 90 days after EPA approves the PWS application for waiver of the treatment study.

(6) *Criteria under which a previous treatment study is acceptable (grandfathered studies).* A PWS that has conducted studies of precursor removal that meet all the criteria in paragraphs (e)(6) (i) and (ii) of this section may use the results of that study in lieu of conducting another treatment study.

(i) The PWS used analytical methods specified in table 7 of § 141.142(b)(1) of this subpart and used the analytical and quality control procedures described in ''DBP/ICR Analytical Methods Manual'', EPA 814–B–96–002.

(ii) The PWS followed a protocol similar to that specified and supplies the data specified in ''ICR Bench- and Pilot-scale Treatment Study Manual'' (EPA 814–B–96–003, April 1996).

(7) *Process for a PWS to obtain EPA approval of criteria applicability.* A PWS wanting to avoid the requirements for a treatment study under the provisions of paragraphs (e) (3) through (6) of this section shall submit the applicable information in paragraphs (e)(7) (i) through (iv) of this section and in ''ICR Bench- and Pilot-scale Treatment Study Manual'' (EPA 814–B–96–003, April 1996) and all monitoring data required under §§ 141.142(a) and 141.143(a) of this subpart to EPA, Technical Support Division, ICR Precursor Removal Studies Coordinator, 26 W. Martin Luther King Drive, Cincinnati, OH 45268.

(i) *Approval of request to avoid treatment studies.* A PWS that believes it qualifies to avoid the requirements for a treatment study under the provisions of paragraph (e)(3) (i) through (iii) of this section shall submit the information showing the applicable criterion for not conducting the study has been met not later than November 14, 1997. A PWS wanting to avoid the requirements for a treatment study under the provisions of paragraph (e)(3)(iv) of this section shall submit the supporting information, including any pilot- or full-scale data showing effective precursor removal, not later than November 14, 1997. A PWS that applies to avoid a treatment study under the provisions of paragraph (e) (4) through (6) of this section and subsequently qualifies to avoid a treatment study under the provisions of paragraph (e)(3) (i) through (iii) of this section may elect to avoid a treatment study under the provisions of paragraph (e)(3) (i) through (iii) of this section. If the PWS elects to avoid a treatment study under the provisions of paragraph (e)(3) (i) through (iii) of this section, the PWS shall notify all PWSs that were associated with the application to avoid a treatment study under the provisions of paragraph (e) (4) through (6) of this section.

(ii) *Approval of request to conduct joint studies.* A PWS that believes it qualifies to avoid the requirements for a treatment study under the joint study provisions of paragraph (e)(4) of this section shall submit a letter of intent to EPA with the information in paragraphs (e)(7)(ii) (A) through (F) of this section for all treatment plants to be included in the joint study not later than May 14, 1997. The letter shall be signed by all PWSs planning to participate in the joint study. All PWSs shall submit a combined application for joint studies approval to EPA (including 12 months of treatment plant influent TOC or finished water TOC results or UFCTOX results, as appropriate, for each treatment plant to be included in the joint study) not later than November 14, 1997.

(A) Data to support their common water resource designation.

(B) Information to demonstrate that treatment plants have similar treatment trains.

(C) Information that treatment plants are in the same size category.

(D) The treatment plant influent TOC or finished water TOC results, or UFCTOX results, as appropriate, from the first six months of monitoring.

(E) What studies will be conducted (i.e., combination of bench/pilot and GAC/membrane).

(F) Any additional supporting data.

(iii) *Approval of request for alternative to treatment studies.* A PWS that believes it qualifies to avoid the requirements for a treatment study under the provisions for an alternative in paragraph (e)(5) of this section shall submit a letter of intent expressing its intention to contribute funds to the cooperative research effort not later than May 14, 1997. The letter shall identify the other treatment plants using the same water resource which will be conducting studies. Each PWS shall submit an application for approval of alternative to treatment studies to EPA (including 12 months of treatment plant influent TOC or finished water TOC results or UFCTOX results, as appropriate) not later than November 14, 1997. EPA shall notify the PWS whether a treatment study is required (because there is no other appropriately sized treatment plant using the same water resource conducting a treatment study) or if the PWS can avoid the study by contributing funds to the cooperative research effort specified in paragraph (e)(5) of this section.

(iv) *Approval of request to use grandfathered studies.* A PWS that believes it qualifies to avoid the requirements for a treatment study under the grandfathered study provisions of paragraph (e)(6) of this section shall submit the following information not later than February 14, 1997: a description of the study, the equipment used, the experimental protocol, the analytical methods, the quality assurance plan, and any reports resulting from the study. EPA shall review the information and inform the PWS whether or not the prior study meets the ICR requirements. Not later than November 14, 1997, the PWS must submit study data in the format specified in ''ICR Manual for Bench- and Pilot-scale Treatment Studies'', EPA 814–B–96–003, April 1996. An approved grandfathered study can be justification for common water resource PWSs contributing to the cooperative research effort under the provisions of paragraph (e)(5) of this section, but may not be used as joint treatment studies unless it incorporates the requirements listed in §141.141(e)(4) of this section and the PWS submits written concurrence of the PWS which conducted the study.

(f) *Effective dates.* (1) A PWS shall respond to the Notice of Applicability sent by EPA within 35 calendar days of receipt of that notice. The PWS's response to the Notice shall indicate what requirements in subpart M apply to each treatment plant operated by the PWS. If a PWS meets the applicability criteria in paragraph (b) of this section and has not received a Notice of Applicability from EPA by June 28, 1996, that PWS must request a Notice of Applicability from EPA by contacting the ICR Utilities Coordinator, TSD, USEPA, 26 West Martin Luther King Drive, Cincinnati, OH 45268, not later than July 15, 1996.

(2) A PWS required to monitor under both paragraphs (c) and (d) of this section shall begin monitoring to comply with the provisions of §141.142 (Disinfection Byproduct and Related Monitoring) and §141.143 (Microbiological Monitoring) of this subpart in the same month. The PWS must submit the sampling plans required by §§141.142(c)(2)(ii) and 141.143(c)(3)(ii) of this subpart at the same time.

(3) *Disinfection Byproduct and Related Monitoring.* A PWS operating a treatment plant required to comply with §141.142 of this subpart shall begin monitoring in the calendar month following approval of the DBP and related monitoring sampling plan submitted under the provisions of §141.142(c)(2)(ii) of this subpart. Once a PWS has begun monitoring, it shall continue to monitor for 18 consecutive months.

(4) *Microbiological Monitoring.* A PWS operating a treatment plant identified in paragraph (d) of this section shall begin monitoring under the provisions of §141.143 of this subpart in the calendar month following approval of the sampling plan submitted under the provisions of §141.143(c)(3)(ii) of this subpart. Once a PWS has begun monitoring, it shall continue to monitor for 18 consecutive months.

(5) DBP precursor removal studies. (i) *TOC, UFCTOX, THM4, and HAA5 monitoring.* A PWS required to comply with §141.144 of this subpart shall begin TOC, UFCTOX, THM4, and HAA5 monitoring specified in paragraph (e)(2) of this section not later than August 14, 1996 and continue this monitoring for 12 consecutive months for TOC and

UFCTOX and four consecutive quarters for THM4 and HAA5.

(ii) A PWS required to conduct a disinfection byproduct precursor removal study (treatment study) under the provisions of paragraph (e)(1) of this section shall begin conducting such treatment studies not later than April 14, 1998 and submit the report(s) of the completed study to EPA not later than July 14, 1999.

§ 141.142 Disinfection byproduct and related monitoring.

(a) *Monitoring requirements.* Samples taken under the provisions of this section shall be taken according to the procedures described in the ''ICR Sampling Manual,'' EPA 814–B–96–001, April 1996. If a treatment plant configuration results in two required sampling points from any table in this section when in fact it is a single location, duplicate analyses are not required for the same location and time. A PWS that uses purchased finished water shall determine whether any monitoring of treatment plant influent is required under paragraphs (a) (2) through (5) of this section because of certain treatment (e.g., use of hypochlorite or chlorine dioxide) of the water provided by the selling PWS.

(1) A PWS shall obtain a complete set of samples at the frequency and location noted in tables 1a and 1b of this section for treatment plants required to test under § 141.141(b) of this subpart. Samples shall be taken according to the sampling plan approved under the provisions of paragraph (c)(2)(ii) of this section.

(i) Samples of finished water shall be collected at a point after which all treatment processes for a particular treatment plant are complete (including the clearwell and final point of chlorination) and before the distribution system begins. A PWS that purchases finished water shall collect a sample before additional disinfectant is added to the purchased finished water. A PWS shall collect a sample of purchased finished water only if the PWS redisinfects the purchased finished water. A sample of finished water is a sample representing the final product water from a particular treatment plant.

(ii) A sample of treatment plant influent for a PWS that treats untreated water shall be taken at a location at the upstream end of a treatment plant where waters from all intakes are blended prior to any treatment or chemical addition. For treatment plants that have multiple intakes and add chemicals at the intake, the sample of treatment plant influent shall be a flow proportional composite of intake samples collected before chemical addition and before pretreatment. If the intakes are expected to have the same source water quality, one representative intake sample may be taken. If a disinfectant is added at or before the intake (e.g., for zebra mussel control), the sample shall be taken in the vicinity of the intake so that the sample is not contaminated by the disinfectant. A sample of treatment plant influent for a PWS that treats purchased finished water is taken at a location just before the purchased finished water is treated. An intake sample is collected after the intake but before blending with waters from other intakes and before addition of chemicals or any treatment.

TABLE 1A.—MONTHLY MONITORING REQUIREMENTS FOR TREATMENT PLANTS

Sampling point	Monthly analyses [1]
Treatment plant influent for non-finished water.	pH, Alkalinity, Turbidity, Temperature, Calcium and Total Hardness, TOC, UV_{254}, Bromide, Ammonia.
Treatment plant influent for purchased finished water [2].	pH, Alkalinity, Turbidity, Temperature, Calcium and Total Hardness, TOC, UV_{254}, Disinfectant residual [3].
Before first point of oxidant addition	Chlorine demand test.
Washwater return between washwater treatment plant and point of addition to process train [4].	pH, Alkalinity, Turbidity, Temperature, Calcium and Total hardness, TOC, UV_{254}, Bromide, Ammonia, Disinfectant residual [3] if disinfectant is used.
Additional water sources added to process train after treatment plant influent. The sample point is before additional water is blended with the process train.	pH, Alkalinity, Turbidity, Temperature, Calcium and Total hardness, TOC, UV_{254}, Bromide, Ammonia, Disinfectant residual [3] if disinfectant is used.

TABLE 1A.—MONTHLY MONITORING REQUIREMENTS FOR TREATMENT PLANTS—Continued

Sampling point	Monthly analyses [1]
Before Filtration	pH, Alkalinity, Turbidity, Temperature, Calcium and Total Hardness, TOC, and UV_{254}.
After Filtration	pH, Alkalinity, Turbidity, Temperature, Calcium and Total Hardness, TOC, and UV_{254}.
Before each Point of Disinfection [5]	pH, Alkalinity, Turbidity, Temperature, Calcium and Total Hardness, TOC, and UV_{254}.
After every unit process that is downstream from the addition of chlorine or chloramines.	Disinfectant Residual [3].
Finished water sample point (Plant effluent).	pH, Alkalinity, Turbidity, Temperature, Calcium and Total Hardness, TOC, UV_{254}, Disinfectant Residual [3].
Entry point to distribution system [6]	pH, Alkalinity, Turbidity, Temperature, Calcium and Total Hardness, TOC, UV_{254}, Disinfectant Residual [3].

[1] TOC: total organic carbon. UV_{254}: absorbance of ultraviolet light at 254 nanometers.

[2] Samples of purchased finished water shall be taken prior to addition of any more disinfectant.

[3] Free chlorine residual and total chlorine residual shall be measured in treatment systems using free chlorine. Total chlorine residual, but not free chlorine residual, shall be measured in treatment systems using chloramines as the residual disinfectant.

[4] Washwater return shall be sampled prior to blending with the process train.

[5] For utilities using ozone or chlorine dioxide, Tables 4 and 5, respectively, of this section, show additional monitoring requirements at this sampling point. Addition of ammonia for the purpose of converting free chlorine to chloramines is considered a point of disinfectant addition. PWSs that disinfect just before filtration may use the ''before filtration'' sampling point analytical results to meet the monitoring requirement for this point.

[6] Entry point to distribution system only required for treatment plants that blend finished water with finished water from other treatment plant(s) prior to entry point of distribution system. For most treatment plants, the finished water sample point and the entry point to the distribution system are the same.

TABLE 1B.—QUARTERLY MONITORING REQUIREMENTS FOR TREATMENT PLANTS

Sampling point	Quarterly analyses [1]
Treatment plant influent for non-finished water.	TOX.
Treatment plant influent for purchased finished water.	THM4, HAA6 [7], HAN, CP, HK, CH, TOX.
Washwater Return between washwater treatment plant and point of addition to process train.	TOX.
After filtration if disinfectant is applied at any point in the treatment plant prior to filtration.	THM4, HAA6 [7], HAN, CP, HK, CH, TOX.
Finished water sample point (Plant Effluent).	THM4, HAA6 [7], HAN, CP, HK, CH, TOX.
Entry point to distribution system [2]	THM4, HAA6 [7], HAN, CP, HK, CH, TOX.
SDS [3]	THM4, HAA6 [7], HAN, CP, HK, CH, TOX, pH, Alkalinity, Turbidity, Temperature, Calcium and Total Hardness, Disinfectant Residual [5].
Four monitoring points in distribution system [4,6].	THM4, HAA6 [7], HAN, CP, HK, CH, TOX, pH, Alkalinity, Turbidity, Temperature, Calcium and Total Hardness, Disinfectant Residual [5].

[1] TOC: total organic carbon. THM4: trihalomethane (four). HAA6: haloacetic acids (six). HAN: Haloacetonitriles. CP: chloropicrin. HK: haloketones. CH: chloral hydrate. TOX: total organic halide. For THM4, HAA6, HAN, and HK, analytical results for individual analytes shall be reported.

[2] Entry point to distribution system only required for treatment plants that blend finished water with finished water from other treatment plant(s) prior to entry point of distribution system. For most treatment plants, the finished water sample point and the entry point to the distribution system are the same.

[3] Simulated Distribution System (SDS) sample shall be collected at the finished water sampling point (or entry point to distribution system if finished water from two or more plants are blended prior to entering the distribution system) and analyzed using the method specified in § 141.142. PWSs using purchased finished water are not required to take an SDS sample at treatment plants that use only purchased finished water.

[4] For each treatment plant, one distribution system equivalent sample location (known as DSE) shall be chosen to correspond to the SDS sample, one sample location shall be chosen to be representative of maximum residence time for the treatment plant, and the remaining two sample locations shall be representative of the average residence time in the distribution system for the treatment plant. PWSs using purchased finished water shall take three samples representing the average residence time in the distribution system for the treatment plant and one representing the maximum residence time for the treatment plant (no DSE sample required).

[5] Free chlorine residual and total chlorine residual shall be measured in treatment systems using free chlorine. Total chlorine residual, but not free chlorine residual, shall be measured in treatment systems using chloramines as the residual disinfectant.

[6] A PWS may use TTHM compliance monitoring locations and analytical results under § 141.30 of this part to the extent that such locations and analytical results are consistent with the requirements of this section.

[7] PWSs are encouraged to also analyze for the additional haloacetic acids bromodichloro-, chlorodibromo-, and tribromo-acetic acid, and report the results as part of the reports specified in paragraph (c)(1) of this section.

(2) *Additional requirements for PWSs using chloramines.* For each treatment plant that uses chloramines for treatment or disinfection residual maintenance, a PWS shall also conduct the additional sampling identified in table 2 of this section. A PWS shall send samples of cyanogen chloride taken under the provisions of this paragraph for analysis to EPA, following the procedures contained in the ''ICR Sampling Manual,'' EPA 814–B–96–001, April 1996.

TABLE 2.—ADDITIONAL QUARTERLY MONITORING FOR TREATMENT PLANTS USING CHLORAMINES

Sampling point	Quarterly analyses
Treatment plant influent for purchased finished water [1]	Cyanogen Chloride[2].
Finished water sample point (plant effluent)	Cyanogen Chloride[2].
Distribution system sample point representing a maximum residence time in distribution system relative to the treatment plant.	Cyanogen Chloride[2].

[1] Applicable only when wholesale water provider is using chloramines.
[2] EPA shall provide all analytical results to the PWS. The PWS shall report all results in its monthly report.

(3) *Additional requirements for PWSs using hypochlorite solutions.* For each treatment plant that uses hypochlorite solutions for treatment or disinfection residual maintenance, a PWS shall also conduct the additional sampling identified in table 3 of this section.

TABLE 3.—ADDITIONAL QUARTERLY MONITORING FOR TREATMENT PLANTS USING HYPOCHLORITE SOLUTIONS

Sampling point	Quarterly analyses
Treatment plant influent for non-finished water	Chlorate.
Treatment plant influent for purchased finished water [1]	Chlorate.
Hypochlorite Stock Solution	pH, Temperature, Free Residual Chlorine, Chlorate.
Finished Water Sample Point (Plant Effluent)	Chlorate.

[1] Applicable only when wholesale water provider is using hypochlorite solutions.

(4) *Additional requirements for PWSs using ozone.* For each treatment plant that uses ozone for treatment, a PWS shall also conduct the additional sampling identified in tables 4a and 4b of this section. A PWS shall collect samples for bromate taken under the provisions of this paragraph in duplicate, with the PWS analyzing one aliquot and submitting the other aliquot for analysis to EPA, following the procedures contained in the ''ICR Sampling Manual,'' EPA 814–B–96–001, April 1996. A PWS shall submit samples for aldehydes taken under the provisions of this paragraph for analysis to EPA, following the procedures contained in the ''ICR Sampling Manual,'' EPA 814–B–96–001, April 1996.

TABLE 4a.—ADDITIONAL MONTHLY MONITORING FOR TREATMENT PLANTS USING OZONE

Sampling point	Monthly analyses
Ozone Contactor Influent	Bromide, bromate [2,3], and ammonia.
Each Ozone Contact Chamber Effluent [1]	Ozone residual.
Ozone Contactor Effluent	Bromate[2].
Finished Water Sample Point (Plant Effluent)	Bromate[2].

[1] Each ozone contactor can be subdivided into its contact chambers. Measure ozone residual in effluent of all contact chambers until <0.05 mg/l is measured in two consecutive chambers.
[2] EPA shall provide all analytical results to the PWS. The PWS shall report all results in its monthly report.
[3] PWSs are not required to analyze a bromate sample at this location. However, PWSs are still required to submit a sample to EPA for analysis.

Table 4B.—ADDITIONAL QUARTERLY MONITORING FOR TREATMENT PLANTS USING OZONE

Sampling point	Quarterly analyses
Ozone Contactor Influent	Aldehydes [1] and AOC/BDOC [2].
Ozone Contactor Effluent	Aldehydes [1] and AOC/BDOC [2].
Finished Water Sample Point (Plant Effluent)	Aldehydes [1] and AOC/BDOC [2].

[1] EPA shall measure the following aldehydes: formaldehyde, acetaldehyde, propanal, butanal, pentanal, glyoxal, and methyl glyoxal. EPA may analyze for other aldehydes. EPA shall provide all analytical results to the PWS. The PWS shall report all results in its monthly report.

[2] Analysis and submission of data for both assimilable organic carbon (AOC) and biodegradable organic carbon (BDOC) are optional. Analytical methods for AOC and BDOC are listed in ''DBP/ICR Analytical Methods Manual,'' EPA 814–B–96–002, April 1996.

(5) *Additional sampling requirements for PWSs using chlorine dioxide.* For each treatment plant that uses chlorine dioxide for treatment or disinfection residual maintenance, a PWS shall also conduct the additional sampling identified in tables 5a and 5b of this section. A PWS shall collect samples for bromate taken under the provisions of this paragraph in duplicate, with the PWS analyzing one aliquot and submitting the other aliquot for analysis to EPA, following the procedures contained in the ''ICR Sampling Manual,'' EPA 814–B–96–001, April 1996. A PWS shall submit samples for aldehydes taken under the provisions of this paragraph for analysis to EPA, following the procedures contained in the ''ICR Sampling Manual,'' EPA 814–B–96–001, April 1996.

TABLE 5A.—ADDITIONAL MONTHLY MONITORING FOR TREATMENT PLANTS USING CHLORINE DIOXIDE

Sampling point	Monthly analyses
Treatment plant influent for purchased finished water [1]	Chlorine Dioxide Residual, Chlorite, Chlorate.
Before first chlorine dioxide application	Chlorate, bromate [2,3].
Before application of ferrous salts, sulfur reducing agents, or GAC.	Chlorine Dioxide Residual, Chlorite, Chlorate, pH.
Finished water sample point (plant effluent)	Chlorine Dioxide Residual, Chlorite, Chlorate, Bromate [2].
Three distribution system sampling points (1 near first customer, 1 in middle of distribution system, and 1 representative of maximum residence time in the distribution system).	Chlorine Dioxide Residual, Chlorite, Chlorate, pH, and Temperature.

[1] Applicable only when wholesale water provider is using chlorine dioxide.

[2] EPA shall provide all analytical results to the PWS. The PWS shall report all results in its monthly report.

[3] PWSs are not required to analyze a bromate sample at this location. However, PWSs are still required to submit a sample to EPA for analysis.

TABLE 5b.—ADDITIONAL QUARTERLY MONITORING FOR TREATMENT PLANTS USING CHLORINE DIOXIDE

Sampling point	Quarterly analyses
Before First Chlorine Dioxide Application	Aldehydes [1] and AOC/BDOC [2].
Before First Point of Downstream Chlorine/Chloramine Application After Chlorine Dioxide Addition.	Aldehydes [1] and AOC/BDOC [2].
Finished Water Sample Point (Plant Effluent)	Aldehydes [1] and AOC/BDOC [2].

[1] EPA shall measure the following aldehydes: formaldehyde, acetaldehyde, propanal, butanal, pentanal, glyoxal, and methyl glyoxal. EPA may analyze for other aldehydes. EPA shall provide all analytical results to the PWS. The PWS shall report all results in its monthly report.

[2] Analysis and submission of data for both assimilable organic carbon (AOC) and biodegradable organic carbon (BDOC) are optional. Analytical methods for AOC and BDOC are listed in ''DBP/ICR Analytical Methods Manual,'' EPA 814–B–96–002, April 1996.

(6) *Additional requirements.* A PWS shall also report the applicable information in tables 6a through 6e of this section. A PWS is required to provide the information in paragraphs (a)(6) (i) through (iii) of this section for each unit process listed in table 6c. The PWS may provide the information in paragraphs (a)(6) (iv) and (v) of this section for each unit process listed in table 6c. T_{10} and T_{50} tracer studies shall be conducted as specified in ''Guidance Manual for Compliance with the Filtration and Disinfection Requirements for Public Water Systems using Surface Water Sources'', appendix C.

(i) Unit process flow (MGD) at time of sampling.

(ii) T_{10} (minutes). A PWS shall determine T_{10} based on a one-time tracer study in the clearwell of all treatment plants required to conduct microbiological monitoring under the provisions of § 141.141(d) of this subpart. The PWS may use results of a tracer study conducted to meet the requirements of subpart H (Filtration and Disinfection) of this part to meet this requirement. For subsequent T_{10} determinations, the PWS shall use a flow-proportional interpolation of the clearwell tracer study. For unit processes other than a clearwell, a PWS shall either estimate T_{10} or use an interpolation of tracer study T_{10} using multiple flows for each unit process in which a disinfectant residual exists.

(iii) Chemicals in use at time of sampling. Report chemical name, chemical dose at time of sampling, and measurement formula. Measurement formulas (e.g., mg/l as Aluminum) shall be provided to determine the correct amount of the chemical compound being added.

(iv) Short circuiting factor (optional). The short circuiting factor is an assumed value for the ratio of T_{10} to nominal contact time (volume divided by flow).

(v) T_{50} (minutes) (optional). T_{50} should be reported only if based on a tracer study.

TABLE 6a.—PUBLIC WATER SYSTEM INFORMATION

Permanent data	Design data	Monthly data
Public Water System:		
Utility Name		Sampling Dates: From (date) To (date).
Public Water Supply Identification Number (PWSID)		Retail population on day of sampling.
Water Industry Data Base (WIDB) Number [Optional]		Wholesale population on day of sampling.
Official Contact Person:		Monthly average Retail flow (MGD).
Name		Monthly average Wholesale flow (MGD).
Mailing Address		
Phone Number [optional]		
FAX Number [optional]		
ICR Contact Person:		
Name		
Mailing Address		
Phone Number [optional]		
FAX Number [optional]		
E-Mail Address [optional]		
Treatment Plant: [1]		
Plant name	Plant type (e.g., Conventional Filtration, Direct Filtration, In-Line Filtration, Two Stage Softening, Disinfection Only/Groundwater, Other Groundwater treatment)	Hours of operation (hours per day)
ICR plant number assigned by EPA [2]		Sludge solids production (lb/day)
PWSID number of treatment plant [3]		Percent solids in sludge (%)
State approved (permitted) plant capacity (MGD)		
Historical minimum water temperature (°C)		
Installed sludge handling capacity (lb/day)		
Process Train:		
Name	Process Train Type (e.g., Conventional Filtration, Direct Filtration, In-Line Filtration, Two Stage Softening, Disinfection Only/Groundwater, Other Groundwater treatment)	

[1] A PWS that operates more than one treatment plant shall report treatment plant information in this table for each treatment plant.

[2] EPA shall assign ICR plant number after the PWS submits sampling plan.

[3] PWSID of treatment plant if different from the PWSID reported in ''Public Water System''.

TABLE 6b.—PLANT INFLUENT INFORMATION

Permanent data	Monthly data
Water Resource [1]	
Name of resource: Type of resource (One of the following): 1 Flowing stream 2 Reservoir/Lake 3 Ground water classified as under the direct influence of surface water (GWUDI) 4 Ground water 5 Purchased finished water 6 Non-Fresh (such as salt water)	If Reservoir/Lake: Mean Residence Time (days).
Intake-Surface Water [2]	
Location of intake: [3] Latitude (deg/min/sec) Longitude (deg/min/sec) Hydrologic unit code (8 digit), if known [4] Stream Reach Code (3 digit) (if known) River mile number (mile) (if known) Is watershed control practiced? (yes/no)	Flow on day of sampling (MGD).
Intake-Ground Water [5] [6]	
Location of intake: Latitude (deg/min/sec) Longitude (deg/min/sec) Hydrological unit code (8 digit), if known [4] Is wellhead protection practiced? (yes/no)	Flow on day of sampling (MGD).
Intake-Purchased Finished Water [7]	
Name of supplying utility ..... PWSID of supplying utility	Flow on day of sampling (MGD).
Plant Influent [8]	
	Monthly average flow (MGD). Flow at time of sampling (MGD).

[1] Each treatment plant shall have at least one water resource. Each water resource shall have at least one intake. A treatment plant that uses more than one water resource shall report water resource information in this table for each water resource.
[2] Intake-Surface Water describes the physical location of an intake structure located in a river, lake, or other surface water resource or, for ground water under the direct influence of surface water, the physical location of a well.
[3] The location of the intake will allow cross referencing into other data bases containing information on possible contamination threats to the intake.
[4] The hydrologic unit code will allow cross referencing into other data bases containing information on possible contamination threats to the intake.
[5] An Intake-Ground Water describes the physical location of a well or well field (if multiple wells draw from a common aquifer.

[6] A PWS is not required to report information for ground water that is not treated.
[7] A PWS is required to report information for purchased finished water only if that water is further treated.
[8] Multiple ''Intakes'' combine into one ''Plant Influent.'' Each treatment plant has only one treatment plant influent. The treatment plant influent shall mark the point in the treatment plant where the ''Plant Influent'' sample shall be collected as described in Tables 1, 2, 3 and 5 of this section.

TABLE 6c.—UNIT PROCESS INFORMATION

Design data	Monthly data
Presedimentation Basin [1]	
Tube Settler Brand Name Plate Settler Brand Name Baffling type[2]	Liquid volume (gallons). Surface area (ft^2). Projected Tube Settler Surface Area (ft^2). Projected Plate Settler Surface Area (ft^2).
Ozone Contact Basin	
Information for the complete ozone contact basin: Type of Ozone Contactor (One of the following) 1 Bubble Diffusion 2 Turbine Number of Chambers Information for each ozone contact chamber: Chamber sequence number Liquid volume (ft^3) Surface area (ft^2) Water/Ozone flow regime (one of the following) 1 Counter-current 2 Co-current	Information for the complete ozone contact basin: Ozone CT (mg min/l).[10] Ozone Giardia Inactivation (logs). Ozone Virus Inactivation (logs). Ozone concentration in feed gas (% by weight). Total Ozone Gas Flow Rate to Contactor (SCFM).[3] Type of feed gas used to generate ozone (one of the following). 1 Air. 2 Oxygen. Total Ozone Applied Dose (mg/l). Information for each ozone contact chamber: Percent ozone gas flow split to this chamber (%). Hydrogen peroxide dose (mg/l).
Washwater Return Point [8]	
Indicate which washwater treatment processes are being used on day of sampling Is there treatment (yes/no):	Flow of returned washwater at time of sampling (MGD). 24 hr average flow prior to sampling (MGD).

If yes: Plain sedimentation (yes/no) Coagulation/sedimentation (yes/no) Filtration (yes/no) Disinfection (yes/no) Other Treatment (Text)	
Rapid Mix	
Type of mixer (one of the following): 1 Mechanical 2 Hydraulic 3 Static 4 Other Baffling type [2]	Mean velocity gradient "G" (sec^{-1}).[4] Liquid volume (gallons).
Flocculation Basin	
Type of mixer (one of the following): 1 Mechanical 2 Hydraulic Number of stages Baffling type [2]	Mean velocity gradient "G" (sec^{-1}) in each stage.[4] Liquid volume of each stage (gallons).
Sedimentation Basin	
Tube settler brand name Plate settler brand name Baffling type [2]	Liquid volume (gallons). Surface area (ft^2). Projected tube settler surface area (ft^2). Projected plate settler surface area (ft^2).
Solids Contact Clarifier	
Brand name: Type (check all that apply): Rectangular basin Upflow Reactor-clarifier Sludge blanket Tube settler brand name Plate settler brand name	Liquid volume (gallons). Surface area of settling zone (ft^2). Projected tube settler surface area (ft^2). Projected plate settler surface area (ft^2).

TABLE 6c.—UNIT PROCESS INFORMATION—Continued

Design data	Monthly data
Baffling type [2]	
Adsorption Clarifier	
Brand Name Baffling type [2]	Liquid volume (gallons). Surface area (ft^2).
Dissolved Air Flotation	
Baffling type [2]	Liquid volume (gallons). Surface area (ft^2). Percent recycle rate (%). Recycle stream pressure (psi).
Recarbonation Basin	
Baffling type [2]	Liquid volume (gallons). Surface area (ft^2).
Filtration	
Media Type (one of the following): 1 Dual media (Anthracite/Sand) 2 GAC over sand 3 Tri media (Anthracite/Sand/Garnet) 4 Sand 5 Deep bed monomedia anthracite 6 Deep bed monomedia GAC 7 Greensand 8 Other Design depth of GAC (inch) Type and manufacturer of activated carbon Design media depth (inch) Minimum water depth to top of media (ft) Depth from top of media to top of backwash trough (ft)	Liquid volume (gallons). Surface area (ft^2). Average filter run time (hr).
Slow Sand Filtration	
Media type Media depth	Surface area (ft^2). Average filter run length.

Media size	Cleaning method.
Diatomaceous Earth Filter	
	Effective DE filter surface (ft^2). Precoat (lb/ft^2). Bodyfeed (mg/l). Run length (hours).
Granular Activated Carbon—Post-Filter Adsorber	
Manufacturer of activated carbon Type of activated carbon	Liquid volume (gallons). Surface area (ft^2). Carbon volume (ft^3). Empty bed contact time (minutes). Operating reactivation frequency (days).
Membranes	
Model name: Type (one of the following): 1 Reverse osmosis 2 Nanofiltration 3 Ultrafiltration 4 Microfiltration 5 Electrodialysis Number of stages Molecular weight cutoff (daltons) Design flux (gpd/ft^2) Design pressure (psi)	Surface area (ft^2). Percent recovery (%). Operating pressure (psi). Operating flux (gpd/ft^2). Cleaning method (one of the following) Hydraulic. Chemical. Cleaning frequency (days).
Air Stripping	
Packing height (ft) Design air to water ratio (volume/volume) Type of packing (Name) Nominal size of packing (inch)	Horizontal cross-section area (ft^2). Air flow (SCFM).[3]
Ion Exchange	
Resin (Name) Resin manufacturer Design exchange capacity (equ/ft^3)[3] Bed depth (ft)	Liquid volume (gallons). Surface area (ft^2).

TABLE 6c.—UNIT PROCESS INFORMATION—Continued

Design data	Monthly data
Disinfection Contact Basin [5 6]	
Baffling type [2]	Liquid volume (gallons). Surface area (ft^2).
Clearwell [7]	
Baffling type [2] Minimum liquid volume (gallons) Covered or Open	Liquid volume (gallons). Surface area (ft^2).
Additional Water Sources [9]	
Type of water source: Purchased Finished water Untreated ground water Treated ground water Untreated surface water Treated surface water Other	Flow of additional source (MGD).[6]
Other Treatment	
Purpose	Surface area (ft^2) [optional]. Liquid Volume (gallons) [optional].

[1] A reservoir to which oxidants, disinfectants, or coagulants are added is considered a presedimentation basin.

[2] Baffling type classified as one of the following: 1 (Unbaffled (mixed tank)), 2 (Poor (inlet/outlet only)), 3 (Average (Inlet/Outlet and intermediate)), 4 (Superior (Serpentine)), or 5 (Perfect (Plug flow)). Information on classifying baffling types can be found in ''Guidance Manual for Compliance with the Filtration and Disinfection Requirements for Public Water Systems using Surface Water Sources'', Appendix C.

[3] ''SCFM'' is standard cubic feet per minute. ''Equ/ft^3'' is equivalents per cubic foot.

[4] The mean velocity gradient is typically computed as G=square root of (P/uV) where P=power expended, u=viscosity, and V=liquid volume.

[5] The disinfection contact basin shall have a stable liquid level.

[6] Disinfection Contact Basin can be used to represent a pipe with a long contact time.

[7] A clear well may have a variable liquid level.

[8] The ''Washwater Return'' shall mark the point in the process train where washwater joins the main flow.

[9] Additional water sources includes water that is added to the process train after the influent.

[10] Ozone CT calculated using the procedure contained in ''Guidance Manual for Compliance with the Filtration and Disinfection Requirements for Public Water Systems using Surface Water Sources'', Appendix O, 1991.

TABLE 6d.—ADDITIONAL PROCESS TRAIN INFORMATION

Design data	Monthly data
Disinfectant Addition	
	Disinfectants in use at time of sampling. Dose (mg/l). Chemical formula (e.g., mg/l as chlorine).
Finished Water Sample Point (Plant Effluent) [1] [2]	
	Monthly average flow (MGD). Flow at time of sampling (MGD).

[1] This shall mark the end of a treatment plant.
[2] Unless the finished water of this treatment plant is blended with finished water from another treatment plant, this point is also the entry point to the distribution system.

TABLE 6e.—FINISHED WATER DISTRIBUTION INFORMATION

Design data	Monthly data
Entry Point to Distribution System [1]	
	Monthly average flow (MGD). Flow at time of sampling (MGD).
Wholesale Information [2]	
Name of purchaser ..... PWSID of purchaser	Flow at time of sampling (MGD).
Distribution System	
Typical maximum residence time (days) Average residence time (days) Design volume of distribution system storage (million gallon) Total surface area of open reservoirs in distribution system storage (ft^2)	Maximum residence time (days). Average residence time (days). Number of disinfection booster stations in operation at time of sampling: Chlorine. Chloramine. Chlorine dioxide. Range of distribution system disinfectant dosages. Chlorine: High (mg/l) Low (mg/l). Chloramine: High (mg/l) Low (mg/l). Chlorine dioxide: High (mg/l) Low (mg/l).

[1] Multiple treatment plants can feed into one entry point to the distribution system. If there is only one treatment plant then "Finished Water Sample Point (Plant Effluent)" and "Entry Point to Distribution System" are the same.
[2] The supplying public water system shall report "Wholesale Information" for each public water system which purchases finished water.

(b) Analytical methods. (1) A PWS shall use the methods identified in table 7 of this section for conducting analyses required by this subpart.

TABLE 7.—ANALYTICAL METHODS APPROVED FOR SUBPART M

Analyte	Methodology [1]		
	40 CFR reference [2]	EPA method	Standard method [3]
pH, alkalinity, calcium hardness, temperature	§ 141.23(k)(1)		
Turbidity	§ 141.74(a)(1)		
Disinfectant residuals: free chlorine, total chlorine, chlorine dioxide, ozone.	§ 141.74(a)(2)		4500–Cl B [9]
Trihalomethanes: chloroform, bro	§ 141.24(e)	551.1 [4]	
modichloromethane, dibro			
mochloromethane, bromoform			
Haloacetic acids: mono-, di-, and trichloroacetic acids; mono- and dibromoacetic acid; bromochloroacetic acid.		552.1,[5] 552.2 [4]	6251 B
Chloral hydrate		551.1 [4]	
Haloacetonitriles: di- and trichloroacetonitrile; bromochloroacetonitrile; dibromoacetonitrile.		551.1 [4]	
Haloketones: 1,1-Dichloropropanone; 1,1,1-trichloropropanone.		551.1 [4]	
Chloropicrin		551.1 [4]	
Chlorite		300.0 [6]	
Chlorate		300.0 [6]	
Bromide		300.0 [6]	
Bromate		300.0 [6]	
Total Organic Halide (TOX)			5320 B
Total Organic Carbon			5310 B, 5310 C, 5310 D
UV absorbance at 254 nm			5910
Simulated Distribution System Test (SDS)			5710 C
Total Hardness			2340 B,[7] 2340 C
Ammonia	§ 136.3, Table 1b [8]	350.1 [6]	4500–NH_3 D, 4500–NH_3 G
Chlorine Demand Test			2350 B

[1] Analyses shall be conducted by using mandatory analytical and quality control procedures contained in ''DBP/ICR Analytical Methods Manual'', EPA 814–B–96–002.

[2] Currently approved methodology for drinking water compliance monitoring is listed in Title 40 of the Code of Federal Regulations in the sections referenced in this column. The 18th and 19th editions of *Standard Methods for the Examination of Water and Wastewater,* American Public Health Association, 1015 Fifteenth Street NW, Washington, D.C. 20005, are equivalent for the methods cited in these sections. Therefore, either edition may be used.

[3] Except where noted, all methods refer to the 19th edition of *Standard Methods for the Examination of Water and Wastewater,* American Public Health Association, 1015 Fifteenth Street NW, Washington, D.C. 20005.

[4] Analytical method reprinted in ''Reprints of EPA Methods for Chemical Analyses Under the Information Collection Rule'', EPA 814–B–96–006. Originally published in ''Methods for the Determination of Organic Compounds in Drinking Water—Supplement III,'' EPA/600/R–95/131, August 1995, PB95–261616.

[5] Analytical method reprinted in ''Reprints of EPA Methods for Chemical Analyses Under the Information Collection Rule'', EPA 814–B–96–006. Originally published in ''Methods for the Determination of Organic Compounds in Drinking Water—Supplement II,'' EPA/600/R–92/129, August 1992, PB92–207703.

[6] Analytical method reprinted in ''Reprints of EPA Methods for Chemical Analyses Under the Information Collection Rule'', EPA 814–B–96–006. Originally published in ''Methods for the Determination of Inorganic Substances in Environmental Samples,'' EPA/600/R–93/100, August 1993, PB94–121811.

[7] The following methods, cited at § 141.23(k)(1) of this part, can be used to determine calcium and magnesium concentrations for use in conjunction with Standard Method 2340 B: EPA Method 200.7, Standard Method 3111 B, Standard Method 3120 B, or ASTM Method D511–93 B.

[8] PWSs may use only the automated electrode method from § 136.3, Table 1b.

[9] Standard Method 4500–Cl B is approved only for determining free chlorine residual concentrations in hypochlorite stock solutions. This method may not be used for any other disinfectant residual analyses.

(2) Analyses under this section shall be conducted by laboratories that have received approval from EPA to perform sample analysis for compliance with this rule. Laboratories that wish to become approved shall contact EPA in writing at USEPA, Technical Support Division, ICR Laboratory Coordinator, 26 W. Martin Luther King Drive, Cincinnati, OH 45268 not later than November 14, 1996. Requirements for approval are included in ''DBP/ICR Analytical Methods Manual'', EPA 814–B–96–002.

(c) *Reporting.* (1) A PWS shall report required data and information collected under the provisions of paragraph (a) of this section to EPA, using an EPA-specified computer readable format. A PWS shall submit a monthly report that indicates the analytical results of all samples collected, including quarterly samples taken in that same month, and all process train data. These reports shall be submitted on a diskette no later than the fourth month following sampling. In addition to the information in tables 1 through 6 in paragraph (a) of this section, reports shall include PWSID, ICR plant identification, sample date, analysis date, laboratory identification numbers, analytical methods used, sample identification numbers, quality assurance code, internal standards, surrogate standards, and preserved sample pH, if appropriate.

(2) *Additional Requirements.* A PWS shall submit a DBP and related monitoring sampling plan for EPA approval, using software provided by EPA, for each treatment plant specified in § 141.141(b)(2) of this subpart that indicates sampling point locations and monitoring to be conducted at each point, and process treatment train information. This sampling plan shall be submitted to EPA at the same time and on the same diskette as the microbiological sampling plan required by § 141.143(c)(3) and no later than eight weeks after the PWS receives the Notice of ICR Final Applicability Determination from EPA, using the procedure specified in ''ICR Sampling Manual'', EPA 814–B–96–001, April 1996.

(3) All reports required by this section shall be submitted to USEPA (ICR4600), ICR Data Center, Room 1111 East Tower, 401 M Street SW., Washington, DC 20460.

(4) The PWS shall keep all data for at least three years following data submission to EPA.

(d) *Incorporation by reference.* The documents and methods listed in paragraphs (d) (1) and (2) of this section are incorporated by reference for purposes specified in this section. This incorporation by reference was approved by the Director of the Federal Register in accordance with 5 U.S.C. 552(a) and 1 CFR part 51. Copies may be inspected at USEPA, Drinking Water Docket (4101), 401 M Street SW., Washington, DC 20460, or at Office of the Federal Register, 800 North Capitol Street, NW., Suite 700, Washington, DC.

(1) ''Standard Methods for the Examination of Water and Wastewater,'' 19th edition, 1995. Available from the American Public Health Association, 1015 Fifteenth Street, NW., Washington, DC 20005.

(2) ''Guidance Manual for Compliance with the Filtration and Disinfection Requirements for Public Water Systems using Surface Water Sources'', Appendices C and O, 1991. Available from American Water Works Association, 6666 West Quincy Avenue, Denver, CO 80235.

§ 141.143 Microbial monitoring.

(a) Monitoring requirements. (1) *Parameters.* A PWS shall sample for the following parameters for the period specified in § 141.141(d) of this subpart and at the location specified and using the analytical methods specified in paragraphs (a)(2) and (b), respectively, of this section. For each sample, a PWS shall determine the densities of total coliforms, fecal coliforms or *Escherichia coli, Giardia, Cryptosporidium,* and total culturable viruses for each treatment plant required to monitor under the provisions of § 141.141(b) of this subpart.

(2) *Monitoring locations.* (i) A PWS shall collect one sample of the treatment plant influent at the frequency specified in § 141.141(d) of this subpart.

(A) A sample of treatment plant influent shall be taken at a location at the upstream end of a treatment plant where waters from all intakes are

blended prior to any treatment or chemical addition.

(B) For treatment plants that have multiple intakes and add chemicals at the intake, the PWS shall take an intake sample of the water resource with the poorest microbiological quality (or, if that cannot be determined, the water resource with the highest flow) collected before chemical addition and before pretreatment. If the intakes are expected to have the same source water quality, one representative intake sample may be taken. If a disinfectant is added at or before the intake (e.g., for zebra mussel control), the sample shall be taken in the vicinity of the intake in such manner that the sample is not contaminated by the disinfectant.

(ii) A PWS that, during any of the first twelve months of monitoring at the treatment plant influent, detects 10 or more *Giardia* cysts, or 10 or more *Cryptosporidium* oocysts, or one or more total culturable viruses, in one liter of water; or calculates a numerical value of the *Giardia* or *Cryptosporidium* concentration equal to or greater than 1000 per 100 liters or virus concentration equal to or greater than 100 per 100 liters; or detects no pathogens in the sample and calculates a numerical value of the detection limit for *Giardia* or *Cryptosporidium* concentration equal to or greater than 1000 per 100 liters or virus concentration equal to or greater than 100 per 100 liters; shall also collect one sample of finished water per month at each such treatment plant, beginning in the first calendar month after the PWS learns of such a result. The sample of finished water shall be collected at a point after which all treatment processes for a particular treatment plant are complete (including the clearwell and final point of disinfection) and before the distribution system begins. For each sample of finished water, PWSs shall determine the density of total coliforms, fecal coliforms or *E. coli, Giardia, Cryptosporidium,* and total culturable viruses. A PWS shall continue finished water monitoring monthly until 18 months of treatment plant influent monitoring has been completed.

(iii) In lieu of conducting finished water monitoring of *Giardia* and *Cryptosporidium* specified in paragraph (a)(2)(ii) of this section, a PWS may notify EPA in its response to the notice of applicability required by paragraph (c)(3)(i) of this section that the PWS will comply with the alternative monitoring requirements in paragraphs (a)(2)(iii) (A) and (B) of this section. The PWS shall still conduct finished water monitoring for all other microorganisms, except for *Giardia* and *Cryptosporidium* monitoring in the finished water.

(A) The PWS measures the particle counts in the treatment plant influent, at points immediately prior to filtration and after filtration (but before the addition of post-filtration chemicals). Particle counting shall be conducted on the same treatment train as is sampled for monitoring conducted under the provisions of § 141.142(a) of this subpart. Such samples shall be collected monthly during the entire 18-month monitoring period, using the procedures contained in the ''ICR Sampling Manual'', EPA 814–B–96–001, April 1996. The PWS may use either grab or continuous particle counting. Particle counting shall be conducted during the same time as protozoa monitoring required by paragraph (a)(2)(iii)(B) of this section.

(*1*) If grab sampling is conducted, the PWS shall collect 12 samples per location at the treatment plant influent, filter influent, and filter effluent, over either a 24-hour period or the duration of the filter run, whichever is shorter.

(*2*) If continuous particle counting is conducted, the PWS shall collect 12 instrument readings per location, evenly spaced in time, at the treatment plant influent, filter influent, and filter effluent, over either a 24-hour period or the duration of the filter run, whichever is shorter.

(*3*) For each sample, the PWS shall measure particle counts per milliliter in the size ranges of 3µm-5µm, 5µm-7µm, 7µm-10µm, 10µm–15µm, and >15µm, and shall report to EPA the mean value in each size range of the 12 values collected over the sampling period.

(B) The PWS collects and analyzes at least four consecutive months of *Giardia* and *Cryptosporidium* samples at the same locations specified in paragraph (a)(2)(iii)(A) of this section, within the first 12 months of the 18 months

of sampling. The PWS shall collect *Giardia* and *Cryptosporidium* samples during the same time period as it is conducting particle counting. The minimum sample volume for *Giardia* and *Cryptosporidium* analyses shall be 100 liters for treatment plant influent and 1,000 liters for water that has undergone any treatment. The PWS may use results of monitoring for *Giardia* and *Cryptosporidium* in the treatment plant influent specified in paragraph (a)(2) of this section to meet the requirements of this paragraph as long as such monitoring meets the requirements of both this paragraph and paragraph (a)(2) of this section.

(iv) If a PWS has monitored total coliforms, fecal coliforms, or *E. coli* in the treatment plant influent for at least five days/week for any period of six consecutive months beginning after January 1, 1994 and 90% of all samples taken in that six-month period contained no greater than 100 total coliforms/100 ml, or 20 fecal coliforms/100 ml, or 20 *E. coli*/100 ml, the PWS may request to not conduct virus monitoring for that treatment plant, for the duration of the requirement. Even if approved, the PWS may subsequently be required to monitor under the criteria in paragraph (a)(2)(iv)(A) of this section. This request shall be submitted as part of the response to the notice of applicability required by paragraph (c)(3)(i) of this section.

(A) If the PWS is subsequently required to monitor the finished water under the provisions of paragraph (a)(2)(ii) of this section, the PWS shall monitor, along with the other specified organisms, total culturable viruses, as specified in paragraph (a)(2)(i) of this section for treatment plant influent and as specified in paragraph (a)(2)(ii) of this section for finished water, until 18 months of microbial monitoring is completed.

(B) A PWS may use coliform data collected under § 141.71(a)(1) of this part for this purpose but, if this is done, the PWS shall submit two separate monitoring reports. One report, to meet the requirements of § 141.71(a)(1) of this part, shall continue to be submitted as required by subpart H of this part. The other report shall be submitted to meet the requirements of paragraph (c)(3) of this section.

(C) If a PWS does not provide EPA with six months of suitable coliform results as part of its response to the notice of applicability, the PWS shall begin virus monitoring. If a PWS begins virus monitoring and subsequently provides EPA with six months of coliform results that are at or below the indicated density limit, and EPA approves the request to not conduct virus monitoring, the PWS may avoid subsequent treatment plant virus monitoring.

(b) Analytical Methods. (1) A PWS shall use the methods listed in paragraphs (b)(1) (i) through (v) of this section for monitoring under this subpart.

(i) Fecal coliforms—specified at § 141.74(a)(1) of this part, except that whenever paired source water samples and finished water samples are to be collected, only the fecal coliform procedure (Standard Method 9221E), as specified in § 141.74(a)(1) of this part, using EC Medium, can be used. The time between sample collection and initiation of sample analysis shall not exceed eight hours. Samples shall be chilled, but not frozen, and shipped at a temperature of less than 10°C. Samples not processed immediately at the laboratory shall be refrigerated. The laboratory must invalidate samples that arrive frozen or at a temperature greater than 10°C.

(ii) Total coliforms—specified at § 141.74(a)(2) of this part. The time between sample collection and initiation of sample analysis shall not exceed eight hours. Samples shall be chilled, but not frozen, and shipped at a temperature of less than 10°C. Samples not processed immediately at the laboratory shall be refrigerated. The laboratory must invalidate samples that arrive frozen or at a temperature greater than 10°C.

(iii) *E. coli*—as specified by § 141.21(f)(6) (i) through (iii) of this part, except that the density shall be reported. PWSs using the EC+MUG and ONPG-MUG tests shall use either a 5-tube or 10-tube 10-ml configuration, with serial dilutions of the original sample as needed, and report the Most Probable Number. PWSs may also use a commercial multi-test system for *E.*

coli enumeration, as long as they use M-Endo medium for the initial isolation of the organisms, pick every colony on the plate with the appearance of a total coliform, and streak it for purification before subjecting the colony to a multi-test system. The time between sample collection and initiation of sample analysis, regardless of method used, shall not exceed eight hours. Samples shall be chilled, but not frozen, and shipped at a temperature of less than 10°C. Samples not processed immediately at the laboratory shall be refrigerated. The laboratory must invalidate samples that arrive frozen or at a temperature greater than 10°C.

(iv) *Giardia* and *Cryptosporidium*—ICR Protozoan Method, as described in ''ICR Microbial Laboratory Manual'', EPA 600/R–95/178, April 1996.

(v) Total culturable viruses—Virus Monitoring Protocol, as described in ''ICR Microbial Laboratory Manual'', EPA 600/R–95/178, April 1996.

(2) *Laboratories.* A PWS shall use EPA-approved laboratories to analyze for *Giardia, Cryptosporidium,* and total culturable viruses. A PWS shall use laboratories certified for microbiology analyses by either EPA or a State under the EPA or State drinking water program for the analysis of total coliforms, fecal coliforms, and *E. coli.* Laboratories that wish to become approved shall contact EPA in writing at USEPA, Technical Support Division, ICR Laboratory Coordinator, 26 W. Martin Luther King Drive, Cincinnati, OH 45268 not later than August 14, 1996. Laboratory approval criteria for *Giardia, Cryptosporidium,* and total culturable viruses are found in the ''ICR Microbial Laboratory Manual'', EPA 600/R–95/178, April 1996.

(3) A PWS shall send EPA a virus archive sample prepared as described in Chapter VIII of ''ICR Microbial Laboratory Manual'', EPA 600/R–95/178, April 1996, for each water sample identified in paragraph (b)(3) (i) or (ii) of this section.

(i) Samples of treatment plant influent and finished water, for every month after the PWS learns that viruses were detected in any previous sample of finished water.

(ii) Samples of treatment plant influent and finished water, regardless of whether viruses are detected in the finished water, for every month after the PWS learns that a density of at least 10 viruses/L was detected in any previous treatment plant influent water sample.

(iii) A PWS may arrange to have virus samples shipped directly to EPA by its virus laboratory for archiving.

(iv) Samples shall be sent on dry ice to ICR Virus Archiving Coordinator following the procedures specified in ''ICR Microbial Laboratory Manual'', EPA 600/R–95/178, April 1996.

(c) *Reporting.* (1) A PWS shall report data and information required under paragraphs (a) and (b) of this section using an EPA-specified computer readable format. A PWS shall submit a monthly report on a diskette, no later than the fourth month following sampling, that indicates the analytical results of all samples collected. Reports shall include PWSID, ICR plant identification, sample date, analysis date, laboratory identification numbers, analytical methods used, sample identification numbers, analytical batch numbers, quality assurance code, and processing batch numbers, if appropriate.

(2)(i) For a PWS using the alternative to *Giardia* and *Cryptosporidium* monitoring in paragraph (a)(2)(iii) of this section, the PWS shall report to EPA the mean value in each size range of the 12 particle counting values collected over the sampling period. In addition, during the four consecutive months when the PWS collects *Giardia* and *Cryptosporidium* samples specified in paragraph (a)(2)(iii)(B) of this section, the PWS shall report to EPA, for each measured site, the densities of *Giardia* and *Cryptosporidium* at each measured site. This information shall be submitted at the same time as the report required by paragraph (c)(1) of this section.

(ii) A PWS that is not required to monitor for total culturable viruses under the provisions of paragraph (a)(2)(iv) of this section shall report to EPA the dates and results of all total coliform, fecal coliform, or *E. coli* monitoring used by the PWS to determine that additional virus monitoring is unnecessary. The report shall indicate all data collected during the six-month time period, and how the data were

used to calculate compliance with this requirement.

(3) *Additional Requirements.* A PWS shall submit a microbiological sampling plan for EPA approval, using software provided by EPA, for each treatment plant specified in § 141.141(b) of this subpart that indicates sampling point locations and monitoring to be conducted at each point. This sampling plan shall be submitted to EPA at the same time and on the same diskette as the DBP and related monitoring sampling plan required by § 141.142(c)(2) and no later than eight weeks after the PWS receives the Notice of ICR Final Applicability Determination from EPA, using the procedure specified in ''ICR Sampling Manual'', EPA 814–B–96–001, April 1996.

(4) All reports required by this section shall be submitted to USEPA (ICR4600), ICR Data Center, Room 1111 East Tower, 401 M Street SW., Washington, DC 20460.

(5) The PWS shall keep all data for at least three years following data submission to EPA.

§ 141.144 Disinfection byproduct precursor removal studies.

(a) *TOC, UFCTOX, THM4, and HAA5 applicability monitoring.* A PWS required to comply with this section shall conduct TOC, UFCTOX, THM4, and HAA5 monitoring specified in § 141.141(e)(2) of this subpart. A PWS may use monitoring results from samples required by § 141.142(a) of this subpart to meet this requirement to the extent that all requirements in each section are met.

(b) *Treatment study requirements.* A PWS identified in § 141.141(b) of this subpart shall conduct disinfection byproduct precursor removal studies (treatment studies). The treatment study shall use bench-and/or pilot-scale systems for at least one of the two appropriate candidate technologies (GAC or membrane processes) for the reduction of organic DBP precursors. The treatment studies shall be designed to yield representative performance data and allow the development of national treatment cost estimates for different levels of organic disinfection byproduct control. The treatment objective of the studies is the achievement of levels of byproducts less than 40 μg/L TTHM and 30 μg/L HAA5, as an annual average. The treatment study shall be conducted with the effluent from treatment processes already in place that remove disinfection byproduct precursors and TOC, to simulate the most likely treatment scenario. PWSs are permitted to optimize these processes or pilot additional processes appropriate for pretreatment for treatment studies. In order to minimize the formation of DBPs, the test water for both the bench- and pilot-scale tests shall be obtained from a location before the first point at which oxidants or disinfectants that form halogenated disinfection byproducts are added. If the use of these oxidants or disinfectants precedes any full-scale treatment process that removes disinfection byproduct precursors, then bench- and pilot-scale treatment processes that represent these full-scale treatment processes are required prior to the GAC or membrane process. A PWS should exercise sound judgement in its selection of treatment process to study and the point at which to obtain water for study. Depending upon the type of treatment study, the study shall be conducted in accordance with the following criteria.

(1) Bench-scale tests are continuous flow tests using rapid small scale column test (RSSCT) for GAC and small scale membrane test apparatus as specified in ''ICR Manual for Bench- and Pilot-scale Treatment Studies'' (EPA 814–B–96–003, April 1996).

(i) GAC bench-scale testing shall include information on the experimental conditions and results necessary to adequately determine the scaled-up breakthrough curves under the conditions of each RSSCT. At least two empty bed contact times (EBCTs) shall be tested using the RSSCT. These RSSCT EBCTs shall be designed to represent a full-scale EBCT of 10 min and a full-scale EBCT of 20 min. Additional EBCTs may be tested. The RSSCT testing is described in the ''ICR Bench- and Pilot-scale Treatment Study Manual'' (EPA 814–B–96–003, April 1996). The RSSCT tests at each EBCT shall be run quarterly to ascertain the impact of seasonal variation. Thus a total of four RSSCTs at each EBCT should be run.

When seasonal variation is not significant, as is the case in most ground waters, the quarterly tests should be run to investigate other variables, as described in the ''ICR Bench- and Pilot-scale Treatment Study Manual'' (EPA 814–B–96–003, April 1996). The RSSCT shall be run until the effluent TOC concentration is at least 70% of the average influent TOC concentration or the effluent TOC reaches a plateau at greater than 50% of the influent TOC (i.e., the effluent TOC does not increase over a two-month full-scale-equivalent time period by more than 10% of the average influent TOC concentration) or a RSSCT operation time that represents the equivalent of one year of full-scale operation, whichever is shorter. The average influent TOC is defined as the running average of the influent TOC at the time of effluent sampling. If, after completion of the first quarter RSSCTs, the PWS finds that the effluent TOC reaches 70% of the average influent TOC within 20 full-scale equivalent days on the EBCT=10 min test and within 30 full-scale equivalent days on the EBCT=20 min test, the last three quarterly tests shall be conducted using membrane bench-scale testing with only one membrane, as described in paragraph (b)(1)(ii) of this section.

(ii) Membrane bench-scale testing shall include information on the experimental conditions and results necessary to determine the water quality produced by the membrane treatment and a preliminary estimate of productivity. The testing procedures and monitoring and reporting requirements are described in the ''ICR Bench- and Pilot-scale Treatment Study Manual'' (EPA 814–B–96–003, April 1996). A minimum of two different membrane types with nominal molecular weight cutoffs of less than 1000 shall be investigated. Membrane tests shall be conducted quarterly over one year to determine the seasonal variation. Thus, a total of four bench-scale tests with each membrane shall be run. If seasonal variation is not significant, as is the case of most ground waters, the quarterly tests should be run to evaluate the impact of other variables, such as pretreatment, or additional membranes could be tested. Alternatively, a PWS may choose to conduct a long-term, single element study using a single membrane type in lieu of evaluating two membranes in four quarterly short-term tests, using the protocol in the ''ICR Bench- and Pilot-scale Treatment Study Manual'' (EPA 814–B–96–003, April 1996).

(2) A PWS shall conduct pilot-scale testing as continuous flow tests. For GAC, the PWS shall use GAC of particle size representative of that used in full-scale practice, a pilot GAC column with a minimum inner diameter of 2.0 inches, and hydraulic loading rate (volumetric flow rate/column cross-sectional area) representative of that used in full-scale practice. The PWS shall design a pilot-scale membrane system as a staged array of elements as described in ''ICR Manual for Bench- and Pilot-scale Treatment Studies'', EPA 814–B–96–003, April 1996.

(i) GAC pilot-scale testing. (A) The pilot testing procedures and monitoring and reporting requirements are prescribed in the ''ICR Bench- and Pilot-scale Treatment Study Manual'' (EPA 814–B–96–003, April 1996).

(B) At least two EBCTs shall be tested, EBCT=10 min and EBCT=20 min, using the pilot-scale plant. Additional EBCTs may be tested.

(C) The pilot tests at each EBCT shall continue until the effluent TOC concentration is at least 70% of the average influent TOC concentration on two consecutive TOC sample dates that are at least two weeks apart or the effluent TOC reaches a plateau at greater than 50% of the influent TOC (i.e., the effluent TOC does not increase over a two-month period by more than 10% of the average influent TOC concentration). If either of these criteria is met for the 20-minute EBCT prior to six months run time, a second pilot test at each EBCT shall be conducted following the same sampling requirements. In all cases the maximum length of the pilot study (one or two tests) is one year. The average influent TOC is defined as the running average of the influent TOC at the time of sampling. The pilot-scale testing shall be timed to capture seasonal variation. If seasonal variation is not significant, as is the case with most ground waters, the pilot-scale test runs shall be designed

to evaluate the impact of other variables, such as pretreatment.

(ii) Membrane pilot-scale testing.

(A) The membrane pilot testing procedures and monitoring and reporting requirements are prescribed in the ''ICR Bench- and Pilot-scale Treatment Study Manual'' (EPA 814–B–96–003, April 1996).

(B) The membrane test system shall be designed to yield information on loss of productivity (fouling), pretreatment requirements, cleaning requirements, and permeate quality and operated at a recovery representative of full-scale operation.

(C) The pilot-scale testing shall be run for one year.

(3) Chlorination under simulated distribution system (SDS) conditions shall be used prior to the measurement of THM4, HAA6, TOX, and chlorine demand. These conditions are described in ''ICR Manual for Bench- and Pilot-scale Treatment Studies'' (EPA 814–B–96–003, April 1996) and represent the average conditions in the distribution system at that time with regard to holding time, temperature, pH, and chlorine residual. If chlorine is not used as the final disinfectant in practice, then a chlorine dose shall be set to yield a free chlorine residual of 1.0 to 0.5 mg/l after a holding time, temperature, and pH equal to those representative of the distribution system averages.

(c) *Analytical Methods.* All analyses required by paragraphs (a) and (b) of this section shall be conducted using the methods and the mandatory analytical and quality control procedures contained in either ''DBP/ICR Analytical Methods Manual'' (EPA 814–B–96–002, April 1996) or ''ICR Manual for Bench- and Pilot-scale Treatment Studies'' (EPA 814–B–96–003, April 1996). In addition, TOC analyses required by paragraph (a) of this section shall be conducted by a laboratory approved under the provisions of §141.142(b)(2) of this subpart.

(d) *Reporting.* (1) TOC and UFCTOX reporting. A PWS shall submit the monthly results of 12 months of TOC or UFCTOX monitoring required by paragraph (a)(1) of this section and the annual average of those monthly results not later than October 14, 1997. This report is not required to be submitted electronically. Although a PWS may use monitoring results from samples required by §141.142(a) of this subpart to meet this requirement, it shall submit separate reports to meet this reporting requirement and the reporting requirement in §141.142(c)(1) of this subpart.

(2) A PWS shall report all data collected under the provisions of paragraph (b) of this section. In addition, a PWS shall report the information for water resource and full-scale and pilot- or bench-scale pretreatment processes that precede the bench/pilot systems. These data and information shall be reported in the format specified in ''ICR Manual for Bench- and Pilot-scale Treatment Studies'' (EPA 814–B–96–003, April 1996) not later than July 14, 1999.

(3) All reports required by this section shall be submitted to USEPA, Technical Support Division, ICR Precursor Removal Studies Coordinator, 26 West Martin Luther King Drive, Cincinnati, OH 45268.

PART 142—NATIONAL PRIMARY DRINKING WATER REGULATIONS IMPLEMENTATION

Subpart A—General Provisions

Sec.
142.1 Applicability.
142.2 Definitions.
142.3 Scope.
142.4 State and local authority.

Subpart B—Primary Enforcement Responsibility

142.10 Requirements for a determination of primary enforcement responsibility.
142.11 Initial determination of primary enforcement responsibility.
142.12 Revision of State programs.
142.13 Public hearing.
142.14 Records kept by States.
142.15 Reports by States.
142.16 Special primacy requirements.
142.17 Review of State programs and procedures for withdrawal of approved primacy programs.
142.18 EPA review of State monitoring determinations.
142.19 EPA review of State implementation of national primary drinking water regulations for lead and copper.

Subpart C—Review of State-Issued Variances and Exemptions

142.20 State-issued variances and exemptions.
142.21 State consideration of a variance or exemption request.
142.22 Review of State variances, exemptions and schedules.
142.23 Notice to State.
142.24 Administrator's rescission.

Subpart D—Federal Enforcement

142.30 Failure by State to assure enforcement.
142.31 [Reserved]
142.32 Petition for public hearing.
142.33 Public hearing.
142.34 Entry and inspection of public water systems.

Subpart E—Variances Issued by the Administrator

142.40 Requirements for a variance.
142.41 Variance request.
142.42 Consideration of a variance request.
142.43 Disposition of a variance request.
142.44 Public hearings on variances and schedules.
142.45 Action after hearing.
142.46 Alternative treatment techniques.

Subpart F—Exemptions Issued by the Administrator

142.50 Requirements for an exemption.
142.51 Exemption request.
142.52 Consideration of an exemption request.
142.53 Disposition of an exemption request.
142.54 Public hearings on exemption schedules.
142.55 Final schedule.
142.56 Extension of date for compliance.
142.57 Bottled water, point-of-use, and point-of-entry devices.

Subpart G—Identification of Best Technology, Treatment Techniques or Other Means Generally Available

142.60 Variances from the maximum contaminant level for total trihalomethanes.
142.61 Variances from the maximum contaminant level for fluoride.
142.62 Variances and exemptions from the maximum contaminant levels for organic and inorganic chemicals.
142.63 Variances and exemptions from the maximum contaminant level for total coliforms.
142.64 Variances and exemptions from the requirements of part 141, subpart H—Filtration and Disinfection.

Subpart H—Indian Tribes

142.72 Requirements for Tribal eligibility.
142.76 Request by an Indian Tribe for a determination of eligibility.
142.78 Procedure for processing an Indian Tribe's application.

Subpart I—Administrator's Review of State Decisions that Implement Criteria Under Which Filtration Is Required

142.80 Review procedures.
142.81 Notice to the State.

Subpart J—Procedures for PWS Administrative Compliance Orders

142.201 Purpose.
142.202 Definitions.
142.203 Proposed administrative compliance orders.
142.204 Notice of proposed administrative compliance orders.
142.205 Opportunity for public hearings; opportunity for State conferences.
142.206 Conduct of public hearings.
142.207 Issuance, amendment or withdrawal of administrative compliance order.
142.208 Administrative assessment of civil penalty for violation of administrative compliance order.

AUTHORITY: 42 U.S.C. 300g, 300g–1, 300g–2, 300g–3, 300g–4, 300g–5, 300g–6, 300j–4, and 300j–9.

SOURCE: 41 FR 2918, Jan. 20, 1976, unless otherwise noted.

Subpart A—General Provisions

§142.1 Applicability.

This part sets forth, pursuant to sections 1413 through 1416, 1445, and 1450 of the Public Health Service Act, as amended by the Safe Drinking Water Act, Public Law 93–523, regulations for the implementation and enforcement of the national primary drinking water regulations contained in part 141 of this chapter.

§142.2 Definitions.

As used in this part, and except as otherwise specifically provided:

Act means the Public Health Service Act.

Administrator means the Administrator of the United States Environmental Protection Agency or his authorized representative.

Agency means the United States Environmental Protection Agency.

Approved State primacy program consists of those program elements listed in § 142.11(a) that were submitted with the initial State application for primary enforcement authority and approved by the EPA Administrator and all State program revisions thereafter that were approved by the EPA Administrator.

Contaminant means any physical, chemical, biological, or radiological substance or matter in water.

Federal agency means any department, agency, or instrumentality of the United States.

Indian Tribe means any Indian Tribe having a Federally recognized governing body carrying out substantial governmental duties and powers over a defined area.

Interstate Agency means an agency of two or more States established by or under an agreement or compact approved by the Congress, or any other agency of two or more States or Indian Tribes having substantial powers or duties pertaining to the control of pollution as determined and approved by the Administrator.

Maximum contaminant level means the maximum permissible level of a contaminant in water which is delivered to the free flowing outlet of the ultimate user of a public water system; except in the case of turbidity where the maximum permissible level is measured at the point of entry to the distribution system. Contaminants added to the water under circumstances controlled by the user, except for those resulting from corrosion of piping and plumbing caused by water quality are excluded from this definition.

Municipality means a city, town, or other public body created by or pursuant to State law, or an Indian Tribe which does not meet the requirements of subpart H of this part.

National primary drinking water regulation means any primary drinking water regulation contained in part 141 of this chapter.

Person means an individual; corporation; company; association; partnership; municipality; or State, federal, or Tribal agency.

Primary enforcement responsibility means the primary responsibility for administration and enforcement of primary drinking water regulations and related requirements applicable to public water systems within a State.

Public water system means a system for the provision to the public of piped water for human consumption, if such system has at least fifteen service connections or regularly serves an average of at least twenty-five individuals daily at least 60 days out of the year. Such term includes (1) any collection, treatment, storage, and distribution facilities under control of the operator of such system and used primarily in connection with such system, and (2) any collection or pretreatment storage facilities not under such control which are used primarily in connection with such system.

Sanitary survey means an onsite review of the water source, facilities, equipment, operation and maintenance of a public water system for the purpose of evaluating the adequacy of such source, facilities, equipment, operation and maintenance for producing and distributing safe drinking water.

State means one of the States of the United States, the District of Columbia, the Commonwealth of Puerto Rico, the Virgin Islands, Guam, American Samoa, the Commonwealth of the Northern Mariana Islands, the Trust Territory of the Pacific Islands, or an eligible Indian tribe.

State primary drinking water regulation means a drinking water regulation of a State which is comparable to a national primary drinking water regulation.

State program revision means a change in an approved State primacy program.

Supplier of water means any person who owns or operates a public water system.

Treatment technique requirement means a requirement of the national primary drinking water regulations which specifies for a contaminant a specific treatment technique(s) known to the Administrator which leads to a reduction in the level of such contaminant sufficient to comply with the requirements of part 141 of this chapter.

[41 FR 2918, Jan. 20, 1976, as amended at 53 FR 37410, Sept. 26, 1988; 54 FR 52137, Dec. 20, 1989; 59 FR 64344, Dec. 14, 1994]

§142.3 Scope.

(a) Except where otherwise provided, this part applies to each public water system in each State; except that this part shall not apply to a public water system which meets all of the following conditions:

(1) Which consists only of distribution and storage facilities (and does not have any collection and treatment facilities);

(2) Which obtains all of its water from, but is not owned or operated by, a public water system to which such regulations apply;

(3) Which does not sell water to any person; and

(4) Which is not a carrier which conveys passengers in interstate commerce.

(b) In order to qualify for primary enforcement responsibility, a State's program for enforcement of primary drinking water regulations must apply to all other public water systems in the State, except for:

(1) Public water systems on carriers which convey passengers in interstate commerce;

(2) Public water systems on Indian land with respect to which the State does not have the necessary jurisdiction or its jurisdiction is in question; or

(3) Public water systems owned or maintained by a Federal agency where the Administrator has waived compliance with national primary drinking water regulations pursuant to section 1447(b) of the Act.

(c) Section 1451 of the SDWA authorizes the Administrator to delegate primary enforcement responsibility for public water systems to Indian Tribes. An Indian Tribe must meet the statutory criteria at 42 U.S.C. 300j–11(b)(1) before it is eligible to apply for Public Water System Supervision grants and primary enforcement responsibility. All primary enforcement responsibility requirements of parts 141 and 142 apply to Indian Tribes except where specifically noted.

[41 FR 2918, Jan. 20, 1976, as amended at 53 FR 37410, Sept. 26, 1988; 59 FR 64344, Dec. 14, 1994]

§142.4 State and local authority.

Nothing in this part shall diminish any authority of a State or political subdivision to adopt or enforce any law or regulation respecting drinking water regulations or public water systems, but no such law or regulation shall relieve any person of any requirements otherwise applicable under this part.

Subpart B—Primary Enforcement Responsibility

§142.10 Requirements for a determination of primary enforcement responsibility.

A State has primary enforcement responsibility for public water systems in the State during any period for which the Administrator determines, based upon a submission made pursuant to §142.11, and submission under §142.12, that such State, pursuant to appropriate State legal authority:

(a) Has adopted drinking water regulations which are no less stringent than the national primary drinking water regulations (NPDWRs) in effect under part 141 of this chapter;

(b) Has adopted and is implementing adequate procedures for the enforcement of such State regulations, such procedures to include:

(1) Maintenance of an inventory of public water systems.

(2) A systematic program for conducting sanitary surveys of public water systems in the State, with priority given to sanitary surveys of public water systems not in compliance with State primary drinking water regulations.

(3)(i) The establishment and maintenance of a State program for the certification of laboratories conducting analytical measurements of drinking water contaminants pursuant to the requirements of the State primary drinking water regulations including the designation by the State of a laboratory officer, or officers, certified by the Administrator, as the official(s) responsible for the State's certification program. The requirements of this paragraph may be waived by the Administrator for any State where all analytical measurements required by the

State's primary drinking water regulations are conducted at laboratories operated by the State and certified by the Agency. Until such time as the Agency establishes a National quality assurance program for laboratory certification the State shall maintain an interim program for the purpose of approving those laboratories from which the required analytical measurements will be acceptable.

(ii) Upon a showing by an Indian Tribe of an intergovernmental or other agreement to have all analytical tests performed by a certified laboratory, the Administrator may waive this requirement.

(4) Assurance of the availability to the State of laboratory facilities certified by the Administrator and capable of performing analytical measurements of all contaminants specified in the State primary drinking water regulations. Until such time as the Agency establishes a National quality assurance program for laboratory certification the Administrator will approve such State laboratories on an interim basis.

(5) The establishment and maintenance of an activity to assure that the design and construction of new or substantially modified public water system facilities will be capable of compliance with the State primary drinking water regulations.

(6) Statutory or regulatory enforcement authority adequate to compel compliance with the State primary drinking water regulations in appropriate cases, such authority to include:

(i) Authority to apply State primary drinking water regulations to all public water systems in the State covered by the national primary drinking water regulations, except for interstate carrier conveyances and systems on Indian land with respect to which the State does not have the necessary jurisdiction or its jurisdiction is in question.

(ii) Authority to sue in courts of competent jurisdiction to enjoin any threatened or continuing violation of the State primary drinking water regulations.

(iii) Right of entry and inspection of public water systems, including the right to take water samples, whether or not the State has evidence that the system is in violation of an applicable legal requirement.

(iv) Authority to require suppliers of water to keep appropriate records and make appropriate reports to the State.

(v) Authority to require public water systems to give public notice that is no less stringent than the EPA requirements in §§ 141.32 and 142.16(a).

(vi) Authority to assess civil or criminal penalties for violation of the State's primary drinking water regulations and public notification requirements, including the authority to assess daily penalties or multiple penalties when a violation continues;

(c) Has established and will maintain record keeping and reporting of its activities under paragraphs (a), (b) and (d) in compliance with §§ 142.14 and 142.15;

(d) If it permits variances or exemptions, or both, from the requirements of the State primary drinking water regulations, it shall do so under conditions and in a manner no less stringent than the requirements under sections 1415 and 1416 of the Act. In granting variances, the State must adopt the Administrator's findings of best available technology, treatment techniques, or other means available as specified in subpart G of this part. (States with primary enforcement responsibility may adopt procedures different from those set forth in subparts E and F of this part, which apply to the issuance of variances and exemptions by the Administrator in States that do not have primary enforcement responsibility, provided, that the State procedures meet the requirements of this paragraph); and

(e) Has adopted and can implement an adequate plan for the provision of safe drinking water under emergency circumstances.

(f) An Indian Tribe shall not be required to exercise criminal enforcement jurisdiction to meet the requirements for primary enforcement responsibility.

[41 FR 2918, Jan. 20, 1976, as amended at 43 FR 5373, Feb. 8, 1978; 52 FR 20675, June 2, 1987; 52 FR 41550, Oct. 28, 1987; 53 FR 37410, Sept. 26, 1988; 54 FR 15188, Apr. 17, 1989; 54 FR 52138, Dec. 20, 1989]

§142.11 Initial determination of primary enforcement responsibility.

(a) A State may apply to the Administrator for a determination that the State has primary enforcement responsibility for public water systems in the State pursuant to section 1413 of the Act. The application shall be as concise as possible and include a side-by-side comparison of the Federal requirements and the corresponding State authorities, including citations to the specific statutes and administrative regulations or ordinances and, wherever appropriate, judicial decisions which demonstrate adequate authority to meet the requirements of §142.10. The following information is to be included with the State application.

(1) The text of the State's primary drinking water regulations, with references to those State regulations that vary from comparable regulations set forth in part 141 of this chapter, and a demonstration that any different State regulation is at least as stringent as the comparable regulation contained in part 141.

(2) A description, accompanied by appropriate documentation, of the State's procedures for the enforcement of the State primary drinking water regulations. The submission shall include:

(i) A brief description of the State's program to maintain a current inventory of public water systems.

(ii) A brief description of the State's program for conducting sanitary surveys, including an explanation of the priorities given to various classes of public water systems.

(iii) A brief description of the State's laboratory approval or certification program, including the name(s) of the responsible State laboratory officer(s) certified by the Administrator.

(iv) Identification of laboratory facilities, available to the State, certified or approved by the Administrator and capable of performing analytical measurements of all contaminants specified in the State's primary drinking water regulations.

(v) A brief description of the State's program activity to assure that the design and construction of new or substantially modified public water system facilities will be capable of compliance with the requirements of the State primary drinking water regulations.

(vi) Copies of State statutory and regulatory provisions authorizing the adoption and enforcement of State primary drinking water regulations, and a brief description of State procedures for administrative or judicial action with respect to public water systems not in compliance with such regulations.

(3) A statement that the State will make such reports and will keep such records as may be required pursuant to §§142.14 and 142.15.

(4) If the State permits variances or exemptions from its primary drinking water regulations, the text of the State's statutory and regulatory provisions concerning variances and exemptions.

(5) A brief description of the State's plan for the provision of safe drinking water under emergency conditions.

NOTE: In satisfaction of this requirement, for public water supplies from groundwater sources, EPA will accept the contingency plan for providing alternate drinking water supplies that is part of a State's Wellhead Protection Program, where such program has been approved by EPA pursuant to section 1428 of the SDWA.

(6)(i) A statement by the State Attorney General (or the attorney for the State primacy agency if it has independent legal counsel) or the attorney representing the Indian tribe that certifies that the laws and regulations adopted by the State or tribal ordinances to carry out the program were duly adopted and are enforceable. State statutes and regulations cited by the State Attorney General and tribal ordinances cited by the attorney representing the Indian tribe shall be in the form of lawfully adopted State statutes and regulations or tribal ordinances at the time the certification is made and shall be fully effective by the time the program is approved by EPA. To qualify as ''independent legal counsel,'' the attorney signing the statement required by this section shall have full authority to independently represent the State primacy agency or Indian tribe in court on all matters pertaining to the State or tribal program.

(ii) After EPA has received the documents required under paragraph (a) of this section, EPA may selectively require supplemental statements by the State Attorney General (or the attorney for the State primacy agency if it has independent legal counsel) or the attorney representing the Indian tribe. Each supplemental statement shall address all issues concerning the adequacy of State authorities to meet the requirements of § 142.10 that have been identified by EPA after thorough examination as unresolved by the documents submitted under paragraph (a) of this section.

(b)(1) The administrator shall act on an application submitted pursuant to § 142.11 within 90 days after receiving such application, and shall promptly inform the State in writing of this action. If he denies the application, his written notification to the State shall include a statement of reasons for the denial.

(2) A final determination by the Administrator that a State has met or has not met the requirements for primary enforcement responsibility shall take effect in accordance with the public notice requirements and related procedures under § 142.13.

(3) When the Administrator's determination becomes effective pursuant to § 142.13, it shall continue in effect unless terminated pursuant to § 142.17.

[41 FR 2918, Jan. 20, 1976, as amended at 54 FR 52138, Dec. 20, 1989; 60 FR 33661, June 28, 1995]

§ 142.12 Revision of State programs.

(a) *General requirements.* Either EPA or the primacy State may initiate actions that require the State to revise its approved State primacy program. To retain primary enforcement responsibility, States must adopt all new and revised national primary drinking water regulations promulgated in part 141 of this chapter and any other requirements specified in this part.

(1) Whenever a State revises its approved primacy program to adopt new or revised Federal regulations, the State must submit a request to the Administrator for approval of the program revision, using the procedures described in paragraphs (b), (c), and (d) of this section. The Administrator shall approve or disapprove each State request for approval of a program revision based on the requirements of the Safe Drinking Water Act and of this part.

(2) For all State program revisions not covered under § 142.12(a)(1), the review procedures outlined in § 142.17(a) shall apply.

(b) *Timing of State requests for approval of program revisions to adopt new or revised Federal regulations.* (1) Complete and final State requests for approval of program revisions to adopt new or revised EPA regulations must be submitted to the Administrator within 18 months after promulgation of the new or revised EPA regulations, unless the State requests an extension and the Administrator has approved the request pursuant to paragraph (b)(2) of this section. If the State expects to submit a final State request for approval of a program revision to EPA more than 18 months after promulgation of the new or revised EPA regulations, the State shall request an extension of the deadline before the expiration of the 18-month period.

(2) The final date for submission of a complete and final State request for a program revision may be extended by EPA for up to a two-year period upon a written application by the State to the Administrator. In the extension application the State must demonstrate it is requesting the extension because it cannot meet the original deadline for reasons beyond its control despite a good faith effort to do so. The application must include a schedule for the submission of a final request by a certain time and provide sufficient information to demonstrate that the State:

(i)(A) Currently lacks the legislative or regulatory authority to enforce the new or revised requirements, or

(B) Currently lacks the program capability adequate to implement the new or revised requirements; or

(C) Is requesting the extension to group two or more program revisions in a single legislative or regulatory action; and

(ii) Is implementing the EPA requirements to be adopted by the State in its program revision pursuant to paragraph (b)(3) of this section within the

scope of its current authority and capabilities.

(3) To be granted an extension, the State must agree with EPA to meet certain requirements during the extension period, which may include the following types of activities as determined appropriate by the Administrator on a case-by-case basis:

(i) Informing public water systems of the new EPA (and upcoming State) requirements and that EPA will be overseeing implementation of the requirements until EPA approves the State program revision;

(ii) Collecting, storing and managing laboratory results, public notices, and other compliance and operation data required by the EPA regulations;

(iii) Assisting EPA in the development of the technical aspects of enforcement actions and conducting informal follow-up on violations (telephone calls, letters, etc.);

(iv) Providing technical assistance to public water systems;

(v) Providing EPA with all information prescribed by § 142.15 of this part on State reporting; and

(vi) For States whose request for an extension is based on a current lack of program capability adequate to implement the new requirements, taking steps agreed to by EPA and the State during the extension period to remedy the deficiency.

(c) *Contents of a State request for approval of a program revision.* (1) The State request for EPA approval of a program revision shall be concise and must include:

(i) The documentation necessary (pursuant to § 142.11(a)) to update the approved State primacy program, and identification of those elements of the approved State primacy program that have not changed because of the program revision. The documentation shall include a side-by-side comparison of the Federal requirements and the corresponding State authorities, including citations to the specific statutes and administrative regulations or ordinances and, wherever appropriate, judicial decisions which demonstrate adequate authority to meet the requirements of § 142.10 as they apply to the program revision.

(ii) Any additional materials that are listed in § 142.16 of this part for a specific EPA regulation, as appropriate; and

(iii) For a complete and final State request only, unless one of the conditions listed in paragraph (c)(2) of this section are met, a statement by the State Attorney General (or the attorney for the State primacy agency if it has independent legal counsel) or the attorney representing the Indian tribe that certifies that the laws and regulations adopted by the State or tribal ordinances to carry out the program revision were duly adopted and are enforceable. State statutes and regulations cited by the State Attorney General and tribal ordinances cited by the attorney for the Indian tribe shall be in the form of lawfully adopted State statutes and regulations or tribal ordinances at the time the certification is made and shall be fully effective by the time the request for program revision is approved by EPA. To qualify as ''independent legal counsel,'' the attorney signing the statement required by this section shall have full authority to independently represent the State primacy agency or tribe in court on all matters pertaining to the State or tribal program.

(2) An Attorney General's statement will be required as part of the State request for EPA approval of a program revision unless EPA specifically waives this requirement for a specific regulation at the time EPA promulgates the regulation, or by later written notice from the Administrator to the State.

(3) After EPA has received the documents required under paragraph (c)(1) of this section, EPA may selectively require supplemental statements by the State Attorney General (or the attorney for the State primacy agency if it has independent legal counsel) or the attorney representing the Indian tribe. Each supplemental statement shall address all issues concerning the adequacy of State authorities to meet the requirements of § 142.10 that have been identified by EPA after thorough examination as unresolved by the documents submitted under paragraph (c)(1) of this section.

(d) *Procedures for review of a State request for approval of a program revision—*

(1) *Preliminary request.* (i) The State may submit to the Administrator for his or her review a preliminary request for approval of each program revision, containing the information listed in paragraph (c)(1) of this section, *in draft form.* The preliminary request does not require an Attorney General's statement in draft form, but does require draft State statutory or regulatory changes and a side-by-side comparison of State authorities with EPA requirements to demonstrate that the State program revision meets EPA requirements under § 142.10 of this part. The preliminary request should be submitted to the Administrator as soon as practicable after the promulgation of the EPA regulations.

(ii) The Administrator will review the preliminary request submitted in accordance with paragraph (d)(1)(i) of this section and make a tentative determination on the request. The Administrator will send the tentative determination and other comments or suggestions to the State for its use in developing the State's final request under paragraph (d)(2) of this section.

(2) *Final request.* The State must submit a complete and final request for approval of a program revision to the Administrator for his or her review and approval. The request must contain the information listed in paragraph (c)(1) of this section *in complete and final form,* in accordance with any tentative determination EPA may have issued. Complete and final State requests for program revisions shall be submitted within 18 months of the promulgation of the new or revised EPA regulations, as specified in paragraph (b) of this section.

(3) *EPA's determination on a complete and final request.* (i) The Administrator shall act on a State's request for approval of a program revision within 90 days after determining that the State request is complete and final and shall promptly notify the State of his/her determination.

(ii) If the Administrator disapproves a final request for approval of a program revision, the Administrator will notify the State in writing. Such notification will include a statement of the reasons for disapproval.

(iii) A final determination by the Administrator on a State's request for approval of a program revision shall take effect in accordance with the public notice requirements and related procedures under § 142.13.

[54 FR 52138, Dec. 20, 1989]

§ 142.13 Public hearing.

(a) The Administrator shall provide an opportunity for a public hearing before a final determination pursuant to § 142.11 that the State meets or does not meet the requirements for obtaining primary enforcement responsibility, or a final determination pursuant to § 142.12(d)(3) to approve or disapprove a State request for approval of a program revision, or a final determination pursuant to § 142.17 that a State no longer meets the requirements for primary enforcement responsibility.

(b) The Administrator shall publish notice of any determination specified in paragraph (a) of this section in the FEDERAL REGISTER and in a newspaper or newspapers of general circulation in the State involved within 15 days after making such determination, with a statement of his reasons for the determination. Such notice shall inform interested persons that they may request a public hearing on the Administrator's determination. Such notice shall also indicate one or more locations in the State where information submitted by the State pursuant to § 142.11 is available for inspection by the general public. A public hearing may be requested by any interested person other than a Federal agency. Frivolous or insubstantial requests for hearing may be denied by the Administrator.

(c) Requests for hearing submitted pursuant to paragraph (b) of this section shall be submitted to the Administrator within 30 days after publication of notice of opportunity for hearing in the FEDERAL REGISTER. Such requests shall include the following information:

(1) The name, address and telephone number of the individual, organization or other entity requesting a hearing.

(2) A brief statement of the requesting person's interest in the Administrator's determination and of information that the requesting person intends to submit at such hearing.

(3) The signature of the individual making the request; or, if the request is made on behalf of an organization or other entity, the signature of a responsible official of the organization or other entity.

(d) The Administrator shall give notice in the FEDERAL REGISTER and in a newspaper or newspapers of general circulation in the State involved of any hearing to be held pursuant to a request submitted by an interested person or on his own motion. Notice of the hearing shall also be sent to the person requesting a hearing, if any, and to the State involved. Notice of the hearing shall include a statement of the purpose of the hearing, information regarding the time and location or locations for the hearing and the address and telephone number of an office at which interested persons may obtain further information concerning the hearing. At least one hearing location specified in the public notice shall be within the involved State. Notice of hearing shall be given not less than 15 days prior to the time scheduled for the hearing.

(e) Hearings convened pursuant to paragraph (d) of this section shall be conducted before a hearing officer to be designated by the Administrator. The hearing shall be conducted by the hearing officer in an informal, orderly and expeditious manner. The hearing officer shall have authority to call witnesses, receive oral and written testimony and take such other action as may be necessary to assure the fair and efficient conduct of the hearing. Following the conclusion of the hearing, the hearing officer shall forward the record of the hearing to the Administrator.

(f) After reviewing the record of the hearing, the Administrator shall issue an order affirming the determination referred to in paragraph (a) of this section or rescinding such determination. If the determination is affirmed, it shall become effective as of the date of the Administrator's order.

(g) If no timely request for hearing is received and the Administrator does not determine to hold a hearing on his own motion, the Administrator's determination shall become effective 30 days after notice is issued pursuant to paragraph (b) of this section.

(h) If a determination of the Administrator that a State no longer meets the requirements for primary enforcement responsibility becomes effective, the State may subsequently apply for a determination that it meets such requirements by submitting to the Administrator information demonstrating that it has remedied the deficiencies found by the Administrator without adversely sacrificing other aspects of its program required for primary enforcement responsibility.

[41 FR 2918, Jan. 20, 1976, as amended at 54 FR 52140, Dec. 20, 1989; 60 FR 33661, June 28, 1995]

§ 142.14 Records kept by States.

(a) Each State which has primary enforcement responsibility shall maintain records of tests, measurements, analyses, decisions, and determinations performed on each public water system to determine compliance with applicable provisions of State primary drinking water regulations.

(1) Records of microbiological analyses shall be retained for not less than 1 year. Actual laboratory reports may be kept or data may be transferred to tabular summaries, provided that the information retained includes:

(i) The analytical method used;

(ii) The number of samples analyzed each month;

(iii) The analytical results, set forth in a form which makes possible comparison with the limits specified in §§ 141.63, 141.71, and 141.72 of this chapter.

(2) Records of microbiological analyses of repeat or special samples shall be retained for not less than one year in the form of actual laboratory reports or in an appropriate summary form.

(3) Records of turbidity measurements shall be kept for not less than one year. The information retained must be set forth in a form which makes possible comparison with the limits specified in §§ 141.71 and 141.73 of this chapter. Until June 29, 1993, for any public water system which is providing filtration treatment and until December 30, 1991, for any public water system not providing filtration treatment and not required by the State to

provide filtration treatment, records kept must be set forth in a form which makes possible comparison with the limits contained in § 141.13.

(i) Date and place of sampling.

(ii) Date and results of analyses.

(4)(i) Records of disinfectant residual measurements and other parameters necessary to document disinfection effectiveness in accordance with §§ 141.72 and 141.74 of this chapter and the reporting requirements of § 141.75 of this chapter shall be kept for not less than one year.

(ii) Records of decisions made on a system-by-system and case-by-case basis under provisions of part 141, subpart H, shall be made in writing and kept at the State.

(A) Records of decisions made under the following provisions shall be kept for 40 years (or until one year after the decision is reversed or revised) and a copy of the decision must be provided to the system:

(*1*) Section 141.73(a)(1)—Any decision to allow a public water system using conventional filtration treatment or direct filtration to substitute a turbidity limit greater than 0.5 NTU;

(*2*) Section 141.73(b)(1)—Any decision to allow a public water system using slow sand filtration to substitute a turbidity limit greater than 1 NTU;

(*3*) Section 141.74(b)(2)—Any decision to allow an unfiltered public water system to use continuous turbidity monitoring;

(*4*) Section 141.74(b)(6)(i)—Any decision to allow an unfiltered public water system to sample residual disinfectant concentration at alternate locations if it also has ground water source(s);

(*5*) Section 141.74(c)(1)—Any decision to allow a public water system using filtration treatment to use continuous turbidity monitoring; or a public water system using slow sand filtration or filtration treatment other than conventional treatment, direct filtration or diatomaceous earth filtration to reduce turbidity sampling to once per day; or for systems serving 500 people or fewer to reduce turbidity sampling to once per day;

(*6*) Section 141.74(c)(3)(i)—Any decision to allow a filtered public water system to sample disinfectant residual concentration at alternate locations if it also has ground water source(s);

(*7*) Section 141.75(a)(2)(ix)—Any decision to allow reduced reporting by an unfiltered public water system; and

(*8*) Section 141.75(b)(2)(iv)—Any decision to allow reduced reporting by a filtered public water system.

(B) Records of decisions made under the following provisions shall be kept for one year after the decision is made:

(*1*) Section 141.71(b)(1)(i)—Any decision that a violation of monthly CT compliance requirements was caused by circumstances that were unusual and unpredictable.

(*2*) Section 141.71(b)(1)(iv)—Any decision that a violation of the disinfection effectiveness criteria was not caused by a deficiency in treatment of the source water;

(*3*) Section 141.71(b)(5)—Any decision that a violation of the total coliform MCL was not caused by a deficiency in treatment of the source water;

(*4*) Section 141.74(b)(1)—Any decision that total coliform monitoring otherwise required because the turbidity of the source water exceeds 1 NTU is not feasible, except that if such decision allows a system to avoid monitoring without receiving State approval in each instance, records of the decision shall be kept until one year after the decision is rescinded or revised.

(C) Records of decisions made under the following provisions shall be kept for the specified period or 40 years, whichever is less.

(*1*) Section 141.71(a)(2)(i)—Any decision that an event in which the source water turbidity which exceeded 5 NTU for an unfiltered public water system was unusual and unpredictable shall be kept for 10 years.

(*2*) Section 141.71(b)(1)(iii)—Any decision by the State that failure to meet the disinfectant residual concentration requirements of § 141.72(a)(3)(i) was caused by circumstances that were unusual and unpredictable, shall be kept unless filtration is installed. A copy of the decision must be provided to the system.

(*3*) Section 141.71(b)(2)—Any decision that a public water system's watershed control program meets the requirements of this section shall be kept

until the next decision is available and filed.

(*4*) Section 141.70(c)—Any decision that an individual is a qualified operator for a public water system using a surface water source or a ground water source under the direct influence of surface water shall be maintained until the qualification is withdrawn. The State may keep this information in the form of a list which is updated periodically. If such qualified operators are classified by category, the decision shall include that classification.

(*5*) Section 141.71(b)(3)—Any decision that a party other than the State is approved by the State to conduct on-site inspections shall be maintained until withdrawn. The State may keep this information in the form of a list which is updated periodically.

(*6*) Section 141.71(b)(4)—Any decision that an unfiltered public water system has been identified as the source of a waterborne disease outbreak, and, if applicable, that it has been modified sufficiently to prevent another such occurrence shall be kept until filtration treatment is installed. A copy of the decision must be provided to the system.

(*7*) Section 141.72—Any decision that certain interim disinfection requirements are necessary for an unfiltered public water system for which the State has determined that filtration is necessary, and a list of those requirements, shall be kept until filtration treatment is installed. A copy of the requirements must be provided to the system.

(*8*) Section 141.72(a)(2)(ii)—Any decision that automatic shut-off of delivery of water to the distribution system of an unfiltered public water system would cause an unreasonable risk to health or interfere with fire protection shall be kept until rescinded.

(*9*) Section 141.72(a)(4)(ii)—Any decision by the State, based on site-specific considerations, that an unfiltered system has no means for having a sample transported and analyzed for HPC by a certified laboratory under the requisite time and temperature conditions specified by § 141.74(a)(3) and that the system is providing adequate disinfection in the distribution system, so that the disinfection requirements contained in § 141.72(a)(4)(i) do not apply, and the basis for the decision, shall be kept until the decision is reversed or revised. A copy of the decision must be provided to the system.

(*10*) Section 141.72(b)(3)(ii)—Any decision by the State, based on site-specific conditions, that a filtered system has no means for having a sample transported and analyzed for HPC by a certified laboratory under the requisite time and temperature conditions specified by § 141.74(a)(3) and that the system is providing adequate disinfection in the distribution system, so that the disinfection requirements contained in § 141.72(b)(3)(i) do not apply, and the basis for the decision, shall be kept until the decision is reversed or revised. A copy of the decision must be provided to the system.

(*11*) Section 141.73(d)—Any decision that a public water system, having demonstrated to the State that an alternative filtration technology, in combination with disinfection treatment, consistently achieves 99.9 percent removal and/or inactivation of *Giardia lamblia* cysts and 99.99 percent removal and/or inactivation of viruses, may use such alternative filtration technology, shall be kept until the decision is reversed or revised. A copy of the decision must be provided to the system.

(*12*) Section 141.74(b), table 3.1—Any decision that a system using either preformed chloramines or chloramines formed by the addition of ammonia prior to the addition of chlorine has demonstrated that 99.99 percent removal and/or inactivation of viruses has been achieved at particular CT values, and a list of those values, shall be kept until the decision is reversed or revised. A copy of the list of required values must be provided to the system.

(*13*) Section 141.74(b)(3)(v)—Any decision that a system using a disinfectant other than chlorine may use $CT_{99.9}$ values other than those in tables 2.1 or 3.1 and/or other operational parameters to determine if the minimum total inactivation rates required by § 141.72(a)(1) are being met, and what those values or parameters are, shall be kept until the decision is reversed or revised. A copy of the list of required values or

parameters must be provided to the system.

(*14*) Section 142.16(b)(2)(i)(B)—Any decision that a system using a ground water source is under the direct influence of surface water.

(iii) Records of any determination that a public water system supplied by a surface water source or a ground water source under the direct influence of surface water is not required to provide filtration treatment shall be kept for 40 years or until withdrawn, whichever is earlier. A copy of the determination must be provided to the system.

(5) Records of each of the following decisions made pursuant to the total coliform provisions of part 141 shall be made in writing and retained by the State.

(i) Records of the following decisions must be retained for 5 years.

(A) Section 141.21(b)(1)—Any decision to waive the 24-hour time limit for collecting repeat samples after a total coliform-positive routine sample if the public water system has a logistical problem in collecting the repeat sample that is beyond the system's control, and what alternative time limit the system must meet.

(B) Section 141.21(b)(5)—Any decision to allow a system to waive the requirement for five routine samples the month following a total coliform-positive sample. If the waiver decision is made as provided in § 141.21(b)(5), the record of the decision must contain all the items listed in that paragraph.

(C) Section 141.21(c)—Any decision to invalidate a total coliform-positive sample. If the decision to invalidate a total coliform-positive sample as provided in § 141.21(c)(1)(iii) is made, the record of the decision must contain all the items listed in that paragraph.

(ii) Records of each of the following decisions must be retained in such a manner so that each system's current status may be determined.

(A) Section 141.21(a)(2)—Any decision to reduce the total coliform monitoring frequency for a community water system serving 1,000 persons or fewer, that has no history of total coliform contamination in its current configuration and had a sanitary survey conducted within the past five years showing that the system is supplied solely by a protected groundwater source and is free of sanitary defects, to less than once per month, as provided in § 141.21(a)(2); and what the reduced monitoring frequency is. A copy of the reduced monitoring frequency must be provided to the system.

(B) Section 141.21(a)(3)(i)—Any decision to reduce the total coliform monitoring frequency for a non-community water system using only ground water and serving 1,000 persons or fewer to less than once per quarter, as provided in § 141.21(a)(3)(i), and what the reduced monitoring frequency is. A copy of the reduced monitoring frequency must be provided to the system.

(C) Section 141.21(a)(3)(ii)—Any decision to reduce the total coliform monitoring frequency for a non-community water system using only ground water and serving more than 1,000 persons during any month the system serves 1,000 persons or fewer, as provided in § 141.21(a)(3)(ii). A copy of the reduced monitoring frequency must be provided to the system.

(D) Section 141.21(a)(5)—Any decision to waive the 24-hour limit for taking a total coliform sample for a public water system which uses surface water, or ground water under the direct influence of surface water, and which does not practice filtration in accordance with part 141, subpart H, and which measures a source water turbidity level exceeding 1 NTU near the first service connection as provided in § 141.21(a)(5).

(E) Section 141.21(d)(1)—Any decision that a non-community water system is using only protected and disinfected ground water and therefore may reduce the frequency of its sanitary survey to less than once every five years, as provided in § 141.21(d), and what that frequency is. A copy of the reduced frequency must be provided to the system.

(F) Section 141.21(d)(2)—A list of agents other than the State, if any, approved by the State to conduct sanitary surveys.

(G) Section 141.21(e)(2)—Any decision to allow a public water system to forgo fecal coliform or *E. coli* testing on a total coliform-positive sample if that system assumes that the total coliform-positive sample is fecal coliform-

positive or *E. coli*-positive, as provided in § 141.21(e)(2).

(6) Records of analysis for other than microbiological contaminants (including total coliform, fecal coliform, and heterotrophic plate count), residual disinfectant concentration, other parameters necessary to determine disinfection effectiveness (including temperature and pH measurements), and turbidity shall be retained for not less than 12 years and shall include at least the following information:

(i) Date and place of sampling.

(ii) Date and results of analyses.

(b) Records required to be kept pursuant to paragraph (a) of this section must be in a form admissible as evidence in State enforcement proceedings.

(c) Each State which has primary enforcement responsibility shall maintain current inventory information for every public water system in the State and shall retain inventory records of public water systems for not less than 12 years.

(d) Each State which has primary enforcement responsibility shall retain, for not less than 12 years, files which shall include for each such public water system in the State:

(1) Reports of sanitary surveys;

(2) Records of any State approvals;

(3) Records of any enforcement actions.

(4) A record of the most recent vulnerability determination, including the monitoring results and other data supporting the determination, the State's findings based on the supporting data and any additional bases for such determination; except that it shall be kept in perpetuity or until a more current vulnerability determination has been issued.

(5) A record of all current monitoring requirements and the most recent monitoring frequency decision pertaining to each contaminant, including the monitoring results and other data supporting the decision, the State's findings based on the supporting data and any additional bases for such decision; except that the record shall be kept in perpetuity or until a more recent monitoring frequency decision has been issued.

(6) A record of the most recent asbestos repeat monitoring determination, including the monitoring results and other data supporting the determination, the State's findings based on the supporting data and any additional bases for the determination and the repeat monitoring frequency; except that these records shall be maintained in perpetuity or until a more current repeat monitoring determination has been issued.

(7) Records of annual certifications received from systems pursuant to part 141, subpart K demonstrating the system's compliance with the treatment techniques for acrylamide and/or epichlorohydrin in § 14.111.

(8) Records of the currently applicable or most recent State determinations, including all supporting information and an explanation of the technical basis for each decision, made under the following provisions of 40 CFR, part 141, subpart I for the control of lead and copper:

(i) Section 141.82(b)—decisions to require a water system to conduct corrosion control treatment studies;

(ii) Section 141.82(d)—designations of optimal corrosion control treatment;

(iii) Section 141.82(f)—designations of optimal water quality parameters;

(iv) Section 141.82(h)—decisions to modify a public water system's optimal corrosion control treatment or water quality parameters;

(v) Section 141.83(b)(2)—determinations of source water treatment; and

(vi) Section 141.83(b)(4)—designations of maximum permissible lead and copper concentrations in source water.

(vii) Section 141.84(e)—determinations that a system does not control entire lead service lines.

(viii) Section 141.84(f)—determinations establishing a shorter lead service line replacement schedule than required by § 141.84.

(9) Records of reports and any other information submitted by PWSs under § 141.90;

(10) Records of state activities, and the results thereof, to verify compliance with State determinations issued under §§ 141.82(f), 141.82(h), 141.83(b)(2), and 141.83(b)(4) and compliance with lead service line replacement schedules under § 141.84.

(11) Records of each system's currently applicable or most recently designated monitoring requirements. If, for the records identified in §§ 142.14(d)(8)(i) through 142.14(d)(8)(viii) above, no change is made to State decision during a 12 year retention period, the State shall maintain the record until a new decision, determination or designation has been issued.

(e) Each State which has primary enforcement responsibility shall retain records pertaining to each variance and exemption granted by it for a period of not less than 5 years following the expiration of such variance or exemption.

(f) Records required to be kept under this section shall be available to the Regional Administrator upon request. The records required to be kept under this section shall be maintained and made available for public inspection by the State, or, the State at its option may require suppliers of water to make available for public inspection those records maintained in accordance with § 141.33.

[41 FR 2918, Jan. 20, 1976, as amended at 54 FR 27537, June 29, 1989; 55 FR 25065, June 19, 1990; 56 FR 3595, Jan. 30, 1991; 56 FR 26562, June 7, 1991]

§ 142.15 Reports by States.

Each State which has primary enforcement responsibility shall submit to the Administrator the following information:

(a) Each State which has primary enforcement responsibility shall submit quarterly reports to the Administrator on a schedule and in a format prescribed by the Administrator, consisting of the following information:

(1) New violations by public water systems in the State during the previous quarter of State regulations adopted to incorporate the requirements of national primary drinking water regulations;

(2) New enforcement actions taken by the State during the previous quarter against public water systems with respect to State regulations adopted to incorporate the requirements of national primary drinking water regulations;

(3) Notification of any new variance or exemption granted during the previous quarter. The notice shall include a statement of reasons for the granting of the variance or exemption, including documentation of the need for the variance or exemption and the finding that the granting of the variance or exemption will not result in an unreasonable risk to health. The State may use a single notification statement to report two or more similar variances or exemptions.

(b) Each State which has primary enforcement responsibility shall submit annual reports to the Administrator on a schedule and in a format prescribed by the Administrator, consisting of the following information:

(1) All additions or corrections to the State's inventory of public water systems;

(2) A summary of the status of each variance and exemption currently in effect.

(c) *Special reports.* (1) *Surface Water Treatment Rule.* (i)(A) A list identifying the name, PWS identification number and date of the determination for each public water system supplied by a surface water source or a ground water source under the direct influence of surface water, which the State has determined is not required to provide filtration treatment.

(B) A list identifying the name and PWS identification number of each public water system supplied by a surface water source or ground water source under the direct influence of surface water, which the State has determined, based on an evaluation of site-specific considerations, has no means of having a sample transported and analyzed for HPC by a certified laboratory under the requisite time and temperature conditions specified in § 141.74(a)(3) and is providing adequate disinfection in the distribution system, regardless of whether the system is in compliance with the criteria of § 141.72 (a)(4)(i) or (b)(3)(i) of this chapter, as allowed by § 141.72 (a)(4)(ii) and (b)(3)(ii). The list must include the effective date of each determination.

(ii) Notification within 60 days of the end of the calendar quarter of any determination that a public water system using a surface water source or a ground water source under the direct influence of surface water is not required to provide filtration treatment.

The notification must include a statement describing the system's compliance with each requirement of the State's regulations that implement §141.71 and a summary of comments, if any, received from the public on the determination. A single notification may be used to report two or more such determinations.

(2) *Total coliforms.* A list of public water systems which the State is allowing to monitor less frequently than once per month for community water systems or less frequently than once per quarter for non-community water systems as provided in §141.21(a), including the effective date of the reduced monitoring requirement for each system.

(3) The results of monitoring for unregulated contaminants shall be reported quarterly.

(4) States shall report to EPA by May 15, August 15, November 15 and February 15 of each year the following information related to each system's compliance with the treatment techniques for lead and copper under 40 CFR part 141, subpart I during the preceding calendar quarter. Specifically, States shall report the name and PWS identification number of:

(i) Each public water system which exceeded the lead and copper action levels and the date upon which the exceedance occurred;

(ii) Each public water system required to complete the corrosion control evaluation specified in §141.82(c) and the date the State received the results of the evaluations from each system;

(iii) Each public water system for which the State has designated optimal corrosion control treatment under §141.82(d), the date of the determination, and each system that completed installation of treatment as certified under §141.90(c)(3);

(iv) Each public water system for which the State has designated optimal water quality parameters under §141.82(f) and the date of the determination;

(v) Each public water system which the State has required to install source water treatment under §141.83(b)(2), the date of the determination, and each system that completed installation of treatment as certified under §141.90(d)(2);

(vi) Each public water system for which the State has specified maximum permissible source water levels under §141.83(b)(4); and

(vii) Each public water system required to begin replacing lead service lines as specified in §141.84, each public water system for which the State has established a replacement schedule under §141.84(f), and each system reporting compliance with its replacement schedule under §141.90(e)(2).

(d) The reports submitted pursuant to this section shall be made available by the State to the public for inspection at one or more locations within the State.

[41 FR 2918, Jan. 20, 1976, as amended at 43 FR 5373, Feb. 8, 1978; 54 FR 27539, June 29, 1989; 55 FR 52140, Dec. 20, 1989; 55 FR 25065, June 19, 1990; 56 FR 3595, Jan. 30, 1991; 56 FR 26562, June 7, 1991]

§142.16 Special primacy requirements.

(a) *State public notification requirements.* If a State exercises the option specified in §141.32(b)(4) to authorize less frequent notice for minor monitoring violations, it must adopt a program revision enforceable under State authorities which promulgates rules specifying either: (1) Which monitoring violations are minor and the frequency of public notification for such violations; or (2) criteria for determining which monitoring violations are minor and the frequency of public notification.

(b) *Requirements for States to adopt 40 CFR part 141, subpart H Filtration and Disinfection.* In addition to the general primacy requirements enumerated elsewhere in this part, including the requirement that State provisions are no less stringent than the federal requirements, an application for approval of a State program revision that adopts 40 CFR part 141, subpart H Filtration and Disinfection, must contain the information specified in this paragraph (b), except that States which require without exception all public water systems using a surface water source or a ground water source under the direct influence of surface water to provide filtration need not demonstrate that the State program has provisions that

apply to systems which do not provide filtration treatment. However, such States must provide the text of the State statutes or regulations which specifies that all public water systems using a surface water source or a ground water source under the direct influence of surface water must provide filtration.

(1) *Enforceable requirements.* In addition to adopting criteria no less stringent than those specified in part 141, subpart H of this chapter, the State's application must include enforceable design and operating criteria for each filtration treatment technology allowed or a procedure for establishing design and operating conditions on a system-by-system basis (e.g., a permit system).

(2) *State practices or procedures.* (i) A State application for program revision approval must include a description of how the State will accomplish the following:

(A) Section 141.70(c) (qualification of operators)—Qualify operators of systems using a surface water source or a ground water source under the direct influence of surface water.

(B) Determine which systems using a ground water source are under the direct influence of surface water by June 29, 1994 for community water systems and by June 29, 1999 for non-community water systems.

(C) Section 141.72(b)(1) (achieving required *Giardia lamblia* and virus removal in filtered systems)—Determine that the combined treatment process incorporating disinfection treatment and filtration treatment will achieve the required removal and/or inactivation of *Giardia lamblia* and viruses.

(D) Section 141.74(a) (State approval of parties to conduct analyses)—approve parties to conduct pH, temperature, turbidity, and residual disinfectant concentration measurements.

(E) Determine appropriate filtration treatment technology for source waters of various qualities.

(ii) For a State which does not require all public water systems using a surface water source or ground water source under the direct influence of surface water to provide filtration treatment, a State application for program revision approval must include a description of how the State will accomplish the following:

(A) Section 141.71(b)(2) (watershed control program)—Judge the adequacy of watershed control programs.

(B) Section 141.71(b)(3) (approval of on-site inspectors)—Approve on-site inspectors other than State personnel and evaluate the results of on-site inspections.

(iii) For a State which adopts any of the following discretionary elements of part 141 of this chapter, the application must describe how the State will:

(A) Section 141.72 (interim disinfection requirements)—Determine interim disinfection requirements for unfiltered systems which the State has determined must filter which will be in effect until filtration is installed.

(B) Section 141.72 (a)(4)(ii) and (b)(3)(ii) (determination of adequate disinfection in system without disinfectant residual)—Determine that a system is unable to measure HPC but is still providing adequate disinfection in the distribution system, as allowed by § 141.72(a)(4)(ii) for systems which do not provide filtration treatment and § 141.72(b)(3)(ii) for systems which do provide filtration treatment.

(C) Section 141.73 (a)(1) and (b)(1) (alternative turbidity limit)—Determine whether an alternative turbidity limit is appropriate and what the level should be as allowed by § 141.73(a)(1) for a system using conventiona1 filtration treatment or direct filtration and by § 141.73(b)(1) for a system using slow sand filtration.

(D) Section 141.73(d) (alternative filtration technologies)—Determine that a public water system has demonstrated that an alternate filtration technology, in combination with disinfection treatment, achieves adequate removal and/or disinfection of *Giardia lamblia* and viruses.

(E) Section 141.74(a)(5) (alternate analytical method for chlorine)—Approve DPD colorimetric test kits for free and combined chlorine measurement or approve calibration of automated methods by the Indigo Method for ozone determination.

(F) Section 141.74 (b)(2) and (c)(1) (approval of continuous turbidity monitoring)—Approve continuous turbidity monitoring, as allowed by § 141.74(b)(2)

for a public water system which does not provide filtration treatment and § 141.74(c)(1) for a system which does provide filtration treatment.

(G) Section 141.74 (b)(6)(i) and (c)(3)(i) (approval of alternate disinfectant residual concentration sampling plans)—Approve alternate disinfectant residual concentration sampling plans for systems which have a combined ground water and surface water or ground water and ground water under the direct influence of a surface water distribution system, as allowed by § 141.74(b)(6)(i) for a public water system which does not provide filtration treatment and § 141.74(c)(3)(i) for a public water system which does provide filtration treatment.

(H) Section 141.74(c)(1) (reduction of turbidity monitoring)—Decide whether to allow reduction of turbidity monitoring for systems using slow sand filtration, an approved alternate filtration technology or serving 500 people or fewer.

(I) Section 141.75 (a)(2)(ix) and (b)(2)(iv) (reduced reporting)—Determine whether reduced reporting is appropriate, as allowed by § 141.75(a)(2)(ix) for a public water system which does not provide filtration treatment and § 141.75(b)(2)(iv) for a public water system which does provide filtration treatment.

(iv) For a State which does not require all public water systems using a surface water source or ground water source under the direct influence of surface water to provide filtration treatment and which uses any of the following discretionary provisions, the application must describe how the State will:

(A) Section 141.71(a)(2)(i) (source water turbidity requirements)—Determine that an exceedance of turbidity limits in source water was caused by circumstances that were unusual and unpredictable.

(B) Section 141.71(b)(1)(i) (monthly CT compliance requirements)—Determine whether failure to meet the requirements for monthly CT compliance in § 141.72(a)(1) was caused by circumstances that were unusual and unpredictable.

(C) Section 141.71(b)(1)(iii) (residual disinfectant concentration requirements)—Determine whether failure to meet the requirements for residual disinfectant concentration entering the distribution system in § 141.72(a)(3)(i) was caused by circumstances that were unusual and unpredictable.

(D) Section 141.71(b)(1)(iv) (distribution system disinfectant residual concentration requirements)—Determine whether failure to meet the requirements for distribution system residual disinfectant concentration in § 141.72(a)(4) was related to a deficiency in treatment.

(E) Section 141.71(b)(4) (system modification to prevent waterborne disease outbreak)—Determine that a system, after having been identified as the source of a waterborne disease outbreak, has been modified sufficiently to prevent another such occurrence.

(F) Section 141.71(b)(5) (total coliform MCL)—Determine whether a total coliform MCL violation was caused by a deficiency in treatment.

(G) Section 141.72(a)(1) (disinfection requirements)—Determine that different ozone, chloramine, or chlorine dioxide $CT_{99.9}$ values or conditions are adequate to achieve required disinfection.

(H) Section 141.72(a)(2)(ii) (shut-off of water to distribution system)—Determine whether a shut-off of water to the distribution system when the disinfectant residual concentration entering the distribution system is less than 0.2 mg/1 will cause an unreasonable risk to health or interfere with fire protection.

(I) Section 141.74(b)(1) (coliform monitoring)—Determine that coliform monitoring which otherwise might be required is not feasible for a system.

(J) Section 141.74(b), table 3.1 (disinfection with chloramines)—Determine the conditions to be met to insure 99.99 percent removal and/or inactivation of viruses in systems which use either preformed chloramines or chloramines for which ammonia is added to the water before chlorine, as allowed by table 3.1.

(c) *Total coliform requirements.* In addition to meeting the general primacy requirements of this part, an application for approval of a State program revision that adopts the requirements of the national primary drinking water

regulation for total coliforms must contain the following information:

(1) The application must describe the State's plan for determining whether sample siting plans are acceptable (including periodic reviews), as required by § 141.21(a)(1).

(2) The national primary drinking water regulation for total coliforms in part 141 gives States the option to impose lesser requirements in certain circumstances, which are listed below. If a State chooses to exercise any of these options, its application for approval of a program revision must include the information listed below (the State need only provide the information listed for those options it has chosen to use).

(i) Section 141.21(a)(2) (Reduced monitoring requirements for community water systems serving 1,000 or fewer persons)—A description of how the State will determine whether it is appropriate to reduce the total coliform monitoring frequency for such systems using the criteria in § 141.21(a)(2) and how it will determine the revised frequency.

(ii) Section 141.21(a)(3)(i) (Reduced monitoring requirements for non-community water systems using ground water and serving 1,000 persons or fewer)—A description of how the State will determine whether it is appropriate to reduce the total coliform monitoring frequency for such systems using the criteria in § 141.21(a)(3)(i) and how it will determine the revised frequency.

(iii) Section 141.21(a)(3)(ii) (Reduced monitoring for non-community water systems using ground water and serving more than 1,000 persons)—A description of how the State will determine whether it is appropriate to reduce the total coliform monitoring frequency for non-community water systems using only ground water and serving more than 1,000 persons during any month the system serves 1,000 persons or fewer and how it will determine the revised frequency.

(iv) Section 141.21(a)(5) (Waiver of time limit for sampling after a turbidity sampling result exceeds 1 NTU)—A description of how the State will determine whether it is appropriate to waive the 24-hour time limit.

(v) Section 141.21(b)(1) (Waiver of time limit for repeat samples)—A description of how the State will determine whether it is appropriate to waive the 24-hour time limit and how it will determine what the revised time limit will be.

(vi) Section 141.21(b)(3) (Alternative repeat monitoring requirements for systems with a single service connection)—A description of how the State will determine whether it is appropriate to allow a system with a single service connection to use an alternative repeat monitoring scheme, as provided in § 141.21(b)(3), and what the alternative requirements will be.

(vii) Section 141.21(b)(5) (Waiver of requirement to take five routine samples the month after a system has a total coliform-positive sample)—A description of how the State will determine whether it is appropriate to waive the requirement for certain systems to collect five routine samples during the next month it serves water to the public, using the criteria in § 141.21(b)(5).

(viii) Section 141.21(c) (Invalidation of total coliform-positive samples)—A description of how the State will determine whether it is appropriate to invalidate a total coliform-positive sample, using the criteria in § 141.21(c).

(ix) Section 141.21(d) (Sanitary surveys)—A description of the State's criteria and procedures for approving agents other than State personnel to conduct sanitary surveys.

(x) Section 141.21(e)(2) (Waiver of fecal coliform or *E. coli* testing on a total coliform-positive sample)—A description of how the State will determine whether it is appropriate to waive fecal coliform or *E. coli* testing on a total coliform-positive sample.

(d) Requirements for States to adopt 40 CFR part 141, subpart I—Control of Lead and Copper. An application for approval of a State program revision which adopts the requirements specified in 40 CFR part 141, subpart I, must contain (in addition to the general primacy requirements enumerated elsewhere in this part, including the requirement that State regulations be at least as stringent as the federal requirements) a description of how the State will accomplish the following program requirements:

(1) Sections 141.82(d), 141.82(f), 141.82(h)—Designating optimal corrosion control treatment methods, optimal water quality parameters and modifications thereto.

(2) Sections 141.83(b)(2) and 141.83(b)(4)—Designating source water treatment methods, maximum permissible source water levels for lead and copper and modifications thereto.

(3) Section 141.90(e)—Verifying compliance with lead service line replacement schedules and of PWS demonstrations of limited control over lead service lines.

(e) An application for approval of a State program revision which adopts the requirements specified in §§141.11, 141.23, 141.24, 141.32, 141.40, 141.61 and 141.62 must contain the following (in addition to the general primacy requirements enumerated elsewhere in this part, including the requirement that State regulations be at least as stringent as the federal requirements):

(1) If a State chooses to issue waivers from the monitoring requirements in §§141.23, 141.24, and 141.40, the State shall describe the procedures and criteria which it will use to review waiver applications and issue waiver determinations.

(i) The procedures for each contaminant or class of contaminants shall include a description of:

(A) The waiver application requirements;

(B) The State review process for ''use'' waivers and for ''susceptibility'' waivers; and

(C) The State decision criteria, including the factors that will be considered in deciding to grant or deny waivers. The decision criteria must include the factors specified in §§141.24(f)(8), 141.24(h)(6), and 141.40(n)(4).

(ii) The State must specify the monitoring data and other documentation required to demonstrate that the contaminant is eligible for a ''use'' and/or ''susceptibility'' waiver.

(2) A monitoring plan for the initial monitoring period by which the State will assure all systems complete the required initial monitoring within the regulatory deadlines.

NOTE: States may update their monitoring plan submitted under the Phase II Rule or simply note in their application that they will use the same monitoring plan for the Phase V Rule.

(i) The initial monitoring plan must describe how systems will be scheduled during the initial monitoring period and demonstrate that the analytical workload on certified laboratories for each of the three years has been taken into account, to assure that the State's plan will result in a high degree of monitoring compliance and that as a result there is a high probability of compliance and will be updated as necessary.

(ii) The State must demonstrate that the initial monitoring plan is enforceable under State law.

[54 FR 15188, Apr. 17, 1989, as amended at 54 FR 27539, June 29, 1989; 55 FR 25065, June 19, 1990; 56 FR 3595, Jan. 30, 1991; 56 FR 26563, June 7, 1991; 57 FR 31847, July 17, 1992; 59 FR 33864, June 30, 1994]

§142.17 Review of State programs and procedures for withdrawal of approved primacy programs.

(a)(1) At least annually the Administrator shall review, with respect to each State determined to have primary enforcement responsibility, the compliance of the State with the requirements set forth in 40 CFR part 142, subpart B, and the approved State primacy program. At the time of this review, the State shall notify the Administrator of any State-initiated program changes (i.e., changes other than those to adopt new or revised EPA regulations), and of any transfer of all or part of its program from the approved State agency to any other State agency.

(2) When, on the basis of the Administrator's review or other available information, the Administrator determines that a State no longer meets the requirements set forth in 40 CFR part 142, subpart B, the Administrator shall initiate proceedings to withdraw primacy approval. Among the factors the Administrator intends to consider as relevant to this determination are the following, where appropriate: whether the State has requested and has been granted, or is awaiting EPA's decision on, an extension under §142.12(b)(2) of the deadlines for meeting those requirements; and whether the State is taking corrective actions that may have been required by the Administrator. The Administrator shall notify

the State in writing that EPA is initiating primacy withdrawal proceedings and shall summarize in the notice the information available that indicates that the State no longer meets such requirements.

(3) The State notified pursuant to paragraph (a)(2) of this section may, within 30 days of receiving the Administrator's notice, submit to the Administrator evidence demonstrating that the State continues to meet the requirements for primary enforcement responsibility.

(4) After reviewing the submission of the State, if any, made pursuant to paragraph (a)(3) of this section, the Administrator shall make a final determination either that the State no longer meets the requirements of 40 CFR part 142, subpart B, or that the State continues to meet those requirements, and shall notify the State of his or her determination. Any final determination that the State no longer meets the requirements of 40 CFR part 142, subpart B, shall not become effective except as provided in § 142.13.

(b) If a State which has primary enforcement responsibility decides to relinquish that authority, it may do so by notifying the Administrator in writing of the State's decision at least 90 days before the effective date of the decision.

[54 FR 52140, Dec. 20, 1989, as amended at 60 FR 33661, June 28, 1995]

§ 142.18 EPA review of State monitoring determinations.

(a) A Regional Administrator may annul a State monitoring determination for the types of determinations identified in §§ 141.23(b), 141.23(c), 141.24(f), 141.24(h), and 141.40(n) in accordance with the procedures in paragraph (b) of this section.

(b) When information available to a Regional Administrator, such as the results of an annual review, indicate a State determination fails to apply the standards of the approved State program, he may propose to annul the State monitoring determination by sending the State and the affected PWS a draft Rescission Order. The draft order shall:

(1) Identify the PWS, the State determination, and the provisions at issue;

(2) Explain why the State determination is not in compliance with the State program and must be changed; and

(3) Describe the actions and terms of operation the PWS will be required to implement.

(c) The State and PWS shall have 60 days to comment on the draft Rescission Order.

(d) The Regional Administrator may not issue a Rescission Order to impose conditions less stringent than those imposed by the State.

(e) The Regional Administrator shall also provide an opportunity for comment upon the draft Rescission Order, by

(1) Publishing a notice in a newspaper in general circulation in communities served by the affected system; and

(2) Providing 30 days for public comment on the draft order.

(f) The State shall demonstrate that the determination is reasonable, based on its approved State program.

(g) The Regional Administrator shall decide within 120 days after issuance of the draft Rescission Order to:

(1) Issue the Rescission Order as drafted;

(2) Issue a modified Rescission Order; or

(3) Cancel the Rescission Order.

(h) The Regional Administrator shall set forth the reasons for his decision, including a responsiveness summary addressing significant comments from the State, the PWS and the public.

(i) The Regional Administrator shall send a notice of his final decision to the State, the PWS and all parties who commented upon the draft Rescission Order.

(j) The Rescission Order shall remain in effect until cancelled by the Regional Administrator. The Regional Administrator may cancel a Rescission Order at any time, so long as he notifies those who commented on the draft order.

(k) The Regional Administrator may not delegate the signature authority for a final Rescission Order or the cancellation of an order.

(l) Violation of the actions, or terms of operation, required by a Rescission

Order is a violation of the Safe Drinking Water Act.

[56 FR 3595, Jan. 30, 1991]

§142.19 EPA review of State implementation of national primary drinking water regulations for lead and copper.

(a) Pursuant to the procedures in this section, the Regional Administrator may review state determinations establishing corrosion control or source water treatment requirements for lead or copper and may issue an order establishing federal treatment requirements for a public water system pursuant to §141.82 (d) and (f) and §141.83(b) (2) and (4) where the Regional Administrator finds that:

(1) A State has failed to issue a treatment determination by the applicable deadline;

(2) A State has abused its discretion in making corrosion control or source water treatment determinations in a substantial number of cases or in cases affecting a substantial population, or

(3) The technical aspects of State's determination would be indefensible in an expected federal enforcement action taken against a system.

(b) If the Regional Administrator determines that review of state determination(s) under this section may be appropriate, he shall request the State to forward to EPA the state determination and all information that was considered by the State in making its determination, including public comments, if any, within 60 days of the Regional Adminstrator's request.

(c) Proposed review of state determinations:

(1) Where the Regional Administrator finds that review of a state determination under paragraph (a) of this section is appropriate, he shall issue a proposed review order which shall:

(i) Identify the public water system(s) affected, the State determination being reviewed and the provisions of state and/or federal law at issue;

(ii) Identify the determination that the State failed to carry out by the applicable deadline, or identify the particular provisions of the State determination which, in the Regional Administrator's judgment, fail to carry out properly applicable treatment requirements, and explain the basis for the Regional Administrator's conclusion;

(iii) Identify the treatment requirements which the Regional Administrator proposes to apply to the affected system(s), and explain the basis for the proposed requirements;

(iv) Request public comment on the proposed order and the supporting record.

(2) The Regional Administrator shall provide notice of the proposed review order by:

(i) Mailing the proposed order to the affected public water system(s), the state agency whose order is being reviewed, and any other parties of interest known to the Regional Administrator; and

(ii) Publishing a copy of the proposed order in a newspaper of general circulation in the affected communities.

(3) The Regional Administrator shall make available for public inspection during the comment period the record supporting the proposed order, which shall include all of the information submitted by the State to EPA under paragraph (b) of this section, all other studies, monitoring data and other information considered by the Agency in developing the proposed order.

(d) Final review order:

(1) Based upon review of all information obtained regarding the proposed review order, including public comments, the Regional Administrator shall issue a final review order within 120 days after issuance of the proposed order which affirms, modifies, or withdraws the proposed order. The Regional Administrator may extend the time period for issuing the final order for good cause. If the final order modifies or withdraws the proposed order, the final order shall explain the reasons supporting the change.

(2) The record of the final order shall consist of the record supporting the proposed order, all public comments, all other information considered by the Regional Administrator in issuing the final order and a document responding to all significant public comments submitted on the proposed order. If new points are raised or new material supplied during the public comment period, the Regional Administrator may

support the responses on those matters by adding new materials to the record. The record shall be complete when the final order is issued.

(3) Notice of the final order shall be provided by mailing the final order to the affected system(s), the State, and all parties who commented on the proposed order.

(4) Upon issuance of the final order, its terms constitute requirements of the national primary drinking water regulation for lead and/or copper until such time as the Regional Administrator issues a new order (which may include recision of the previous order) pursuant to the procedures in this section. Such requirements shall supersede any inconsistent treatment requirements established by the State pursuant to the national primary drinking water regulations for lead and copper.

(5) The Regional Administrator may not issue a final order to impose conditions less stringent than those imposed by the State.

(e) The Regional Administrator may not delegate authority to sign the final order under this section.

(f) Final action of the Regional Administrator under paragraph (d) of this section shall constitute action of the Administrator for purposes of 42 U.S.C. § 300j–7(a)(2).

[56 FR 26563, June 7, 1991]

Subpart C—Review of State-Issued Variances and Exemptions

§ 142.20 State-issued variances and exemptions.

States with primary enforcement responsibility may issue variances and exemptions from the requirements of primary drinking water regulations under conditions and in a manner which are not less stringent than the conditions under which, and the manner in which, variances and exemptions may be granted under sections 1415 and 1416 of the Act. In States that do not have primary enforcement responsibility, variances and exemptions from the requirements of applicable national primary drinking water regulations may be granted by the Administrator pursuant to subparts E and F.

§ 142.21 State consideration of a variance or exemption request.

A State with primary enforcement responsibility shall act on any variance or exemption request submitted to it, within 90 days of receipt of the request.

§ 142.22 Review of State variances, exemptions and schedules.

(a) Not later than 18 months after the effective date of the interim national primary drinking water regulations the Administrator shall complete a comprehensive review of the variances and exemptions granted (and schedules prescribed pursuant thereto) by the States with primary enforcement responsibility during the one-year period beginning on such effective date. The Administrator shall conduct such subsequent reviews of exemptions and schedules as he deems necessary to carry out the purposes of this title, but at least one review shall be completed within each 3–year period following the completion of the first review under this paragraph.

(b) Notice of a proposed review shall be published in the FEDERAL REGISTER. Such notice shall (1) provide information respecting the location of data and other information respecting the variances and exemptions to be reviewed (including data and other information concerning new scientific matters bearing on such variances and exemptions), and (2) advise of the opportunity to submit comments on the variances and exemptions reviewed and on the need for continuing them. Upon completion of any such review, the Administrator shall publish in the FEDERAL REGISTER the results of his review, together with findings responsive to any comments submitted in connection with such review.

§ 142.23 Notice to State.

(a) If the Administrator finds that a State has, in a substantial number of instances, abused its discretion in granting variances or exemptions under section 1415(a) or section 1416(a) of the Act or failed to prescribe schedules in accordance with section 1415(a) or section 1416(b) of the Act, he shall notify the State of his findings. Such notice shall:

(1) Identify each public water system for which the finding was made;

(2) Specify the reasons for the finding; and

(3) As appropriate, propose revocation of specific variances or exemptions, or propose revised schedules for specific public water systems.

(b) The Administrator shall also notify the State of a public hearing to be held on the provisions of the notice required by paragraph (a) of this section. Such notice shall specify the time and location for the hearing. If, upon notification of a finding by the Administrator, the State takes adequate corrective action, the Administrator shall rescind his notice to the State of a public hearing, provided that the Administrator is notified of the corrective action prior to the hearing.

(c) The Administrator shall publish notice of the public hearing in the FEDERAL REGISTER and in a newspaper or newspapers of general circulation in the involved State including a summary of the findings made pursuant to paragraph (a) of this section, a statement of the time and location for the hearing, and the address and telephone number of an office at which interested persons may obtain further information concerning the hearing.

(d) Hearings convened pursuant to paragraphs (b) and (c) of this section shall be conducted before a hearing officer to be designated by the Administrator. The hearing shall be conducted by the hearing officer in an informal, orderly and expeditious manner. The hearing officer shall have authority to call witnesses, receive oral and written testimony and take such other action as may be necessary to assure the fair and efficient conduct of the hearing. Following the conclusion of the hearing, the hearing officer shall forward the record of the hearing to the Administrator.

(e) Within 180 days after the date notice is given pursuant to paragraph (b) of this section, the Administrator shall:

(1) Rescind the finding for which the notice was given and promptly notify the State of such rescission, or

(2) Promulgate with any modifications as appropriate such revocation and revised schedules proposed in such notice and promptly notify the State of such action.

(f) A revocation or revised schedule shall take effect 90 days after the State is notified under paragraph (e)(2) of this section.

§142.24 Administrator's rescission.

If, upon notification of a finding by the Administrator under §142.23, the State takes adequate corrective action before the effective date of the revocation or revised schedule, the Administrator shall rescind the application of his finding to that variance, exemption or schedule.

Subpart D—Federal Enforcement

§142.30 Failure by State to assure enforcement.

(a) The Administrator shall notify a State and the appropriate supplier of water whenever he finds during a period in which the State has primary enforcement responsibility for public water systems that a public water system within such State is not in compliance with any primary drinking water regulation contained in part 141 of this chapter or with any schedule or other requirements imposed pursuant to a variance or exemption granted under section 1415 or 1416 of the Act: *Provided,* That the State will be deemed to have been notified of a violation referred to in a report submitted by the State.

(b) The Administrator shall provide advice and technical assistance to such State and public water system as may be appropriate to bring the system into compliance by the earliest feasible time.

[41 FR 2918, Jan. 20, 1976, as amended at 52 FR 20675, June 2, 1987]

§142.31 [Reserved]

§142.32 Petition for public hearing.

(a) If the Administrator makes a finding of noncompliance pursuant to §142.30 with respect to a public water system in a State which has primary

enforcement responsibility, the Administrator may, for the purpose of assisting that State in carrying out such responsibility and upon the petition of such State or public water system or persons served by such system, hold, after appropriate notice, public hearings for the purpose of gathering information as described in § 142.33.

(b) A petition for a public hearing pursuant to paragraph (a) of this section shall be filed with the Administrator and shall include the following information:

(1) The name, address and telephone number of the individual or other entity requesting a hearing.

(2) If the petition is filed by a person other than the State or public water system, a statement that the person is served by the system.

(3) A brief statement of information that the requesting person intends to submit at the requested hearing.

(4) The signature of the individual submitting the petition; or, if the petition is filed on behalf of a State, public water system or other entity, the signature of a responsible official of the State or other entity.

§ 142.33 Public hearing.

(a) If the Administrator grants the petition for public hearing, he shall give appropriate public notice of such hearing. Such notice shall be by publication in the FEDERAL REGISTER and in a newspaper of general circulation or by other appropriate communications media covering the area served by such public water system.

(b) A hearing officer designated by the Administrator shall gather during the public hearing information from technical or other experts, Federal, State, or other public officials, representatives of the public water system, persons served by the system, and other interested persons on:

(1) The ways in which the system can within the earliest feasible time be brought into compliance, and

(2) The means for the maximum feasible protection of the public health during any period in which such system is not in compliance.

(c) On the basis of the hearing and other available information the Administrator shall issue recommendations which shall be sent to the State and public water system and shall be made available to the public and communications media.

§ 142.34 Entry and inspection of public water systems.

(a) Any supplier of water or other person subject to a national primary drinking water regulation shall, at any time, allow the Administrator, or a designated representative of the Administrator, upon presenting appropriate credentials and a written notice of inspection, to enter any establishment, facility or other property of such supplier or other person to determine whether such supplier or other person has acted or is acting in compliance with the requirements of the Act or subchapter D of this chapter. Such inspection may include inspection, at reasonable times, of records, files, papers, processes, controls and facilities, or testing of any feature of a public water system, including its raw water source.

(b) Prior to entry into any establishment, facility or other property within a State which has primary enforcement responsibility, the Administrator shall notify, in writing, the State agency charged with responsibility for safe drinking water of his intention to make such entry and shall include in his notification a statement of reasons for such entry. The Administrator shall, upon a showing by the State agency that such an entry will be detrimental to the administration of the State's program of primary enforcement responsibility, take such showing into consideration in determining whether to make such entry. The Administrator shall in any event offer the State agency the opportunity of having a representative accompany the Administrator or his representative on such entry.

(c) No State agency which receives notice under paragraph (b) of this section may use the information contained in the notice to inform the person whose property is proposed to be entered of the proposed entry; if a State so uses such information, notice to the agency under paragraph (b) of

this section is not required for subsequent inspections of public water systems until such time as the Administrator determines that the agency has provided him satisfactory assurances that it will no longer so use information contained in a notice received under paragraph (b) of this section.

Subpart E—Variances Issued by the Administrator

§ 142.40 Requirements for a variance.

(a) The Administrator may grant one or more variances to any public water system within a State that does not have primary enforcement responsibility from any requirement respecting a maximum contaminant level of an applicable national primary drinking water regulation upon a finding that:

(1) Because of characteristics of the raw water sources which are reasonably available to the system, the system cannot meet the requirements respecting the maximum contaminant levels of such drinking water regulations despite application of the best technology, treatment techniques, or other means, which the Administrator finds are generally available (taking costs into consideration); and

(2) The granting of a variance will not result in an unreasonable risk to the health of persons served by the system.

(b) The Administrator may grant one or more variances to any public water system within a State that does not have primary enforcement responsibility from any requirement of a specified treatment technique of an applicable national primary drinking water regulation upon a finding that the public water system applying for the variance has demonstrated that such treatment technique is not necessary to protect the health of persons because of the nature of the raw water source of such system.

§ 142.41 Variance request.

A supplier of water may request the granting of a variance pursuant to this subpart for a public water system within a State that does not have primary enforcement responsibility by submitting a request for a variance in writing to the Administrator. Suppliers of water may submit a joint request for variances when they seek similar variances under similar circumstances. Any written request for a variance or variances shall include the following information:

(a) The nature and duration of variance requested.

(b) Relevant analytical results of water quality sampling of the system, including results of relevant tests conducted pursuant to the requirements of the national primary drinking water regulations.

(c) For any request made under § 142.40(a):

(1) Explanation in full and evidence of the best available treatment technology and techniques.

(2) Economic and legal factors relevant to ability to comply.

(3) Analytical results of raw water quality relevant to the variance request.

(4) A proposed compliance schedule, including the date each step toward compliance will be achieved. Such schedule shall include as a minimum the following dates:

(i) Date by which arrangement for alternative raw water source or improvement of existing raw water source will be completed.

(ii) Date of initiation of the connection of the alternative raw water source or improvement of existing raw water source.

(iii) Date by which final compliance is to be achieved.

(5) A plan for the provision of safe drinking water in the case of an excessive rise in the contaminant level for which the variance is requested.

(6) A plan for additional interim control measures during the effective period of variance.

(d) For any request made under § 142.40(b), a statement that the system will perform monitoring and other reasonable requirements prescribed by the Administrator as a condition to the variance.

(e) Other information, if any, believed to be pertinent by the applicant.

(f) Such other information as the Administrator may require.

[41 FR 2918, Jan. 20, 1976, as amended at 52 FR 20675, June 2, 1987]

§ 142.42 Consideration of a variance request.

(a) The Administrator shall act on any variance request submitted pursuant to § 142.41 within 90 days of receipt of the request.

(b) In his consideration of whether the public water system is unable to comply with a contaminant level required by the national primary drinking water regulations because of the nature of the raw water source, the Administrator shall consider such factors as the following:

(1) The availability and effectiveness of treatment methods for the contaminant for which the variance is requested.

(2) Cost and other economic considerations such as implementing treatment, improving the quality of the source water or using an alternate source.

(c) A variance may only be issued to a system after the system's application of the best technology, treatment techniques, or other means, which the Administrator finds are available (taking costs into consideration).

(d) In his consideration of whether a public water system should be granted a variance to a required treatment technique because such treatment is unnecessary to protect the public health, the Administrator shall consider such factors as the following:

(1) Quality of the water source including water quality data and pertinent sources of pollution.

(2) Source protection measures employed by the public water system.

[41 FR 2918, Jan. 20, 1976, as amended at 52 FR 20675, June 2, 1987]

§ 142.43 Disposition of a variance request.

(a) If the Administrator decides to deny the application for a variance, he shall notify the applicant of his intention to issue a denial. Such notice shall include a statement of reasons for the proposed denial, and shall offer the applicant an opportunity to present, within 30 days of receipt of the notice, additional information or argument to the Administrator. The Administrator shall make a final determination on the request within 30 days after receiving any such additional information or argument. If no additional information or argument is submitted by the applicant the application shall be denied.

(b) If the Administrator proposes to grant a variance request submitted pursuant to § 142.41, he shall notify the applicant of his decision in writing. Such notice shall identify the variance, the facility covered, and shall specify the period of time for which the variance will be effective.

(1) For the type of variance specified in § 142.40(a) such notice shall provide that the variance will be terminated when the system comes into compliance with the applicable regulation, and may be terminated upon a finding by the Administrator that the system has failed to comply with any requirements of a final schedule issued pursuant to § 142.44.

(2) For the type of variance specified in § 142.40(b) such notice shall provide that the variance may be terminated at any time upon a finding that the nature of the raw water source is such that the specified treatment technique for which the variance was granted is necessary to protect the health of persons or upon a finding that the public water system has failed to comply with monitoring and other requirements prescribed by the Administrator as a condition to the granting of the variance.

(c) For a variance specified in § 142.40(a)(1) the Administrator shall propose a schedule for:

(1) Compliance (including increments of progress) by the public water system with each contaminant level requirement covered by the variance; and,

(2) Implementation by the public water system of such additional control measures as the Administrator may require for each contaminant covered by the variance.

(d) The proposed schedule for compliance shall specify dates by which steps towards compliance are to be taken, including at the minimum, where applicable:

(1) Date by which arrangement for an alternative raw water source or improvement of existing raw water source will be completed.

(2) Date of initiation of the connection for the alternative raw water

source or improvement of the existing raw water source.

(3) Date by which final compliance is to be achieved.

(e) The proposed schedule may, if the public water system has no access to an alternative raw water source, and can effect or anticipate no adequate improvement of the existing raw water source, specify an indefinite time period for compliance until a new and effective treatment technology is developed at which time a new compliance schedule shall be prescribed by the Administrator.

(f) The proposed schedule for implementation of additional interim control measures during the period of variance shall specify interim treatment techniques, methods and equipment, and dates by which steps toward meeting the additional interim control measures are to be met.

(g) The schedule shall be prescribed by the Administrator at the time of granting of the variance, subsequent to provision of opportunity for hearing pursuant to §142.44.

[41 FR 2918, Jan. 20, 1976, as amended at 52 FR 20675, June 2, 1987]

§142.44 Public hearings on variances and schedules.

(a) Before a variance and schedule proposed by the Administrator pursuant to §142.43 may take effect, the Administrator shall provide notice and opportunity for public hearing on the variance and schedule. A notice given pursuant to the preceding sentence may cover the granting of more than one variance and a hearing held pursuant to such notice shall include each of the variances covered by the notice.

(b) Public notice of an opportunity for hearing on a variance and schedule shall be circulated in a manner designed to inform interested and potentially interested persons of the proposed variance and schedule, and shall include at least the following:

(1) Posting of a notice in the principal post office of each municipality or area served by the public water system, and publishing of a notice in a newspaper or newspapers of general circulation in the area served by the public water system; and

(2) Mailing of a notice to the agency of the State in which the system is located which is responsible for the State's water supply program, and to other appropriate State or local agencies at the Administrator's discretion.

(3) Such notice shall include a summary of the proposed variance and schedule and shall inform interested persons that they may request a public hearing on the proposed variance and schedule.

(c) Requests for hearing may be submitted by any interested person other than a Federal agency. Frivolous or insubstantial requests for hearing may be denied by the Administrator. Requests must be submitted to the Administrator within 30 days after issuance of the public notices provided for in paragraph (b) of this section. Such requests shall include the following information:

(1) The name, address and telephone number of the individual, organization or other entity requesting a hearing;

(2) A brief statement of the interest of the person making the request in the proposed variance and schedule, and of information that the requester intends to submit at such hearing;

(3) The signature of the individual making the request, or, if the request is made on behalf of an organization or other entity, the signature of a responsible official of the organization or other entity.

(d) The Administrator shall give notice in the manner set forth in paragraph (b) of this section of any hearing to be held pursuant to a request submitted by an interested person or on his own motion. Notice of the hearing shall also be sent to the persons requesting the hearing, if any. Notice of the hearing shall include a statement of the purpose of the hearing, information regarding the time and location for the hearing, and the address and telephone number of an office at which interested persons may obtain further information concerning the hearing. At least one hearing location specified in the public notice shall be within the involved State. Notice of hearing shall be given not less than 15 days prior to the time scheduled for the hearing.

(e) A hearing convened pursuant to paragraph (d) of this section shall be

conducted before a hearing officer to be designated by the Administrator. The hearing shall be conducted by the hearing officer in an informal, orderly and expeditious manner. The hearing officer shall have authority to call witnesses, receive oral and written testimony and take such other action as may be necessary to assure the fair and efficient conduct of the hearing. Following the conclusion of the hearing, the hearing officer shall forward the record of the hearing to the Administrator.

(f) The variance and schedule shall become effective 30 days after notice of opportunity for hearing is given pursuant to paragraph (b) of this section if no timely request for hearing is submitted and the Administrator does not determine to hold a public hearing on his own motion.

[41 FR 2918, Jan. 20, 1976, as amended at 52 FR 20675, June 2, 1987]

§ 142.45 Action after hearing.

Within 30 days after the termination of the public hearing held pursuant to § 142.44, the Administrator shall, taking into consideration information obtained during such hearing and relevant information, confirm, revise or rescind the proposed variance and schedule.

[52 FR 20675, June 2, 1987]

§ 142.46 Alternative treatment techniques.

The Administrator may grant a variance from any treatment technique requirement of a national primary drinking water regulation to a supplier of water, whether or not the public water system for which the variance is requested is located in a State which has primary enforcement responsibility, upon a showing from any person that an alternative treatment technique not included in such requirement is at least as efficient in lowering the level of the contaminant with respect to which such requirements was prescribed. A variance under this paragraph shall be conditioned on the use of the alternative treatment technique which is the basis of the variance.

Subpart F—Exemptions Issued by the Administrator

§ 142.50 Requirements for an exemption.

The Administrator may exempt any public water system within a State that does not have primary enforcement responsibility from any requirement respecting a maximum contaminant level or any treatment technique requirement, or from both, of an applicable national primary drinking water regulation upon a finding that:

(a) Due to compelling factors (which may include economic factors), the public water system is unable to comply with such contaminant level or treatment technique requirement;

(b) The public water system was in operation on the effective date of such contaminant level or treatment technique requirement; and

(c) The granting of the exemption will not result in an unreasonable risk to health.

§ 142.51 Exemption request.

A supplier of water may request the granting of an exemption pursuant to this subpart for a public water system within a State that does not have primary enforcement responsibility by submitting a request for exemption in writing to the Administrator. Suppliers of water may submit a joint request for exemptions when they seek similar exemptions under similar circumstances. Any written request for an exemption or exemptions shall include the following information:

(a) The nature and duration of exemption requested.

(b) Relevant analytical results of water quality sampling of the system, including results of relevant tests conducted pursuant to the requirements of the national primary drinking water regulations.

(c) Explanation of the compelling factors such as time or economic factors which prevent such system from achieving compliance.

(d) Other information, if any, believed by the applicant to be pertinent to the application.

(e) A proposed compliance schedule, including the date when each step toward compliance will be achieved.

(f) Such other information as the Administrator may require.

§142.52 Consideration of an exemption request.

(a) The Administrator shall act on any exemption request submitted pursuant to §142.51 within 90 days of receipt of the request.

(b) In his consideration of whether the public water system is unable to comply due to compelling factors, the Administrator shall consider such factors as the following:

(1) Construction, installation, or modification of the treatment equipment or systems.

(2) The time needed to put into operation a new treatment facility to replace an existing system which is not in compliance.

(3) Economic feasibility of compliance.

§142.53 Disposition of an exemption request.

(a) If the Administrator decides to deny the application for an exemption, he shall notify the applicant of his intention to issue a denial. Such notice shall include a statement of reasons for the proposed denial, and shall offer the applicant an opportunity to present, within 30 days of receipt of the notice, additional information or argument to the Administrator. The Administrator shall make a final determination on the request within 30 days after receiving any such additional information or argument. If no additional information or argument is submitted by the applicant, the application shall be denied.

(b) If the Administrator grants an exemption request submitted pursuant to §142.51, he shall notify the applicant of his decision in writing. Such notice shall identify the facility covered, and shall specify the termination date of the exemption. Such notice shall provide that the exemption will be terminated when the system comes into compliance with the applicable regulation, and may be terminated upon a finding by the Administrator that the system has failed to comply with any requirements of a final schedule issued pursuant to §142.55.

(c) The Administrator shall propose a schedule for:

(1) Compliance (including increments of progress) by the public water system with each contaminant level requirement and treatment technique requirement covered by the exemption; and

(2) Implementation by the public water system of such control measures as the Administrator may require for each contaminant covered by the exemption.

(d) The schedule shall be prescribed by the Administrator at the time the exemption is granted, subsequent to provision of opportunity for hearing pursuant to §142.54.

[41 FR 2918, Jan. 20, 1976, as amended at 52 FR 20675, June 2, 1987]

§142.54 Public hearings on exemption schedules.

(a) Before a schedule proposed by the Administrator pursuant to §142.53 may take effect, the Administrator shall provide notice and opportunity for public hearing on the schedule. A notice given pursuant to the preceding sentence may cover the proposal of more than one such schedule and a hearing held pursuant to such notice shall include each of the schedules covered by the notice.

(b) Public notice of an opportunity for hearing on an exemption schedule shall be circulated in a manner designed to inform interested and potentially interested persons of the proposed schedule, and shall include at least the following:

(1) Posting of a notice in the principal post office of each municipality or area served by the public water system, and publishing of a notice in a newspaper or newspapers of general circulation in the area served by the public water system.

(2) Mailing of a notice to the agency of the State in which the system is located which is responsible for the State's water supply program and to other appropriate State or local agencies at the Administrator's discretion.

(3) Such notices shall include a summary of the proposed schedule and shall inform interested persons that

they may request a public hearing on the proposed schedule.

(c) Requests for hearing may be submitted by any interested person other than a Federal agency. Frivolous or insubstantial requests for hearing may be denied by the Administrator. Requests must be submitted to the Administrator within 30 days after issuance of the public notices provided for in paragraph (b) of this section. Such requests shall include the following information:

(1) The name, address and telephone number of the individual, organization or other entity requesting a hearing;

(2) A brief statement of the interest of the person making the request in the proposed schedule and of information that the requesting person intends to submit at such hearing; and

(3) The signature of the individual making the request, or, if the request is made on behalf of an organization or other entity, the signature of a responsibile official of the organization or other entity.

(d) The Administrator shall give notice in the manner set forth in paragraph (b) of this section of any hearing to be held pursuant to a request submitted by an interested person or on his own motion. Notice of the hearing shall also be sent to the person requesting the hearing, if any. Notice of the hearing shall include a statement of the purpose of the hearing, information regarding the time and location of the hearing, and the address and telephone number of an office at which interested persons may obtain further information concerning the hearing. At least one hearing location specified in the public notice shall be within the involved State. Notice of the hearing shall be given not less than 15 days prior to the time scheduled for the hearing.

(e) A hearing convened pursuant to paragraph (d) of this section shall be conducted before a hearing officer to be designated by the Administrator. The hearing shall be conducted by the hearing officer in an informal, orderly and expeditious manner. The hearing officer shall have authority to call witnesses, receive oral and written testimony and take such action as may be necessary to assure the fair and efficient conduct of the hearing. Following the conclusion of the hearing, the hearing officer shall forward the record of the hearing to the Administrator.

[41 FR 2918, Jan. 20, 1976, as amended at 52 FR 20675, June 2, 1987]

§ 142.55 Final schedule.

(a) Within 30 days after the termination of the public hearing pursuant to § 142.54, the Administrator shall, taking into consideration information obtained during such hearing, revise the proposed schedule as necessary and prescribe the final schedule for compliance and interim measures for the public water system granted an exemption under § 142.52.

(b) Such schedule must require compliance as follows:

(1) In the case of an exemption granted with respect to a contaminant level or treatment technique requirement prescribed by the national primary drinking water regulations promulgated under section 1421(a) of the Safe Drinking Water Act, not later than June 19, 1987, and

(2) In the case of an exemption granted with respect to a contaminant level or treatment technique requirement prescribed by national primary drinking water regulations, other than a regulation referred to in section 1412(a), 12 months after the issuance of the exemption.

(c) If the public water system has entered into an enforceable agreement to become a part of a regional public water system, as determined by the Administrator, such schedule shall require compliance by the public water system with each contaminant level and treatment technique requirement prescribed by:

(1) Interim national primary drinking water regulations pursuant to part 141 of this chapter, by no later than January 1, 1983; and

(2) Revised national primary drinking water regulations pursuant to part 141 of this chapter, by no later than nine years after the effective date of such regulations.

[41 FR 2918, Jan. 20, 1976, as amended at 52 FR 20675, June 2, 1987]

§142.56 Extension of date for compliance.

(a) The final date for compliance provided in any schedule in the case of any exemption may be extended by the State (in the case of a State which has primary enforcement responsibility) or by the Administrator (in any other case) for a period not to exceed 3 years after the date of the issuance of the exemption if the public water system establishes that:

(1) The system cannot meet the standard without capital improvements which cannot be completed within the period of such exemption;

(2) In the case of a system which needs financial assistance for the necessary improvements, the system has entered into an agreement to obtain such financial assistance; or

(3) The system has entered into an enforceable agreement to become a part of a regional public water system; and the system is taking all practicable steps to meet the standard.

(b) In the case of a system which does not serve more than 500 service connections and which needs financial assistance for the necessary improvements, an exemption granted under paragraph (a) (1) or (2) may be renewed for one or more additional 2-year periods if the system establishes that it is taking all practicable steps to meet the requirements of paragraph (a) of this section.

[52 FR 20676, June 2, 1987]

§142.57 Bottled water, point-of-use, and point-of-entry devices.

(a) A State may require a public water system to use bottled water, point-of-use devices, or point-of-entry devices as a condition of granting an exemption from the requirements of §§141.61 (a) and (c), and 141.62 of this chapter.

(b) Public water systems using bottled water as a condition of obtaining an exemption from the requirements of §§141.61 (a) and (c) and 141.62(b) must meet the requirements in §142.62(g).

(c) Public water systems that use point-of-use or point-of-entry devices as a condition for receiving an exemption must meet the requirements in §141.62(h).

[56 FR 3596, Jan. 30, 1991, as amended at 56 FR 30280, July 1, 1991]

Subpart G—Identification of Best Technology, Treatment Techniques or Other Means Generally Available

§142.60 Variances from the maximum contaminant level for total trihalomethanes.

(a) The Administrator, pursuant to section 1415(a)(1)(A) of the Act, hereby identifies the following as the best technology, treatment techiques or other means generally available for achieving compliance with the maximum contaminant level for total trihalomethanes (§141.12(c)):

(1) Use of chloramines as an alternate or supplemental disinfectant or oxidant.

(2) Use of chlorine dioxide as an alternate or supplemental disinfectant or oxidant.

(3) Improved existing clarification for THM precursor reduction.

(4) Moving the point of chlorination to reduce TTHM formation and, where necessary, substituting for the use of chlorine as a pre-oxidant chloramines, chlorine dioxide or potassium permanganate.

(5) Use of powdered activated carbon for THM precursor or TTHM reduction seasonally or intermittently at dosages not to exceed 10 mg/L on an annual average basis.

(b) The Administrator in a state that does not have primary enforcement responsibility or a state with primary enforcement responsibility (primacy state) that issues variances shall require a community water system to install and/or use any treatment method identified in §142.60(a) as a condition for granting a variance unless the Administrator or primacy state determines that such treatment method identified in §142.60(a) is not available and effective for TTHM control for the system. A treatment method shall not be considered to be ''available and effective'' for an individual system if the treatment method would not be technically appropriate and technically

feasible for that system or would only result in a marginal reduction in TTHM for the system. If, upon application by a system for a variance, the Administrator or primacy state that issues variances determines that none of the treatment methods identified in § 142.60(a) is available and effective for the system, that system shall be entitled to a variance under the provisions of section 1415(a)(1)(A) of the Act. The Administrator's or primacy state's determination as to the availability and effectiveness of such treatment methods shall be based upon studies by the system and other relevant information. If a system submits information intending to demonstrate that a treatment method is not available and effective for TTHM control for that system, the Administrator or primacy state shall make a finding whether this information supports a decision that such treatment method is not available and effective for that system before requiring installation and/or use of such treatment method.

(c) Pursuant to § 142.43 (c) through (g) or corresponding state regulations, the Administrator or primacy state that issues variances shall issue a schedule of compliance that may require the system being granted the variance to examine the following treatment methods (1) to determine the probability that any of these methods will significantly reduce the level of TTHM for that system, and (2) if such probability exists, to determine whether any of these methods are technically feasible and economically reasonable, and that the TTHM reductions obtained will be commensurate with the costs incurred with the installation and use of such treatment methods for that system:

Introduction of off-line water storage for THM precursor reduction.

Aeration for TTHM reduction, where geographically and environmentally appropriate.

Introduction of clarification where not currently practiced.

Consideration of alternative sources of raw water.

Use of ozone as an alternate or supplemental disinfectant or oxidant.

(d) If the Administrator or primacy state that issues variances determines that a treatment method identified in § 142.60(c) is technically feasible, economically reasonable and will achieve TTHM reductions commensurate with the costs incurred with the installation and/or use of such treatment method for the system, the Administrator or primacy state shall require the system to install and/or use that treatment method in connection with a compliance schedule issued under the provisions of section 1415(a)(1)(A) of the Act. The Administrator's or primacy state's determination shall be based upon studies by the system and other relevant information. In no event shall the Administrator require a system to install and/or use a treatment method not described in § 142.60 (a) or (c) to obtain or maintain a variance from the TTHM Rule or in connection with any variance compliance schedule.

[48 FR 8414, Feb. 28, 1983]

§ 142.61 Variances from the maximum contaminant level for fluoride.

(a) The Administrator, pursuant to section 1415(a)(1)(A) of the Act, hereby identifies the following as the best technology, treatment techniques or other means generally available for achieving compliance with the Maximum Contaminant Level for fluoride.

(1) Activated alumina absorption, centrally applied

(2) Reverse osmosis, centrally applied

(b) The Administrator in a state that does not have primary enforcement responsibility or a state with primary enforcement responsibility (primacy state) that issues variances shall require a community water system to install and/or use any treatment method identified in § 142.61(a) as a condition for granting a variance unless the Administrator or the primacy state determines that such treatment method identified in § 142.61(a) as a condition for granting a variance is not available and effective for fluoride control for the system. A treatment method shall not be considered to be ''available and effective'' for an individual system if the treatment method would not be technically appropriate and technically feasible for that system. If, upon application by a system for a variance, the Administrator or primacy state that issues variances determines that none of the treatment

methods identified in §142.61(a) are available and effective for the system, that system shall be entitled to a variance under the provisions of section 1415(a)(1)(A) of the Act. The Administrator's or primacy state's determination as to the availability and effectiveness of such treatment methods shall be based upon studies by the system and other relevant information. If a system submits information to demonstrate that a treatment method is not available and effective for fluoride control for that system, the Administrator or primacy state shall make a finding whether this information supports a decision that such treatment method is not available and effective for that system before requiring installation and/or use of such treatment method.

(c) Pursuant to §142.43 (c)–(g) or corresponding state regulations, the Administrator or primacy state that issues variances shall issue a schedule of compliance that may require the system being granted the variance to examine the following treatment methods (1) to determine the probability that any of these methods will significantly reduce the level of fluoride for that system, and (2) if such probability exists, to determine whether any of these methods are technically feasible and economically reasonable, and that the fluoride reductions obtained will be commensurate with the costs incurred with the installation and use of such treatment methods for that system:

(1) Modification of lime softening;
(2) Alum coagulation;
(3) Electrodialysis;
(4) Anion exchange resins;
(5) Well field management;
(6) Alternate source;
(7) Regionalization.

(d) If the Administrator or primary state that issues variances determines that a treatment method identified in §142.61(c) or other treatment method is technically feasible, economically reasonable, and will achieve fluoride reductions commensurate with the costs incurred with the installation and/or use of such treatment method for the system, the Administrator or primacy state shall require the system to install and/or use that treatment method in connection with a compliance schedule issued under the provisions of section 1415(a)(1)(A) of the Act. The Administrator's or primacy state's determination shall be based upon studies by the system and other relevant information.

[51 FR 11411, Apr. 2, 1986]

§142.62 Variances and exemptions from the maximum contaminant levels for organic and inorganic chemicals.

(a) The Administrator, pursuant to section 1415(a)(1)(A) of the Act hereby identifies the technologies listed in paragraphs (a)(1) through (a)(54) of this section as the best technology, treatment techniques, or other means available for achieving compliance with the maximum contaminant levels for organic chemicals listed in §141.61 (a) and (c):

Contaminant	Best available technologies		
	PTA [1]	GAC [2]	OX [3]
(1) Benzene	X	X	
(2) Carbon tetrachloride	X	X	
(3) 1,2-Dichloroethane	X	X	
(4) Trichloroethylene	X	X	
(5) para-Dichlorobenzene	X	X	
(6) 1,1-Dichloroethylene	X	X	
(7) 1,1,1-Trichloroethane	X	X	
(8) Vinyl chloride	X		
(9) cis-1,2-Dichloroethylene	X	X	
(10) 1,2-Dichloropropane	X	X	
(11) Ethylbenzene	X	X	
(12) Monochlorobenzene	X	X	
(13) o-Dichlorobenzene	X	X	
(14) Styrene	X	X	
(15) Tetrachloroethylene	X	X	
(16) Toluene	X	X	
(17) trans-1,2-Dichloroethylene	X	X	
(18) Xylense (total)	X	X	
(19) Alachlor		X	

Contaminant	Best available technologies		
	PTA [1]	GAC [2]	OX [3]
(20) Aldicarb		X	
(21) Aldicarb sulfoxide		X	
(22) Aldicarb sulfone		X	
(23) Atrazine		X	
(24) Carbofuran		X	
(25) Chlordane		X	
(26) Dibromochloropropane	X	X	
(27) 2,4-D		X	
(28) Ethylene dibromide	X	X	
(29) Heptachlor		X	
(30) Heptachlor epoxide		X	
(31) Lindane		X	
(32) Methoxychlor		X	
(33) PCBs		X	
(34) Pentachlorophenol		X	
(35) Toxaphene		X	
(36) 2,4,5-TP		X	
(37) Benzo[a]pyrene		X	
(38) Dalapon		X	
(39) Dichloromethane	X		
(40) Di(2-ethylhexyl)adipate	X	X	
(41) Di(2-ethylhexyl)phthalate		X	
(42) Dinoseb		X	
(43) Diquat		X	
(44) Endothall		X	
(45) Endrin		X	
(46) Glyphosate			X
(47) Hexachlorobenzene		X	
(48) Hexachlorocyclopentadiene	X	X	
(49) Oxamyl (Vydate)		X	
(50) Picloram		X	
(51) Simazine		X	
(52) 1,2,4-Trichlorobenzene	X	X	
(53) 1,1,2-Trichloroethane	X	X	
(54) 2,3,7,8-TCDD (Dioxin)		X	

[1] Packed Tower Aeration
[2] Granular Activated Carbon
[3] Oxidation (Chlorination or Ozonation)

(b) The Administrator, pursuant to section 1415(a)(1)(A) of the Act, hereby identifies the following as the best technology, treatment techniques, or other means available for achieving compliance with the maximum contaminant levels for the inorganic chemicals listed in § 141.62:

BAT FOR INORGANIC COMPOUNDS LISTED IN § 141.62(B)

Chemical name	BAT(s)
Antimony	2,7
Asbestos	2,3,8
Barium	5,6,7,9
Beryllium	1,2,5,6,7
Cadmium	2,5,6,7
Chromium	2,5,6 [2],7
Cyanide	5,7,10
Mercury	2 [1],4,6 [1],7 [1]
Nickel	5,6,7
Nitrite	5,7,9
Nitrate	5,7
Selenium	1,2 [3],6,7,9
Thallium	1,5

[1] BAT only if influent Hg concentrations ≤10μg/1.
[2] BAT for Chromium III only.
[3] BAT for Selenium IV only.

Key to BATS in Table

1=Activated Alumina
2=Coagulation/Filtration (not BAT for systems <500 service connections)
3=Direct and Diatomite Filtration
4=Granular Activated Carbon
5=Ion Exchange
6=Lime Softening (not BAT for systems <500 service connections)
7=Reverse Osmosis
8=Corrosion Control
9=Electrodialysis
10=Chlorine
11=Ultraviolet

(c) A State shall require community water systems and non-transient, non-community water systems to install and/or use any treatment method identified in § 142.62 (a) and (b) as a condition for granting a variance except as provided in paragraph (d) of this section. If, after the system's installation of the treatment method, the system

cannot meet the MCL, that system shall be eligible for a variance under the provisions of section 1415(a)(1)(A) of the Act.

(d) If a system can demonstrate through comprehensive engineering assessments, which may include pilot plant studies, that the treament methods identified in § 142.62 (a) and (b) would only achieve a *de minimis* reduction in contaminants, the State may issue a schedule of compliance that requires the system being granted the variance to examine other treatment methods as a condition of obtaining the variance.

(e) If the State determines that a treatment method identified in paragraph (d) of this section is technically feasible, the Administrator or primacy State may require the system to install and/or use that treatment method in connection with a compliance schedule issued under the provisions of section 1415(a)(1)(A) of the Act. The State's determination shall be based upon studies by the system and other relevant information.

(f) The State may require a public water system to use bottled water, point-of-use devices, point-of-entry devices or other means as a condition of granting a variance or an exemption from the requirements of §§ 141.61 (a) and (c) and 141.62, to avoid an unreasonable risk to health. The State may require a public water system to use bottled water and point-of-use devices or other means, *but not point-of-entry devices*, as a condition for granting an exemption from corrosion control treatment requirements for lead and copper in §§ 141.81 and 141.82 to avoid an unreasonable risk to health. The State may require a public water system to use point-of-entry devices as a condition for granting an exemption from the source water and lead service line replacement requirements for lead and copper under §§ 141.83 or 141.84 to avoid an unreasonable risk to health.

(g) Public water systems that use bottled water as a condition for receiving a variance or an exemption from the requirements of §§ 141.61 (a) and (c) and 141.62, or an exemption from the requirements of §§ 141.81–141.84 must meet the requirements specified in either paragraph (g)(1) or (g)(2) and paragraph (g)(3) of this section:

(1) The Administrator or primacy State must require and approve a monitoring program for bottled water. The public water system must develop and put in place a monitoring program that provides reasonable assurances that the bottled water meets all MCLs. The public water system must monitor a representative sample of the bottled water for all contaminants regulated under §§ 141.61 (a) and (c) and 141.62 during the first three-month period that it supplies the bottled water to the public, and annually thereafter. Results of the monitoring program shall be provided to the State annually.

(2) The public water system must receive a certification from the bottled water company that the bottled water supplied has been taken from an ''approved source'' as defined in 21 CFR 129.3(a); the bottled water company has conducted monitoring in accordance with 21 CFR 129.80(g) (1) through (3); and the bottled water does not exceed any MCLs or quality limits as set out in 21 CFR 103.35, part 110, and part 129. The public water system shall provide the certification to the State the first quarter after it supplies bottled water and annually thereafter. At the State's option a public water system may satisfy the requirements of this subsection if an approved monitoring program is already in place in another State.

(3) The public water system is fully responsible for the provision of sufficient quantities of bottled water to every person supplied by the public water system via door-to-door bottled water delivery.

(h) Public water systems that use point-of-use or point-of-entry devices as a condition for obtaining a variance or an exemption from NPDWRs must meet the following requirements:

(1) It is the responsibility of the public water system to operate and maintain the point-of-use and/or point-of-entry treatment system.

(2) Before point-of-use or point-of-entry devices are installed, the public water system must obtain the approval of a monitoring plan which ensures

that the devices provide health protection equivalent to that provided by central water treatment.

(3) The public water system must apply effective technology under a State-approved plan. The microbiological safety of the water must be maintained at all times.

(4) The State must require adequate certification of performance, field testing, and, if not included in the certification process, a rigorous engineering design review of the point-of-use and/or point-of-entry devices.

(5) The design and application of the point-of-use and/or point-of-entry devices must consider the potential for increasing concentrations of heterotrophic bacteria in water treated with activated carbon. It may be necessary to use frequent backwashing, post-contactor disinfection, and Heterotrophic Plate Count monitoring to ensure that the microbiological safety of the water is not compromised.

(6) The State must be assured that buildings connected to the system have sufficient point-of-use or point-of-entry devices that are properly installed, maintained, and monitored such that all consumers will be protected.

(7) In requiring the use of a point-of-entry device as a condition for granting an exemption from the treatment requirements for lead and copper under §§ 141.83 or 141.84, the State must be assured that use of the device will not cause increased corrosion of lead and copper bearing materials located between the device and the tap that could increase contaminant levels at the tap.

[56 FR 3596, Jan. 30, 1991, as amended at 56 FR 26563, June 7, 1991; 57 FR 31848, July 17, 1992; 59 FR 33864, June 30, 1994; 59 FR 34325, July 1, 1994]

§ 142.63 Variances and exemptions from the maximum contaminant level for total coliforms.

(a) No variances or exemptions from the maximum contaminant level in § 141.63 of this chapter are permitted.

(b) EPA has stayed the effective date of this section relating to the total coliform MCL of § 141.63(a) of this chapter for systems that demonstrate to the State that the violation of the total coliform MCL is due to a persistent growth of total coliforms in the distribution system rather than fecal or pathogenic contamination, a treatment lapse or deficiency, or a problem in the operation or maintenance of the distribution system.

[54 FR 27568, June 29, 1989, as amended at 56 FR 1557, Jan. 15, 1991]

§ 142.64 Variances and exemptions from the requirements of part 141, subpart H—Filtration and Disinfection.

(a) No variances from the requirements in part 141, subpart H are permitted.

(b) No exemptions from the requirements in § 141.72 (a)(3) and (b)(2) to provide disinfection are permitted.

[54 FR 27540, June 29, 1989]

Subpart H—Indian Tribes

SOURCE: 53 FR 37411, Sept. 26, 1988, unless otherwise noted.

§ 142.72 Requirements for Tribal eligibility.

The Administrator is authorized to treat an Indian Tribe as eligible to apply for primary enforcement responsibility for the Public Water System Program if it meets the following criteria:

(a) The Indian Tribe is recognized by the Secretary of the Interior.

(b) The Indian Tribe has a tribal governing body which is currently ''carrying out substantial governmental duties and powers'' over a defined area, (*i.e.*, is currently performing governmental functions to promote the health, safety, and welfare of the affected population within a defined geographic area).

(c) The Indian Tribe demonstrates that the functions to be performed in regulating the public water systems that the applicant intends to regulate are within the area of the Indian Tribal government's jurisdiction.

(d) The Indian Tribe is reasonably expected to be capable, in the Administrator's judgment, of administering (in a manner consistent with the terms

and purposes of the Act and all applicable regulations) an effective Public Water System program.

[53 FR 37411, Sept. 26, 1988, as amended at 59 FR 64344, Dec. 14, 1994]

§ 142.76 Request by an Indian Tribe for a determination of eligibility.

An Indian Tribe may apply to the Administrator for a determination that it meets the criteria of section 1451 of the Act. The application shall be concise and describe how the Indian Tribe will meet each of the requirements of § 142.72. The application shall consist of the following information:

(a) A statement that the Tribe is recognized by the Secretary of the Interior.

(b) A descriptive statement demonstrating that the Tribal governing body is currently carrying out substantial governmental duties and powers over a defined area. The statement should:

(1) Describe the form of the Tribal government;

(2) Describe the types of governmental functions currently performed by the Tribal governing body such as, but not limited to, the exercise of police powers affecting (or relating to) the health, safety, and welfare of the affected population; taxation; and the exercise of the power of eminent domain; and

(3) Identify the sources of the Tribal government's authority to carry out the governmental functions currently being performed.

(c) A map or legal description of the area over which the Indian Tribe asserts jurisdiction; a statement by the Tribal Attorney General (or equivalent official) which describes the basis for the Tribe's jurisdictional assertion (including the nature or subject matter of the asserted jurisdiction); a copy of those documents such as Tribal constitutions, by-laws, charters, executive orders, codes, ordinances, and/or resolutions which the Tribe believes are relevant to its assertions regarding jurisdiction; and a description of the locations of the public water systems the Tribe proposes to regulate.

(d) A narrative statement describing the capability of the Indian Tribe to administer an effective Public Water System program. The narrative statement should include:

(1) A description of the Indian Tribe's previous management experience which may include, the administration of programs and services authorized by the Indian Self-Determination and Education Assistance Act (25 U.S.C. 450 *et seq.*), the Indian Mineral Development Act (25 U.S.C. 2101 *et seq.*), or the Indian Sanitation Facilities Construction Activity Act (42 U.S.C. 2004a).

(2) A list of existing environmental or public health programs administered by the Tribal governing body and a copy of related Tribal laws, regulations and policies.

(3) A description of the Indian Tribe's accounting and procurement systems.

(4) A description of the entity (or entities) which exercise the executive, legislative, and judicial functions of the Tribal government.

(5) A description of the existing, or proposed, agency of the Indian Tribe which will assume primary enforcement responsibility, including a description of the relationship between owners/operators of the public water systems and the agency.

(6) A description of the technical and administrative capabilities of the staff to administer and manage an effective Public Water System Program or a plan which proposes how the Tribe will acquire additional administrative and/or technical expertise. The plan must address how the Tribe will obtain the funds to acquire the additional administrative and technical expertise.

(e) The Administrator may, in his discretion, request further documentation necessary to support a Tribe's eligibility.

(f) If the Administrator has previously determined that a Tribe has met the prerequisites that make it eligible to assume a role similar to that of a state as provided by statute under the Safe Drinking Water Act, the Clean Water Act, or the Clean Air Act, then that Tribe need provide only that information unique to the Public Water System program (paragraphs (c), (d)(5) and (6) of this section).

[53 FR 37411, Sept. 26, 1988, as amended at 59 FR 64344, Dec. 14, 1994]

§ 142.78 Procedure for processing an Indian Tribe's application.

(a) The Administrator shall process a completed application of an Indian Tribe in a timely manner. He shall promptly notify the Indian Tribe of receipt of the application.

(b) A tribe that meets the requirements of § 142.72 is eligible to apply for development grants and primary enforcement responsibility for a Public Water System Program and associated funding under section 1443(a) of the Act and for primary enforcement responsibility for public water systems under section 1413 of the Act.

[53 FR 37411 Sept. 26, 1988, as amended at 59 FR 64345, Dec. 14, 1994]

Subpart I—Administrator's Review of State Decisions that Implement Criteria Under Which Filtration Is Required

SOURCE: 54 FR 27540, June 29, 1989, unless otherwise noted.

§ 142.80 Review procedures.

(a) The Administrator may initiate a comprehensive review of the decisions made by States with primary enforcement responsibility to determine, in accordance with § 141.71 of this chapter, if public water systems using surface water sources must provide filtration treatment. The Administrator shall complete this review within one year of its initiation and shall schedule subsequent reviews as (s)he deems necessary.

(b) EPA shall publish notice of a proposed review in the FEDERAL REGISTER. Such notice must:

(1) Provide information regarding the location of data and other information pertaining to the review to be conducted and other information including new scientific matter bearing on the application of the criteria for avoiding filtration; and

(2) Advise the public of the opportunity to submit comments.

(c) Upon completion of any such review, the Administrator shall notify each State affected by the results of the review and shall make the results available to the public.

§ 142.81 Notice to the State.

(a) If the Administrator finds through periodic review or other available information that a State (1) has abused its discretion in applying the criteria for avoiding filtration under § 141.71 of this chapter in determining that a system does not have to provide filtration treatment, or (2) has failed to prescribe compliance schedules for those systems which must provide filtration in accordance with section 1412(b)(7)(C)(ii) of the Act, (s)he shall notify the State of these findings. Such notice shall:

(1) Identify each public water system for which the Administrator finds the State has abused its discretion;

(2) Specify the reasons for the finding;

(3) As appropriate, propose that the criteria of § 141.71 of this chapter be applied properly to determine the need for a public water system to provide filtration treatment or propose a revised schedule for compliance by the public water system with the filtration treatment requirements;

(b) The Administrator shall also notify the State that a public hearing is to be held on the provisions of the notice required by paragraph (a) of this section. Such notice shall specify the time and location of the hearing. If, upon notification of a finding by the Administrator that the State has abused its discretion under § 141.71 of this chapter, the State takes corrective action satisfactory to the Administrator, the Administrator may rescind the notice to the State of a public hearing.

(c) The Administrator shall publish notice of the public hearing in the FEDERAL REGISTER and in a newspaper of general circulation in the involved State, including a summary of the findings made pursuant to paragraph (a) of this section, a statement of the time and location for the hearing, and the address and telephone number of an office at which interested persons may obtain further information concerning the hearing.

(d) Hearings convened pursuant to paragraphs (b) and (c) of this section shall be conducted before a hearing officer to be designated by the Administrator. The hearing shall be conducted

by the hearing officer in an informal, orderly, and expeditious manner. The hearing officer shall have the authority to call witnesses, receive oral and written testimony, and take such other action as may be necessary to ensure the fair and efficient conduct of the hearing. Following the conclusion of the hearing, the hearing officer may make a recommendation to the Administrator based on the testimony presented at the hearing and shall forward any such recommendation and the record of the hearing to the Administrator.

(e) Within 180 days after the date notice is given pursuant to paragraph (b) of this section, the Administrator shall:

(1) Rescind the notice to the State of a public hearing if the State takes corrective action satisfactory to the Administrator; or

(2) Rescind the finding for which the notice was given and promptly notify the State of such rescission; or

(3) Uphold the finding for which the notice was given. In this event, the Administrator shall revoke the State's decision that filtration was not required or revoke the compliance schedule approved by the State, and promulgate, as appropriate, with any appropriate modifications, a revised filtration decision or compliance schedule and promptly notify the State of such action.

(f) Revocation of a State's filtration decision or compliance schedule and/or promulgation of a revised filtration decision or compliance schedule shall take effect 90 days after the State is notified under paragraph (e)(3) of this section.

Subpart J—Procedures for PWS Administrative Compliance Orders

SOURCE: 56 FR 3755, Jan. 30, 1991, unless otherwise noted.

§ 142.201 Purpose.

This part prescribes procedures for notice and opportunity for public hearings, conferences with primary States and issuance of administrative compliance orders under section 1414(g) of the Safe Drinking Water Act, 42 U.S.C. 300g–3(g).

§ 142.202 Definitions.

(a) The term *Hearing Officer* means an Environmental Protection Agency employee who has been delegated by the Administrator the authority to preside over a public hearing held pursuant to section 1414(g)(2) of the Safe Drinking Water Act, 42 U.S.C. 300g–3(g)(2).

(b) The term *party* means any ''person'' or ''supplier of water'' as defined in section 1401 of the SDWA, 42 U.S.C. 300f, alleged to have violated any regulation implementation section 1412 of the SDWA, 42 U.S.C. 300g–1, any schedule or other requirement imposed pursuant to section 1415 or section 1416 of the SDWA, 42 U.S.C. 300g–4 and 300g–5, or section 1445 of the SDWA, 42 U.S.C. 300j–4, or any regulation implementing section 1445.

§ 142.203 Proposed administrative compliance orders.

If the Administrator finds that a party has violated a regulation, schedule, or other requirement of the SDWA referenced in § 142.202(b), the Administrator may prepare a proposed administrative compliance order that would require the party to comply with the regulation, schedule, or other requirement that is alleged to have been violated. Any such proposed administrative order shall state with reasonable specificity the nature of the violation, and may, if appropriate, specify a reasonable time for compliance.

§ 142.204 Notice of proposed administrative compliance orders.

The Administrator shall simultaneously provide a copy of any proposed administrative compliance order to:

(a) *The party*. The Administrator shall provide a copy of a proposed compliance order to the party personally or by sending it to the party by certified mail, return receipt requested. The Administrator shall provide a copy of a proposed administrative compliance order to an appropriate person, such as the affected location or facility manager, or any other appropriate employee or agent of the party who in the ordinary course of business is authorized to sign for certified mail on behalf of the party. If the party is a federal agency, State or State agency, or a

local unit of government, the Administrator shall provide a copy of a proposed administrative order to its chief executive officer, or its authorized agent for receipt of certified mail. Notification of the party is complete upon acceptance of personal service or when the return receipt is signed. If personal service is ineffective and if certified mail is refused or unclaimed, the Administrator shall notify the party by another appropriate means. In such case, notification is complete upon the execution of substituted service.

(b) *The public.* The Administrator shall make publicly available each proposed administrative compliance order at the time of its proposal.

(c) *The State.* In the case of a State with primary enforcement responsibility for public water systems pursuant to section 1413(a) of the SDWA, 42 U.S.C. 300g–2(a), the Administrator shall provide notice under this subsection by sending a copy of each proposed administrative compliance order by certified mail, return receipt requested to the appropriate State agency of the State involved.

§ 142.205 Opportunity for public hearings; opportunity for State conferences.

(a) The Administrator shall provide the party, the public and the State an opportunity for a public hearing on any proposed administrative compliance order by stating in a letter accompanying each proposed administrative compliance order (or its copy) that a public hearing shall be convened if the party or the State sends written notice of such request to the Administrator within fourteen days of receipt of the proposed administrative compliance order noticed under § 142.204, or if the Administrator determines that within fourteen days of the date of notice the public has expressed a significant interest in the convening of a public hearing. Hearings will be held only for the purposes specified in § 142.206(a). All requests for hearings shall identify which of the purposes specified in § 142.206(a) is the basis for the request. The Administrator may extend the time allowed for submitting requests for good cause.

(b) In the case of a State with primary enforcement responsibility under section 1413(a) of the SDWA, the Administrator shall provide the State with an opportunity to confer regarding any proposed administrative compliance order to a public water supplier by stating in a letter accompanying each mailing of the proposed administrative compliance order sent to the State that such a conference shall be held between the State and the Administrator, if the State requests such a conference within ten days of the dates of receipt of proposed administrative compliance order noticed under § 142.204.

(c) For purposes of this subsection, receipt occurs at the time of personal service or three days after the date of mailing or other means of substituted service, except that if receipt is provided by certified mail, return receipt requested, notice occurs when the return receipt is signed. For the purpose of computation of time, the day of the mailing, Saturdays, Sundays, and federal holidays are excluded.

§ 142.206 Conduct of public hearings.

(a) The purpose of the public hearing shall be to determine whether a proposed administrative order:

(1) Has correctly stated the extent and nature of a party's violation of any regulation, schedule, or other requirement of the SDWA referenced in § 142.202(b) and

(2) Has provided, where appropriate, a reasonable time for the party to comply with applicable requirements of the SDWA and its implementing regulations.

(b) Prior to convening a public hearing under this subsection, the Administrator shall appoint a Hearing Officer. The Hearing Officer shall preside over any public hearing convened under this section. The Hearing Officer shall determine the form and procedures of the public hearing, and shall maintain complete and accurate record of the proceedings in written or other permanent form. The Hearing Officer shall provide the Administrator with the record of any public hearing conducted under this subsection.

(c) The party, any member of the public, or the State may present information to the Hearing Officer at the public hearing (or to the Administrator in writing before the date set for the public hearing) relevant to whether:

(1) The party has violated the applicable regulation, schedule, or other requirement referenced in the proposed administrative compliance order;

(2) The party has violated any other applicable regulation, schedule, or other requirement of the SDWA referenced in §142.202(b); and

(3) The proposed order, where appropriate, provides a reasonable time for the party to comply with applicable requirements of the SDWA and its implementing regulations.

§142.207 Issuance, amendment or withdrawal of administrative compliance order.

(a) Based on the administrative record, the Administrator shall either issue the order as proposed, amend the proposed order or withdraw the proposed order.

(b) Any order issued shall require the party to comply with any applicable regulation, schedule, or other requirement of the SDWA referenced in §142.202(b) and may establish a time or date for compliance which the Administrator determines is reasonable, based on the administrative record.

(c) The Administrator shall determine within a reasonable time whether to issue, amend or withdraw the proposed order and shall promptly notify in writing the party, all members of the public participating under §142.206(c) and the State, in the case of a State with primary enforcement authority over public water systems pursuant to section 1413(a) of the SDWA, or in the case of a State participating under §142.206(c).

§142.208 Administrative assessment of civil penalty for violation of administrative compliance order.

In the event the Administrator decides to seek a penalty under the authority provided in section 1414(g)(3)(B) of the SDWA, 42 U.S.C. 300g–3(g)(3)(B), for violation of, or failure or refusal to comply with, an order, the procedures provided in 40 CFR part 22 shall govern the assessment of such a penalty.

PART 143—NATIONAL SECONDARY DRINKING WATER REGULATIONS

Sec.
143.1 Purpose.
143.2 Definitions.
143.3 Secondary maximum contaminant levels.
143.4 Monitoring.
143.5 Compliance with secondary maximum contaminant level and public notification for fluoride.

AUTHORITY: 42 U.S.C. 300f *et seq.*

SOURCE: 44 FR 42198, July 19, 1979, unless otherwise noted.

§143.1 Purpose.

This part establishes National Secondary Drinking Water Regulations pursuant to section 1412 of the Safe Drinking Water Act, as amended (42 U.S.C. 300g–1). These regulations control contaminants in drinking water that primarily affect the aesthetic qualities relating to the public acceptance of drinking water. At considerably higher concentrations of these contaminants, health implications may also exist as well as aesthetic degradation. The regulations are not Federally enforceable but are intended as guidelines for the States.

§143.2 Definitions.

(a) *Act* means the Safe Drinking Water Act as amended (42 U.S.C. 300f *et seq.).*

(b) *Contaminant* means any physical, chemical, biological, or radiological substance or matter in water.

(c) *Public water system* means a system for the provision to the public of piped water for human consumption, if such a system has at least fifteen service connections or regularly serves an average of at least twenty-five individuals daily at least 60 days out of the year. Such term includes (1) any collection, treatment, storage, and distribution facilities under control of the operator of such system and used primarily in connection with such system, and (2) any collection or pretreatment storage facilities not under such control which are used primarily in connection with such system. A public

water system is either a ''community water system'' or a ''non-community water system.''

(d) *State* means the agency of the State or Tribal government which has jurisdiction over public water systems. During any period when a State does not have responsibi1ity pursuant to section 1443 of the Act, the term ''State'' means the Regional Administrator, U.S. Environmental Protection Agency.

(e) *Supplier of water* means any person who owns or operates a public water system.

(f) *Secondary maximum contaminant levels* means SMCLs which apply to public water systems and which, in the judgement of the Administrator, are requisite to protect the public welfare. The SMCL means the maximum permissible level of a contaminant in water which is delivered to the free flowing outlet of the ultimate user of public water system. Contamimants added to the water under circumstances controlled by the user, except those resulting from corrosion of piping and plumbing caused by water quality, are excluded from this definition.

[44 FR 42198, July 19, 1979, as amended at 53 FR 37412, Sept. 26, 1988]

§ 143.3 Secondary maximum contaminant levels.

The secondary maximum contaminant levels for public water systems are as follows:

Contaminant	Level
Aluminum	0.05 to 0.2 mg/l.
Chloride	250 mg/l.
Color	15 color units.
Copper	1.0 mg/l.
Corrosivity	Non-corrosive.
Fluoride	2.0 mg/l.
Foaming agents	0.5 mg/l.
Iron	0.3 mg/l.
Manganese	0.05 mg/l.
Odor	3 threshold odor number.
pH	6.5–8.5.
Silver	0.1 mg/l.
Sulfate	250 mg/l.
Total dissolved solids (TDS)	500 mg/l.
Zinc	5 mg/l.

These levels represent reasonable goals for drinking water quality. The States may establish higher or lower levels which may be appropriate dependent upon local conditions such as unavailability of alternate source waters or other compelling factors, provided that public health and welfare are not adversely affected.

[44 FR 42198, July 19, 1979, as amended at 51 FR 11412, Apr. 2, 1986; 56 FR 3597, Jan. 30, 1991]

§ 143.4 Monitoring.

(a) It is recommended that the parameters in these regulations should be monitored at intervals no less frequent than the monitoring performed for inorganic chemical contaminants listed in the National Interim Primary Drinking Water Regulations as applicable to community water systems. More frequent monitoring would be appropriate for specific parameters such as pH, color, odor or others under certain circumstances as directed by the State.

(b) Measurement of pH, copper and fluoride to determine compliance under § 143.3 may be conducted with one of the methods in § 141.23(k)(1). Analyses of aluminum, chloride, foaming agents, iron, manganese, odor, silver, sulfate, total dissolved solids (TDS) and zinc to determine compliance under § 143.3 may be conducted with the methods in the following table. Criteria for analyzing aluminum, copper, iron, manganese, silver and zinc samples with digestion or directly without digestion, and other analytical test procedures are contained in *Technical Notes on Drinking Water Methods,* EPA–600/R–94–173, October 1994, which is available at NTIS PB95–104766.

Contaminant	EPA	ASTM [3]	SM4	Other
Aluminum	[2] 200.7		3120B.	
	[2] 200.8		3113B.	
	[2] 200.9		3111D.	
Chloride	[1] 300.0	D4327–91	4110 4500–Cl—D.	
Color			2120B.	
Foaming Agents			5540C.	
Iron	[2] 200.7		3120B.	

Contaminant	EPA	ASTM [3]	SM4	Other
	[2] 200.9		3111B	
			3113B.	
Manganese	[2] 200.7		3120B.	
	[2] 200.8		3111B.	
	[2] 200.9		3113B.	
Odor			2150B.	
Silver	[2] 200.7		3120B	I–3720–85 [5]
	[2] 200.8		3111B.	
	[2] 200.9		3113B.	
Sulfate	[1] 300.0	D4327–91	4110.	
	[1] 375.2		4500–SO_4–F	
			4500–SO_4–C,D.	
TDS			2540C.	
Zinc	[2] 200.7		3120B.	
	[2] 200.8		3111B.	

FOOTNOTES:

[1] ''Methods for the Determination of Inorganic Substances in Environmental Samples'', EPA–600/R–93–100, August 1993. Available at NTIS, PB94–121811.

[2] ''Methods for the Determination of Metals in Environmental Samples—Supplement I'', EPA–600/R–94–111, May 1994. Available at NTIS, PB94–184942.

[3] The procedures shall be done in accordance with the *Annual Book of ASTM Standards,* 1994, Vols. 11.01 and 11.02, American Society for Testing and Materials. This incorporation by reference was approved by the Director of the Federal Register in accordance with 5 U.S.C. 552(a) and 1 CFR Part 51. Copies may be obtained from the American Society for Testing and Materials, 1916 Race Street, Philadelphia, PA 19103. Copies may be inspected at EPA's Drinking Water Docket, 401 M Street, SW., Washington, DC 20460; or at the Office of the Federal Register, 800 North Capitol Street, NW., Suite 700, Washington, DC.

[4] The procedures shall be done in accordance with the 18th edition of *Standard Methods for the Examination of Water and Wastewater,* 1992, American Public Health Association. This incorporation by reference was approved by the Director of the Federal Register in accordance with 5 U.S.C. 552(a) and 1 CFR Part 51. Copies may be obtained from the American Public Health Association, 1015 Fifteenth Street NW., Washington, DC 20005. Copies may be inspected at EPA's Drinking Water Docket, 401 M Street, SW., Washington, DC 20460; or at the Office of the Federal Register, 800 North Capitol Street, NW., Suite 700, Washington, DC.

[5] Available from Books and Open-File Reports Section, U.S. Geological Survey, Federal Center, Box 25425, Denver, CO 80225–0425.

[44 FR 42198, July 19, 1979, as amended at 53 FR 5147, Feb. 19, 1988; 56 FR 30281, July 1, 1991; 59 FR 62470, Dec. 5, 1994]

§ 143.5 Compliance with secondary maximum contaminant level and public notification for fluoride.

(a) Community water systems, as defined in 40 CFR 141.2(e)(i) of this title, that exceed the secondary maximum contaminant level for fluoride as determined by the last single sample taken in accordance with the requirements of § 141.23 of this title or any equivalent state law, but do not exceed the maximum contaminant level for flouride as specified by § 141.62 of this title or any equivalent state law, shall provivde the notice described in paragraph (b) of all billing units annually, all new billing units at the time service begins, and the state public health officer.

(b) The notice required by paragraph (a) shall contain the following language including the language necessary to replace the superscripts:

PUBLIC NOTICE

Dear User,

The U.S. Environmental Protection Agency requires that we send you this notice on the level of fluoride in your drinking water. The drinking water in your community has a fluoride concentration of [1] milligrams per liter (mg/l).

Federal regulations require that fluoride, which occurs naturally in your water supply, not exceed a concentration of 4.0 mg/l in drinking water. This is an enforceable standard called a Maximum Contaminant Level (MCL), and it has been established to protect the public health. Exposure to drinking water levels above 4.0 mg/l for many years may result in some cases of crippling skeletal fluorosis, which is a serious bone disorder.

Federal law also requires that we notify you when monitoring indicates that the fluoride in your drinking water exceeds 2.0 mg/l. This is intended to alert families about dental problems that might affect children under nine years of age. The fluoride concentration of your water exceeds this federal guideline.

Fluoride in children's drinking water at levels of approximately 1 mg/l reduces the number of dental cavities. However, some children exposed to levels of fluoride greater than about 2.0 mg/l may develop dental fluorosis. Dental fluorosis, in its moderate and severe forms, is a brown staining and/or pitting of the *permanent* teeth.

Because dental fluorosis occurs only when *developing* teeth (before they erupt from the gums) are exposed to elevated fluoride levels, households without children are not expected to be affected by this level of fluoride.

Families with children under the age of nine are encouraged to seek other sources of drinking water for their children to avoid the possibility of staining and pitting.

Your water supplier can lower the concentration of fluoride in your water so that you will still receive the benefits of cavity prevention while the possibility of stained and pitted teeth is minimized. Removal of fluoride may increase your water costs. Treatment systems are also commercially available for home use. Information on such systems is available at the address given below. Low fluoride bottled drinking water that would meet all standards is also commercially available.

For further information, contact [2] at your water system.

[1] PWS shall insert the compliance result which triggered notification under this part.

[2] PWS shall insert the name, address, and telephone number of a contact person at the PWS.

(c) The effective date of this section is May 2, 1986.

[51 FR 11412, Apr. 2, 1986; 51 FR 24329, July 3, 1986, as amended at 52 FR 41550, Oct. 28, 1987]

PART 144—UNDERGROUND INJECTION CONTROL PROGRAM

Subpart A—General Provisions

Sec.
144.1 Purpose and scope of part 144.
144.2 Promulgation of Class II programs for Indian lands.
144.3 Definitions.
144.4 Considerations under Federal law.
144.5 Confidentiality of information.
144.6 Classification of wells.
144.7 Identification of underground sources of drinking water and exempted aquifers.
144.8 Noncompliance and program reporting by the Director.

Subpart B—General Program Requirements

144.11 Prohibition of unauthorized injection.
144.12 Prohibition of movement of fluid into underground sources of drinking water.
144.13 Prohibition of Class IV wells.
144.14 Requirements for wells injecting hazardous waste.
144.15 [Reserved]
144.16 Waiver of requirement by Director.
144.17 Records.

Subpart C—Authorization of Underground Injection by Rule

144.21 Existing Class I, II (except enhanced recovery and hydrocarbon storage) and III wells.
144.22 Existing Class II enhanced recovery and hydrocarbon storage wells.
144.23 Class IV wells.
144.24 Class V wells.
144.25 Requiring a permit.
144.26 Inventory requirements.
144.27 Requiring other information.
144.28 Requirements for Class I, II, and III wells authorized by rule.

Subpart D—Authorization by Permit

144.31 Application for a permit; authorization by permit.
144.32 Signatories to permit applications and reports.
144.33 Area permits.
144.34 Emergency permits.
144.35 Effect of a permit.
144.36 Duration of permits.
144.37 Continuation of expiring permits.
144.38 Transfer of permits.
144.39 Modification or revocation and reissuance of permits.
144.40 Termination of permits.
144.41 Minor modifications of permits.

Subpart E—Permit Conditions

144.51 Conditions applicable to all permits.
144.52 Establishing permit conditions.
144.53 Schedule of compliance.
144.54 Requirements for recording and reporting of monitoring results.
144.55 Corrective action.

Subpart F—Financial Responsibility: Class I Hazardous Waste Injection Wells

144.60 Applicability.
144.61 Definitions of terms as used in this subpart.
144.62 Cost estimate for plugging and abandonment.
144.63 Financial assurance for plugging and abandonment.
144.64 Incapacity of owners or operators, guarantors, or financial institutions.
144.65 Use of State-required mechanisms.
144.66 State assumption of responsibility.
144.70 Wording of the instruments.

AUTHORITY: Safe Drinking Water Act, 42 U.S.C. 300f *et seq;* Resource Conservation and Recovery Act, 42 U.S.C. 6901 *et seq.*

SOURCE: 48 FR 14189, Apr. 1, 1983, unless otherwise noted.

Subpart A—General Provisions

§ 144.1 Purpose and scope of part 144.

(a) *Contents of part 144.* The regulations in this part set forth requirements for the Underground Injection Control (UIC) program promulgated under Part C of the Safe Drinking

Water Act (SDWA) (Pub. L. 93–523, as amended; 42 U.S.C. 300f *et seq.*) and, to the extent that they deal with hazardous waste, the Resource Conservation and Recovery Act (RCRA) (Pub. L. 94–580 as amended; 42 U.S.C. 6901 *et seq.).*

(b) *Applicability*. (1) The regulations in this part establish minimum requirements for UIC programs. To the extent set forth in part 145, each State must meet these requirements in order to obtain primary enforcement authority for the UIC program in that State.

(2) In addition to serving as minimum requirements for UIC programs, the regulations in this part constitute a part of the UIC program for States listed in part 147 to be administered directly by EPA.

(c) The information requirements located in the following sections have been cleared by the Office of Management and Budget: Sections 144.11, 144.28(c)(d)(i), 144.31, 14.33, 144.51(j)(m)(n), 144.52(a), 144.54, 144.55, 144.15, 144.23, 144.26, 144.27, 144.28(i)(k), 144.51(o), 146.52. The OMB clearance number is 2040–0042.

(d) *Authority*. (1) Section 1421 of SDWA requires the Administrator to promulgate regulations establishing minimum requirements for effective UIC programs.

(2) Section 1422 of SDWA requires the Administrator to list in the FEDERAL REGISTER ''each State for which in his judgment a State underground injection control program may be necessary to assure that underground injection will not endanger drinking water sources'' and to establish by regulation a program for EPA administration of UIC programs in the absence of an approved State program in a listed State.

(3) Section 1423 of SDWA provides procedures for EPA enforcement of UIC requirements.

(4) Section 1431 authorizes the Administrator to take action to protect the health of persons when a contaminant which is present in or may enter a public water system or underground source of drinking water may present an imminent and substantial endangerment to the health of persons.

(5) Section 1445 of SDWA authorizes the promulgation of regulations for such recordkeeping, reporting, and monitoring requirements ''as the Administrator may reasonably require * * * to assist him in establishing regulations under this title,'' and a ''right of entry and inspection to determine compliance with this title, including for this purpose, inspection, at reasonable time, or records, files, papers, processes, controls, and facilities * * *.''

(6) Section 1450 of SDWA authorizes the Administrator ''to prescribe such regulations as are necessary or appropriate to carry out his functions'' under SDWA.

(e) *Overview of the UIC program.* An UIC program is necessary in any State listed by EPA under section 1422 of the SDWA. Because all States have been listed, the SDWA requires all States to submit an UIC program within 270 days after July 24, 1980, the effective date of 40 CFR part 146, which was the final element of the UIC minimum requirements to be originally promulgated, unless the Administrator grants an extension, which can be for a period not to exceed an additional 270 days. If a State fails to submit an approvable program, EPA will establish a program for that State. Once a program is established, SDWA provides that all underground injections in listed States are unlawful and subject to penalties unless authorized by a permit or a rule. This part sets forth the requirements governing all UIC programs, authorizations by permit or rule and prohibits certain types of injection. The technical regulations governing these authorizations appear in 40 CFR part 146.

(f) *Structure of the UIC program*—(1) *Part 144.* This part sets forth the permitting and other program requirements that must be met by UIC Programs, whether run by a State or by EPA. It is divided into the following subparts:

(i) Subpart A describes general elements of the program, including definitions and classifications.

(ii) Subpart B sets forth the general program requirements, including the performance standards applicable to all injection activities, basic elements that all UIC programs must contain, and provisions for waiving permit of rule requirements under certain circumstances.

(iii) Subpart C sets forth requirements for wells authorized by rule.

(iv) Subpart D sets forth permitting procedures.

(v) Subpart E sets forth specific conditions, or types of conditions, that must at a minimum be included in all permits.

(vi) Subpart F sets forth the financial responsibility requirements for owners and operators of all existing and new Class I hazardous waste injection wells.

(2) *Part 145.* While part 144 sets forth minimum requirements for all UIC Programs, these requirements are specifically identified as elements of a State application for primacy to administer an UIC Program in part 145. Part 145 also sets forth the necessary elements of a State submission and the procedural requirements for approval of State programs.

(3) *Part 124.* The public participation requirements that must be met by UIC Programs, whether administered by the State or by EPA, are set forth in part 124. EPA must comply with all part 124 requirements; State administered programs must comply with part 124 as required by part 145. These requirements carry out the purposes of the public participation requirement of 40 CFR part 25 (Public Participation), and supersede the requirements of that part as they apply to the UIC Program.

(4) *Part 146.* This part sets forth the technical criteria and standards that must be met in permits and authorizations by rule as required by part 144.

(g) *Scope of the permit or rule requirement.* The UIC Permit Program regulates underground injections by five classes of wells (see definition of ''well injection,'' § 144.3). The five classes of wells are set forth in § 144.6. All owners or operators of these injection wells must be authorized either by permit or rule by the Director. In carrying out the mandate of the SDWA, this subpart provides that no injection shall be authorized by permit or rule if it results in the movement of fluid containing any contaminant into Underground Sources of Drinking Water (USDWs—see § 144.3 for definition), if the presence of that contaminant may cause a violation of any primary drinking water regulation under 40 CFR part 142 or may adversely affect the health of persons (§ 144.12). Existing Class IV wells which inject hazardous waste directly into an underground source of drinking water are to be eliminated over a period of six months and new such Class IV wells are to be prohibited (§ 144.13). Class V wells will be inventoried and assessed and regulatory action will be established at a later date.

In the meantime, if remedial action appears necessary, an individual permit may be required (§ 144.25) or the Director must require remedial action or closure by order (§ 144.12(c)). During UIC program development, the Director may identify aquifers and portions of aquifers which are actual or potential sources of drinking water. This will provide an aid to the Director in carrying out his or her duty to protect all USDWs. An aquifer is a USDW if it fits the definition, even if it has not been ''identified.'' The Director may also designate ''exempted aquifers'' using criteria in § 146.04. Such aquifers are those which would otherwise qualify as ''underground sources of drinking water'' to be protected, but which have no real potential to be used as drinking water sources. Therefore, they are not USDWs. No aquifer is an ''exempted aquifer'' until it has been affirmatively designated under the procedures in § 144.7. Aquifers which do not fit the definition of ''underground sources of drinking water'' are not ''exempted aquifers.'' They are simply not subject to the special protection afforded USDWs.

(1) *Specific inclusions.* The following wells are included among those types by injection activities which are covered by the UIC regulations. (This list is not intended to be exclusive but is for clarification only.)

(i) Any injection well located on a drilling platform inside the State's territorial waters.

(ii) Any dug hole or well that is deeper than its largest surface dimension, where the principal function of the hole is emplacement of fluids.

(iii) Any septic tank or cesspool used by generators of hazardous waste, or by owners or operators of hazardous waste management facilities, to dispose of fluids containing hazardous waste.

(iv) Any septic tank, cesspool, or other well used by a multiple dwelling, community, or Regional system for the injection of wastes.

(2) *Specific exclusions.* The following are not covered by these regulations:

(i) Injection wells located on a drilling platform or other site that is beyond the State's territorial waters.

(ii) Individual or single family residential waste disposal systems such as domestic cesspools or septic systems.

(iii) Non-residential cesspools, septic systems or similar waste disposal systems if such systems (A) Are used solely for the disposal of sanitary waste, and (B) have the capacity to serve fewer than 20 persons a day.

(iv) Injection wells used for injection of hydrocarbons which are of pipeline quality and are gases at standard temperature and pressure for the purpose of storage.

(v) Any dug hole which is not used for emplacement of fluids underground.

(3) The prohibition applicable to Class IV wells under §144.13 does not apply to injections of hazardous wastes into aquifers or portions thereof which have been exempted pursuant to §146.04.

(h) *Interim Status under RCRA for Class I Hazardous Waste Injection Wells.* The minimum national standards which define acceptable injection of hazardous waste during the period of interim status under RCRA are set out in the applicable provisions of this part, parts 146 and 147, and §265.430 of this chapter. The issuance of a UIC permit does not automatically terminate RCRA interim status. A Class I well's interim status does, however, automatically terminate upon issuance to that well of a RCRA permit, or upon the well's receiving a RCRA permit-by-rule under §270.60(b) of this chapter. Thus, until a Class I well injecting hazardous waste receives a RCRA permit or RCRA permit-by-rule, the well's interim status requirements are the applicable requirements imposed pursuant to this part and parts 146, 147, and 265 of this chapter, including any requirements imposed in the UIC permit.

[48 FR 14189, Apr. 1, 1983, as amended at 49 FR 20181, May 11, 1984; 52 FR 20676, June 2, 1987; 52 FR 45797, Dec. 1, 1987; 53 FR 28147, July 26, 1988]

§144.2 Promulgation of Class II programs for Indian lands.

Notwithstanding the requirements of this part or parts 124 and 146 of this chapter, the Administrator may promulgate an alternate UIC Program for Class II wells on any Indian reservation or Indian lands. In promulgating such a program the Administrator shall consider the following factors:

(a) The interest and preferences of the tribal government having responsibility for the given reservation or Indian lands;

(b) The consistency between the alternate program and any program in effect in an adjoining jurisdiction; and

(c) Such other factors as are necessary and appropriate to carry out the Safe Drinking Water Act.

§144.3 Definitions.

Terms not defined in this section have the meaning given by the appropriate Act. When a defined term appears in a definition, the defined term is sometimes placed within quotation marks as an aid to readers.

Administrator means the Administrator of the United States Environmental Protection Agency, or an authorized representative.

Application means the EPA standard national forms for applying for a permit, including any additions, revisions or modifications to the forms; or forms approved by EPA for use in approved States, including any approved modifications or revisions.

Appropriate Act and regulations means the Solid Waste Disposal Act, as amended by the Resource Conservation and Recovery Act (RCRA); or Safe Drinking Water Act (SDWA), whichever is applicable; and applicable regulations promulgated under those statutes.

Approved State Program means a UIC program administered by the State or Indian Tribe that has been approved by

EPA according to SDWA sections 1422 and/or 1425.

Aquifer means a geological ''formation,'' group of formations, or part of a formation that is capable of yielding a significant amount of water to a well or spring.

Area of review means the area surrounding an injection well described according to the criteria set forth in § 146.06 or in the case of an area permit, the project area plus a circumscribing area the width of which is either ¼ of a mile or a number calculated according to the criteria set forth in § 146.06.

Contaminant means any physical, chemical, biological, or radiological substance or matter in water.

Director means the Regional Administrator, the State director or the Tribal director as the context requires, or an authorized representative. When there is no approved State or Tribal program, and there is an EPA administered program, ''Director'' means the Regional Administrator. When there is an approved State or Tribal program, ''Director'' normally means the State or Tribal director. In some circumstances, however, EPA retains the authority to take certain actions even when there is an approved State or Tribal program. In such cases, the term ''Director'' means the Regional Administrator and not the State or Tribal director.

Draft permit means a document prepared under § 124.6 indicating the Director's tentative decision to issue or deny, modify, revoke and reissue, terminate, or reissue a ''permit.'' A notice of intent to terminate a permit, and a notice of intent to deny a permit, as discussed in § 124.5 are types of ''draft permits.'' A denial of a request for modification, revocation and reissuance, or termination, as discussed in § 124.5 is not a ''draft permit.''

Drilling mud means a heavy suspension used in drilling an ''injection well,'' introduced down the drill pipe and through the drill bit.

Eligible Indian Tribe is a Tribe that meets the statutory requirements established at 42 U.S.C. 300j-11(b)(1).

Emergency permit means a UIC ''permit'' issued in accordance with § 144.34.

Environmental Protection Agency (''EPA'') means the United States Environmental Protection Agency.

EPA means the United States ''Environmental Protection Agency.''

Exempted aquifer means an ''aquifer'' or its portion that meets the criteria in the definition of ''underground source of drinking water'' but which has been exempted according to the procedures in § 144.7.

Existing injection well means an ''injection well'' other than a ''new injection well.''

Facility or activity means any UIC ''injection well,'' or an other facility or activity that is subject to regulation under the UIC program.

Fluid means any material or substance which flows or moves whether in a semisolid, liquid, sludge, gas, or any other form or state.

Formation means a body of consolidated or unconsolidated rock characterized by a degree of lithologic homogeneity which is prevailingly, but not necessarily, tabular and is mappable on the earth's surface or traceable in the subsurface.

Formation fluid means ''fluid'' present in a ''formation'' under natural conditions as opposed to introduced fluids, such as ''drilling mud.''

Generator means any person, by site location, whose act or process produces hazardous waste identified or listed in 40 CFR part 261.

Ground water means water below the land surface in a zone of saturation.

Hazardous waste means a hazardous waste as defined in 40 CFR 261.3.

Hazardous waste management facility (''HWM facility'') means all contiguous land, and structures, other appurtenances, and improvements on the land used for treating, storing, or disposing of hazardous waste. A facility may consist of several treatment, storage, or disposal operational units (for example, one or more landfills, surface impoundments, or combination of them).

HWM facility means ''Hazardous Waste Management facility''

Indian lands means ''Indian country'' as defined in 18 U.S.C. 1151. That section defines Indian country as:

(a) All land within the limits of any Indian reservation under the jurisdiction of the United States government, notwithstanding the issuance of any patent, and, including rights-of-way running through the reservation;

(b) All dependent Indian communities within the borders of the United States whether within the original or subsequently acquired territory thereof, and whether within or without the limits of a State; and

(c) All Indian allotments, the Indian titles to which have not been extinguished, including rights-of-way running through the same.

Indian Tribe means any Indian Tribe having a Federally recognized governing body carrying out substantial governmental duties and powers over a defined area.

Injection well means a ''well'' into which ''fluids'' are being injected.

Injection zone means a geological ''formation'' group of formations, or part of a formation receiving fluids through a ''well.''

Interstate Agency means an agency of two or more States established by or under an agreement or compact approved by the Congress, or any other agency of two or more States or Indian Tribes having substantial powers or duties pertaining to the control of pollution as determined and approved by the Administrator under the ''appropriate Act and regulations.''

Major facility means any UIC ''facility or activity'' classified as such by the Regional Administrator, or, in the case of approved State programs, the Regional Administrator in conjunction with the State Director.

Manifest means the shipping document originated and signed by the ''generator'' which contains the information required by subpart B of 40 CFR part 262.

New injection wells means an ''injection well'' which began injection after a UIC program for the State applicable to the well is approved or prescribed.

Owner or operator means the owner or operator of any ''facility or activity'' subject to regulation under the UIC program.

Permit means an authorization, license, or equivalent control document issued by EPA or an approved State to implement the requirements of this part, parts 145, 146 and 124. ''Permit'' includes an area permit (§ 144.33) and an emergency permit (§ 144.34). Permit does not include UIC authorization by rule (§ 144.21), or any permit which has not yet been the subject of final agency action, such as a ''draft permit.''

Person means an individual, association, partnership, corporation, municipality, State, Federal, or Tribal agency, or an agency or employee thereof.

Plugging means the act or process of stopping the flow of water, oil or gas into or out of a formation through a borehole or well penetrating that formation.

Project means a group of wells in a single operation.

Radioactive Waste means any waste which contains radioactive material in concentrations which exceed those listed in 10 CFR part 20, appendix B, table II, column 2.

RCRA means the Solid Waste Disposal Act as amended by the Resource Conservation and Recovery Act of 1976 (Pub. L. 94–580, as amended by Pub. L. 95–609, Pub. L. 96–510, 42 U.S.C. 6901 *et seq.*).

Regional Administrator means the Regional Administrator of the appropriate Regional Office of the Environmental Protection Agency or the authorized representative of the Regional Administrator.

Schedule of compliance means a schedule of remedial measures included in a ''permit,'' including an enforceable sequence of interim requirements (for example, actions, operations, or milestone events) leading to compliance with the ''appropriate Act and regulations.''

SDWA means the Safe Drinking Water Act (Pub. L. 93–523, as amended; 42 U.S.C. 300f *et seq.*).

Site means the land or water area where any ''facility or activity'' is physically located or conducted, including adjacent land used in connection with the facility or activity.

State means any of the 50 States, the District of Columbia, Guam, the Commonwealth of Puerto Rico, the Virgin Islands, American Samoa, the Trust Territory of the Pacific Islands, the

Commonwealth of the Northern Mariana Islands, or an Indian Tribe treated as a State.

State Director means the chief administrative officer of any State, interstate, or Tribal agency operating an ''approved program,'' or the delegated representative of the State director. If the responsibility is divided among two or more States, interstate, or Tribal agencies, ''State Director'' means the chief administrative officer of the State, interstate, or Tribal agency authorized to perform the particular procedure or function to which reference is made.

State/EPA agreement means an agreement between the Regional Administrator and the State which coordinates EPA and State activities, responsibilities and programs.

Stratum (plural strata) means a single sedimentary bed or layer, regardless of thickness, that consists of generally the same kind of rock material.

Total dissolved solids means the total dissolved (filterable) solids as determined by use of the method specified in 40 CFR part 136.

Transferee means the owner or operator receiving ownership and/or operational control of the well.

Transferor means the owner or operator transferring ownership and/or operational control of the well.

UIC means the Underground Injection Control program under Part C of the Safe Drinking Water Act, including an ''approved State program.''

Underground injection means a ''well injection.''

Underground source of drinking water (USDW) means an aquifer or its portion:

(a)(1) Which supplies any public water system; or

(2) Which contains a sufficient quantity of ground water to supply a public water system; and

(i) Currently supplies drinking water for human consumption; or

(ii) Contains fewer than 10,000 mg/l total dissolved solids; and

(b) Which is not an exempted aquifer.

USDW means ''underground source of drinking water.''

Well means a bored, drilled or driven shaft, or a dug hole, whose depth is greater than the largest surface dimension.

Well injection means the subsurface emplacement of ''fluids'' through a bored, drilled, or driven ''well;'' or through a dug well, where the depth of the dug well is greater than the largest surface dimension.

[48 FR 14189, Apr. 1, 1983, as amended at 49 FR 45305, Nov. 15, 1984; 52 FR 20676, June 2, 1987; 53 FR 37412, Sept. 26, 1988; 58 FR 63895, Dec. 3, 1993; 59 FR 64345, Dec. 14, 1994]

§ 144.4 Considerations under Federal law.

The following is a list of Federal laws that may apply to the issuance of permits under these rules. When any of these laws is applicable, its procedures must be followed. When the applicable law requires consideration or adoption of particular permit conditions or requires the denial of a permit, those requirements also must be followed.

(a) The *Wild and Scenic Rivers Act,* 16 U.S.C. 1273 *et seq.* Section 7 of the Act prohibits the Regional Administrator from assisting by license or otherwise the construction of any water resources project that would have a direct, adverse effect on the values for which a national wild and scenic river was established.

(b) *The National Historic Preservation Act of 1966,* 16 U.S.C. 470 *et seq.* Section 106 of the Act and implementing regulations (36 CFR part 800) require the Regional Administrator, before issuing a license, to adopt measures when feasible to mitigate potential adverse effects of the licensed activity and properties listed or eligible for listing in the National Register of Historic Places. The Act's requirements are to be implemented in cooperation with State Historic Preservation Officers and upon notice to, and when appropriate, in consultation with the Advisory Council on Historic Preservation.

(c) The *Endangered Species Act,* 16 U.S.C. 1531 *et seq.* Section 7 of the Act and implementing regulations (50 CFR part 402) require the Regional Administrator to ensure, in consultation with the Secretary of the Interior or Commerce, that any action authorized by EPA is not likely to jeopardize the continued existence of any endangered or

threatened species or adversely affect its critical habitat.

(d) *The Coastal Zone Management Act,* 16 U.S.C. 1451 *et seq.* Section 307(c) of the Act and implementing regulations (15 CFR part 930) prohibit EPA from issuing a permit for an activity affecting land or water use in the coastal zone until the applicant certifies that the proposed activity complies with the State Coastal Zone Management program, and the State or its designated agency concurs with the certification (or the Secretary of Commerce overrides the States nonconcurrence).

(e) The *Fish and Wildlife Coordination Act,* 16 U.S.C. 661 *et seq.*, requires the Regional Administrator, before issuing a permit proposing or authorizing the impoundment (with certain exemptions), diversion, or other control or modification of any body of water, consult with the appropriate State agency exercising jurisdiction over wildlife resources to conserve these resources.

(f) *Executive orders.* [Reserved]

(Clean Water Act (33 U.S.C. 1251 *et seq.*), Safe Drinking Water Act (42 U.S.C. 300f *et seq.*), Clean Air Act (42 U.S.C. 7401 *et seq.*), Resource Conservation and Recovery Act (42 U.S.C. 6901 *et seq.*))

[48 FR 14189, Apr. 1, 1983, as amended at 48 FR 39621, Sept. 1, 1983]

§144.5 Confidentiality of information.

(a) In accordance with 40 CFR part 2, any information submitted to EPA pursuant to these regulations may be claimed as confidential by the submitter. Any such claim must be asserted at the time of submission in the manner prescribed on the application form or instructions or, in the case of other submissions, by stamping the words ''confidential business information'' on each page containing such information. If no claim is made at the time of submission, EPA may make the information available to the public without further notice. If a claim is asserted, the information will be treated in accordance with the procedures in 40 CFR part 2 (Public Information).

(b) Claims of confidentiality for the following information will be denied:

(1) The name and address of any permit applicant or permittee;

(2) Information which deals with the existence, absence, or level of contaminants in drinking water.

§144.6 Classification of wells.

Injection wells are classified as follows:

(a) *Class I.* (1) Wells used by generators of hazardous waste or owners or operators of hazardous waste management facilities to inject hazardous waste beneath the lowermost formation containing, within one-quarter mile of the well bore, an underground source of drinking water.

(2) Other industrial and municipal disposal wells which inject fluids beneath the lowermost formation containing, within one quarter mile of the well bore, an underground source of drinking water.

(b) *Class II.* Wells which inject fluids:

(1) Which are brought to the surface in connection with natural gas storage operations, or conventional oil or natural gas production and may be commingled with waste waters from gas plants which are an intergral part of production operations, unless those waters are classified as a hazardous waste at the time of injection.

(2) For enhanced recovery of oil or natural gas; and

(3) For storage of hydrocarbons which are liquid at standard temperature and pressure.

(c) *Class III.* Wells which inject for extraction of minerals including:

(1) Mining of sulfur by the Frasch process;

(2) In situ production of uranium or other metals; this category includes only in-situ production from ore bodies which have not been conventionally mined. Solution mining of conventional mines such as stopes leaching is included in Class V.

(3) Solution mining of salts or potash.

(d) *Class IV.* (1) Wells used by generators of hazardous waste or of radioactive waste, by owners or operators of hazardous waste management facilities, or by owners or operators of radioactive waste disposal sites to dispose of hazardous waste or radioactive waste into a formation which within one-quarter (¼) mile of the well contains

an underground source of drinking water.

(2) Wells used by generators of hazardous waste or of radioactive waste, by owners or operators of hazardous waste management facilities, or by owners or operators of radioactive waste disposal sites to dispose of hazardous waste or radioactive waste above a formation which within one-quarter (¼) mile of the well contains an underground source of drinking water.

(3) Wells used by generators of hazardous waste or owners or operators of hazardous waste management facilities to dispose of hazardous waste, which cannot be classified under paragraph (a)(1) or (d) (1) and (2) of this section (e.g., wells used to dispose of hazardous waste into or above a formation which contains an aquifer which has been exempted pursuant to § 146.04).

(e) *Class V.* Injection wells not included in Classes I, II, III, or IV.

[48 FR 14189, Apr. 1, 1983, as amended at 52 FR 20676, June 2, 1987]

§ 144.7 Identification of underground sources of drinking water and exempted aquifers.

(a) The Director may identify (by narrative description, illustrations, maps, or other means) and shall protect, except where exempted under paragraph (b) of this section, as an underground source of drinking water, all aquifers or parts of aquifers which meet the definition of an ''underground source of drinking water'' in § 144.3. Even if an aquifer has not been specifically identified by the Director, it is an underground source of drinking water if it meets the definition in § 144.3.

(b)(1) The Director may identify (by narrative description, illustrations, maps, or other means) and describe in geographic and/or geometric terms (such as vertical and lateral limits and gradient) which are clear and definite, all aquifers or parts thereof which the Director proposes to designate as exempted aquifers using the criteria in 40 CFR 146.04.

(2) No designation of an exempted aquifer submitted as part of a UIC Program shall be final until approved by the Administrator as part of a UIC program.

(3) Subsequent to program approval or promulgation, the Director may, after notice and opportunity for a public hearing, identify additional exempted aquifers. For approved State programs exemption of aquifers identifed (i) under § 146.04(b) shall be treated as a program revision under § 145.32; (ii) under § 146.04(c) shall become final if the State Director submits the exemption in writing to the Administrator and the Administrator has not disapproved the designation within 45 days. Any disapproval by the Administrator shall state the reasons and shall constitute final Agency action for purposes of judicial review.

(c)(1) For Class III wells, the Director shall require an applicant for a permit which necessitates an aquifer exemption under § 146.04(b)(1) to furnish the data necessary to demonstrate that the aquifer is expected to be mineral or hydrocarbon producing. Information contained in the mining plan for the proposed project, such as a map and general description of the mining zone, general information on the mineralogy and geochemistry of the mining zone, analysis of the amenability of the mining zone to the proposed mining method, and a time-table of planned development of the mining zone shall be considered by the Director in addition to the information required by § 144.31(g).

(2) For Class II wells, a demonstration of commercial producibility shall be made as follows:

(i) For a Class II well to be used for enhanced oil recovery processes in a field or project containing aquifers from which hydrocarbons were previously produced, commercial producibility shall be presumed by the Director upon a demonstration by the applicant of historical production having occurred in the project area or field.

(ii) For Class II wells not located in a field or project containing aquifers from which hydrocarbons were previously produced, information such as logs, core data, formation description, formation depth, formation thickness and formation parameters such as permeability and porosity shall be considered by the Director, to the extent such information is available.

§144.8 Noncompliance and program reporting by the Director.

The Director shall prepare quarterly and annual reports as detailed below. When the State is the permit-issuing authority, the State Director shall submit any reports required under this section to the Regional Administrator. When EPA is the permit-issuing authority, the Regional Administrator shall submit any report required under this section to EPA Headquarters.

(a) *Quarterly reports.* The Director shall submit quarterly narrative reports for major facilities as follows:

(1) *Format.* The report shall use the following format:

(i) Provide an alphabetized list of permittees. When two or more permittees have the same name, the lowest permit number shall be entered first.

(ii) For each entry on the list, include the following information in the following order:

(A) Name, location, and permit number of the noncomplying permittees.

(B) A brief description and date of each instance of noncompliance for that permittee. Instances of noncompliance may include one or more the kinds set forth in paragraph (a)(2) of this section. When a permittee has noncompliance of more than one kind, combine the information into a single entry for each such permittee.

(C) The date(s) and a brief description of the action(s) taken by the Director to ensure compliance.

(D) Status of the instance(s) of noncompliance with the date of the review of the status or the date of resolution.

(E) Any details which tend to explain or mitigate the instance(s) of noncompliance.

(2) *Instances of noncompliance to be reported.* Any instances of noncompliance within the following categories shall be reported in successive reports until the noncompliance is reported as resolved. Once noncompliance is reported as resolved it need not appear in subsequent reports.

(i) *Failure to complete construction elements.* When the permittee has failed to complete, by the date specified in the permit, an element of a compliance schedule involving either planning for construction or a construction step (for example, begin construction, attain operation level); and the permittee has not returned to compliance by accomplishing the required elements of the schedule within 30 days from the date a compliance schedule report is due under the permit.

(ii) *Modifications to schedules of compliance.* When a schedule of compliance in the permit has been modified under §§144.39 or 144.41 because of the permittee's noncompliance.

(iii) *Failure to complete or provide compliance schedule or monitoring reports.* When the permittee has failed to complete or provide a report required in a permit compliance schedule (for example, progress report or notice of noncompliance or compliance) or a monitoring report; and the permittee has not submitted the complete report within 30 days from the date it is due under the permit for compliance schedules, or from the date specified in the permit for monitoring reports.

(iv) *Deficient reports.* When the required reports provided by the permittee are so deficient as to cause misunderstanding by the Director and thus impede the review of the status of compliance.

(v) *Noncompliance with other permit requirements.* Noncompliance shall be reported in the following circumstances:

(A) Whenever the permittee has violated a permit requirement (other than reported under paragraph (a)(2) (i) or (ii) of this section), and has not returned to compliance within 45 days from the date reporting of noncompliance was due under the permit; or

(B) When the Director determines that a pattern of noncompliance exists for a major facility permittee over the most recent four consecutive reporting periods. This pattern includes any violation of the same requirement in two consecutive reporting periods, and any violation of one or more requirements in each of four consecutive reporting periods; or

(C) When the Director determines significant permit noncompliance or other significant event has occurred, such as a migration of fluids into a USDW.

(vi) *All other.* Statistical information shall be reported quarterly on all other instances of noncompliance by major facilities with permit requirements not

otherwise reported under paragraph (a) of this section.

(b) *Annual reports*—(1) *Annual noncompliance report.* Statistical reports shall be submitted by the Director on nonmajor UIC permittees indicating the total number reviewed, the number of noncomplying nonmajor permittees, the number of enforcement actions, and number of permit modifications extending compliance deadlines. The statistical information shall be organized to follow the types of noncompliance listed in paragraph (a) of this section.

(2) *For State-administered UIC Programs only.* In addition to the annual noncompliance report, the State Director shall:

(i) Submit each year a program report to the Administrator (in a manner and form prescribed by the Administrator) consisting of:

(A) A detailed description of the State's implementation of its program;

(B) Suggested changes, if any to the program description (see §145.23(f)) which are necessary to reflect more accurately the State's progress in issuing permits;

(C) An updated inventory of active underground injection operations in the State.

(ii) In addition to complying with the requirements of paragraph (b)(2)(i) of this section, the Director shall provide the Administrator, on February 28th and August 31st of each of the first two years of program operation, the information required in 40 CFR 146.15, 146.25, and 146.35.

(c) *Schedule.* (1) For all quarterly reports. On the last working day of May, August, November, and February, the State Director shall submit to the Regional Administrator information concerning noncompliance with permit requirements by major facilities in the State in accordance with the following schedule. The Regional Administrator shall prepare and submit information for EPA-issued permits to EPA Headquarters in accordance with the same schedule.

QUARTERS COVERED BY REPORTS ON NONCOMPLIANCE BY MAJOR FACILITIES

[Date for completion of reports]	
January, February, and March	[1] May 31
April, May, and June	[1] Aug. 31
July, August, and September	[1] Nov. 30
October, November, and December	[1] Feb. 28

[1] Reports must be made available to the public for inspection and copying on this date.

(2) *For all annual reports.* The period for annual reports shall be for the calendar year ending December 31, with reports completed and available to the public no more than 60 days later.

Subpart B—General Program Requirements

§ 144.11 Prohibition of unauthorized injection.

Any underground injection, except into a well authorized by rule or except as authorized by permit issued under the UIC program, is prohibited. The construction of any well required to have a permit is prohibited until the permit has been issued.

[48 FR 14189, Apr. 1, 1983, as amended at 58 FR 63895, Dec. 3, 1993]

§ 144.12 Prohibition of movement of fluid into underground sources of drinking water.

(a) No owner or operator shall construct, operate, maintain, convert, plug, abandon, or conduct any other injection activity in a manner that allows the movement of fluid containing any contaminant into underground sources of drinking water, if the presence of that contaminant may cause a violation of any primary drinking water regulation under 40 CFR part 142 or may otherwise adversely affect the health of persons. The applicant for a permit shall have the burden of showing that the requirements of this paragraph are met.

(b) For Class I, II and III wells, if any water quality monitoring of an underground source of drinking water indicates the movement of any contaminant into the underground source of drinking water, except as authorized under part 146, the Director shall prescribe such additional requirements for construction, corrective action, operation, monitoring, or reporting (including closure of the injection well) as are

necessary to prevent such movement. In the case of wells authorized by permit, these additional requirements shall be imposed by modifying the permit in accordance with §144.39, or the permit may be terminated under §144.40 if cause exists, or appropriate enforcement action may be taken if the permit has been violated. In the case of wells authorized by rule, see §§144.21 through 144.24. For EPA administered programs, such enforcement action shall be taken in accordance with appropriate sections of the SDWA.

(c) For Class V wells, if at any time the Director learns that a Class V well may cause a violation of primary drinking water regulations under 40 CFR part 142, he or she shall:

(1) Require the injector to obtain an individual permit;

(2) Order the injector to take such actions (including, where required, closure of the injection well) as may be necessary to prevent the violation. For EPA administered programs, such orders shall be issued in accordance with the appropriate provisions of the SDWA; or

(3) Take enforcement action.

(d) Whenever the Director learns that a Class V well may be otherwise adversely affecting the health of persons, he or she may prescribe such actions as may be necessary to prevent the adverse effect, including any action authorized under paragraph (c) of this section.

(e) Notwithstanding any other provision of this section, the Director may take emergency action upon receipt of information that a contaminant which is present in or likely to enter a public water system or underground source of drinking water may present an imminent and substantial endangerment to the health of persons. If the Director is an EPA official, he must first determine that the appropriate State and local authorities have not taken appropriate action to protect the health of such persons, before taking emergency action.

[48 FR 14189, Apr. 1, 1983, as amended at 52 FR 20676, June 2, 1987]

§144.13 Prohibition of Class IV wells.

(a) The following are prohibited, except as provided in paragraph (c) of this section:

(1) The construction of any Class IV well.

(2) The operation or maintenance of any Class IV well not in operation prior to July 18, 1980.

(3) The operation or maintenance of any Class IV well that was in operation prior to July 18, 1980, after six months following the effective date of a UIC program approved or promulgated for the state.

(4) Any increase in the amount of hazardous waste or change in the type of hazardous waste injected into a Class IV well.

(b) The owner or operator of a Class IV well shall comply with the requirements of §144.14, and with the requirements of §144.23 regarding closure of Class IV wells.

(c) Wells used to inject contaminated ground water that has been treated and is being reinjected into the same formation from which it was drawn are not prohibited by this section if such injection is approved by EPA pursuant to provisions for cleanup of releases under the Comprehensive Environmental Response, Compensation, and Liability Act of 1980 (CERCLA), 42 U.S.C. 9601–9657, or pursuant to requirements and provisions under the Resource Conservation and Recovery Act (RCRA), 42 U.S.C. 6901 through 6987.

(d) *Clarification*. The following wells are not prohibited by this action:

(1) Wells used to inject hazardous waste into aquifers or portions thereof that have been exempted pursuant to §146.4, if the exempted aquifer into which waste is injected underlies the lowermost formation containing a USDW. Such wells are Class I wells as specified in §144.6(a)(1), and the owner or operator must comply with the requirements applicable to Class I wells.

(2) Wells used to inject hazardous waste where no USDW exists within one quarter mile of the well bore in any underground formation, provided that the Director determines that such injection is into a formation sufficiently isolated to ensure that injected fluids do not migrate from the injection zone. Such wells are Class I wells

as specified in § 144.6(a)(1), and the owner or operator must comply with the requirements applicable to Class I wells.

[49 FR 20181, May 11, 1984]

§ 144.14 Requirements for wells injecting hazardous waste.

(a) *Applicability.* The regulations in this section apply to all generators of hazardous waste, and to the owners or operators of all hazardous waste management facilities, using any class of well to inject hazardous wastes accompanied by a manifest. (See also § 144.13.)

(b) *Authorization.* The owner or operator of any well that is used to inject hazardous waste required to be accompanied by a manifest or delivery document shall apply for authorization to inject as specified in § 144.31 within 6 months after the approval or promulgation of the State UIC program.

(c) *Requirements.* In addition to complying with the applicable requirements of this part and 40 CFR part 146, the owner or operator of each facility meeting the requirements of paragraph (b) of this section, shall comply with the following:

(1) *Notification.* The owner or operator shall comply with the notification requirements of section 3010 of Public Law 94–580.

(2) *Identification number.* The owner or operator shall comply with the requirements of 40 CFR 264.11.

(3) *Manifest system.* The owner or operator shall comply with the applicable recordkeeping and reporting requirements for manifested wastes in 40 CFR 264.71.

(4) *Manifest discrepancies.* The owner or operator shall comply with 40 CFR 264.72.

(5) *Operating record.* The owner or operator shall comply with 40 CFR 264.73(a), (b)(1), and (b)(2).

(6) *Annual report.* The owner or operator shall comply with 40 CFR 264.75.

(7) *Unmanifested waste report.* The owner or operator shall comply with 40 CFR 264.75.

(8) *Personnel training.* The owner or operator shall comply with the applicable personnel training requirements of 40 CFR 264.16.

(9) *Certification of closure.* When abandonment is completed, the owner or operator must submit to the Director certification by the owner or operator and certification by an independent registered professional engineer that the facility has been closed in accordance with the specifications in § 144.52(a)(6).

(d) *Additional requirements for Class IV wells.* [Reserved]

§ 144.15 [Reserved]

§ 144.16 Waiver of requirement by Director.

(a) *When injection does not occur into, through or above an underground source of drinking water,* the Director may authorize a well or project with less stringent requirements for area of review, construction, mechanical integrity, operation, monitoring, and reporting than required in 40 CFR part 146 or § 144.52 to the extent that the reduction in requirements will not result in an increased risk of movement of fluids into an underground source of drinking water.

(b) When injection occurs through or above an underground source of drinking water, but the radius of endangering influence when computed under § 146.06(a) is smaller or equal to the radius of the well, the Director may authorize a well or project with less stringent requirements for operation, monitoring, and reporting than required in 40 CFR part 146 or § 144.52 to the extent that the reduction in requirements will not result in an increased risk of movement of fluids into an underground source of drinking water.

(c) When reducing requirements under paragraph (a) or (b) of this section, the Director shall prepare a fact sheet under § 124.8 explaining the reasons for the action.

§ 144.17 Records.

The Director or the Administrator may require, by written notice on a selective well-by-well basis, an owner or operator of an injection well to establish and maintain records, make reports, conduct monitoring, and provide other information as is deemed necessary to determine whether the owner or operator has acted or is acting in

compliance with Part C of the SDWA or its implementing regulations.

[58 FR 63895, Dec. 3, 1993]

Subpart C—Authorization of Underground Injection by Rule

§144.21 Existing Class I, II (except enhanced recovery and hydrocarbon storage) and III wells.

(a) An existing Class I, II (except enhanced recovery and hydrocarbon storage) and III injection well is authorized by rule if the owner or operator injects into the existing well within one year after the date at which a UIC program authorized under the SDWA becomes effective for the first time or inventories the well pursuant to the requirements of §144.26. An owner or operator of a well which is authorized by rule pursuant to this section shall rework, operate, maintain, convert, plug, abandon or inject into the well in compliance with applicable regulations.

(b) *Duration of well authorization by rule.* Well authorization under this section expires upon the effective date of a permit issued pursuant to §§144.25, 144.31, 144.33 or 144.34; after plugging and abandonment in accordance with an approved plugging and abandonment plan pursuant to §§144.28(c) and 146.10, and upon submission of a plugging and abandonment report pursuant to §144.28(k); or upon conversion in compliance with §144.28(j).

(c) *Prohibitions on injection.* An owner or operator of a well authorized by rule pursuant to this section is prohibited from injecting into the well:

(1) Upon the effective date of an applicable permit denial;

(2) Upon failure to submit a permit application in a timely manner pursuant to §§144.25 or 144.31;

(3) Upon failure to submit inventory information in a timely manner pursuant to §144.26;

(4) Upon failure to comply with a request for information in a timely manner pursuant to §144.27;

(5) Upon failure to provide alternative financial assurance pursuant to §144.28(d)(7);

(6) Forty-eight hours after receipt of a determination by the Director pursuant to §144.28(f)(3) that the well lacks mechanical integrity, unless the Director requires immediate cessation;

(7) Upon receipt of notification from the Director pursuant to §144.28(l) that the transferee has not demonstrated financial responsibility pursuant to §144.28(d);

(8) For Class I and III wells:

(i) In States with approved programs, five years after the effective date of the UIC program unless a timely and complete permit application is pending the Director's decision; or

(ii) In States with programs administered by EPA, one year after the effective date of the UIC program unless a timely and complete permit application is pending the Director's decision; or

(9) For Class II wells (except enhanced recovery and hydrocarbon storage), five years after the effective date of the UIC program unless a timely and complete permit application is pending the Director's decision.

(d) *Class II and III wells in existing fields or projects.* Notwithstanding the prohibition in §144.11, this section authorizes Class II and Class III wells or projects in existing fields or projects to continue normal operations until permitted, including construction, operation, and plugging and abandonment of wells as part of the operation, provided the owner or operator maintains compliance with all applicable requirements.

(e) *Requirements.* The owner or operator of a well authorized under this section shall comply with the applicable requirements of §144.28 and part 147 of this chapter no later than one year after authorization.

[48 FR 14189, Apr. 1, 1983, as amended at 49 FR 20181, May 11, 1984; 58 FR 63895, Dec. 3, 1993]

§144.22 Existing Class II enhanced recovery and hydrocarbon storage wells.

(a) An existing Class II enhanced recovery or hydrocarbon storage injection well is authorized by rule for the life of the well or project, if the owner or operator injects into the existing well within one year after the date which a UIC program authorized under the SDWA becomes effective for the first time or inventories the well pursuant to the requirements of §144.26.

An owner or operator of a well which is authorized by rule pursuant to this section shall rework, operate, maintain, convert, plug, abandon or inject into the well in compliance with applicable regulations.

(b) *Duration of well authorization by rule.* Well authorization under this section expires upon the effective date of a permit issued pursuant to §§ 144.25, 144.31, 144.33 or 144.34; after plugging and abandonment in accordance with an approved plugging and abandonment plan pursuant to §§ 144.28(c) and 146.10 of this chapter, and upon submission of a plugging and abandonment report pursuant to § 144.28(k); or upon conversion in compliance with § 144.28(j).

(c) *Prohibitions on injection.* An owner or operator of a well authorized by rule pursuant to this section is prohibited from injecting into the well:

(1) Upon the effective date of an applicable permit denial;

(2) Upon failure to submit a permit application in a timely manner pursuant to §§ 144.25 or 144.31;

(3) Upon failure to submit inventory information in a timely manner pursuant to § 144.26;

(4) Upon failure to comply with a request for information in a timely manner pursuant to § 144.27;

(5) Upon failure to provide alternative financial assurance pursuant to § 144.28(d)(7);

(6) Forty-eight hours after receipt of a determination by the Director pursuant to § 144.28(f)(3) that the well lacks mechanical integrity, unless the Director requires immediate cessation; or

(7) Upon receipt of notification from the Director pursuant to § 144.28(l) that the transferee has not demonstrated financial responsibility pursuant to § 144.28(d).

(d) *Requirements.* The owner or operator of a well authorized under this section shall comply with the applicable requirements of § 144.28 and part 147 of this chapter. Such owner or operator shall comply with the casing and cementing requirements no later than 3 years and other requirements no later than 1 year after authorization.

[49 FR 20181, May 11, 1984, as amended at 58 FR 63896, Dec. 3, 1993]

§ 144.23 Class IV wells.

(a) Injection into existing Class IV wells is authorized for up to six months after approval or promulgation of the UIC Program. Such wells are subject to the requirements of §§ 144.13 and 144.14(c).

(b) *Closure.* For EPA administered programs only,

(1) Prior to abandoning any Class IV well, the owner or operator shall plug or otherwise close the well in a manner acceptable to the Regional Administrator.

(2) [Reserved]

(3) The owner or operator of a Class IV well must notify the Regional Administrator of intent to abandon the well at least thirty days prior to abandonment.

[49 FR 20181, May 11, 1984, as amended at 60 FR 33932, June 29, 1995]

§ 144.24 Class V wells.

(a) A Class V injection well is authorized by rule until further requirements under future regulations become applicable.

(b) *Duration of well authorization by rule.* Well authorization under this section expires upon the effective date of a permit issued pursuant to §§ 144.25, 144.31, 144.33 or 144.34, or upon proper closure of the well.

(c) *Prohibition of injection.* An owner or operator of a well which is authorized by rule pursuant to this section is prohibited from injecting into the well:

(1) Upon the effective date of an applicable permit denial;

(2) Upon failure to submit a permit application in a timely manner pursuant to §§ 144.25 or 144.31;

(3) Upon failure to submit inventory information in a timely manner pursuant to § 144.26; or

(4) Upon failure to comply with a request for information in a timely manner pursuant to § 144.27.

[58 FR 63896, Dec. 3, 1993]

§ 144.25 Requiring a permit.

(a) The Director may require the owner or operator of any Class I, II, III or V injection well which is authorized by rule under this subpart to apply for and obtain an individual or area UIC

permit. Cases where individual or area UIC permits may be required include:

(1) The injection well is not in compliance with any requirement of the rule;

NOTE: Any underground injection which violates any authorization by rule is subject to appropriate enforcement action.

(2) The injection well is not or no longer is within the category of wells and types of well operations authorized in the rule;

(3) The protection of USDWs requires that the injection operation be regulated by requirements, such as for corrective action, monitoring and reporting, or operation, which are not contained in the rule.

(4) When the injection well is a Class I, II (except existing enhanced recovery and hydrocarbon storage) or III well, in accordance with a schedule established by the Director pursuant to §144.31(c).

(b) For EPA-administered programs, the Regional Administrator may require an owner or operator of any well which is authorized by rule under this subpart to apply for an individual or area UIC permit under this paragraph only if the owner or operator has been notified in writing that a permit application is required. The owner or operator of a well which is authorized by rule under this subpart is prohibited from injecting into the well upon the effective date of permit denial, or upon failure by the owner or operator to submit an application in a timely manner as specified in the notice. The notice shall include: a brief statement of the reasons for requiring a permit; an application form; a statement setting a time for the owner or operator to file the application; and a statement of the consequences of denial or issuance of the permit, or failure to submit an application, as described in this paragraph.

(c) An owner or operator of a well authorized by rule may request to be excluded from the coverage of this subpart by applying for an individual or area UIC permit. The owner or operator shall submit an application under §144.31 with reasons supporting the request, to the Director. The Director may grant any such requests.

[48 FR 14189, Apr. 1, 1983, as amended at 49 FR 20182, May 11, 1984; 58 FR 63896, Dec. 3, 1993]

§144.26 Inventory requirements.

The owner or operator of an injection well which is authorized by rule under this subpart shall submit inventory information to the Director. Such an owner or operator is prohibited from injecting into the well upon failure to submit inventory information for the well within the time specified in paragraph (d) or (e) of this section.

(a) *Contents.* As part of the inventory, the Director shall require and the owner/operator shall provide at least the following information:

(1) Facility name and location;

(2) Name and address of legal contact;

(3) Ownership of facility;

(4) Nature and type of injection wells; and

(5) Operating status of injection wells.

NOTE: This information is requested on national form ''Inventory of Injection Wells,'' OMB No. 158–R0170.

(b) *Additional contents.* For EPA administered programs only, the owner or operator of a well listed in paragraph (b)(1) of this section shall provide the information listed in paragraph (b)(2) of this section.

(1) This section applies to the following wells:

(i) Class II enhanced recovery wells;

(ii) Class IV wells;

(iii) The following Class V wells:

(A) Sand or other backfill wells [§146.5(e)(8)];

(B) Radioactive waste disposal wells [§146.5(e)(11)];

(C) Geothermal energy recovery wells [§146.5(e)(12)];

(D) Brine return flow wells [§146.5(e)(14)];

(E) Wells used in experimental technologies [§146.5(e)(15)];

(F) Municipal and industrial disposal wells other than Class I; and

(G) Any other Class V wells at the discretion of the Regional Administrator.

(2) The owner or operator of a well listed in paragraph (b)(1) shall provide

a listing of all wells owned or operated setting forth the following information for each well. (A single description of wells at a single facility with substantially the same characteristics is acceptable).

(i) For Class II only, the field name(s);

(ii) Location of each well or project given by Township, Range, Section, and Quarter-Section, or by latitude and longitude to the nearest second, according to the conventional practice in the State;

(iii) Date of completion of each well;

(iv) Identification and depth of the formation(s) into which each well is injecting;

(v) Total depth of each well;

(vi) Casing and cementing record, tubing size, and depth of packer;

(vii) Nature of the injected fluids;

(viii) Average and maximum injection pressure at the wellhead;

(ix) Average and maximum injection rate; and

(x) Date of the last mechanical integrity test, if any.

(c) *Notice.* Upon approval of the UIC Program in a State, the Director shall notify owners or operators of injection wells of their duty to submit inventory information. The method of notification selected by the Director must assure that the owners or operators will be made aware of the inventory requirement.

(d) *Deadlines.* Except as provided in paragraph (e) of this section. (1) The owner or operator of an injection well shall submit inventory information no later than one year after the date of approval or effective date of the UIC program for the State. The Director need not require inventory information from any facility with interim status under RCRA.

(2) For EPA administered programs the information need not be submitted if a complete permit application is submitted within one year of the effective data of the UIC program. The owner or operator of Class IV well shall submit inventory information no later than 60 days after the effective date of the program.

(e) *Deadlines for Class V Wells (EPA-administered programs only).* (1) The owner or operator of a Class V well in which injection took place within one year after the date at which a UIC program authorized under the SDWA first became effective, and who failed to submit inventory for the well within the time specified in paragraph (d) of this section may resume injection 90 days after submittal of the inventory information to the Director unless the owner or operator receives notice that injection may not resume or may resume sooner.

(2) The owner or operator of a Class V well in which injection started after the first anniversary date at which a UIC program authorized under the SDWA became effective, shall submit inventory information no later than one year after May 2, 1994.

(3) The owner or operator of a Class V well in which injection will start after May 2, 1994, shall submit inventory information prior to starting injection.

(4) The owner or operator of a Class V injection well prohibited from injecting for failure to submit inventory information for the well within the time specified in paragraphs (e) (2) and (3) of this section, may resume injection 90 days after submittal of the inventory information to the Director unless the owner or operator receives notice from the Director that injection may not resume or may resume sooner.

[48 FR 14189, Apr. 1, 1983, as amended at 49 FR 20182, May 11, 1984; 58 FR 63896, Dec. 3, 1993]

§ 144.27 Requiring other information.

(a) For EPA administered programs only, in addition to the inventory requirements of § 144.26, the Regional Administrator may require the owner or operator of any well authorized by rule under this subpart to submit information as deemed necessary by the Regional Administrator to determine whether a well may be endangering an underground source of drinking water in violation of § 144.12 of this part.

(b) Such information requirements may include, but are not limited to:

(1) Performance of ground-water monitoring and the periodic submission of reports of such monitoring;

(2) An analysis of injected fluids, including periodic submission of such analyses; and

(3) A description of the geologic strata through and into which injection is taking place.

(c) Any request for information under this section shall be made in writing, and include a brief statement of the reasons for requiring the information. An owner or operator shall submit the information within the time period(s) provided in the notice.

(d) An owner or operator of an injection well authorized by rule under this subpart is prohibited from injecting into the well upon failure of the owner or operator to comply with a request for information within the time period(s) specified by the Director pursuant to paragraph (c) of this section. An owner or operator of a well prohibited from injection under this section shall not resume injection except under a permit issued pursuant to §§144.25, 144.31, 144.33 or 144.34.

[49 FR 20182, May 11, 1984, as amended at 58 FR 63896, Dec. 3, 1993]

§144.28 Requirements for Class I, II, and III wells authorized by rule.

The following requirements apply to the owner or operator of a Class I, II or III well authorized by rule under this subpart, as provided by §§144.21(e) and 144.22(d).

(a) The owner or operator shall comply with all applicable requirements of this subpart and subpart B of this part. Any noncompliance with these requirements constitutes a violation of the Safe Drinking Water Act and is grounds for enforcement action, except that the owner or operator need not comply with these requirements to the extent and for the duration such noncompliance is authorized by an emergency permit under §144.34.

(b) *Twenty-four hour reporting.* The owner or operator shall report any noncompliance which may endanger health or the environment, including:

(1) Any monitoring or other information which indicates that any contaminant may cause an endangerment to a USDW; or

(2) Any noncompliance or malfunction of the injection system which may cause fluid migration into or between USDWs.

Any information shall be provided orally within 24 hours from the time the owner or operator becomes aware of the circumstances. A written submission shall also be provided within five days of the time the owner or operator becomes aware of the circumstances. The written submission shall contain a description of the noncompliance and its cause, the period of noncompliance, including exact dates and times, and if the noncompliance has not been corrected, the anticipated time it is expected to continue; and steps taken or planned to reduce, eliminate, and prevent recurrence of the noncompliance.

(c) *Plugging and abandonment plan.* (1) The owner or operator shall prepare, maintain, and comply with a plan for plugging and abandonment of the well or project that meets the requirements of §146.10 of this chapter and is acceptable to the Director. For purposes of this paragraph, temporary intermittent cessation of injection operations is not abandonment.

(2) For EPA administered programs:

(i) The owner or operator shall submit the plan, on a form provided by the Regional Administrator, no later than one year after the effective date of the UIC program in the state.

(ii) The owner or operator shall submit any proposed significant revision to the method of plugging reflected in the plan no later than the notice of plugging required by §144.28(j)(2) (i.e., 45 days prior to plugging unless shorter notice is approved).

(iii) The plan shall include the following information:

(A) The nature and quantity and material to be used in plugging;

(B) The location and extent (by depth) of the plugs;

(C) Any proposed test or measurement to be made;

(D) The amount, size, and location (by depth) of casing to be left in the well;

(E) The method and location where casing is to be parted; and

(F) [Reserved]

(G) The estimated cost of plugging the well.

(iv) After a cessation of operations of two years the owner or operator shall plug and abandon the well in accordance with the plan unless he:

(A) Provides notice to the Regional Administrator;

(B) Describe actions or procedures, satisfactory to the Regional Administrator, that the owner or operator will take to ensure that the well will not endanger USDWs during the period of temporary abandonment. These actions and procedures shall include compliance with the technical requirements applicable to active injection wells unless waived by the Regional Administrator.

(v) The owner or operator of any well that has been temporarily abandoned [ceased operations for more than two years and has met the requirements of paragraphs (c)(2) (A) and (B) of this section] shall notify the Regional Administrator prior to resuming operation of the well.

(d) *Financial responsibility.* (1) The owner, operator and/or, for EPA-administered programs, the transferor of a Class I, II or III well, is required to demonstrate and maintain financial responsibility and resources to close, plug and abandon the underground injection operation in a manner prescribed by the Director until:

(i) The well has been plugged and abandoned in accordance with an approved plugging and abandonment plan pursuant to §§ 144.28(c) and 146.10 and submission of a plugging and abandonment report has been made pursuant to § 144.28(k);

(ii) The well has been converted in compliance with the requirements of § 144.28(j); or

(iii) For EPA-administered programs, the transferor has received notice from the Director that the transferee has demonstrated financial responsibility for the well. The owner or operator shall show evidence of such financial responsibility to the Director by the submission of a surety bond, or other adequate assurance, such as a financial statement.

(2) For EPA-administered programs, the owner or operator shall submit such evidence no later than one year after the effective date of the UIC program in the State. Where the ownership or operational control of the well is transferred more than one year after the effective date of the UIC program, the transferee shall submit such evidence no later than the date specified in the notice required pursuant to § 144.28(l)(2).

(3) For EPA administered programs the Regional Administrator may require the owner or operator to submit a revised demonstration of financial responsibility if the Regional Administrator has reason to believe that the original demonstration is no longer adequate to cover the cost of closing, plugging and abandoning the well.

(4) For EPA administered programs the owner or operator of a well injecting hazardous waste must comply with the financial responsibility requirements of subpart F of this part.

(5) For EPA-administered programs, an owner or operator must notify the Regional Administrator by certified mail of the commencement of any voluntary or involuntary proceeding under Title 11 (Bankruptcy) of the United States Code which names the owner or operator as debtor, within 10 business days after the commencement of the proceeding. Any party acting as guarantor for the owner or operator for the purpose of financial responsibility must so notify the Regional Administrator, if the guarantor is named as debtor in any such proceeding.

(6) In the event of commencement of a proceeding specified in paragraph (d)(5) of this section, an owner or operator who has furnished a financial statement for the purpose of demonstrating financial responsibility under this section shall be deemed to be in violation of this paragraph until an alternative financial assurance demonstration acceptable to the Regional Administrator is provided either by the owner or operator or by its trustee in bankruptcy, receiver, or other authorized party. All parties shall be prohibited from injecting into the well until such alternate financial assurance is provided.

(e) *Casing and cementing requirements.* For enhanced recovery and hydrocarbon storage wells:

(1) The owner or operator shall case and cement the well to prevent movement of fluids into or between underground sources of drinking water. In determining and specifying casing and cementing requirements, the following factors shall be considered:

(i) Depth to the injection zone;

(ii) Depth to the bottom of all USDWs; and

(iii) Estimated maximum and average injection pressures.

(2) In addition, in determining and specifying casing and cementing requirements the Director may consider information on:

(i) Nature of formation fluids;

(ii) Lithology of injection and confining zones;

(iii) External pressure, internal pressure, and axial loading;

(iv) Hole size;

(v) Size and grade of all casing strings; and

(vi) Class of cement.

(3) The requirements in paragraphs (e) (1) and (2) of this section need not apply if:

(i) Regulatory controls for casing and cementing existed at the time of drilling of the well and the well is in compliance with those controls; and

(ii) Well injection will not result in the movement of fluids into an underground source of drinking water so as to create a significant risk to the health of persons.

(4) When a State did not have regulatory controls for casing and cementing prior to the time of the submission of the State program to the Administrator, the Director need not apply the casing and cementing requirements in paragraph (e)(1) of this section if he submits as a part of his application for primacy, an appropriate plan for casing and cementing of existing, newly converted, and newly drilled wells in existing fields, and the Administrator approves the plan.

(f) *Operating requirements.* (1) Injection between the outermost casing protecting underground sources of drinking water and the well bore is prohibited.

(2) The owner or operator of a Class I, II or III injection well authorized by rule shall establish and maintain mechanical integrity as defined in § 146.8 of this chapter until the well is properly plugged in accordance with an approved plugging and abandonment plan pursuant to §§ 144.28(c) and 146.10, and a plugging and abandonment report pursuant to § 144.28(k) is submitted, or until the well is converted in compliance with § 144.28(j). For EPA-administered programs, the Regional Administrator may require by written notice that the owner or operator comply with a schedule describing when mechanical integrity demonstrations shall be made.

(3) When the Director determines that a Class I (non-hazardous), II or III injection well lacks mechanical integrity pursuant to § 146.8 of this chapter, the Director shall give written notice of his determination to the owner or operator. Unless the Director requires immediate cessation, the owner or operator shall cease injection into the well within 48 hours of receipt of the Director's determination. The Director may allow plugging of the well in accordance with the requirements of § 146.10 of this chapter, or require the owner or operator to perform such additional construction, operation, monitoring, reporting and corrective action as is necessary to prevent the movement of fluid into or between USDWs caused by the lack of mechanical integrity. The owner or operator may resume injection upon receipt of written notification from the Director that the owner or operator has demonstrated mechanical integrity pursuant to § 146.8 of this chapter.

(4) The Director may allow the owner or operator of a well which lacks mechanical integrity pursuant to § 146.8(a)(1) of this chapter to continue or resume injection if the owner or operator has made a satisfactory demonstration that there is no movement of fluid into or between USDWs.

(5) For Class I wells, unless an alternative to a packer has been approved under § 146.12(c) of this chapter, the owner or operator shall fill the annulus between the tubing and the long string of casings with a fluid approved by the Director and maintain a pressure, also approved by the Director, on the annulus. For EPA administered programs, the owner or operator of a Class I well completed with tubing and packer shall fill the annulus between tubing and casing with a noncorrosive fluid and maintain a positive pressure on the annulus. For other Class I wells, the owner or operator shall insure that the alternative completion method will reliably provide a comparable level of

protection to underground sources of drinking water.

(6) Injection pressure.

(i) For Class I and III wells:

(A) Except during stimulation, the owner or operator shall not exceed an injection pressure at the wellhead which shall be calculated so as to assure that the pressure during injection does not initiate new fractures or propagate existing fractures in the injection zone; and

(B) The owner or operator shall not inject at a pressure which will initiate fractures in the confining zone or cause the movement of injection or formation fluids into an underground source of drinking water.

(ii) For Class II wells:

(A) The owner or operator shall not exceed a maximum injection pressure at the wellhead which shall be calculated so as to assure that the pressure during injection does not initiate new fractures of propagate existing fractures in the confining zone adjacent to the USDWs; and

(B) The owner or operator shall not inject at a pressure which will cause the movement of injection or formation fluids into an underground source of drinking water.

(g) *Monitoring requirements.* The owner or operator shall perform the monitoring as described in this paragraph. For EPA administered programs, monitoring of the nature of the injected fluids shall comply with applicable analytical methods cited and described in table I of 40 CFR 136.3 or in appendix III of 40 CFR part 261 or by other methods that have been approved by the Regional Administrator.

(1) The owner or operator of a Class I well shall:

(i) Analyze the nature of the injected fluids with sufficient frequency to yield data representative of their characteristics;

(ii) Install and use continuous recording devices to monitor injection pressure, flow rate and volume, and the pressure on the annulus between the tubing and the long string of casing;

(iii) Install and use monitoring wells within the area of review if required by the Director, to monitor any migration of fluids into and pressure in the underground sources of drinking water. The type, number and location of the wells, the parameters to be measured, and the frequency of monitoring must be approved by the Director.

(2) For Class II wells:

(i) The owner or operator shall monitor the nature of the injected fluids with sufficient frequency to yield data representative of their characteristics. For EPA administered programs, this frequency shall be at least once within the first year of the authorization and thereafter when changes are made to the fluid.

(ii) The owner or operator shall observe the injection pressure, flow rate, and cumulative volume at least with the following frequencies:

(A) Weekly for produced fluid disposal operations;

(B) Monthly for enhanced recovery operations;

(C) Daily during the injection of liquid hydrocarbons and injection for withdrawal of stored hydrocarbons; and

(D) Daily during the injection phase of cyclic steam operations.

(iii) The owner or operator shall record one observation of injection pressure, flow rate and cumulative volume at reasonable intervals no greater than thirty days.

(iv) For enhanced recovery and hydrocarbon storage wells:

(A) The owner or operator shall demonstrate mechanical integrity pursuant to § 146.8 of this chapter at least once every five years during the life of the injection well.

(B) For EPA administered programs, the Regional Administrator by written notice may require the owner or operator to comply with a schedule describing when such demonstrations shall be made.

(C) For EPA administered programs, the owner or operator of any well required to be tested for mechanical integrity shall notify the Regional Administrator at least 30 days prior to any required mechanical integrity test. The Regional Administrator may allow a shorter notification period if it would be sufficient to enable EPA to witness the mechanical integrity testing if it chose. Notification may be in the form of a yearly or quarterly schedule of planned mechanical integrity tests, or it may be on an individual basis.

(v) The owner or operator of a hydrocarbon storage or enhanced recovery wells may monitor them by manifold monitoring on a field or project basis rather than on an individual well basis if such facilities consist of more than one injection well, operate with a common manifold, and provided the owner or operator demonstrates to the Director that manifold monitoring is comparable to individual well monitoring.

(3)(i) For Class III wells the owner or operator shall provide to the Director a qualitative analysis and ranges in concentrations of all constituents of injected fluids at least once within the first year of authorization and thereafter whenever the injection fluid is modified to the extent that the initial data are incorrect or incomplete. The owner or operator may request Federal confidentiality as specified in 40 CFR part 2. If the information is proprietary the owner or operator may in lieu of the ranges in concentrations choose to submit maximum concentrations which shall not be exceeded. In such a case the owner or operator shall retain records of the undisclosed concentrations and provide them upon request to the Regional Administrator as part of any enforcement investigation; and

(ii) Monitor injection pressure and either flow rate or volume semi-monthly, or meter and record daily injected and produced fluid volumes as appropriate;

(iii) Monitor the fluid level in the injection zone semi-monthly, where appropriate;

(iv) All Class III wells may be monitored on a field or project basis rather than an individual well basis by manifold monitoring. Manifold monitoring may be used in cases of facilities consisting of more than one injection well, operating with a common manifold. Separate monitoring systems for each well are not required provided the owner or operator demonstrates to the Director that manifold monitoring is comparable to individual well monitoring.

(h) *Reporting requirements.* The owner or operator shall submit reports to the Director as follows:

(1) For Class I wells, quarterly reports on:

(i) The physical, chemical, and other relevant characteristics of the injection fluids;

(ii) Monthly average, maximum, and minimum values for injection pressure, flow rate and volume, and annular pressure;

(iii) The results from ground-water monitoring wells prescribed in paragraph (g)(1)(iii) of this section;

(iv) The results of any test of the injection well conducted by the owner or operator during the reported quarter if required by the Director; and

(v) Any well work over performed during the reported quarter.

(2) For Class II wells:

(i) An annual report to the Director summarizing the results of all monitoring, as required in paragraph (g)(2) of this section. Such summary shall include monthly records of injected fluids, and any major changes in characteristics or sources of injected fluids. Previously submitted information may be included by reference.

(ii) The owner or operator of hydrocarbon storage and enhanced recovery projects may report on a field or project basis rather than on an individual well basis where manifold monitoring is used.

(3) For Class III wells:

(i) Quarterly reporting on all monitoring, as required in paragraph (g)(3) of this section;

(ii) Quarterly reporting of the results of any periodic tests required by the Director that are performed during the reported quarter;

(iii) Monitoring may be reported on a project or field basis rather than an individual well basis where manifold monitoring is used.

(i) *Retention of records.* The owner or operator shall retain records of all monitoring information, including the following:

(1) Calibration and maintenance records and all original strip chart recordings for continuous monitoring instrumentation, and copies of all reports required by this section, for a period of at least three years from the date of the sample, measurement, or report. This period may be extended by request of the Director at any time; and

(2) The nature and composition of all injected fluids until three years after the completion of any plugging and abandonment procedures specified under § 144.52(l)(6). The Director may require the owner or operator to deliver the records to the Director at the conclusion of the retention period. For EPA administered programs, the owner or operator shall continue to retain the records after the three year retention period unless he delivers the records to the Regional Administrator or obtains written approval from the Regional Administrator to discard the records.

(j) *Notice of abandonment.* (1) The owner or operator shall notify the Director, according to a time period required by the Director, before conversion or abandonment of the well.

(2) For EPA-administered programs, the owner or operator shall notify the Regional Administrator at least 45 days before plugging and abandonment. The Regional Administrator, at his discretion, may allow a shorter notice period.

(k) *Plugging and abandonment report.* For EPA-administered programs, within 60 days after plugging a well or at the time of the next quarterly report (whichever is less) the owner or operator shall submit a report to the Regional Administrator. If the quarterly report is due less than 15 days before completion of plugging, then the report shall be submitted within 60 days. The report shall be certified as accurate by the person who performed the plugging operation. Such report shall consist of either:

(1) A statement that the well was plugged in accordance with the plan previously submitted to the Regional Administrator; or

(2) Where actual plugging differed from the plan previously submitted, an updated version of the plan, on the form supplied by the Regional Administrator, specifying the different procedures used.

(l) *Change of ownership or operational control.* For EPA-administered programs:

(1) The transferor of a Class I, II or III well authorized by rule shall notify the Regional Administrator of a transfer of ownership or operational control of the well at least 30 days in advance of the proposed transfer.

(2) The notice shall include a written agreement between the transferor and the transferee containing a specific date for transfer of ownership or operational control of the well; and a specific date when the financial responsibility demonstration of § 144.28(d) will be met by the transferee.

(3) The transferee is authorized to inject unless he receives notification from the Director that the transferee has not demonstrated financial responsibility pursuant to § 144.28(d).

(m) *Requirements for Class I hazardous waste wells.* The owner or operator of any Class I well injecting hazardous waste shall comply with § 144.14(c). In addition, for EPA-administered programs the owner or operator shall properly dispose of, or decontaminate by removing all hazardous waste residues, all injection well equipment.

[49 FR 20182, May 11, 1984, as amended at 58 FR 63897, Dec. 3, 1993]

Subpart D—Authorization by Permit

§ 144.31 Application for a permit; authorization by permit.

(a) *Permit application.* Unless an underground injection well is authorized by rule under subpart C of this part, all injection activities including construction of an injection well are prohibited until the owner or operator is authorized by permit. An owner or operator of a well currently authorized by rule must apply for a permit under this section unless well authorization by rule was for the life of the well or project. Authorization by rule for a well or project for which a permit application has been submitted terminates for the well or project upon the effective date of the permit. Procedures for applications, issuance and administration of emergency permits are found exclusively in § 144.34. A RCRA permit applying the standards of part 264, subpart C of this chapter will constitute a UIC permit for hazardous waste injection wells for which the technical standards in part 146 of this chapter are not generally appropriate.

(b) *Who applies?* When a facility or activity is owned by one person but is operated by another person, it is the operator's duty to obtain a permit.

(c) *Time to apply.* Any person who performs or proposes an underground injection for which a permit is or will be required shall submit an application to the Director in accordance with the UIC program as follows:

(1) For existing wells, as expeditiously as practicable and in accordance with the schedule in any program description under § 145.23(f) or (for EPA administered programs) on a schedule established by the Regional Administrator, but no later than 4 years from the approval or promulgation of the UIC program, or as required under § 144.14(b) for wells injecting hazardous waste. For EPA administered programs the owner or operator of Class I or III wells shall submit a complete permit application no later than 1 year after the effective date of the program.

(2) For new injection wells, except new wells in projects authorized under § 144.21(d) or authorized by an existing area permit under § 144.33(c), a reasonable time before construction is expected to begin.

(d) *Completeness.* The Director shall not issue a permit before receiving a complete application for a permit except for emergency permits. An application for a permit is complete when the Director receives an application form and any supplemental information which are completed to his or her satisfaction. The completeness of any application for a permit shall be judged independently of the status of any other permit application or permit for the same facility or activity. For EPA-administered programs, an application which is reviewed under § 124.3 is complete when the Director receives either a complete application or the information listed in a notice of deficiency.

(e) *Information requirements.* All applicants for permits shall provide the following information to the Director, using the application form provided by the Director.

(1) The activities conducted by the applicant which require it to obtain permits under RCRA, UIC, the National Pollution Discharge Elimination system (NPDES) program under the Clean Water Act, or the Prevention of Significant Deterioration (PSD) program under the Clean Air Act.

(2) Name, mailing address, and location of the facility for which the application is submitted.

(3) Up to four SIC codes which best reflect the principal products or services provided by the facility.

(4) The operator's name, address, telephone number, ownership status, and status as Federal, State, private, public, or other entity.

(5) Whether the facility is located on Indian lands.

(6) A listing of all permits or construction approvals received or applied for under any of the following programs:

(i) Hazardous Waste Management program under RCRA.

(ii) UIC program under SDWA.

(iii) NPDES program under CWA.

(iv) Prevention of Significant Deterioration (PSD) program under the Clean Air Act.

(v) Nonattainment program under the Clean Air Act.

(vi) National Emission Standards for Hazardous Pollutants (NESHAPS) preconstruction approval under the Clean Air Act.

(vii) Ocean dumping permits under the Marine Protection Research and Sanctuaries Act.

(viii) Dredge and fill permits under section 404 of CWA.

(ix) Other relevant environmental permits, including State permits.

(7) A topographic map (or other map if a topographic map is unavailable) extending one mile beyond the property boundaries of the source depicting the facility and each of its intake and discharge structures; each of its hazardous waste treatment, storage, or disposal facilities; each well where fluids from the facility are injected underground; and those wells, springs, and other surface water bodies, and drinking water wells listed in public records or otherwise known to the applicant within a quarter mile of the facility property boundary.

(8) A brief description of the nature of the business.

(9) For EPA-administered programs, the applicant shall identify and submit on a list with the permit application

the names and addresses of all owners of record of land within one-quarter mile of the facility boundary. This requirement may be waived by the Regional Administrator where the site is located in a populous area and the Regional Administrator determines that the requirement would be impracticable.

(10) A plugging and abandonment plan that meets the requirements of § 146.10 of this chapter and is acceptable to the Director.

(f) *Recordkeeping.* Applicants shall keep records of all data used to complete permit applications and any supplemental information submitted under § 144.31 for a period of at least 3 years from the date the application is signed.

(g) *Information Requirements for Class I Hazardous Waste Injection Wells Permits.* (1) The following information is required for each active Class I hazardous waste injection well at a facility seeking a UIC permit:

(i) Dates well was operated.

(ii) Specification of all wastes which have been injected in the well, if available.

(2) The owner or operator of any facility containing one or more active hazardous waste injection wells must submit all available information pertaining to any release of hazardous waste or constituents from any active hazardous waste injection well at the facility.

(3) The owner or operator of any facility containing one or more active Class I hazardous waste injection wells must conduct such preliminary site investigations as are necessary to determine whether a release is occurring, has occurred, or is likely to have occurred.

[48 FR 14189, Apr. 1, 1983, as amended at 49 FR 20185, May 11, 1984; 52 FR 45797, Dec. 1, 1987; 52 FR 46963, Dec. 10, 1987; 58 FR 63897, Dec. 3, 1993]

§ 144.32 Signatories to permit applications and reports.

(a) *Applications.* All permit applications, except those submitted for Class II wells (see paragraph (b) of this section), shall be signed as follows:

(1) *For a corporation:* by a responsible corporate officer. For the purpose of this section, a responsible corporate officer means; (i) A president, secretary, treasurer, or vice president of the corporation in charge of a principal business function, or any other person who performs similar policy- or decision-making functions for the corporation, or (ii) the manager of one or more manufacturing, production, or operating facilities employing more than 250 persons or having gross annual sales or expenditures exceeding $25 million (in second-quarter 1980 dollars), if authority to sign documents has been assigned or delegated to the manager in accordance with corporate procedures.

NOTE: EPA does not require specific assignments or delegations of authority to responsible corporate officers identified in § 144.32(a)(1)(i). The Agency will presume that these responsible corporate officers have the requisite authority to sign permit applications unless the corporation has notified the Director to the contrary. Corporate procedures governing authority to sign permit applications may provide for assignment or delegation to applicable corporate positions under § 144.32(a)(1)(ii) rather than to specific individuals.

(2) *For a partnership or sole proprietorship:* by a general partner or the proprietor, respectively; or

(3) *For a municipality, State, Federal, or other public agency:* by either a principal executive officer or ranking elected official. For purposes of this section, a principal executive officer of a Federal agency includes: (i) The chief executive officer of the agency, or (ii) a senior executive officer having responsibility for the overall operations of a principal geographic unit of the agency (e.g., Regional Administrators of EPA).

(b) *Reports.* All reports required by permits, other information requested by the Director, and all permit applications submitted for Class II wells under § 144.31 shall be signed by a person described in paragraph (a) of this section, or by a duly authorized representative of that person. A person is a duly authorized representative only if:

(1) The authorization is made in writing by a person described in paragraph (a) of this section;

(2) The authorization specifies either an individual or a position having responsibility for the overall operation of the regulated facility or activity, such

as the position of plant manager, operator of a well or a well field, superintendent, or position of equivalent responsibility. (A duly authorized representative may thus be either a named individual or any individual occupying a named position); and

(3) The written authorization is submitted to the Director.

(c) *Changes to authorization*. If an authorization under paragraph (b) of this section is no longer accurate because a different individual or position has responsibility for the overall operation of the facility, a new authorization satisfying the requirements of paragraph (b) of this section must be submitted to the Director prior to or together with any reports, information, or applications to be signed by an authorized representative.

(d) *Certification*. Any person signing a document under paragraph (a) or (b) of this section shall make the following certification:

I certify under penalty of law that this document and all attachments were prepared under my direction or supervision in accordance with a system designed to assure that qualified personnel properly gather and evaluate the information submitted. Based on my inquiry of the person or persons who manage the system, or those persons directly responsible for gathering the information, the information submitted is, to the best of my knowledge and belief, true, accurate, and complete. I am aware that there are significant penalties for submitting false information, including the possibility of fine and imprisonment for knowing violations.

(Clean Water Act (33 U.S.C. 1251 *et seq.*), Safe Drinking Water Act (42 U.S.C. 300f *et seq.*), Clean Air Act (42 U.S.C. 7401 *et seq.*), Resource Conservation and Recovery Act (42 U.S.C. 6901 *et seq.*))

[48 FR 14189, Apr. 1, 1983, as amended at 48 FR 39621, Sept. 1, 1983]

§144.33 Area permits.

(a) The Director may issue a permit on an area basis, rather than for each well individually, provided that the permit is for injection wells:

(1) Described and identified by location in permit application(s) if they are existing wells, except that the Director may accept a single description of wells with substantially the same characteristics;

(2) Within the same well field, facility site, reservoir, project, or similar unit in the same State;

(3) Operated by a single owner or operator; and

(4) Used to inject other than hazardous waste.

(b) Area permits shall specify:

(1) The area within which underground injections are authorized, and

(2) The requirements for construction, monitoring, reporting, operation, and abandonment, for all wells authorized by the permit.

(c) The area permit may authorize the permittee to construct and operate, convert, or plug and abandon wells within the permit area provided:

(1) The permittee notifies the Director at such time as the permit requires;

(2) The additional well satisfies the criteria in paragraph (a) of this section and meets the requirements specified in the permit under paragraph (b) of this section; and

(3) The cumulative effects of drilling and operation of additional injection wells are considered by the Director during evaluation of the area permit application and are acceptable to the Director.

(d) If the Director determines that any well constructed pursuant to paragraph (c) of this section does not satisfy any of the requirements of paragraphs (c) (1) and (2) of this section the Director may modify the permit under §144.39, terminate under §144.40, or take enforcement action. If the Director determines that cumulative effects are unacceptable, the permit may be modified under §144.39.

§144.34 Emergency permits.

(a) *Coverage*. Notwithstanding any other provision of this part or part 124, the Director may temporarily permit a specific underground injection if:

(1) An imminent and substantial endangerment to the health of persons will result unless a temporary emergency permit is granted; or

(2) A substantial and irretrievable loss of oil or gas resources will occur unless a temporary emergency permit is granted to a Class II well; and

(i) Timely application for a permit could not practicably have been made; and

(ii) The injection will not result in the movement of fluids into underground sources of drinking water; or

(3) A substantial delay in production of oil or gas resources will occur unless a temporary emergency permit is granted to a new Class II well and the temporary authorization will not result in the movement of fluids into an underground source of drinking water.

(b) *Requirements for issuance.* (1) Any temporary permit under paragraph (a)(1) of this section shall be for no longer term than required to prevent the hazard.

(2) Any temporary permit under paragraph (a)(2) of this section shall be for no longer than 90 days, except that if a permit application has been submitted prior to the expiration of the 90-day period, the Director may extend the temporary permit until final action on the application.

(3) Any temporary permit under paragraph (a)(3) of this section shall be issued only after a complete permit application has been submitted and shall be effective until final action on the application.

(4) Notice of any temporary permit under this paragraph shall be published in accordance with § 124.11 within 10 days of the issuance of the permit.

(5) The temporary permit under this section may be either oral or written. If oral, it must be followed within 5 calendar days by a written temporary emergency permit.

(6) The Director shall condition the temporary permit in any manner he or she determines is necessary to ensure that the injection will not result in the movement of fluids into an underground source of drinking water.

[48 FR 14189, Apr. 1, 1983, as amended at 49 FR 20185, May 11, 1984]

§ 144.35 Effect of a permit.

(a) Except for Class II and III wells, compliance with a permit during its term constitutes compliance, for purposes of enforcement, with Part C of the SDWA. However, a permit may be modified, revoked and reissued, or terminated during its term for cause as set forth in §§ 144.39 and 144.40.

(b) The issuance of a permit does not convey any property rights of any sort, or any exclusive privilege.

(c) The issuance of a permit does not authorize any injury to persons or property or invasion of other private rights, or any infringement of State or local law or regulations.

§ 144.36 Duration of permits.

(a) Permits for Class I and Class V wells shall be effective for a fixed term not to exceed 10 years. UIC permits for Class II and III wells shall be issued for a period up to the operating life of the facility. The Director shall review each issued Class II or III well UIC permit at least once every 5 years to determine whether it should be modified, revoked and reissued, terminated, or a minor modification made as provided in §§ 144.39, 144.40, and 144.41.

(b) Except as provided in § 144.37, the term of a permit shall not be extended by modification beyond the maximum duration specified in this section.

(c) The Director may issue any permit for a duration that is less than the full allowable term under this section.

§ 144.37 Continuation of expiring permits.

(a) *EPA permits.* When EPA is the permit-issuing authority, the conditions of an expired permit continue in force under 5 U.S.C. 558(c) until the effective date of a new permit if:

(1) The permittee has submitted a timely application which is a complete application for a new permit; and

(2) The Regional Administrator, through no fault of the permittee does not issue a new permit with an effective date on or before the expiration date of the previous permit (for example, when issuance is impracticable due to time or resource constraints).

(b) *Effect.* Permits continued under this section remain fully effective and enforceable.

(c) *Enforcement.* When the permittee is not in compliance with the conditions of the expiring or expired permit the Regional Administrator may choose to do any or all of the following:

(1) Initiate enforcement action based upon the permit which has been continued;

(2) Issue a notice of intent to deny the new permit. If the permit is denied, the owner or operator would then be

required to cease the activities authorized by the continued permit or be subject to enforcement action for operating without a permit;

(3) Issue a new permit under part 124 with appropriate conditions; or

(4) Take other actions authorized by these regulations.

(d) *State continuation.* An EPA issued permit does not continue in force beyond its time expiration date under Federal law if at that time a State is the permitting authority. A State authorized to administer the UIC program may continue either EPA or State-issued permits until the effective date of the new permits, if State law allows. Otherwise, the facility or activity is operating without a permit from the time of expiration of the old permit to the effective date of the State-issued new permit.

§144.38 Transfer of permits.

(a) *Transfers by modification.* Except as provided in paragraph (b) of this section, a permit may be transferred by the permittee to a new owner or operator only if the permit has been modified or revoked and reissued (under §144.39(b)(2)), or a minor modification made (under §144.41(d)), to identify the new permittee and incorporate such other requirements as may be necessary under the Safe Drinking Water Act.

(b) *Automatic transfers.* As an alternative to transfers under paragraph (a) of this section, any UIC permit for a well not injecting hazardous waste may be automatically transferred to a new permittee if:

(1) The current permittee notifies the Director at least 30 days in advance of the proposed transfer date referred to in paragraph (b)(2) of this section;

(2) The notice includes a written agreement between the existing and new permittees containing a specific date for transfer or permit responsibility, coverage, and liability between them, and the notice demonstrates that the financial responsibility requirements of §144.52(a)(7) will be met by the new permittee; and

(3) The Director does not notify the existing permittee and the proposed new permittee of his or her intent to modify or revoke and reissue the permit. A modification under this paragraph may also be a minor modification under §144.41. If this notice is not received, the transfer is effective on the date specified in the agreement mentioned in paragraph (b)(2) of this section.

§144.39 Modification or revocation and reisssuance of permits.

When the Director receives any information (for example, inspects the facility, receives information submitted by the permittee as required in the permit (see §144.51 of this chapter), receives a request for modification or revocation and reissuance under §124.5, or conducts a review of the permit file) he or she may determine whether or not one or more of the causes listed in paragraphs (a) and (b) of this section for modification or revocation and reissuance or both exist. If cause exists, the Director may modify or revoke and reissue the permit accordingly, subject to the limitations of paragraph (c) of this section, and may request an updated application if necessary. When a permit is modified, only the conditions subject to modification are reopened. If a permit is revoked and reissued, the entire permit is reopened and subject to revision and the permit is reissued for a new term. See §124.5(c)(2) of this chapter. If cause does not exist under this section or §144.41 of this chapter, the Director shall not modify or revoke and reissue the permit. If a permit modification satisfies the criteria in §144.41 for ''minor modifications'' the permit may be modified without a draft permit or public review. Otherwise, a draft permit must be prepared and other procedures in part 124 must be followed.

(a) *Causes for modification.* The following are causes for modification. For Class I hazardous waste injection wells, Class II, or Class III wells the following may be causes for revocation and reissuance as well as modification; and for all other wells the following may be cause for revocation or reissuance as well as modification when the permittee requests or agrees.

(1) *Alterations.* There are material and substantial alterations or additions to the permitted facility or activity which occurred after permit issuance

which justify the application of permit conditions that are different or absent in the existing permit.

(2) *Information.* The Director has received information. Permits other than for Class II and III wells may be modified during their terms for this cause only if the information was not available at the time of permit issuance (other than revised regulations, guidance, or test methods) and would have justified the application of different permit conditions at the time of issuance. For UIC area permits (§ 144.33), this cause shall include any information indicating that cumulative effects on the environment are unacceptable.

(3) *New regulations.* The standards or regulations on which the permit was based have been changed by promulgation of new or amended standards or regulations or by judicial decision after the permit was issued. Permits other than for Class I hazardous waste injection wells, Class II, or Class III wells may be modified during their terms for this cause only as follows:

(i) For promulgation of amended standards or regulations, when:

(A) The permit condition requested to be modified was based on a promulgated part 146 regulation; and

(B) EPA has revised, withdrawn, or modified that portion of the regulation on which the permit condition was based, and

(C) A permittee requests modification in accordance with § 124.5 within ninety (90) days after FEDERAL REGISTER notice of the action on which the request is based.

(ii) For judicial decisions, a court of competent jurisdiction has remanded and stayed EPA promulgated regulations if the remand and stay concern that portion of the regulations on which the permit condition was based and a request is filed by the permittee in accordance with § 124.5 within ninety (90) days of judicial remand.

(4) *Compliance schedules.* The Director determines good cause exists for modification of a compliance schedule, such as an act of God, strike, flood, or materials shortage or other events over which the permittee has little or no control and for which there is no reasonably available remedy. See also § 144.41(c) (minor modifications).

(b) *Causes for modification or revocation and reissuance.* The following are causes to modify or, alternatively, revoke and reissue a permit:

(1) Cause exists for termination under § 144.40, and the Director determines that modification or revocation and reissuance is appropriate.

(2) The Director has received notification (as required in the permit, see § 144.41(d)) of a proposed transfer of the permit. A permit also may be modified to reflect a transfer after the effective date of an automatic transfer (§ 144.38(b)) but will not be revoked and reissued after the effective date of the transfer except upon the request of the new permittee.

(3) A determination that the waste being injected is a hazardous waste as defined in § 261.3 either because the definition has been revised, or because a previous determination has been changed.

(c) *Facility siting.* Suitability of the facility location will not be considered at the time of permit modification or revocation and reissuance unless new information or standards indicate that a threat to human health or the environment exists which was unknown at the time of permit issuance.

[48 FR 14189, Apr. 1, 1983, as amended at 53 FR 28147, July 26, 1988]

§ 144.40 Termination of permits.

(a) The Director may terminate a permit during its term, or deny a permit renewal application for the following causes:

(1) Noncompliance by the permittee with any condition of the permit;

(2) The permittee's failure in the application or during the permit issuance process to disclose fully all relevant facts, or the permittee's misrepresentation of any relevant facts at any time; or

(3) A determination that the permitted activity endangers human health or the environment and can only be regulated to acceptable levels by permit modification or termination;

(b) The Director shall follow the applicable procedures in part 124 in terminating any permit under this section.

§144.41 Minor modifications of permits.

Upon the consent of the permittee, the Director may modify a permit to make the corrections or allowances for changes in the permitted activity listed in this section, without following the procedures of part 124. Any permit modification not processed as a minor modification under this section must be made for cause and with part 124 draft permit and public notice as required in §144.39. Minor modifications may only:

(a) Correct typographical errors;

(b) Require more frequent monitoring or reporting by the permittee;

(c) Change an interim compliance date in a schedule of compliance, provided the new date is not more than 120 days after the date specified in the existing permit and does not interfere with attainment of the final compliance date requirement; or

(d) Allow for a change in ownership or operational control of a facility where the Director determines that no other change in the permit is necessary, provided that a written agreement containing a specific date for transfer of permit responsibility, coverage, and liability between the current and new permittees has been submitted to the Director.

(e) Change quantities or types of fluids injected which are within the capacity of the facility as permitted and, in the judgment of the Director, would not interfere with the operation of the facility or its ability to meet conditions described in the permit and would not change its classification.

(f) Change construction requirements approved by the Director pursuant to §144.52(a)(1) (establishing UIC permit conditions), provided that any such alteration shall comply with the requirements of this part and part 146.

(g) Amend a plugging and abandonment plan which has been updated under §144.52(a)(6).

Subpart E—Permit Conditions

§144.51 Conditions applicable to all permits.

The following conditions apply to all UIC permits. All conditions applicable to all permits shall be incorporated into the permits either expressly or by reference. If incorporated by reference, a specific citation to these regulations (or the corresponding approved State regulations) must be given in the permit.

(a) *Duty to comply.* The permittee must comply with all conditions of this permit. Any permit noncompliance constitutes a violation of the Safe Drinking Water Act and is grounds for enforcement action; for permit termination, revocation and reissuance, or modification; or for denial of a permit renewal application; except that the permittee need not comply with the provisions of this permit to the extent and for the duration such noncompliance is authorized in an emergency permit under §144.34.

(b) *Duty to reapply.* If the permittee wishes to continue an activity regulated by this permit after the expiration date of this permit, the permittee must apply for and obtain a new permit.

(c) *Need to halt or reduce activity not a defense.* It shall not be a defense for a permittee in an enforcement action that it would have been necessary to halt or reduce the permitted activity in order to maintain compliance with the conditions of this permit.

(d) *Duty to mitigate.* The permittee shall take all reasonable steps to minimize or correct any adverse impact on the environment resulting from noncompliance with this permit.

(e) *Proper operation and maintenance.* The permittee shall at all times properly operate and maintain all facilities and systems of treatment and control (and related appurtenances) which are installed or used by the permittee to achieve compliance with the conditions of this permit. Proper operation and maintenance includes effective performance, adequate funding, adequate operator staffing and training, and adequate laboratory and process controls, including appropriate quality assurance procedures. This provision requires the operation of back-up or auxiliary facilities or similar systems only when necessary to achieve compliance with the conditions of the permit.

(f) *Permit actions.* This permit may be modified, revoked and reissued, or terminated for cause. The filing of a request by the permittee for a permit modification, revocation and reissuance, or termination, or a notification of planned changes or anticipated noncompliance, does not stay any permit condition.

(g) *Property rights.* This permit does not convey any property rights of any sort, or any exclusive privilege.

(h) *Duty to provide information.* The permittee shall furnish to the Director, within a time specified, any information which the Director may request to determine whether cause exists for modifying, revoking and reissuing, or terminating this permit, or to determine compliance with this permit. The permittee shall also furnish to the Director, upon request, copies of records required to be kept by this permit.

(i) *Inspection and entry.* The permittee shall allow the Director, or an authorized representative, upon the presentation of credentials and other documents as may be required by law, to:

(1) Enter upon the permittee's premises where a regulated facility or activity is located or conducted, or where records must be kept under the conditions of this permit;

(2) Have access to and copy, at reasonable times, any records that must be kept under the conditions of this permit;

(3) Inspect at reasonable times any facilities, equipment (including monitoring and control equipment), practices, or operations regulated or required under this permit; and

(4) Sample or monitor at reasonable times, for the purposes of assuring permit compliance or as otherwise authorized by the SDWA, any substances or parameters at any location.

(j) *Monitoring and records.* (1) Samples and measurements taken for the purpose of monitoring shall be representative of the monitored activity.

(2) The permittee shall retain records of all monitoring information, including the following:

(i) Calibration and maintenance records and all original strip chart recordings for continuous monitoring instrumentation, copies of all reports required by this permit, and records of all data used to complete the application for this permit, for a period of at least 3 years from the date of the sample, measurement, report, or application. This period may be extended by request of the Director at any time; and

(ii) The nature and composition of all injected fluids until three years after the completion of any plugging and abandonment procedures specified under § 144.52(a)(6), or under part 146 subpart G as appropriate. The Director may require the owner or operator to deliver the records to the Director at the conclusion of the retention period. For EPA administered programs, the owner or operator shall continue to retain the records after the three year retention period unless he delivers the records to the Regional Administrator or obtains written approval from the Regional Administrator to discard the records.

(3) Records of monitoring information shall include:

(i) The date, exact place, and time of sampling or measurements;

(ii) The individual(s) who performed the sampling or measurements;

(iii) The date(s) analyses were performed;

(iv) The individual(s) who performed the analyses;

(v) The analytical techniques or methods used; and

(vi) The results of such analyses.

(k) *Signatory requirement.* All applications, reports, or information submitted to the Administrator shall be signed and certified. (See § 144.32.)

(l) *Reporting requirements.* (1) *Planned changes.* The permittee shall give notice to the Director as soon as possible of any planned physical alterations or additions to the permitted facility.

(2) *Anticipated noncompliance.* The permittee shall give advance notice to the Director of any planned changes in the permitted facility or activity which may result in noncompliance with permit requirements.

(3) *Transfers.* This permit is not transferable to any person except after notice to the Director. The Director may require modification or revocation and reissuance of the permit to change the name of the permittee and incorporate such other requirements as may

be necessary under the Safe Drinking Water Act. (See § 144.38; in some cases, modification or revocation and reissuance is mandatory.)

(4) *Monitoring reports.* Monitoring results shall be reported at the intervals specified elsewhere in this permit.

(5) *Compliance schedules.* Reports of compliance or noncompliance with, or any progress reports on, interim and final requirements contained in any compliance schedule of this permit shall be submitted no later than 30 days following each schedule date.

(6) *Twenty-four hour reporting.* The permittee shall report any noncompliance which may endanger health or the environment, including:

(i) Any monitoring or other information which indicates that any contaminant may cause an endangerment to a USDW; or

(ii) Any noncompliance with a permit condition or malfunction of the injection system which may cause fluid migration into or between USDWs.

Any information shall be provided orally within 24 hours from the time the permittee becomes aware of the circumstances. A written submission shall also be provided within 5 days of the time the permittee becomes aware of the circumstances. The written submission shall contain a description of the noncompliance and its cause, the period of noncompliance, including exact dates and times, and if the noncompliance has not been corrected, the anticipated time it is expected to continue; and steps taken or planned to reduce, eliminate, and prevent reoccurrence of the noncompliance.

(7) *Other noncompliance.* The permittee shall report all instances of noncompliance not reported under paragraphs (l) (4), (5), and (6) of this section, at the time monitoring reports are submitted. The reports shall contain the information listed in paragraph (l)(6) of this section.

(8) *Other information.* Where the permittee becomes aware that it failed to submit any relevant facts in a permit application, or submitted incorrect information in a permit application or in any report to the Director, it shall promptly submit such facts or information.

(m) *Requirements prior to commencing injection.* Except for all new wells authorized by an area permit under § 144.33(c), a new injection well may not commence injection until construction is complete, and

(1) The permittee has submitted notice of completion of construction to the Director; and

(2)(i) The Director has inspected or otherwise reviewed the new injection well and finds it is in compliance with the conditions of the permit; or

(ii) The permittee has not received notice form the Director of his or her intent to inspect or otherwise review the new injection well within 13 days of the date of the notice in paragraph (m)(1) of this section, in which case prior inspection or review is waived and the permittee may commence injection. The Director shall include in his notice a reasonable time period in which he shall inspect the well.

(n) The permittee shall notify the Director at such times as the permit requires before conversion or abandonment of the well or in the case of area permits before closure of the project.

(o) A Class I, II or III permit shall include and a Class V permit may include, conditions which meet the applicable requirements of § 146.10 of this chapter to insure that plugging and abandonment of the well will not allow the movement of fluids into or between USDWs. Where the plan meets the requirements of § 146.10 of this chapter, the Director shall incorporate it into the permit as a permit condition. Where the Director's review of an application indicates that the permittee's plan is inadequate, the Director may require the applicant to revise the plan, prescribe conditions meeting the requirements of this paragraph, or deny the permit. For purposes of this paragraph, temporary or intermittent cessation of injection operations is not abandonment.

(p) *Plugging and abandonment report.* For EPA-administered programs, within 60 days after plugging a well or at the time of the next quarterly report (whichever is less) the owner or operator shall submit a report to the Regional Administrator. If the quarterly report is due less than 15 days before completion of plugging, then the report

shall be submitted within 60 days. The report shall be certified as accurate by the person who performed the plugging operation. Such report shall consist of either:

(1) A statement that the well was plugged in accordance with the plan previously submitted to the Regional Administrator; or

(2) Where actual plugging differed from the plan previously submitted, and updated version of the plan on the form supplied by the regional administrator, specifying the differences.

(q) *Duty to establish and maintain mechanical integrity.* (1) The owner or operator of a Class I, II or III well permitted under this part shall establish prior to commencing injection or on a schedule determined by the Director, and thereafter maintain mechanical integrity as defined in § 146.8 of this chapter. For EPA-administered programs, the Regional Administrator may require by written notice that the owner or operator comply with a schedule describing when mechanical integrity demonstrations shall be made.

(2) When the Director determines that a Class I, II, or III well lacks mechanical integrity pursuant to § 146.8 of this chapter, he shall give written notice of his determination to the owner or operator. Unless the Director requires immediate cessation, the owner or operator shall cease injection into the well within 48 hours of receipt of the Director's determination. The Director may allow plugging of the well pursuant to the requirements of § 146.10 of this chapter or require the permittee to perform such additional construction, operation, monitoring, reporting and corrective action as is necessary to prevent the movement of fluid into or between USDWs caused by the lack of mechanical integrity. The owner or operator may resume injection upon written notification from the Director that the owner or operator has demonstrated mechanical integrity pursuant to § 146.8 of this chapter.

(3) The Director may allow the owner or operator of a well which lacks mechanical integrity pursuant to § 146.8(a)(1) of this chapter to continue or resume injection, if the owner or operator has made a satisfactory demonstration that there is no movement of fluid into or between USDWs.

[48 FR 14189, Apr. 1, 1983, as amended at 49 FR 20185, May 11, 1984; 53 FR 28147, July 26, 1988; 58 FR 63898, Dec. 3, 1993]

§ 144.52 Establishing permit conditions.

(a) In addition to conditions required in § 144.51, the Director shall establish conditions, as required on a case-by-case basis under § 144.36 (duration of permits), § 144.53(a) (schedules of compliance), §144.54 (monitoring), and for EPA permits only § 144.53(b) (alternate schedules of compliance), and § 144.4 (considerations under Federal law). Permits for owners or operators of hazardous waste injection wells shall include conditions meeting the requirements of § 144.14 (requirements for wells injecting hazardous waste), § 144.52 (a)(7) and (a)(9), and subpart G of part 146. Permits for other wells shall contain the following requirements, when applicable.

(1) *Construction requirements* as set forth in part 146. Existing wells shall achieve compliance with such requirements according to a compliance schedule established as a permit condition. The owner or operator of a proposed new injection well shall submit plans for testing, drilling, and construction as part of the permit application. Except as authorized by an area permit, no constuction may commence until a permit has been issued containing construction requirements (see § 144.11). New wells shall be in compliance with these requirements prior to commencing injection operations. Changes in construction plans during construction may be approved by the Administrator as minor modifications (§ 144.41). No such changes may be physically incorporated into construction of the well prior to approval of the modification by the Director.

(2) *Corrective action* as set forth in §§ 144.55 and 146.7

(3) *Operation requirements* as set forth in 40 CFR part 146; the permit shall establish any maximum injection volumes and/or pressures necessary to assure that fractures are not initiated in the confining zone, that injected fluids do not migrate into any underground

source of drinking water, that formation fluids are not displaced into any underground source of drinking water, and to assure compliance with the part 146 operating requirements.

(4) *Requirements for wells managing hazardous waste,* as set forth in § 144.14.

(5) Monitoring and reporting requirements as set forth in 40 CFR part 146. The permittee shall be required to identify types of tests and methods used to generate the monitoring data. For EPA administered programs, monitoring of the nature of injected fluids shall comply with applicable analytical methods cited and described in table I of 40 CFR 136.3 or in appendix III of 40 CFR part 261 or in certain circumstances by other methods that have been approved by the Regional Administrator.

(6) After a cessation of operations of two years the owner or operator shall plug and abandon the well in accordance with the plan unless he:

(i) Provides notice to the Regional Administrator;

(ii) Describes actions or procedures, satisfactory to the Regional Administrator, that the owner or operator will take to ensure that the well will not endanger USDWs during the period of temporary abandonment. These actions and procedures shall include compliance with the technical requirements applicable to active injection wells unless waived by the Regional Administrator.

(7) *Financial responsibility.* (i) The permittee, including the transferor of a permit, is required to demonstrate and maintain financial responsibility and resources to close, plug, and abandon the underground injection operation in a manner prescribed by the Director until:

(A) The well has been plugged and abandoned in accordance with an approved plugging and abandonment plan pursuant to §§ 144.51(o) and 146.10 of this chapter, and submitted a plugging and abandonment report pursuant to § 144.51(p); or

(B) The well has been converted in compliance with the requirements of § 144.51(n); or

(C) The transferor of a permit has received notice from the Director that the owner or operator receiving transfer of the permit, the new permittee, has demonstrated financial responsibility for the well.

(ii) The permittee shall show evidence of such financial responsibility to the Director by the submission of a surety bond, or other adequate assurance, such as a financial statement or other materials acceptable to the Director. For EPA administered programs, the Regional Administrator may on a periodic basis require the holder of a lifetime permit to submit an estimate of the resources needed to plug and abandon the well revised to reflect inflation of such costs, and a revised demonstration of financial responsibility, if necessary. The owner or operator of a well injecting hazardous waste must comply with the financial responsibility requirements of subpart F of this part.

(8) *Mechanical integrity.* A permit for any Class I, II or III well or injection project which lacks mechanical integrity shall include, and for any Class V well may include, a condition prohibiting injection operations until the permittee shows to the satisfaction of the Director under § 146.08 that the well has mechanical integrity.

(9) *Additional conditions.* The Director shall impose on a case-by-case basis such additional conditions as are necessary to prevent the migration of fluids into underground sources of drinking water.

(b)(1) In addition to conditions required in all permits the Director shall establish conditions in permits as required on a case-by-case basis, to provide for and assure compliance with all applicable requirements of the SDWA and parts 144, 145, 146 and 124.

(2) For a State issued permit, an applicable requirement is a State statutory or regulatory requirement which takes effect prior to final administrative disposition of the permit. For a permit issued by EPA, an applicable requirement is a statutory or regulatory requirement (including any interim final regulation) which takes effect prior to the issuance of the permit (except as provided in § 124.86(c) for UIC permits being processed under subpart E or F of part 124). Section 124.14 (reopening of comment period) provides a

means for reopening EPA permit proceedings at the discretion of the Director where new requirements become effective during the permitting process and are of sufficient magnitude to make additional proceedings desirable. For State and EPA administered programs, an applicable requirement is also any requirement which takes effect prior to the modification or revocation and reissuance of a permit, to the extent allowed in § 144.39.

(3) New or reissued permits, and to the extent allowed under § 144.39 modified or revoked and reissued permits, shall incorporate each of the applicable requirements referenced in § 144.52.

(c) *Incorporation.* All permit conditions shall be incorporated either expressly or by reference. If incorporated by reference, a specific citation to the applicable regulations or requirements must be given in the permit.

[48 FR 14189, Apr. 1, 1983, as amended at 49 FR 20185, May 11, 1984; 53 FR 28147, July 26, 1988; 58 FR 63898; Dec. 3, 1993]

§ 144.53 Schedule of compliance.

(a) *General.* The permit may, when appropriate, specify a schedule of compliance leading to compliance with the SDWA and parts 144, 145, 146, and 124.

(1) *Time for compliance.* Any schedules of compliance shall require compliance as soon as possible, and in no case later than 3 years after the effective date of the permit.

(2) *Interim dates.* Except as provided in paragraph (b)(1)(ii) of this section, if a permit establishes a schedule of compliance which exceeds 1 year from the date of permit issuance, the schedule shall set forth interim requirements and the dates for their achievement.

(i) The time between interim dates shall not exceed 1 year.

(ii) If the time necessary for completion of any interim requirement is more than 1 year and is not readily divisible into stages for completion, the permit shall specify interim dates for the submission of reports of progress toward completion of the interim requirements and indicate a projected completion date.

(3) *Reporting.* The permit shall be written to require that if paragraph (a)(1) of this section is applicable, progress reports be submitted no later than 30 days following each interim date and the final date of compliance.

(b) *Alternative schedules of compliance.* A permit applicant or permittee may cease conducting regulated activities (by plugging and abandonment) rather than continue to operate and meet permit requirements as follows:

(1) If the permittee decides to cease conducting regulated activities at a given time within the term of a permit which has already been issued:

(i) The permit may be modified to contain a new or additional schedule leading to timely cessation of activities; or

(ii) The permittee shall cease conducting permitted activities before noncompliance with any interim or final compliance schedule requirement already specified in the permit.

(2) If the decision to cease conducting regulated activities is made before issuance of a permit whose term will include the termination date, the permit shall contain a schedule leading to termination which will ensure timely compliance with applicable requirements.

(3) If the permittee is undecided whether to cease conducting regulated activities, the Director may issue or modify a permit to contain two schedules as follows:

(i) Both schedules shall contain an identical interim deadline requiring a final decision on whether to cease conducting regulated activities no later than a date which ensures sufficient time to comply with applicable requirements in a timely manner if the decision is to continue conducting regulated activities;

(ii) One schedule shall lead to timely compliance with applicable requirements;

(iii) The second schedule shall lead to cessation of regulated activities by a date which will ensure timely compliance with applicable requirements;

(iv) Each permit containing two schedules shall include a requirement that after the permittee has made a final decision under paragraph (b)(3)(i)

of this section it shall follow the schedule leading to compliance if the decision is to continue conducting regulated activities, and follow the schedule leading to termination if the decision is to cease conducting regulated activities.

(4) The applicant's or permittee's decision to cease conducting regulated activities shall be evidenced by a firm public commitment satisfactory to the Director, such as a resolution of the board of directors of a corporation.

§144.54 Requirements for recording and reporting of monitoring results.

All permits shall specify:

(a) Requirements concerning the proper use, maintenance, and installation, when appropriate, of monitoring equipment or methods (including biological monitoring methods when appropriate);

(b) Required monitoring including type, intervals, and frequency sufficient to yield data which are representative of the monitored activity including when appropriate, continuous monitoring;

(c) Applicable reporting requirements based upon the impact of the regulated activity and as specified in part 146. Reporting shall be no less frequent than specified in the above regulations.

§144.55 Corrective action.

(a) *Coverage.* Applicants for Class I, II, (other than existing), or III injection well permits shall identify the location of all known wells within the injection well's area of review which penetrate the injection zone, or in the case of Class II wells operating over the fracture pressure of the injection formation, all known wells within the area of review penetrating formations affected by the increase in pressure. For such wells which are improperly sealed, completed, or abandoned, the applicant shall also submit a plan consisting of such steps or modifications as are necessary to prevent movement of fluid into underground sources of drinking water (''corrective action''). Where the plan is adequate, the Director shall incorporate it into the permit as a condition. Where the Director's review of an application indicates that the permittee's plan is inadequate (based on the factors in §146.07), the Director shall require the applicant to revise the plan, prescribe a plan for corrective action as a condition of the permit under paragraph (b) of this section, or deny the application. The Director may disregard the provisions of §146.06 (Area of Review) and §146.07 (Corrective Action) when reviewing an application to permit an existing Class II well.

(b) *Requirements*—(1) *Existing injection wells.* Any permit issued for an existing injection well (other than Class II) requiring corrective action shall include a compliance schedule requiring any corrective action accepted or prescribed under paragraph (a) of this section to be completed as soon as possible.

(2) *New injection wells.* No owner or operator of a new injection well may begin injection until all required corrective action has been taken.

(3) *Injection pressure limitation.* The Director may require as a permit condition that injection pressure be so limited that pressure in the injection zone does not exceed hydrostatic pressure at the site of any improperly completed or abandoned well within the area of review. This pressure limitation shall satisfy the corrective action requirement. Alternatively, such injection pressure limitation can be part of a compliance schedule and last until all other required corrective action has been taken.

(4) *Class III wells only.* When setting corrective action requirements the Director shall consider the overall effect of the project on the hydraulic gradient in potentially affected USDWs, and the corresponding changes in potentiometric surface(s) and flow direction(s) rather than the discrete effect of each well. If a decision is made that corrective action is not necessary based on the determinations above, the monitoring program required in §146.33(b) shall be designed to verify the validity of such determinations.

Subpart F—Financial Responsibility: Class I Hazardous Waste Injection Wells

SOURCE: 49 FR 20186, May 11, 1984, unless otherwise noted.

§ 144.60 Applicability.

(a) The requirements of §§ 144.62, 144.63, and 144.70 apply to owners and operators of all existing and new Class I Hazardous waste injection wells, except as provided otherwise in this section.

§ 144.61 Definitions of terms as used in this subpart.

(a) *Plugging and abandonment plan* means the plan for plugging and abandonment prepared in accordance with the requirements of §§ 144.28 and 144.51.

(b) *Current plugging cost estimate* means the most recent of the estimates prepared in accordance with § 144.62 (a), (b) and (c).

(c) *Parent corporation* means a corporation which directly owns at least 50 percent of the voting stock of the corporation which is the injection well owner or operator; the latter corporation is deemed a *subsidiary* of the parent corporation.

(d) The following terms are used in the specifications for the financial test for plugging and abandonment. The definitions are intended to represent the common meanings of the terms as they are generally used by the business community.

Assets means all existing and all probable future economic benefits obtained or controlled by a particular entity.

Current assets means cash or other assets or resources commonly identified as those which are reasonably expected to be realized in cash or sold or consumed during the normal operating cycle of the business.

Current liabilities means obligations whose liquidation is reasonably expected to require the use of existing resources properly classifiable as current assets or the creation of other current liabilities.

Independently audited refers to an audit performed by an independent certified public accountant in accordance with generally accepted auditing standards.

Liabilities means probable future sacrifices of economic benefits arising from present obligations to transfer assets or provide services to other entities in the future as a result of past transactions or events.

Net working capital means current assets minus current liabilities.

Net worth means total assets minus total liabilities and is equivalent to owner's equity.

Tangible net worth means the tangible assets that remain after deducting liabilities; such assets would not include intangibles such as goodwill and rights to patents or royalties.

§ 144.62 Cost estimate for plugging and abandonment.

(a) The owner or operator must prepare a written estimate, in current dollars, of the cost of plugging the injection well in accordance with the plugging and abandonment plan as specified in §§ 144.28 and 144.51. The plugging and abandonment cost estimate must equal the cost of plugging and abandonment at the point in the facility's operating life when the extent and manner of its operation would making plugging and abandonment the most expensive, as indicated by its plugging and abandonment plan.

(b) The owner or operator must adjust the plugging and abandonment cost estimate for inflation within 30 days after each anniversary of the date on which the first plugging and abandonment cost estimate was prepared. The adjustment must be made as specified in paragraphs (b) (1) and (2) of this section, using an inflation factor derived from the annual Oil and Gas Field Equipment Cost Index. The inflation factor is the result of dividing the latest published annual Index by the Index for the previous year.

(1) The first adjustment is made by multiplying the plugging and abandonment cost estimate by the inflation factor. The result is the adjusted plugging and abandonment cost estimate.

(2) Subsequent adjustments are made by multiplying the latest adjusted plugging and abandonment cost estimate by the latest inflation factor.

(c) The owner or operator must revise the plugging and abandonment cost estimate whenever a change in the plugging and abandonment plan increases the cost of plugging and abandonment. The revised plugging and abandonment cost estimate must be adjusted for inflation as specified in § 144.62(b).

(d) The owner or operator must keep the following at the facility during the operating life of the facility: the latest plugging and abandonment cost estimate prepared in accordance with §144.62 (a) and (c) and, when this estimate has been adjusted in accordance with §144.62(b), the latest adjusted plugging and abandonment cost estimate.

§144.63 Financial assurance for plugging and abandonment.

An owner or operator of each facility must establish financial assurance for the plugging and abandonment of each existing and new Class I hazardous waste injection well. He must choose from the options as specified in paragraphs (a) through (f) of this section.

(a) *Plugging and abandonment trust fund.* (1) An owner or operator may satisfy the requirements of this section by establishing a plugging and abandonment trust fund which conforms to the requirements of this paragraph and submitting an originally signed duplicate of the trust agreement to the Regional Administrator. An owner or operator of a Class I well injecting hazardous waste must submit the originally signed duplicate of the trust agreement to the Regional Administrator with the permit application or for approval to operate under rule. The trustee must be an entity which has the authority to act as a trustee and whose trust operations are regulated and examined by a Federal or State agency.

(2) The wording of the trust agreement must be identical to the wording specified in §144.70(a)(1), and the trust agreement must be accompanied by a formal certification of acknowledgment (for example, see §144.70(a)(2)). Schedule A of the trust agreement must be updated within 60 days after a change in the amount of the current plugging and abandonment cost estimate covered by the agreement.

(3) Payments into the trust fund must be made annually by the owner or operator over the term of the initial permit or over the remaining operating life of the injection well as estimated in the plugging and abandonment plan, whichever period is shorter; this period is hereafter referred to as the ''pay-in period.'' The payments into the plugging and abandonment trust fund must be made as follows:

(i) For a new well, the first payment must be made before the initial injection of hazardous waste. A receipt from the trustee for this payment must be submitted by the owner or operator to the Regional Administrator before this initial injection of hazardous waste. The first payment must be at least equal to the current plugging and abandonment cost estimate, except as provided in §144.70(g), divided by the number of years in the pay-in period. Subsequent payments must be made no later than 30 days after each anniversary date of the first payment. The amount of each subsequent payment must be determined by this formula:

$$\text{Next payment} = \frac{PE - CV}{Y}$$

where PE is the current plugging and abandonment cost estimate, CV is the current value of the trust fund, and Y is the number of years remaining in the pay-in period.

(ii) If an owner or operator establishes a trust fund as specified in §144.63(a) of this chapter, and the value of that trust fund is less than the current plugging and abandonment cost estimate when a permit is awarded for the injection well, the amount of the current plugging and abandonment cost estimate still to be paid into the trust fund must be paid in over the pay-in period as defined in paragraph (a)(3) of this section. Payments must continue to be made no later than 30 days after each anniversary date of the first payment made pursuant to part 144 of this chapter. The amount of each payment must be determined by this formula:

$$\text{Next payment} = \frac{PE - CV}{Y}$$

where PE is the current plugging and abandonment cost estimate, CV is the current value of the trust fund, and Y is the number of years remaining in the pay-in period.

(4) The owner or operator may accelerate payments into the trust fund or he may deposit the full amount of the current plugging and abandonment

cost estimate at the time the fund is established. However, he must maintain the value of the fund at no less than the value that the fund would have if annual payments were made as specified in paragraph (a)(3) of this section.

(5) If the owner or operator establishes a plugging and abandonment trust fund after having used one or more alternate mechanisms specified in this section or in § 144.63 of this chapter, his first payment must be in at least the amount that the fund would contain if the trust fund were established initially and annual payments made according to specifications of this paragraph.

(6) After the pay-in period is completed, whenever the current plugging and abandonment cost estimate changes, the owner or operator must compare the new estimate with the trustee's most recent annual valuation of the trust fund. If the value of the fund is less than the amount of the new estimate, the owner or operator, within 60 days after the change in the cost estimate, must either deposit an amount into the fund so that its value after this deposit at least equals the amount of the current plugging and abandonment cost estimate, or obtain other financial assurance as specified in this section to cover the difference.

(7) If the value of the trust fund is greater than the total amount of the current plugging and abandonment cost estimate, the owner or operator may submit a written request to the Regional Administrator for release of the amount in excess of the current plugging and abandonment cost estimate.

(8) If an owner or operator substitutes other financial assurance as specified in this section for all or part of the trust fund, he may submit a written request to the Regional Administrator for release of the amount in excess of the current plugging and abandonment cost estimate covered by the trust fund.

(9) Within 60 days after receiving a request from the owner or operator for release of funds as specified in paragraph (a) (7) or (8) of this section, the Regional Administrator will instruct the trustee to release to the owner or operator such funds as the Regional Administrator specifies in writing.

(10) After beginning final plugging and abandonment, an owner or operator or any other person authorized to perform plugging and abandonment may request reimbursement for plugging and abandonment expenditures by submitting itemized bills to the Regional Administrator. Within 60 days after receiving bills for plugging and abandonment activities, the Regional Administrator will determine whether the plugging and abandonment expenditures are in accordance with the plugging and abandonment plan or otherwise justified, and if so, he will instruct the trustee to make reimbursement in such amounts as the Regional Administrator specifies in writing. If the Regional Administrator has reason to believe that the cost of plugging and abandonment will be significantly greater than the value of the trust fund, he may withhold reimbursement of such amounts as he deems prudent until he determines, in accordance with § 144.63(i), that the owner or operator is no longer required to maintain financial assurance for plugging and abandonment.

(11) The Regional Administrator will agree to termination of the trust when:

(i) An owner or operator substitutes alternate financial assurance as specified in this section; or

(ii) The Regional Administrator releases the owner or operator from the requirements of this section in accordance with § 144.63(i).

(b) *Surety bond guaranteeing payment into a plugging and abandonment trust fund.* (1) An owner or operator must satisfy the requirements of this section by obtaining a surety bond which conforms to the requirements of this paragraph and submitting the bond to the Regional Administrator with the application for a permit or for approval to operate under rule. The bond must be effective before the initial injection of hazardous waste. The surety company issuing the trust must, at a minimum, be among those listed as acceptable sureties on Federal bonds in Circular 570 of the U.S. Department of the Treasury.

(2) The wording of the surety bond must be identical to the wording in § 144.70(b).

(3) The owner or operator who uses a surety bond to satisfy the requirements of this section must also establish a standby trust fund. Under the terms of the bond, all payments made thereunder will be deposited by the surety directly into the standby trust fund in accordance with instructions from the Regional Administrator. This standby trust fund must meet the requirements specified in § 144.63(a), except that:

(i) An originally signed duplicate of the trust agreement must be submitted to the Regional Administrator with the surety bond; and

(ii) Until the standby trust fund is funded pursuant to the requirements of this section, the following are not required by these requirements:

(A) Payments into the trust fund as specified in § 144.63(a);

(B) Updating of Schedule A of the trust agreement [see § 144.70(a)] to show current plugging and abandonment cost estimates;

(C) Annual valuations as required by the trust agreement; and

(D) Notices of nonpayment as required by the trust agreement.

(4) The bond must guarantee that the owner or operator will:

(i) Fund the standby trust fund in an amount equal to the penal sum of the bond before beginning of plugging and abandonment of the injection well; or

(ii) Fund the standby trust fund in an amount equal to the penal sum within 15 days after an order to begin plugging and abandonment is issued by the Regional Administrator or a U.S. district court or other court of competent jurisdiction; or

(iii) Provide alternate financial assurance as specified in this section, and obtain the Regional Administrator's written approval of the assurance provided, within 90 days after receipt by both the owner or operator and the Regional Administrator of a notice of cancellation of the bond from the surety.

(5) Under the terms of the bond, the surety will become liable on the bond obligation when the owner or operator fails to perform as guaranteed by the bond.

(6) The penal sum of the bond must be in amount at least equal to the current plugging and abandonment cost estimate, except as provided in § 144.63(g).

(7) Whenever the current plugging and abandonment cost estimate increases to an amount greater than the penal sum, the owner or operator, within 60 days after the increase, must either cause the penal sum to be increased to an amount at least equal to the current plugging and abandonment cost estimate and submit evidence of such increase to the Regional Administrator, or obtain other financial assurance as specified in this section to cover the increase. Whenever the current plugging and abandonment cost estimate decreases, the penal sum may be reduced to the amount of the current plugging and abandonment cost estimate following written approval by the Regional Administrator.

(8) Under the terms of the bond, the surety may cancel the bond by sending notice of cancellation by certified mail to the owner or operator and to the Regional Administrator. Cancellation may not occur, however, during 120 days beginning on the date of the receipt of the notice of cancellation by both owner or operator and the Regional Administrator as evidenced by the returned receipts.

(9) The owner or operator may cancel the bond if the Regional Administrator has given prior written consent based on his receipt of evidence of alternate financial assurance as specified in this section.

(c) *Surety bond guaranteeing performance of plugging and abandonment.* (1) An owner or operator may satisfy the requirements of this section by obtaining a surety bond which conforms to the requirements of this paragraph and submitting the bond to the Regional Administrator. An owner or operator of a new facility must submit the bond to the Regional Administrator with the permit application or for approval to operate under rule. The bond must be effective before injection of hazardous waste is started. The surety company issuing the bond must, at a minimum, be among those listed as acceptable

sureties on Federal bonds in Circular 570 of the U.S. Department of the Treasury.

(2) The wording of the surety bond must be identical to the wording specified in § 144.70(c).

(3) The owner or operator who uses a surety bond to satisfy the requirements of this section must also establish a standby trust fund. Under the terms of the bond, all payments made thereunder will be deposited by the surety directly into the standby trust fund in accordance with instructions from the Regional Administrator. The standby trust must meet the requirements specified in § 144.63(a), except that:

(i) An original signed duplicate of the trust agreement must be submitted to the Regional Administrator with the surety bond; and

(ii) Unless the standby trust fund is funded pursuant to the requirements of this section, the following are not required by these regulations:

(A) Payments into the trust fund as specified in § 144.63(a);

(B) Updating of Schedule A of the trust agreement [see § 144.70(a)] to show current plugging and abandonment cost estimates;

(C) Annual valuations as required by the trust agreement; and

(D) Notices of nonpayment as required by the trust agreement.

(4) The bond must guarantee that the owner or operator will:

(i) Perform plugging and abandonment in accordance with the plugging and abandonment plan and other requirements of the permit for the injection well whenever required to do so; or

(ii) Provide alternate financial assurance as specified in this section, and obtain the Regional Administrator's written approval of the assurance provided, within 90 days after receipt by both the owner or operator and the Regional Administrator of a notice of cancellation of the bond from the surety.

(5) Under the terms of the bond, the surety will become liable on the bond obligation when the owner or operator fails to perform as guaranteed by the bond. Following a determination that the owner or operator has failed to perform plugging and abandonment in accordance with the plugging and abandonment plan and other permit requirements when required to do so, under terms of the bond the surety will perform plugging and abandonment as guaranteed by the bond or will deposit the amount of the penal sum into the standby trust fund.

(6) The penal sum of the bond must be in an amount at least equal to the current plugging and abandonment cost estimate.

(7) Whenever the current plugging and abandonment cost estimate increases to an amount greater than the penal sum, the owner or operator, within 60 days after the increase, must either cause the penal sum to be increased to an amount at least equal to the current plugging and abandonment cost estimate and submit evidence of such increase to the Regional Administrator, or obtain other financial assurance as specified in this section. Whenever the plugging and abandonment cost estimate decreases, the penal sum may be reduced to the amount of the current plugging and abandonment cost estimate following written approval by the Regional Administrator.

(8) Under the terms of the bond, the surety may cancel the bond by sending notice of cancellation by certified mail to the owner or operator and to the Regional Administrator. Cancellation may not occur, however, during the 120 days beginning on the date of receipt of the notice of cancellation by both the owner or operator and the Regional Administrator, as evidenced by the return receipts.

(9) The owner or operator may cancel the bond if the Regional Administrator has given prior written consent. The Regional Administrator will provide such written consent when:

(i) An owner or operator substitute alternate financial assurance as specified in this section; or

(ii) The Regional Administrator releases the owner or operator from the requirements of this section in accordance with § 144.63(i).

(10) The surety will not be liable for deficiencies in the performance of plugging and abandonment by the owner or operator after the Regional Administrator releases the owner or operator

from the requirements of this section in accordance with § 144.63(i).

(d) *Plugging and abandonment letter of credit.* (1) An owner or operator may satisfy the requirements of this section by obtaining an irrevocable standby letter of credit which conforms to the requirements of this paragraph and submitting the letter to the Regional Administrator. An owner or operator of an injection well must submit the letter of credit to the Regional Administrator during submission of the permit application or for approval to operate under rule. The letter of credit must be effective before initial injection of hazardous waste. The issuing institution must be an entity which has the authority to issue letters of credit and whose letter-of-credit operations are regulated and examined by a Federal or State agency.

(2) The wording of the letter of credit must be identical to the wording specified in § 144.70(d).

(3) An owner or operator who uses a letter of credit to satisfy the requirements of this section must also establish a standby trust fund. Under the terms of the letter of credit, all amounts paid pursuant to a draft by the Regional Administrator will be deposited by the issuing institution directly into the standby trust fund in accordance with instructions from the Regional Administrator. This standby trust fund must meet the requirements of the trust fund specified in § 144.63(a), except that:

(i) An originally signed duplicate of the trust agreement must be submitted to the Regional Administrator with the letter of credit; and

(ii) Unless the standby trust fund is funded pursuant to the requirements of this section, the following are not required by these regulations:

(A) Payments into the trust fund as specified in § 144.63(a);

(B) Updating of Schedule A of the trust agreement (see § 144.70(a)) to show current plugging and abandonment cost estimates;

(C) Annual valuations as required by the trust agreement; and

(D) Notices of nonpayment as required by the trust agreement.

(4) The letter of credit must be accompanied by a letter from the owner or operator referring to the letter of credit by number, issuing institution, and date, and providing the following information: the EPA Identification Number, name, and address of the facility, and the amount of funds assured for plugging and abandonment of the well by the letter of credit.

(5) The letter of credit must be irrevocable and issued for a period of at least 1 year. The letter of credit must provide that the expiration date will be automatically extended for a period of at least 1 year unless, at least 120 days before the current expiration date, the issuing institution notifies both the owner or operator and the Regional Administrator by certified mail of a decision not to extend the expiration date. Under the terms of the letter of credit, the 120 days will begin on the date when both the owner or operator and the Regional Administrator have received the notice, as evidenced by the return receipts.

(6) The letter of credit must be issued in an amount at least equal to the current plugging and abandonment cost estimate, except as provided in § 144.63(g).

(7) Whenever the current plugging and abandonment cost estimate increases to an amount greater than the amount of the credit, the owner or operator, within 60 days after the increase, must either cause the amount of the credit to be increased so that it at least equals the current plugging and abandonment cost estimate and submit evidence of such increase to the Regional Administrator, or obtain other financial assurance as specified in this section to cover the increase. Whenever the current plugging and abandonment cost estimate decreases, the amount of the credit may be reduced to the amount of the current plugging and abandonment cost estimate following written approval by the Regional Administrator.

(8) Following a determination that the owner or operator has failed to perform final plugging and abandonment in accordance with the plugging and abandonment plan and other permit requirements when required to do so, the Regional Administrator may draw on the letter of credit.

(9) If the owner or operator does not establish alternate financial assurance as specified in this section and obtain written approval of such alternate assurance from the Regional Administrator within 90 days after receipt by both the owner or operator and the Regional Administrator of a notice from the issuing institution that it has decided not to extend the letter of credit beyond the current expiration date, the Regional Administrator will draw on the letter of credit. The Regional Administrator may delay the drawing if the issuing institution grants an extension of the term of the credit. During the last 30 days of any such extension the Regional Administrator will draw on the letter of credit if the owner or operator has failed to provide alternate financial assurance as specified in this section and obtain written approval of such assurance from the Regional Administrator.

(10) The Regional Administrator will return the letter of credit to the issuing institution for termination when:

(i) An owner or operator substitutes alternate financial assurance as specified in this section; or

(ii) The Regional Administrator releases the owner or operator from the requirements of this section in accordance with § 144.63(i).

(e) *Plugging and abandonment insurance.* (1) An owner or operator may satisfy the requirements of this section by obtaining plugging and abandonment insurance which conforms to the requirements of this paragraph and submitting a certificate of such insurance to the Regional Administrator. An owner or operator of a new injection well must submit the certificate of insurance to the Regional Administrator with the permit application or for approval operate under rule. The insurance must be effective before injection starts. At a minimum, the insurer must be licensed to transact the business of insurance, or eligible to provide insurance as an excess or surplus lines insurer, in one or more States.

(2) The wording of the certificate of insurance must be identical to the wording specified in § 144.70(e).

(3) The plugging and abandonment insurance policy must be issued for a face amount at least equal to the current plugging and abandonment estimate, except as provided in § 144.63(g). The term ''face amount'' means the total amount the insurer is obligated to pay under the policy. Actual payments by the insurer will not change the face amount, although the insurers future liability will be lowered by the amount of the payments.

(4) The plugging and abandonment insurance policy must guarantee that funds will be available whenever final plugging and abandonment occurs. The policy must also guarantee that once plugging and abandonment begins, the issurer will be responsible for paying out funds, up to an amount equal to the face amount of the policy, upon the direction of the Regional Administrator, to such party or parties as the Regional Administrator specifies.

(5) After beginning plugging and abandonment, an owner or operator or any other person authorized to perform plugging and abandonment may request reimbursement for plugging and abandonment expenditures by submitting itemized bills to the Regional Administrator. Within 60 days after receiving bills for plugging and abandonment activities, the Regional Administrator will determine whether the plugging and abandonment expenditures are in accordance with the plugging and abandonment plan or otherwise justified, and if so, he will instruct the insurer to make reimbursement in such amounts as the Regional Administrator specifies in writing. If the Regional Administrator has reason to believe that the cost of plugging and abandonment will be significantly greater than the face amount of the policy, he may withhold reimbursement of such amounts as he deems prudent until he determines, in accordance with § 144.63(i), that the owner or operator is no longer required to maintain financial assurance for plugging and abandonment of the injection well.

(6) The owner or operator must maintain the policy in full force and effect until the Regional Administrator consents to termination of the policy by the owner or operator as specified in paragraph (e)(10) of this section. Failure to pay the premium, without substitution of alternate financial assurance as specified in this section, will

constitute a significant violation of these regulations, warranting such remedy as the Regional Administrator deems necessary. Such violation will be deemed to begin upon receipt by the Regional Administrator of a notice of future cancellation, termination, or failure to renew due to nonpayment of the premium, rather than upon the date of expiration.

(7) Each policy must contain provisions allowing assignment to a successor owner or operator. Such assignment may be conditional upon consent of the insurer, provided such consent is not unreasonably refused.

(8) The policy must provide that the insurer may not cancel, terminate, or fail to renew the policy except for failure to pay the premium. The automatic renewal of the policy must, at a minimum, provide the insured with the option of renewal at the face amount of the expiring policy. If there is a failure to pay the premium, the insurer may elect to cancel, terminate, or fail to renew the policy by sending notice by certified mail to the owner or operator and the Regional Administrator. Cancellation, termination, or failure to renew may not occur, however, during 120 days beginning with the date of receipt of the notice by both the Regional Administrator and the owner or operator, as evidenced by the return of receipts. Cancellation, termination, or failure to renew may not occur and the policy will remain in full force and effect in the event that on or before the date of expiration:

(i) The Regional Administrator deems the injection well abandoned; or

(ii) The permit is terminated or revoked or a new permit is denied; or

(iii) Plugging and abandonment is ordered by the Regional Administrator or a U.S. district court or other court of competent jurisdiction; or

(iv) The owner or operator is named as debtor in a voluntary or involuntary proceeding under title 11 (Bankruptcy), U.S. Code; or

(v) The premium due is paid.

(9) Whenever the current plugging and abandonment cost estimate increases to an amount greater than the face amount of the policy, the owner or operator, within 60 days after the increase, must either cause the face amount to be increased to an amount at least equal to the current plugging and abandonment estimate and submit evidence of such increase to the Regional Administrator, or obtain other financial assurance as specified in this section to cover the increase. Whenever the current plugging and abandonment cost estimate decreases, the face amount may be reduced to the amount of the current plugging and abandonment cost estimate following written approval by the Regional Administrator.

(10) The Regional Administrator will give written consent to the owner or operator that he may terminate the insurance policy when:

(i) An owner or operator substitutes alternate financial assurance as specified in this section; or

(ii) The Regional Administrator releases the owner or operator from the requirements of this section in accordance with § 144.63(i).

(f) *Financial test and corporate guarantee for plugging and abandonment.* (1) An owner or operator may satisfy the requirements of this section by demonstrating that he passes a financial test as specified in this paragraph. To pass this test the owner or operator must meet the criteria of either paragraph (f)(1)(i) or (f)(1)(ii) of this section:

(i) The owner or operator must have:

(A) Two of the following three ratios: A ratio of total liabilities to net worth less than 2.0; a ratio of the sum of net income plus depreciation, depletion, and amortization to total liabilities greater than 0.1; and a ratio of current assets to current liabilities greater than 1.5; and

(B) Net working capital and tangible net worth each at least six times the sum of the current plugging and abandonment cost estimate; and

(C) Tangible net worth of at least $10 million; and

(D) Assets in the United States amounting to at least 90 percent of his total assets or at least six times the sum of the current plugging and abandonment cost estimate.

(ii) The owner or operator must have:

(A) A current rating for his most recent bond issuance of AAA, AA, A or BBB as issued by Standard and Poor's

or Aaa, Aa, A, or Baa as issued by Moody's; and

(B) Tangible net worth at least six times the sum of the current plugging and abandonment cost estimate; and

(C) Tangible net worth of at least $10 million; and

(D) Assets located in the United States amounting to at least 90 percent of his total assets or at least six times the sum of the current plugging and abandonment cost estimates.

(2) The phrase ''current plugging and abandonment cost estimate'' as used in paragraph (f)(1) of this section refers to the cost estimate required to be shown in paragraphs 1 through 4 of the letter from the owner's or operator's chief financial officer § 144.70(f).

(3) To demonstrate that he meets this test, the owner or operator must submit the following items to the Regional Administrator:

(i) A letter signed by the owner's or operator's chief financial officer and worded as specified in § 144.70(f); and

(ii) A copy of the independent certified public accountant's report on examination of the owner's or operator's financial statements for the latest completed fiscal year; and

(iii) A special report from the owner's or operator's independent certified public accountant to the owner or operator stating that:

(A) He has compared the data which the letter from the chief financial officer specifies as having been derived from the independently audited, year-end financial statements for the latest fiscal year with the amounts in such financial statements; and

(B) In connection with that procedure, no matters came to his attention which caused him to believe that the specified data should be adjusted.

(4) An owner or operator of a new injection well must submit the items specified in paragraph (f)(3) of this section to the Regional Administrator within 90 days after the close of each succeeding fiscal year. This information must consist of all three items specified in paragraph (f)(3) of this section.

(5) After the initial submission of items specified in paragraph (f)(3) of this section, the owner or operator must send updated information to the Regional Administrator within 90 days after the close of each succeeding fiscal year. This information must consist of all three items specified in paragraph (f)(3) of this section.

(6) If the owner or operator no longer meets the requirements of paragraph (f)(1) of this section, he must send notice to the Regional Administrator of intent to establish alternate financial assurance as specified in this section. The notice must be sent by certified mail within 90 days after the end of the fiscal year for which the year-end financial data show that the owner or operator no longer meets the requirements. The owner or operator must provide the alternate financial assurance within 120 days after the end of such fiscal year.

(7) The Regional Administrator may, based on a reasonable belief that the owner or operator may no longer meet the requirements of paragraph (f)(1) of this section, require reports of financial condition at any time from the owner or operator in addition to those specified in paragraph (f)(3) of this section. If the Regional Administrator finds, on the basis of such reports or other information, that the owner or operator no longer meets the requirements of paragraph (f)(1) of this section, the owner or operator must provide alternate financial assurance as specified in this section within 30 days after notification of such a finding.

(8) The Regional Administrator may disallow use of this test on the basis of qualifications in the opinion expressed by the independent certified public accountant in his report on examination of the owner's or operator's financial statements [see paragraph (f)(3)(ii) of this section]. An adverse opinion or disclaimer of opinion will be cause for disallowance. The Regional Administrator will evaluate other qualifications on an individual basis. The owner or operator must provide alternate financial assurance as specified in this section within 30 days after notification of the disallowance.

(9) The owner or operator is no longer required to submit the items specified in paragraph (f)(3) of this section when:

(i) An owner or operator substitutes alternate financial assurance as specified in this section; or

(ii) The Regional Administrator releases the owner or operator from the requirements of this section in accordance with § 144.63(i).

(10) An owner or operator may meet the requirements of this section by obtaining a written guarantee, hereafter referred to as ''corporate guarantee.'' The guarantee must be the parent corporation of the owner or operator. The guarantee must meet the requirements for owners or operators in paragraphs (f)(1) through (f)(8) of this section and must comply with the terms of the corporate guarantee. The wording of the corporate guarantee must be identical to the wording specified in § 144.70(h). The corporate guarantee must accompany the items sent to the Regional Administrator as specified in paragraph (f)(3) of this section. The terms of the corporate guarantee must provide that:

(i) If the owner or operator fails to perform plugging and abandonment of the injection well covered by the corporate guarantee in accordance with the plugging and abandonment plan and other permit requirements whenever required to do so, the guarantee will do so or establish a trust fund as specified in § 144.63(a) in the name of the owner or operator.

(ii) The corporate guarantee will remain in force unless the guarantor sends notice of cancellation by certified mail to the owner or operator and the Regional Administrator, as evidenced by the return receipts. Cancellation may not occur, however, during the 120 days beginning on the date of receipt of the notice of cancellation by both the owner or operator and the Regional Administrator, as evidenced by the return receipts.

(iii) If the owner or operator fails to provide alternate financial assurance as specified in this section and obtain the written approval of such alternate assurance from the Regional Administrator within 90 days after receipt by both the owner or operator and the Regional Administrator of a notice of cancellation of the corporate guarantee from the guarantor, the guarantor will provide such alternative financial assurance in the name of the owner or operator.

(g) *Use of multiple financial mechanisms.* An owner or operator may satisfy the requirements of this section by establishing more than one financial mechanism per injection well. These mechanisms are limited to trust funds, surety bonds, guaranteeing payment into a trust fund, letters of credit, and insurance. The mechanisms must be as specified in paragraphs (a), (b), (d), and (e), respectively, of this section, except that it is the combination of mechanisms, rather than the single mechanism, which must provide financial assurance for an amount at least equal to the adjusted plugging and abandonment cost. If an owner or operator uses a trust fund in combination with a surety bond or letter of credit, he may use that trust fund as the standby trust fund for the other mechanisms. A single standby trust may be established for two or more mechanisms. The Regional Administrator may invoke any or all of the mechanisms to provide for plugging and abandonment of the injection well.

(h) *Use of a financial mechanism for multiple facilities.* An owner or operator may use a financial assurance mechanism specified in this section to meet the requirements of this section for more than one injection well. Evidence of financial assurance submitted to the Regional Administrator must include a list showing, for each injection well, the EPA Identification Number, name, address, and the amount of funds for plugging and abandonment assured by the mechanism. If the injection wells covered by the mechanism are in more than one Region, identical evidence of financial assurance must be submitted to and maintained with the Regional Administrators of all such Regions. The amount of funds available through the mechanism must be no less than the sum of funds that would be available if a separate mechanism had been established and maintained for each injection well. In directing funds available through the mechanism for plugging and abandonment of any of the injection wells covered by the mechanism, the Regional Administrator may direct only the amount of funds designated for that injection well, unless

the owner or operator agrees to use additional funds available under the mechanism.

(i) *Release of the owner or operator from the requirements of this section.* Within 60 days after receiving certifications from the owner or operator and an independent registered professional engineer that plugging and abandonment has been accomplished in accordance with the plugging and abandonment plan, the Regional Administrator will notify the owner or operator in writing that he is no longer required by this section to maintain financial assurance for plugging and abandonment of the injection well, unless the Regional Administrator has reason to believe that plugging and abandonment has not been in accordance with the plugging and abandonment plan.

§ 144.64 Incapacity of owners or operators, guarantors, or financial institutions.

(a) An owner or operator must notify the Regional Administrator by certified mail of the commencement of a voluntary or involuntary proceeding under title 11 (Bankruptcy), U.S. Code, naming the owner or operator as debtor, within 10 business days after the commencement of the proceeding. A guarantor of a corporate guarantee as specified in § 144.63(f) must make such a notification if he is named as debtor, as required under the terms of the guarantee (§ 144.70(f)).

(b) An owner or operator who fulfills the requirements of § 144.63 by obtaining a letter of credit, surety bond, or insurance policy will be deemed to be without the required financial assurance or liability coverage in the event of bankruptcy, insolvency, or a suspension or revocation of the license or charter of the issuing institution. The owner or operator must establish other financial assurance or liability coverage within 60 days after such an event.

§ 144.65 Use of State-required mechanisms.

(a) For a facility located in a State where EPA is administering the requirements of this subpart but where the State has plugging and abandonment regulations that include requirements for financial assurance of plugging and abandonment, an owner or operator may use State-required financial mechanisms to meet the requirements of this subpart if the Regional Administrator determines that the State mechanisms are at least equivalent to the mechanisms specified in this subpart. The Regional Administrator will evaluate the equivalency of the mechanisms mainly in terms of (1) certainty of the availability of funds for the required plugging and abandonment activities and (2) the amount of funds that will be made available. The Regional Administrator may also consider other factors. The owner or operator must submit to the Regional Administrator evidence of the establishment of the mechanism together with a letter requesting that the State-required mechanism be considered acceptable for meeting the requirements of this subpart. The submittal must include the following information: The facility's EPA Identification Number, name and address, and the amounts of funds for plugging and abandonment coverage assured by the mechanism. The Regional Administrator will notify the owner or operator of his determination regarding the mechanism's acceptability. The Regional Administrator may require the owner or operator to submit additional information as is deemed necessary for making this determination.

(b) If a State-required mechanism is found acceptable as specified in paragraph (a) of this section except for the amount of funds available, the owner or operator may satisfy the requirements of this subpart by increasing the funds available through the State-required mechanism or using additional mechanisms as specified in this subpart. The amounts of funds available through the State and Federal mechanisms must at least equal the amounts required by this subpart.

§ 144.66 State assumption of responsibility.

(a) If a State either assumes legal responsibility for an owner's or operator's compliance with the plugging and abandonment requirements of these regulations or assures that funds will be available from State sources to

cover these requirements, the owner or operator will be in compliance with the requirements of this subpart if the Regional Administrator determines that the State's assumption of responsibility is at least equivalent to the mechanisms specified in this subpart. The Regional Administrator will evaluate the equivalency of State guarantees mainly in terms of (1) certainty of the availability of funds for the required plugging and abandonment coverage and (2) the amount of funds that will be made available. The Regional Administrator may also consider other factors. The owner or operator must submit to the Regional Administrator a letter from the State describing the nature of the State's assumption of responsibility together with a letter from the owner or operator requesting that the State's asumption of responsibility be considered acceptable for meeting the requirements of this subpart. The letter from the State must include, or have attached to it, the following information: the facility's EPA Identification Number, name and address, and the amounts of funds for plugging and abandonment coverage that are guaranteed by the State. The Regional Administrator will notify the owner or operator of his determination regarding the acceptability of the State's guarantee in lieu of mechanisms specified in this subpart. The Regional Administrator may require the owner or operator to submit additional information as is deemed necessary to make this determination. Pending this determination, the owner or operator will be deemed to be in compliance with §144.63.

(b) If a State's assumption of responsibility is found acceptable as specified in paragraph (a) of this section except for the amount of funds available, the owner or operator may satisfy the requirements of this subpart by use of both the State's assurance and additional financial mechanisms as specified in this subpart. The amount of funds available through the State and Federal mechanisms must at least equal the amount required by this subpart.

§144.70 Wording of the instruments.

(a)(1) A trust agreement for a trust fund, as specified in §144.63(a) of this chapter, must be worded as follows, except that instructions in brackets are to be replaced with the relevant information and the brackets deleted:

TRUST AGREEMENT

TRUST AGREEMENT, the ''Agreement,'' entered into as of [date] by and between [name of the owner or operator], a [name of State] [insert ''corporation,'' ''partnership,'' ''association,'' or ''proprietorship''], the ''Grantor,'' and [name of corporate trustee], [insert ''incorporated in the State of _____'' or ''a national bank''], the ''Trustee.''

Whereas, the United States Environmental Protection Agency, ''EPA,'' an agency of the United States Government, has established certain regulations applicable to the Grantor, requiring that an owner or operator of an injection well shall provide assurance that funds will be available when needed for plugging and abandonment of the injection well,

Whereas, the Grantor has elected to establish a trust to provide all or part of such financial assurance for the facility(ies) identified herein,

Whereas, the Grantor, acting through its duly authorized officers, has selected the Trustee to be the trustee under this agreement, and the Trustee is willing to act as trustee,

Now, therefore, the Grantor and the Trustee agree as follows:

Section 1. Definitions. As used in this Agreement:

(a) The term ''Grantor'' means the owner or operator who enters into this Agreement and any successors or assigns of the Grantor.

(b) The term ''Trustee'' means the Trustee who enters into this Agreement and any successor Trustee.

(c) Facility or activity means any ''underground injection well'' or any other facility or activity that is subject to regulation under the Underground Injection Control Program.

Section 2. Identification of Facilities and Cost Estimates. This Agreement pertains to the facilities and cost estimates identified on attached Schedule A [on Schedule A, for each facility list the EPA Identification Number, name, address, and the current plugging and abandonment cost estimate, or portions thereof, for which financial assurance is demonstrated by this Agreement].

Section 3. Establishment of Fund. The Grantor and the Trustee hereby establish a trust fund, the ''Fund,'' for the benefit of EPA. The Grantor and the Trustee intend that no third party have access to the Fund except as herein provided. The Fund is established initially as consisting of the property, which

is acceptable to the Trustee, described in Schedule B attached hereto. Such property and any other property subsequently transferred to the Trustee is referred to as the Fund, together with all earnings and profits thereon, less any payments or distributions made by the Trustee pursuant to this Agreement. The Fund shall be held by the Trustee, IN TRUST, as hereinafter provided. The Trustee shall not be responsible nor shall it undertake any responsibility for the amount or adequacy of, nor any duty to collect from the Grantor, any payments necessary to discharge any liabilities of the Grantor established by EPA.

Section 4. Payment for Plugging and Abandonment. The Trustee shall make payments from the Fund as the EPA Regional Administrator shall direct, in writing, to provide for the payment of the costs of plugging and abandonment of the injection wells covered by this Agreement. The Trustee shall reimburse the Grantor or other persons as specified by the EPA Regional Administrator from the Fund for plugging and abandonment expenditures in such amounts as the EPA Regional Administrator shall direct in writing. In addition, the Trustee shall refund to the Grantor such amounts as the EPA Regional Administrator specifies in writing. Upon refund, such funds shall no longer constitute part of the Fund as defined herein.

Section 5. Payments Comprising the Fund. Payments made to the Trustee for the Fund shall consist of cash or securities acceptable to the Trustee.

Section 6. Trustee Management. The Trustee shall invest and reinvest the principal and income of the Fund and keep the Fund invested as a single fund, without distinction between principal and income, in accordance with general investment policies and guidelines which the Grantor may communicate in writing to the Trustee from time to time, subject, however, to the provisions of this Section. In investing, reinvesting, exchanging, selling, and managing the Fund, the Trustee shall discharge his duties with respect to the trust fund solely in the interest of the beneficiary and with the care, skill, prudence, and diligence under the circumstances then prevailing which persons of prudence, acting in a like capacity and familiar with such matters, would use in the conduct of an enterprise of a like character and with like aims; *except that*:

(i) Securities or other obligations of the Grantor, or any other owner or operator of the facilities, or any of their affiliates as defined in the Investment Company Act of 1940, as amended, 15 U.S.C. 80a–2.(a), shall not be acquired or held, unless they are securities or other obligations of the Federal or a State government;

(ii) The Trustee is authorized to invest the Fund in time or demand deposits of the Trustee, to the extent insured by an agency of the Federal or State government; and

(iii) The Trustee is authorized to hold cash awaiting investment or distribution uninvested for a reasonable time and without liability for the payment of interest thereon.

Section 7. Commingling and Investment. The Trustee is expressly authorized in its discretion:

(a) To transfer from time to time any or all of the assets of the Fund to any common, commingled, or collective trust fund created by the Trustee in which the Fund is eligible to participate, subject to all of the provisions thereof, to be commingled with the assets of other trusts participating therein; and

(b) To purchase shares in any investment company registered under the Investment Company Act of 1940, 15 U.S.C. 80a–1 *et seq.*, including one which may be created, managed, underwritten, or to which investment advice is rendered or the shares of which are sold by the Trustee. The Trustee may vote shares in its discretion.

Section 8. Express Powers of Trustee. Without in any way limiting the powers and discretions conferred upon the Trustee by the other provisions of this Agreement or by law, the Trustee is expressly authorized and empowered:

(a) To sell, exchange, convey, transfer, or otherwise dispose of any property held by it, by public or private sale. No person dealing with the Trustee shall be bound to see to the application of the purchase money or to inquire into the validity or expediency of any such sale or other disposition;

(b) To make, execute, acknowledge, and deliver any and all documents of transfer and conveyance and any and all other instruments that may be necessary or appropriate to carry out the powers herein granted;

(c) To register any securities held in the Fund in its own name or in the name of a nominee and to hold any security in bearer form or in book entry, or to combine certificates representing such securities with certificates of the same issue held by the Trustee in other fiduciary capacities, or to deposit or arrange for the deposit of such securities in a qualified central depository even though, when so deposited, such securities may be merged and held in bulk in the name of the nominee of such depositary with other securities deposited therein by another person, or to deposit or arrange for the deposit of any securities issued by the United States Government, or any agency or instrumentality thereof, with a Federal Reserve bank, but the books and records of the Trustee shall at all times show that all such securities are part of the Fund;

(d) To deposit any cash in the Fund in interest-bearing accounts maintained or savings certificates issued by the Trustee, in its

separate corporate capacity, or in any other banking institution affiliated with the Trustee, to the extent insured by an agency of the Federal or State government; and

(e) To compromise or otherwise adjust all claims in favor of or against the Fund.

Section 9. Taxes and Expenses. All taxes of any kind that may be assessed or levied against or in respect of the Fund and all brokerage commissions incurred by the Fund shall be paid from the Fund. All other expenses incurred by the Trustee in connection with the administration of this Trust, including fees for legal services rendered to the Trustee, the compensation of the Trustee to the extent not paid directly by the Grantor, and all other proper charges and disbursements of the Trustee shall be paid from the Fund.

Section 10. Annual Valuation. The Trustee shall annually, at least 30 days prior to the anniversary date of establishment of the Fund, furnish to the Grantor and to the appropriate EPA Regional Administrator a statement confirming the value of the Trust. Any securities in the Fund shall be valued at market value as of no more than 60 days prior to the anniversary date of establishment of the Fund. The failure of the Grantor to object in writing to the Trustee within 90 days after the statement has been furnished to the Grantor and the EPA Regional Administrator shall constitute a conclusively binding assent by the Grantor, barring the Grantor from asserting any claim or liability against the Trustee with respect to matters disclosed in the statement.

Section 11. Advice of Counsel. The Trustee may from time to time consult with counsel, who may be counsel to the Grantor, with respect to any question arising as to the construction of this Agreement of any action to be taken hereunder. The Trustee shall be fully protected, to the extent permitted by law, in acting upon the advice of counsel.

Section 12. Trustee Compensation. The Trustee shall be entitled to reasonable compensation for its services as agreed upon in writing from time to time with the Grantor.

Section 13. Successor Trustee. The Trustee may resign or the Grantor may replace the Trustee, but such resignation or replacement shall not be effective until the Grantor has appointed a successor trustee and this successor accepts the appointment. The successor trustee shall have the same powers and duties as those conferred upon the Trustee hereunder. Upon the successor trustee's acceptance of the appointment, the Trustee shall assign, transfer, and pay over to the successor trustee the funds and properties then constituting the Fund. If for any reason the Grantor cannot or does not act in the event of the resignation of the Trustee, the Trustee may apply to a court of competent jurisdiction for the appointment of a successor trustee or for instructions. The successor trustee shall specify the date on which it assumes administration of the trust in a writing sent to the Grantor, the EPA Regional Administrator, and the present Trustee by certified mail 10 days before such change becomes effective. Any expenses incurred by the Trustee as a result of any of the acts contemplated by this Section shall be paid as provided in Section 9.

Section 14. Instructions to the Trustee. All orders, requests, and instructions by the Grantor to the Trustee shall be in writing, signed by such persons as are designated in the attached Exhibit A or such other designees as the Grantor may designate by amendment to Exhibit A. The Trustee shall be fully protected in acting without inquiry in accordance with the Grantor's orders, requests, and instructions. All orders, requests, and instructions by the EPA Regional Administrator to the Trustee shall be in writing, signed by the EPA Regional Administrators of the Regions in which the facilities are located, or their designees, and the Trustee shall act and shall be fully protected in acting in accordance with such orders, requests, and instructions. The Trustee shall have the right to assume, in the absence of written notice to the contrary, that no event constituting a change or a termination of the authority of any person to act on behalf of the Grantor or EPA hereunder has occurred. The Trustee shall have no duty to act in the absence of such orders, requests, and instructions from the Grantor and/or EPA, except as provided for herein.

Section 15. Notice of Nonpayment. The Trustee shall notify the Grantor and the appropriate EPA Regional Administrator, by certified mail within 10 days following the expiration of the 30-day period after the anniversary of the establishment of the Trust, if no payment is received from the Grantor during that period. After the pay-in period is completed, the Trustee shall not be required to send a notice of nonpayment.

Section 16. Amendment of Agreement. This Agreement may be amended by an instrument in writing executed by the Grantor, the Trustee, and the appropriate EPA Regional Administrator, or by the Trustee and the appropriate EPA Regional Administrator if the Grantor ceases to exist.

Section 17. Irrevocability and Termination. Subject to the right of the parties to amend this Agreement as provided in Section 16, this Trust shall be irrevocable and shall continue until terminated at the written agreement of the Grantor, the Trustee, and the EPA Regional Administrator, or by the Trustee and the EPA Regional Administrator if the Grantor ceases to exist. Upon termination of the Trust, all remaining trust property, less final trust administration expenses, shall be delivered to the Grantor.

Section 18. Immunity and Indemnification. The Trustee shall not incur personal liability of any nature in connection with any act or omission, made in good faith, in the administration of this Trust, or in carrying out any directions by the Grantor or the EPA Regional Administrator issued in accordance with this Agreement. The Trustee shall be indemnified and saved harmless by the Grantor or from the Trust Fund, or both, from and against any personal liability to which the Trustee may be subjected by reason of any act or conduct in its official capacity, including all expenses reasonably incurred in its defense in the event the Grantor fails to provide such defense.

Section 19. Choice of Law. This Agreement shall be administered, construed, and enforced according to the laws of the State of [insert name of State].

Section 20. Interpretation. As used in this Agreement, words in the singular include the plural and words in the plural include the singular. The descriptive headings for each Section of this Agreement shall not affect the interpretation or the legal efficacy of this Agreement.

In Witness Whereof the parties have caused this Agreement to be executed by their respective officers duly authorized and their corporate seals to be hereunto affixed and attested as of the date first above written. The parties below certify that the wording of this Agreement is identical to the wording specified in 40 CFR 144.70(a)(1) as such regulations were constituted on the date first above written.

[Signature of Grantor]
By [Title]
Attest:

[Title]

[Seal]

[Signature of Trustee]
By
Attest:

[Title]

[Seal]

(2) The following is an example of the certification of acknowledgment which must accompany the trust agreement for a trust fund as specified in § 144.63(a). State requirements may differ on the proper content of this acknowledgment.

State of _____
County of _____

On this [date], before me personally came [owner or operator] to me known, who, being by me duly sworn, did depose and say that she/he resides at [address], that she/he is [title] of [corporation], the corporation described in and which executed the above instrument; that she/he knows the seal of said corporation; that the seal affixed to such instrument is such corporate seal; that it was so affixed by order to the Board of Directors of said corporation, and that she/he signed her/his name thereto by like order.

[Signature of Notary Public]

(b) A surety bond guaranteeing payment into a trust fund, as specified in § 144.63 of this chapter, must be worded as follows, except that instructions in brackets are to be replaced with the relevant information and the brackets deleted:

FINANCIAL GUARANTEE BOND

Dated bond executed: _____
Effective date: _____

Principal: [legal name and business address of owner or operator].

Type of organization: [insert ''individual,'' ''joint venture,'' ''partnership,'' or ''corporation''].

State of incorporation: _____

Surety(ies): [name(s) and business address(es)].

EPA Identification Number, name, address, and plugging and abandonment amount(s) for each facility guaranteed by this bond [indicate plugging and abandonment amounts separately]: _____

Total penal sum of bond: $_____

Surety's bond number: _____

Know All Persons By These Presents, That we, the Principal and Surity(ies) hereto are firmly bound to the U.S. Environmental Protection Agency (hereinafter called EPA), in the above penal sum for the payment of which we bind ourselves, our heirs, executors, administrators, successors, and assigns jointly and severally; provided that, where the Surety(ies) are corporations acting as co-surties, we, the Sureties, bind ourselves in such sum ''jointly and severally'' only for the purpose of allowing a joint action or actions against any or all of us, and for all other purposes each Surety binds itself, jointly and severally with the Principal, for the payment of such sum only as is set forth opposite the name of such Surety, but if no limit of liability is indicated, the limit of liability shall be the full amount of the penal sum.

Whereas said Principal is required, under the Underground Injection Control Regulations (UIC), to have a permit or comply with requirements to operate under rule in order to own or operate each injection well identified above, and

Whereas said Principal is required to provide financial assurance for plugging and abandonment as a condition of the permit or provisions to operate under rule, and

Whereas said Principal shall establish a standby trust fund as is required when a surety bond is used to provide such financial assurance;

Now, therefore, the conditions of the obligation are such that if the Principal shall faithfully, before the beginning of plugging and abandonment of each injection well identified above, fund the standby trust fund in the amount(s) identified above for the injection well,

Or if the Principal shall fund the standby trust fund in such amount(s) within 15 days after an order to begin plugging and abandonment is issued by an EPA Regional Administrator or a U.S. district court or other court of competent jurisdiction,

Or, if the Principal shall provide alternate financial assurance, as specified in subpart F of 40 CFR part 144, as applicable, and obtain the EPA Regional Administrator's written approval of such assurance, within 90 days after the date of notice of cancellation is received by both the Principal and the EPA Regional Administrator(s) from the Surety(ies), then this obligation shall be null and void, otherwise it is to remain in full force and effect.

The Surety(ies) shall become liable on this bond obligation only when the Principal has failed to fulfill the conditions described above. Upon notification by an EPA Regional Administrator that the Principal has failed to perform as guaranteed by this bond, the Surety(ies) shall place funds in the amount guaranteed for the injection well(s) into the standby trust funds as directed by the EPA Regional Administrator.

The liability of the Surety(ies) shall not be discharged by any payment or succession of payments hereunder, unless and until such payment or payments shall amount in the aggregate to the penal sum of the bond, but in no event shall the obligation of the Surety(ies) hereunder exceed the amount of said penal sum.

The Surety(ies) may cancel the bond by sending notice of cancellation by certified mail to the Principal and to the EPA Regional Administrator(s) for the Region(s) in which the injection well(s) is (are) located, provided, however, that that cancellation shall not occur during the 120 days beginning on the date of receipt of the notice of cancellation by both the Principal and the EPA Regional Administrator(s), as evidenced by the return receipts.

The Principal may terminate this bond by sending written notice to the Surety(ies), provided, however, that no such notice shall become effective until the Surety(ies) receive(s) written authorization for termination of the bond by the EPA Regional Administrator(s) of the Region(s) in which the bonded facility(ies) is (are) located.

[The following paragraph is an optional rider that may be included but is not required.]

Principal and Surety(ies) hereby agree to adjust the penal sum of the bond yearly so that it guarantees a new plugging and abandonment amount, provided that the penal sum does not increase by more than 20 percent in any one year, and no decrease in the penal sum takes place without the written permission of the EPA Regional Administrator(s).

In Witness Whereof, the Principal and Surety(ies) have executed this Financial Guarantee Bond and have affixed their seals on the date set forth above.

The persons whose signatures appear below hereby certify that they are authorized to execute this surety bond on behalf of the Principal and Surety(ies) and that the wording of this surety bond is identical to the wording specified in 40 CFR 144.70(b) as such regulations were constituted on the date this bond was executed.

Principal

[Signature(s)]
[Name(s)]
[Title(s)]
[Corporate seal]

Corporate Surety(ies)

[Name and address]
State of incorporation: _____.
Liability limit: $_____.
[Signature(s)]
[Name(s) and title(s)]
[Corporate seal]
[For every co-surety, provide signature(s), corporate seal, and other information in the same manner as for Surety above.]
Bond premium: $_____.

(c) A surety bond guaranteeing performance of plugging and abandonment, as specified in § 144.63(c), must be worded as follows, except that the instructions in brackets are to be replaced with the relevant information and the brackets deleted:

PERFORMANCE BOND

Date bond executed: _____.
Effective date: _____.
Principal: [legal name and business address of owner or operator].
Type of organization: [insert ''individual,'' ''joint venture,'' ''partnership,'' or ''corporation''].
State of incorporation: _____.
Surety(ies): [name(s) and business address(es)]

EPA Identification Number, name, address, and plugging and abandonment amounts(s) for each injection well guaranteed by this

bond [indicate plugging and abandonment amounts for each well]:

Total penal sum of bond: $_____.

Surety's bond number: _____.

Know All Persons By These Presents, That We, the Principal and Surety(ies) hereto are firmly bound to the U.S. Environmental Protection Agency [hereinafter called EPA], in the above penal sum for the payment of which we bind ourselves, our heirs, executors, administrators, successors, and assigns jointly and severally; provided that, where the Surety(ies) are corporations acting as co-sureties, we, the Sureties, bind ourselves in such sum ''jointly and severally'' only for the purpose of allowing a joint action or actions against any or all of us, and for all other purposes each Surety binds itself, jointly and severally with the Principal, for the payment of such sum only as is set forth opposite the name of such Surety, but if no limit of liability is indicated, the limit of liability shall be the full amount of the penal sum.

Whereas said Principal is required, under the Undergound Injection Control Regulations, as amended, to have a permit or comply with provisions to operate under rule for each injection well identified above, and

Whereas said Principal is required to provide financial assurance for plugging and abandonment as a condition of the permit or approval to operate under rule, and

Whereas said Principal shall establish a standby trust fund as is required when a surety bond is used to provide such financial assurance;

Now, Therefore, the conditions of this obligation are such that if the Principal shall faithfully perform plugging and abandonment, whenever required to do so, of each injection well for which this bond guarantees plugging and abandonment, in accordance with the plugging and abandonment plan and other rquirements of the permit or provisions for operating under rule and other requirements of the permit or provisions for operating under rule as may be amended, pursuant to all applicable laws, statutes, rules and regulations, as such laws, statutes, rules, and regulations may be amended,

Or, if the Principal shall provide alternate financial assurance as specified in subpart F of 40 CFR part 144, and obtain the EPA Regional Administrator's written approval of such assurance, within 90 days after the date of notice of cancellation is received by both the Principal and the EPA Regional Administrator(s) from the Surety(ies), then this obligation shall be null and void, otherwise it is to remain in full force and effect.

The Surety(ies) shall become liable on this bond obligation only when the Principal has failed to fulfill the conditions described above.

Upon notification by an EPA Regional Administrator that the Principal has been found in violation of the plugging and abandonment requirements of 40 CFR part 144, for an injection well which this bond guarantees performances of plugging and abandonment, the Surety(ies) shall either perform plugging and abandonment in accordance with the plugging and abandonment plan and other permit requirements or provisions for operating under rule and other requirements or place the amount for plugging and abandonment into a standby trust fund as directed by the EPA Regional Administrator.

Upon notification by an EPA Regional Administrator that the Principal has failed to provide alternate financial assurance as specified in subpart F of 40 CFR part 144, and obtain written approval of such assurance from the EPA Regional Administrator(s) during the 90 days following receipt by both the Principal and the EPA Regional Administrator(s) of a notice of cancellation of the bond, the Surety(ies) shall place funds in the amount guaranteed for the injection well(s) into the standby trust fund as directed by the EPA Regional Administrator.

The surety(ies) hereby waive(s) notification of amendments to plugging and abandonment plans, permits, applicable laws, statutes, rules, and regulations and agrees that no such amendment shall in any way alleviate its (their) obligation on this bond.

The liability of the Surety(ies) shall not be discharged by any payment or succession of payments hereunder, unless and until such payment or payments shall amount in the aggregate to the penal sum of the bond, but in no event shall the obligation of the Surety(ies) hereunder exceed the amount of said penal sum.

The Surety(ies) may cancel the bond by sending notice by certified mail to the owner or operator and to the EPA Regional Administrator(s) for the Region(s) in which the injection well(s) is (are) located, provided, however, that cancellation shall not occur during the 120 days beginning on the date of receipt of the notice of cancellation by both the Principal and the EPA Regional Administrator(s), as evidenced by the return receipts.

The principal may terminate this bond by sending written notice to the Surety(ies), provided, however, that no such notice shall become effective until the Surety(ies) receive(s) written authorization for termination of the bond by the EPA Regional Administrator(s) of the EPA Region(s) in which the bonded injection well(s) is (are) located.

[The following paragraph is an optional rider that may be included but is not required.]

Principal and Surety(ies) hereby agree to adjust the penal sum of the bond yearly so that it guarantees a new plugging and abandonment amount, provided that the penal

sum does not increase by more than 20 percent in any one year, and no decrease in the penal sum takes place without the written permission of the EPA Regional Administrator(s).

In Witness Whereof, The Principal and Surety(ies) have executed this Performance Bond and have affixed their seals on the date set forth above.

The persons whose signatures appear below hereby certify that they are authorized to execute this surety bond on behalf of the Principal and Surety(ies) and that the wording on this surety bond is identical to the wording specified in 40 CFR 144.70(c) as such regulation was constituted on the date this bond was executed.

Principal.
[Signature(s)]
[Name(s)]
[Title(s)]
[Corporate seal]
[Corporate Surety(ies)]
[Name and address]
State of incorporation:

Liability limit: $_____.
[Signature(s)]
[Name(s) and title(s)]
Corporate seal:
[For every co-surety, provide signature(s), corporate seal, and other information in the same manner as for Surety above.]
Bond premium: $_____.

(d) A letter of credit, as specified in § 144.63(d) of this chapter, must be worded as follows, except that instructions in brackets are to be replaced with the relevant information and the brackets deleted:

IRREVOCABLE STANDBY LETTER OF CREDIT

Regional Administrator(s)
Region(s) _____
U.S. Environmental Protection Agency.
Dear Sir or Madam:

We hereby establish our Irrevocable Standby Letter of Credit No. _____ in your favor, at the request and for the account of [owner's or operator's name and address] up to the aggregate amount of [in words] U.S. dollars $_____, available upon presentation [insert, if more than one Regional Administrator is a beneficiary, ''by any one of you''] of

(1) Your sight draft, bearing reference to this letter of credit No. _____, and

(2) Your signed statement reading as follows: ''I certify that the amount of the draft is payable pursuant to regulations issued under authority of the Safe Drinking Water Act.''

This letter of credit is effective as of [date] and shall expire on [date at least 1 year later], but such expiration date shall be automatically extended for a period of [at least 1 year] on [date] and on each successive expiration date, unless, at least 120 days before the current expiration date, we notify both you and [owner's or operator's name] by certified mail that we have decided not to extend this letter of credit beyond the current expiration date. In the event you are so notified, any unused portion of the credit shall be available upon presentation of your sight draft for 120 days after the date of receipt by both you and [owner's or operator's name], as shown on the signed return receipts.

Whenever this letter of credit is drawn on under and in compliance with the terms of this credit, we shall duly honor such draft upon presentation to us, and we shall deposit the amount of the draft directly into the standby trust fund of [owner's or operator's name] in accordance with your instructions.

We certify that the wording of this letter of credit is identical to the wording specified in 40 CFR 144.70(d) as such regulations were constituted on the date shown immediately below.

[Signature(s) and title(s) of official(s) of issuing institution]
[Date]

This credit is subject to [insert ''the most recent edition of the Uniform Customs and Practice for Documentary Credits, published and copyrighted by the International Chamber of Commerce,'' or ''the Uniform Commercial Code'']].

(e) A certificate of insurance, as specified in § 144.63(e) of this chapter, must be worded as follows, except that instructions in brackets are to be replaced with the relevant information and the brackets deleted:

Certificate of Insurance for Plugging and Abandonment

Name and Address of Insurer (herein called the ''insurer''):

Name and Address of Insurer (herein called the ''insurer''):

Injection Wells covered: [list for each well: The EPA Identification Number, name, address, and the amount of insurance for plugging and abandonment (these amounts for all injection wells covered must total the face amount shown below).]
Face Amount: _____
Policy Number: _____
Effective Date: _____

The insurer hereby certifies that it has issued to the Insured the policy of insurance identified above to provide financial assurance for plugging and abandonment for the injection wells identified above. The Insurer further warrants that such policy conforms in all respects with the requirements of 40 CFR 144.63(e), as applicable and as such regulations were constituted on the date shown

immediately below. It is agreed that any provision of the policy inconsistent with such regulations is hereby amended to eliminate such inconsistency.

Whenever requested by the EPA Regional Administrator(s) of the U.S. Environmental Protection Agency, the Insurer agrees to furnish to the EPA Regional Administrator(s) a duplicate original of the policy listed above, including all endorsements thereon.

I hereby certify that the wording of this certificate is identical to the wording specified in 40 CFR 144.70(e) as such regulations were constituted on the date shown immediately below.

[Authorized signature of Insurer]
[Name of person signing]
[Title of person signing]
[Signature of witness or notary:]

[Date]

(f) A letter from the chief financial officer, as specified in § 144.63(f) of this chapter, must be worded as follows, except that instructions in brackets are to be replaced with the relevant information and the brackets deleted:

LETTER FROM CHIEF FINANCIAL OFFICER

[Address to Regional Administrator of every Region in which injection wells for which financial responsibility is to be demonstrated through the financial test are located.]

I am the chief financial officer of [name and address of firm.] This letter is in support of this firm's use of the financial test to demonstrate financial assurance, as specified in subpart F of 40 CFR part 144.

[Fill out the following four paragraphs regarding injection wells and associated cost estimates. If your firm has no injection wells that belong in a particular paragraph, write ''None'' in the space indicated. For each injection well, include its EPA Identification Number, name, address, and current plugging and abandonment cost estimate.]

1. This firm is the owner or operator of the following injection wells for which financial assurance for plugging and abandonment is demonstrated through the financial test specified in subpart F of 40 CFR part 144. The current plugging and abandonment cost estimate covered by the test is shown for each injection well: _____.

2. This firm guarantees, through the corporate guarantee specified in subpart F of 40 CFR part 144, the plugging and abandonment of the following injection wells owned or operated by subsidaries of this firm. The current cost estimate for plugging and abandonment so guaranteed is shown for each injection well: _____.

3. In States where EPA is not administering the financial requirements of subpart F of 40 CFR part 144, this firm, as owner or operator or guarantor, is demonstrating financial assurance for the plugging and abandoment of the following injection wells through the use of a test equivalent or substantially equivalent to the financial test specified in subpart F of 40 CFR part 144. The current plugging and abandonment cost estimate covered by such a test is shown for each injection well: _____.

4. This firm is the owner or operator of the following injection wells for which financial assurance for plugging and abandonment is not demonstrated either to EPA or a State through the financial test or any other financial assurance mechanism specified in subpart F of 40 CFR part 144 or equivalent or substantially equivalent State mechanisms. The current plugging and abandonment cost estimate not covered by such financial assurance is shown for each injection well: _____.

This firm [insert ''is required'' or ''is not required''] to file a Form 10K with the Securities and Exchange Commission (SEC) for the latest fiscal year.

The fiscal year of this firm ends on [month, day]. The figures for the following items marked with an asterisk are derived from this firm's independently audited, year-end financial statements for the latest completed fiscal year, ended [date].

[Fill in Alternative I if the criteria of paragraph (f)(1)(i) of § 144.63 of this chapter are used. Fill in Alternative II if the criteria of paragraph (f)(1)(ii) of § 144.63 of this chapter are used.]

ALTERNATIVE I

1. (a) Current plugging and abandonment cost	$_____
(b) Sum of the company's financial responsibilities under 40 CFR Parts 264 and 265, Subpart H, currently met using the financial test or corporate guarantee	.....
(c) Total of lines a and b	.....
*2. Total liabilities [if any portion of the plugging and abandonment cost is included in total liabilities, you may deduct the amount of that portion from this line and add that amount to lines 3 and 4]	.....
*3. Tangible net worth	.....
*4. Net worth	.....
*5. Current assets	.....
*6. Current liabilities	.....
*7. Net working capital [line 5 minus line 6]	.....
*8. The sum of net income plus depreciation, depletion and amortization	.....
*9. Total assets in U.S. (required only if less than 90% of firm's assets are located in U.S.)	.....

	Yes	No
10. Is line 3 at least $10 million?	.....	.....
11. Is line 3 at least 6 times line 1(c)?	.....	.....
12. Is line 7 at least 6 times line 1(c)?	.....	.....
*13. Are at least 90% of firm's assets located in the U.S.? If not, complete line 14.	.....	.....

ALTERNATIVE I—Continued

14. Is line 9 at least 6 times line 1(c)?	.....	.....
15. Is line 2 divided by line 4 less than 2.0?	.....	.....
16. Is line 8 divided by line 2 greater than 0.1?	.....	.....
17. Is line 5 divided by line 6 greater than 1.5?	.....	.....

ALTERNATIVE II

1. (a) Current plugging and abandonment cost	$_____
(b) Sum of the company's financial responsibilities under 40 CFR Parts 264 and 265, Subpart H, currently met using the financial test or corporate guarantee	.....
(c) Total of lines a and b	.....
2. Current bond rating of most recent issuance of this firm and name of rating service	.....
3. Date of issuance of bond	.....
4. Date of maturity of bond	.....
*5. Tangible net worth [if any portion of the plugging and abandonment cost estimate is included in ''total liabilities'' on your firm's financial statements, you may add the amount of that portion to this line]	.....
*6. Total assets in U.S. (required only if less than 90% of firm's assets are located in U.S.)	.....

	Yes	No
7. Is line 5 at least $10 million?	.....	.....
8. Is line 5 at least 6 times line 1(c)?	.....	.....
*9. Are at least 90% of the firm's assets located in the U.S.? If not, complete line 10	.....	.....
10. Is line 6 at least 6 times line 1(c)?	.....	.....

I hereby certify that the wording of this letter is identical to the wording specified in 40 CFR 144.70(f) as such regulations were constituted on the date shown immediately below.

[Signature]
[Name]
[Title]
[Date]

(g) A corporate guarantee as specified in § 144.63(e) must be worded as follows except that instructions in brackets are to be replaced with the relevant information and the bracketed material deleted:

GUARANTEE FOR PLUGGING AND ABANDONMENT

Guarantee made this ___ day of _____, 19___, by [name of guaranteeing entity], a business corporation organized under the laws of the State of _____, herein referred to as guarantor, to the United States Environmental Protection Agency (EPA), obligee, on behalf of our subsidiary [owner or operator] of [business address].

Recitals

1. Guarantor meets or exceeds the financial test criteria and agrees to comply with the reporting requirements for guarantors as specified in 40 CFR 144.63(e).

2. [Owner or operator] owns or operates the following Class I hazardous waste injection well covered by this guarantee: [List for each facility: EPA Identification Number, name, and address. Indicate for each whether guarantee is for closure, post-closure care, or both.]

3. ''Plugging and abandonment plan'' as used below refers to the plans maintained as required by 40 CFR part 144 for the plugging and abandonment of injection wells as identified above.

4. For value received from [owner or operator], guarantor guarantees to EPA that in the event that [owner or operator] fails to perform [''plugging and abandonment''] of the above facility(ies) in accordance with the plugging and abandonment plan and other requirements when required to do so, the guarantor will do so or fund a trust fund as specified in 40 CFR 144.63 in the name of [owner or operator] in the amount of the adjusted plugging and abandonment cost estimates prepared as specified in 40 CFR 144.62.

5. Guarantor agrees that, if at the end of any fiscal year before termination of this guarantee, the guarantor fails to meet the financial test criteria, guarantor will send within 90 days, by certified mail, notice to the EPA Regional Administrator(s) for the Region(s) in which the facility(ies) is (are) located and to [owner or operator] that he intends to provide alternate financial assurance as specified in 40 CFR 144.63 in the name of [owner or operator]. Within 30 days after sending such notice, the guarantor will establish such financial assurance if [owner or operator] has not done so.

6. The guarantor agrees to notify the Regional Administrator, by certified mail, of a voluntary or involuntary case under Title 11, U.S. Code, naming guarantor as debtor, within 10 days after its commencement.

7. Guarantor agrees that within 30 days after being notified by an EPA Regional Administrator of a determination that guarantor no longer meets the financial test criteria or that he is disallowed from continuing as a guarantor of plugging and adandonment, he will establish alternate financial assurance, as specified in 40 CFR 144.63, in the name of [owner or operator] if [owner or operator] has not done so.

8. Guarantor agrees to remain bound under this guarantee notwithstanding any or all of the following: amendment or modification of the plugging and abandonment plan, the extension or reduction of the time of performance of plugging and abandonment or any other modification or alteration of an obligation of [owner or operator] pursuant to 40 CFR part 144.

9. Guarantor agrees to remain bound under this guarantee for so long as [owner or operator] must comply with the applicable financial assurance requirements of 40 CFR part 144 for the above-listed facilities, except that guarantor may cancel this guarantee by sending notice by certified mail, to the EPA Regional Administrator(s) for the Region(s) in which the facility(ies) is (are) located and to [owner or operator], such cancellation to become effective no earlier than 120 days after actual receipt of such notice by both EPA and [owner or operator] as evidenced by the return receipts.

10. Guarantor agrees that if [owner or operator] fails to provide alternate financial assurance and obtain written approval of such assurance from the EPA Regional Administrator(s) within 90 days after a notice of cancellation by the guarantor is received by both the EPA Regional Administrator(s) and [owner or operator], guarantor will provide alternate financial assurance as specified in 40 CFR 144.63 in the name of [owner or operator].

11. Guarantor expressly waives notice of acceptance of this guarantee by the EPA or by [owner or operator]. Guarantor also expressly waives notice of amendments or modifications of the plugging and abandonment plan.

I hereby certify that the wording of this guarantee is identical to the wording specified in 40 CFR 144.70(f).

Effective date: _____.

[Name of guarantor]

[Authorized signature for guarantor]

[Type name of person signing]

[Title of person signing]

Signature of witness or notary: _____

[48 FR 14189, Apr. 1, 1983, as amended at 59 FR 29959, June 10, 1994]

PART 145—STATE UIC PROGRAM REQUIREMENTS

Subpart A—General Program Requirements

Sec.
145.1 Purpose and scope.
145.2 Definitions.

Subpart B—Requirements for State Programs

145.11 Requirements for permitting.
145.12 Requirements for compliance evaluation programs.
145.13 Requirements for enforcement authority.
145.14 Sharing of information.

Subpart C—State Program Submissions

145.21 General requirements for program approvals.
145.22 Elements of a program submission.
145.23 Program description.
145.24 Attorney General's statement.
145.25 Memorandum of Agreement with the Regional Administrator.

Subpart D—Program Approval, Revision and Withdrawal

145.31 Approval process.
145.32 Procedures for revision of State programs.
145.33 Criteria for withdrawal of State programs.
145.34 Procedures for withdrawal of State programs.

Subpart E—Indian Tribes

145.52 Requirements for Tribal eligibility.
145.56 Request by an Indian Tribe for a determination of eligibility.
145.58 Procedure for processing an Indian Tribe's application.

AUTHORITY: 42 U.S.C. 300f *et seq.*

SOURCE: 48 FR 14202, Apr. 1, 1983, unless otherwise noted.

Subpart A—General Program Requirements

§ 145.1 Purpose and scope.

(a) This part specifies the procedures EPA will follow in approving, revising, and withdrawing State programs under section 1422 (underground injection control—UIC) of SDWA, and includes the elements which must be part of submissions to EPA for program approval and the substantive provisions which must be present in State programs for them to be approved.

(b) State submissions for program approval must be made in accordance with the procedures set out in subpart C. This includes developing and submitting to EPA a program description (§ 145.23), an Attorney General's Statement (§ 145.24), and a Memorandum of Agreement with the Regional Administrator (§ 145.25).

(c) The substantive provisions which must be included in State programs to obtain approval include requirements for permitting, compliance evaluation, enforcement, public participation, and sharing of information. The requirements are found in subpart B. Many of

the requirements for State programs are made applicable to States by cross-referencing other EPA regulations. In particular, many of the provisions of parts 144 and 124 are made applicable to States by the references contained in §145.11.

(d) Upon submission of a complete program, EPA will conduct a public hearing, if interest is shown, and determine whether to approve or disapprove the program taking into consideration the requirements of this part, the Safe Drinking Water Act and any comments received.

(e) Upon approval of a State program, the Administrator shall suspend the issuance of Federal permits for those activities subject to the approved State program.

(f) Any State program approved by the Administrator shall at all times be conducted in accordance with the requirements of this part.

(g) Nothing in this part precludes a State from:

(1) Adopting or enforcing requirements which are more stringent or more extensive than those required under this part;

(2) Operating a program with a greater scope of coverage than that required under this part. Where an approved State program has a greater scope of coverage than required by Federal law the additional coverage is not part of the federally approved program.

(h) Section 1451 of the SDWA authorizes the Administrator to delegate primary enforcement responsibility for the Underground Injection Control Program to eligible Indian Tribes. An Indian Tribe must establish its eligibility to be treated as a State before it is eligible to apply for Underground Injection Control grants and primary enforcement responsibility. All requirements of parts 124, 144, 145, and 146 that apply to States with UIC primary enforcement responsibility also apply to Indian Tribes except where specifically noted.

[48 FR 14202, Apr. 1, 1983, as amended at 53 FR 37412, Sept. 26, 1988; 59 FR 64345, Dec. 14, 1994]

§145.2 Definitions.

The definitions of part 144 apply to all subparts of this part.

Subpart B—Requirements for State Programs

§145.11 Requirements for permitting.

(a) All State programs under this part must have legal authority to implement each of the following provisions and must be administered in conformance with each; except that States are not precluded from omitting or modifying any provisions to impose more stringent requirements.

(1) Section 144.5(b)—(Confidential information);

(2) Section 144.6—(Classification of injection wells);

(3) Section 144.7—(Identification of underground sources of drinking water and exempted aquifers);

(4) Section 144.8—(Noncompliance reporting);

(5) Section 144.11—(Prohibition of unauthorized injection);

(6) Section 144.12—(Prohibition of movement of fluids into underground sources of drinking water);

(7) Section 144.13—(Elimination of Class IV wells);

(8) Section 144.14—(Requirements for wells managing hazardous waste);

(9) Sections 144.21–144.26—(Authorization by rule);

(10) Section 144.31—(Application for a permit);

(11) Section 144.32—(Signatories);

(12) Section 144.33—(Area Permits);

(13) Section 144.34—(Emergency permits);

(14) Section 144.35—(Effect of permit);

(15) Section 144.36—(Duration);

(16) Section 144.38—(Permit transfer);

(17) Section 144.39—(Permit modification);

(18) Section 144.40—(Permit termination);

(19) Section 144.51—(Applicable permit conditions);

(20) Section 144.52—(Establishing permit conditions);

(21) Section 144.53(a)—(Schedule of compliance);

(22) Section 144.54—(Monitoring requirements);

(23) Section 144.55—(Corrective Action);

(24) Section 124.3(a)—(Application for a permit);

(25) Section 124.5 (a), (c), (d), and (f)—(Modification of permits);

(26) Section 124.6 (a), (c), (d), and (e)—(Draft Permit);

(27) Section 124.8—(Fact sheets);

(28) Section 124.10 (a)(1)(ii), (a)(1)(iii), (a)(1)(v), (b), (c), (d), and (e)—(Public notice);

(29) Section 124.11—(Public comments and requests for hearings);

(30) Section 124.12(a)—(Public hearings); and

(31) Section 124.17 (a) and (c)—(Response to comments).

(b)(1) States need not implement provisions identical to the provisions listed in paragraphs (a) (1) through (31) of this section. Implemented provisions must, however, establish requirements at least as stringent as the corresponding listed provisions. While States may impose more stringent requirements, they may not make one requirement more lenient as a tradeoff for making another requirement more stringent; for example, by requiring that public hearings be held prior to issuing any permit while reducing the amount of advance notice of such a hearing.

(2) State programs may, if they have adequate legal authority, implement any of the provisions of parts 144 and 124. See, for example § 144.37(d) (continuation of permits) and § 124.4 (consolidation of permit processing).

§ 145.12 Requirements for compliance evaluation programs.

(a) State programs shall have procedures for receipt, evaluation, retention and investigation for possible enforcement of all notices and reports required of permittees and other regulated persons (and for investigation for possible enforcement of failure to submit these notices and reports).

(b) State programs shall have inspection and surveillance procedures to determine, independent of information supplied by regulated persons, compliance or noncompliance with applicable program requirements. The State shall maintain:

(1) A program which is capable of making comprehensive surveys of all facilities and activities subject to the State Director's authority to identify persons subject to regulation who have failed to comply with permit application or other program requirements. Any compilation, index, or inventory of such facilities and activities shall be made available to the Regional Administrator upon request;

(2) A program for periodic inspections of the facilities and activities subject to regulation. These inspections shall be conducted in a manner designed to:

(i) Determine compliance or noncompliance with issued permit conditions and other program requirements;

(ii) Verify the accuracy of information submitted by permittees and other regulated persons in reporting forms and other forms supplying monitoring data; and

(iii) Verify the adequacy of sampling, monitoring, and other methods used by permittees and other regulated persons to develop that information;

(3) A program for investigating information obtained regarding violations of applicable program and permit requirements; and

(4) Procedures for receiving and ensuring proper consideration of information submitted by the public about violations. Public effort in reporting violations shall be encouraged and the State Director shall make available information on reporting procedures.

(c) The State Director and State officers engaged in compliance evaluation shall have authority to enter any site or premises subject to regulation or in which records relevant to program operation are kept in order to copy any records, inspect, monitor or otherwise investigate compliance with permit conditions and other program requirements. States whose law requires a search warrant before entry conform with this requirement.

(d) Investigatory inspections shall be conducted, samples shall be taken and other information shall be gathered in a manner [e.g., using proper ''chain of custody'' procedures] that will produce evidence admissible in an enforcement proceeding or in court.

§ 145.13 Requirements for enforcement authority.

(a) Any State agency administering a program shall have available the following remedies for violations of State program requirements:

(1) To restrain immediately and effectively any person by order or by suit in State court from engaging in any

unauthorized activity which is endangering or causing damage to public health or environment;

NOTE: This paragraph requires that States have a mechanism (e.g., an administrative cease and desist order or the ability to seek a temporary restraining order) to stop any unauthorized activity endangering public health or the environment.

(2) To sue in courts of competent jurisdiction to enjoin any threatened or continuing violation of any program requirement, including permit conditions, without the necessity of a prior revocation of the permit;

(3) To assess or sue to recover in court civil penalties and to seek criminal remedies, including fines, as follows:

(i) For all wells except Class II wells, civil penalties shall be recoverable for any program violation in at least the amount of $2,500 per day. For Class II wells, civil penalties shall be recoverable for any program violation in at least the amount of $1,000 per day.

(ii) Criminal fines shall be recoverable in at least the amount of $5,000 per day against any person who willfully violates any program requirement, or for Class II wells, pipeline (production) severance shall be imposable against any person who willfully violates any program requirement.

NOTE: In many States the State Director will be represented in State courts by the State Attorney General or other appropriate legal officer. Although the State Director need not appear in court actions he or she should have power to request that any of the above actions be brought.

(b)(1) The maximum civil penalty or criminal fine (as provided in paragraph (a)(3) of this section) shall be assessable for each instance of violation and, if the violation is continuous, shall be assessable up to the maximum amount for each day of violation.

(2) The burden of proof and degree of knowledge or intent required under State law for establishing violations under paragraph (a)(3) of this section, shall be no greater than the burden of proof or degree of knowledge or intent EPA must provide when it brings an action under the Safe Drinking Water Act.

NOTE: For example, this requirement is not met if State law includes mental state as an element of proof for civil violations.

(c) A civil penalty assessed, sought, or agreed upon by the State Director under paragraph (a)(3) of this section shall be appropriate to the violation.

NOTE: To the extent that State judgments or settlements provide penalties in amounts which EPA believes to be substantially inadequate in comparison to the amounts which EPA would require under similar facts, EPA, when authorized by the applicable statute, may commence separate actions for penalties.

In addition to the requirements of this paragraph, the State may have other enforcement remedies. The following enforcement options, while not mandatory, are highly recommended:

Procedures for assessment by the State of the costs of investigations, inspections, or monitoring surveys which lead to the establishment of violations;

Procedures which enable the State to assess or to sue any persons responsible for unauthorized activities for any expenses incurred by the State in removing, correcting, or terminating any adverse effects upon human health and the environment resulting from the unauthorized activity, or both; and

Procedures for the administrative assessment of penalties by the Director.

(d) Any State administering a program shall provide for public participation in the State enforcement process by providing either:

(1) Authority which allows intervention as of right in any civil or administrative action to obtain remedies specified in paragraph (a) (1), (2) or (3) of this section by any citizen having an interest which is or may be adversely affected; or

(2) Assurance that the State agency or enforcement authority will:

(i) Investigate and provide written responses to all citizen complaints submitted pursuant to the procedures specified in § 145.12(b)(4);

(ii) Not oppose intervention by any citizen when permissive intervention may be authorized by statute, rule, or regulation; and

(iii) Publish notice of and provide at least 30 days for public comment on any proposed settlement of a State enforcement action.

(e) To the extent that an Indian Tribe does not assert or is precluded from asserting criminal enforcement

authority the Administrator will assume primary enforcement responsibility for criminal violations. The Memorandum of Agreement in § 145.25 shall reflect a system where the Tribal agency will refer such violations to the Administrator in an appropriate and timely manner.

(Clean Water Act (33 U.S.C. 1251 *et seq.*), Safe Drinking Water Act (42 U.S.C. 300f *et seq.*), Clean Air Act (42 U.S.C. 7401 *et seq.*), Resource Conservation and Recovery Act (42 U.S.C. 6901 *et seq.*))

[48 FR 14202, Apr. 1, 1983, as amended at 48 FR 39621, Sept. 1, 1983; 53 FR 37412, Sept. 26, 1988]

§ 145.14 Sharing of information.

(a) Any information obtained or used in the administration of a State program shall be available to EPA upon request without restriction. If the information has been submitted to the State under a claim of confidentiality, the State must submit that claim to EPA when providing information under this section. Any information obtained from a State and subject to a claim of confidentiality will be treated in accordance with the regulations in 40 CFR part 2. If EPA obtains from a State information that is not claimed to be confidential, EPA may make that information available to the public without further notice.

(b) EPA shall furnish to States with approved programs the information in its files not submitted under a claim of confidentiality which the State needs to implement its approved program. EPA shall furnish to States with approved programs information submitted to EPA under a claim of confidentiality, which the State needs to implement its approved program, subject to the conditions in 40 CFR part 2.

Subpart C—State Program Submissions

§ 145.21 General requirements for program approvals.

(a) States shall submit to the Administrator a proposed State UIC program complying with § 145.22 of this part within 270 days of the date of promulgation of the UIC regulations on June 24, 1980. The administrator may, for good cause, extend the date for submission of a proposed State UIC program for up to an additional 270 days.

(b) States shall submit to the Administrator 6 months after the date of promulgation of the UIC regulations a report describing the State's progress in developing a UIC program. If the Administrator extends the time for submission of a UIC program an additional 270 days, pursuant to § 145.21(a), the State shall submit a second report six months after the first report is due. The Administrator may prescribe the manner and form of the report.

(c) The requirements of § 145.21 (a) and (b) shall not apply to Indian Tribes.

(d) EPA will establish a UIC program in any State which does not comply with paragraph (a) of this section. EPA will continue to operate a UIC program in such a State until the State receives approval of a UIC program in accordance with the requirements of this part.

NOTE: States which are authorized to administer the NPDES permit program under section 402 of CWA are encouraged to rely on existing statutory authority, to the extent possible, in developing a State UIC program. Section 402(b)(1)(D) of CWA requires that NPDES States have the authority ''to issue permits which control the disposal of pollutants into wells.'' In many instances, therefore, NPDES States will have existing statutory authority to regulate well disposal which satisfies the requirements of the UIC program. Note, however, that CWA excludes certain types of well injections from the definition of ''pollutant.'' If the State's statutory authority contains a similar exclusion it may need to be modified to qualify for UIC program approval.

(e) If a State can demonstrate to EPA's satisfaction that there are no underground injections within the State for one or more classes of injection wells (other than Class IV wells) subject to SDWA and that such injections cannot legally occur in the State until the State has developed an approved program for those classes of injections, the State need not submit a program to regulate those injections and a partial program may be approved. The demonstration of legal prohibition shall be made by either explicitly banning new injections of the class not covered by the State program or providing a certification from the

State Attorney General that such new injections cannot legally occur until the State has developed an approved program for that class. The State shall submit a program to regulate both those classes of injections for which a demonstration is not made and class IV wells.

(f) When a State UIC program is fully approved by EPA to regulate all classes of injections, the State assumes primary enforcement authority under section 1422(b)(3) of SDWA. EPA retains primary enforcement responsibility whenever the State program is disapproved in whole or in part. States which have partially approved programs have authority to enforce any violation of the approved portion of their program. EPA retains authority to enforce violations of State underground injection control programs, except that, when a State has a fully approved program, EPA will not take enforcement actions without providing prior notice to the State and otherwise complying with section 1423 of SDWA.

(g) A State can assume primary enforcement responsibility for the UIC program, notwithstanding §145.21(3), when the State program is unable to regulate activities on Indian lands within the State. EPA will administer the program on Indian lands if the State does not seek this authority.

[48 FR 14202, Apr. 1, 1983, as amended at 53 FR 37412, Sept. 26, 1988]

§145.22 Elements of a program submission.

(a) Any State that seeks to administer a program under this part shall submit to the Administrator at least three copies of a program submission. The submission shall contain the following:

(1) A letter from the Governor of the State requesting program approval;

(2) A complete program description, as required by §145.23, describing how the State intends to carry out its responsibilities under this part;

(3) An Attorney General's statement as required by §145.24;

(4) A Memorandum of Agreement with the Regional Administrator as required by §145.25;

(5) Copies of all applicable State statutes and regulations, including those governing State administrative procedures;

(6) The showing required by §145.31(b) of the State's public participation activities prior to program submission.

(b) Within 30 days of receipt by EPA of a State program submission, EPA will notify the State whether its submission is complete. If EPA finds that a State's submission is complete, the statutory review period (i.e., the period of time allotted for formal EPA review of a proposed State program under the Safe Drinking Water Act) shall be deemed to have begun on the date of receipt of the State's submission. If EPA finds that a State's submission is incomplete, the statutory review period shall not begin until all the necessary information is received by EPA.

(c) If the State's submission is materially changed during the statutory review period, the statutory review period shall begin again upon receipt of the revised submission.

(d) The State and EPA may extend the statutory review period by agreement.

§145.23 Program description.

Any State that seeks to administer a program under this part shall submit a description of the program it proposes to administer in lieu of the Federal program under State law or under an interstate compact. The program description shall include:

(a) A description in narrative form of the scope, structure, coverage and processes of the State program.

(b) A description (including organization charts) of the organization and structure of the State agency or agencies which will have responsibility for administering the program, including the information listed below. If more than one agency is responsible for administration of a program, each agency must have statewide jurisdiction over a class of activities. The responsibilities of each agency must be delineated, their procedures for coordination set forth, and an agency may be designated as a ''lead agency'' to facilitate communications between EPA and the

State agencies having program responsibility. When the State proposes to administer a program of greater scope of coverage than is required by Federal law, the information provided under this paragraph shall indicate the resources dedicated to administering the Federally required portion of the program.

(1) A description of the State agency staff who will carry out the State program, including the number, occupations, and general duties of the employees. The State need not submit complete job descriptions for every employee carrying out the State program.

(2) An itemization of the estimated costs of establishing and administering the program for the first two years after approval, including cost of the personnel listed in paragraph (b)(1) of this section, cost of administrative support, and cost of technical support.

(3) An itemization of the sources and amounts of funding, including an estimate of Federal grant money, available to the State Director for the first two years after approval to meet the costs listed in paragraph (b)(2) of this section, identifying any restrictions or limitations upon this funding.

(c) A description of applicable State procedures, including permitting procedures and any State administrative or judicial review procedures.

(d) Copies of the permit form(s), application form(s), reporting form(s), and manifest format the State intends to employ in its program. Forms used by States need not be identical to the forms used by EPA but should require the same basic information. The State need not provide copies of uniform national forms it intends to use but should note its intention to use such forms.

NOTE: States are encouraged to use uniform national forms established by the Administrator. If uniform national forms are used, they may be modified to include the State Agency's name, address, logo, and other similar information, as appropriate, in place of EPA's.

(e) A complete description of the State's compliance tracking and enforcement program.

(f) A State UIC program description shall also include:

(1) A schedule for issuing permits within five years after program approval to all injection wells within the State which are required to have permits under this part and part 144;

(2) The priorities (according to criteria set forth in 40 CFR 146.09) for issuing permits, including the number of permits in each class of injection well which will be issued each year during the first five years of program operation;

(3) A description of how the Director will implement the mechanical integrity testing requirements of 40 CFR 146.08, including the frequency of testing that will be required and the number of tests that will be reviewed by the Director each year;

(4) A description of the procedure whereby the Director will notify owners and operators of injection wells of the requirement that they apply for and obtain a permit. The notification required by this paragraph shall require applications to be filed as soon as possible, but not later than four years after program approval for all injection wells requiring a permit;

(5) A description of any rule under which the Director proposes to authorize injections, including the text of the rule;

(6) For any existing enhanced recovery and hydrocarbon storage wells which the Director proposes to authorize by rule, a description of the procedure for reviewing the wells for compliance with applicable monitoring, reporting, construction, and financial responsibility requirements of §§ 144.51 and 144.52, and 40 CFR part 146;

(7) A description of and schedule for the State's program to establish and maintain a current inventory of injection wells which must be permitted under State law;

(8) Where the Director had designated underground sources of drinking water in accordance with § 144.7(a), a description and identification of all such designated sources in the State;

(9) A description of aquifers, or parts thereof, which the Director has identified under § 144.7(b) as exempted aquifers, and a summary of supporting data;

(10) A description of and schedule for the State's program to ban Class IV wells prohibited under §144.13; and

(11) A description of and schedule for the State's program to establish an inventory of Class V wells and to assess the need for a program to regulate Class V wells.

§145.24 Attorney General's statement.

(a) Any State that seeks to administer a program under this part shall submit a statement from the State Attorney General (or the attorney for those State or interstate agencies which have independent legal counsel) that the laws of the State, or an interstate compact, provide adequate authority to carry out the program described under §145.23 and to meet the requirements of this part. This statement shall include citations to the specific statutes, administrative regulations, and, where appropriate, judicial decisions which demonstrate adequate authority. State statutes and regulations cited by the State Attorney General or independent legal counsel shall be in the form of lawfully adopted State statutes and regulations at the time the statement is signed and shall be fully effective by the time the program is approved. To qualify as ''independent legal counsel'' the attorney signing the statement required by this section must have full authority to independently represent the State agency in court on all matters pertaining to the State program.

NOTE: EPA will supply States with an Attorney General's statement format on request.

(b) When a State seeks authority over activities on Indian lands, the statement shall contain an appropriate analysis of the State's authority.

§145.25 Memorandum of Agreement with the Regional Administrator.

(a) Any State that seeks to administer a program under this part shall submit a Memorandum of Agreement. The Memorandum of Agreement shall be executed by the State Director and the Regional Administrator and shall become effective when approved by the Administrator. In addition to meeting the requirements of paragraph (b) of this section, the Memorandum of Agreement may include other terms, conditions, or agreements consistent with this part and relevant to the administration and enforcement of the State's regulatory program. The Administrator shall not approve any Memorandum of Agreement which contains provisions which restrict EPA's statutory oversight responsibility.

(b) The Memorandum of Agreement shall include the following:

(1) Provisions for the prompt transfer from EPA to the State of pending permit applications and any other information relevant to program operation not already in the possession of the State Director (e.g., support files for permit issuance, compliance reports, etc.). When existing permits are transferred from EPA to State for administration, the Memorandum of Agreement shall contain provisions specifying a procedure for transferring the administration of these permits. If a State lacks the authority to directly administer permits issued by the Federal government, a procedure may be established to transfer responsibility for these permits.

NOTE: For example, EPA and the State and the permittee could agree that the State would issue a permit(s) identical to the outstanding Federal permit which would simultaneously be terminated.

(2) Provisions specifying classes and categories of permit applications, draft permits, and proposed permits that the State will send to the Regional Administrator for review, comment and, where applicable, objection.

(3) Provisions specifying the frequency and content of reports, documents and other information which the State is required to submit to EPA. The State shall allow EPA to routinely review State records, reports, and files relevant to the administration and enforcement of the approved program. State reports may be combined with grant reports where appropriate.

(4) Provisions on the State's compliance monitoring and enforcement program, including:

(i) Provisions for coordination of compliance monitoring activities by the State and by EPA. These may specify the basis on which the Regional Administrator will select facilities or

activities within the State for EPA inspection. The Regional Administrator will normally notify the State at least 7 days before any such inspection; and

(ii) Procedures to assure coordination of enforcement activities.

(5) When appropriate, provisions for joint processing of permits by the State and EPA, for facilities or activities which require permits from both EPA and the State under different programs. See § 124.4.

(6) Provisions for modification of the Memorandum of Agreement in accordance with this part.

(c) The Memorandum of Agreement, the annual program and grant and the State/EPA Agreement should be consistent. If the State/EPA Agreement indicates that a change is needed in the Memorandum of Agreement, the Memorandum of Agreement may be amended through the procedures set forth in this part. The State/EPA Agreement may not override the Memorandum of Agreement.

NOTE: Detailed program priorities and specific arrangements for EPA support of the State program will change and are therefore more appropriately negotiated in the context of annual agreements rather than in the MOA. However, it may still be appropriate to specify in the MOA the basis for such detailed agreements, e.g., a provision in the MOA specifying that EPA will select facilities in the State for inspection annually as part of the State/EPA agreement.

Subpart D—Program Approval, Revision and Withdrawal

§ 145.31 Approval process.

(a) Prior to submitting an application to the Administrator for approval of a State UIC program, the State shall issue public notice of its intent to adopt a UIC program and to seek program approval from EPA. This public notice shall:

(1) Be circulated in a manner calculated to attract the attention of interested persons. Circulation of the public notice shall include publication in enough of the largest newspapers in the State to attract Statewide attention and mailing to persons on appropriate State mailing lists and to any other persons whom the agency has reason to believe are interested;

(2) Indicate when and where the State's proposed program submission may be reviewed by the public;

(3) Indicate the cost of obtaining a copy of the submission;

(4) Provide for a comment period of not less than 30 days during which interested persons may comment on the proposed UIC program;

(5) Schedule a public hearing on the State program for no less than 30 days after notice of the hearing is published;

(6) Briefly outline the fundamental aspects of the State UIC program; and

(7) Identify a person that an interested member of the public may contact for further information.

(b) After complying with the requirements of paragraph (a) of this section any State may submit a proposed UIC program under section 1422 of SDWA and § 145.22 of this part to EPA for approval. Such a submission shall include a showing of compliance with paragraph (a) of this section; copies of all written comments received by the State; a transcript, recording or summary of any public hearing which was held by the State; and a responsiveness summary which identifies the public participation activities conducted, describes the matters presented to the public, summarizes significant comments received, and responds to these comments. A copy of the responsiveness summary shall be sent to those who testified at the hearing, and others upon request.

(c) After determining that a State's submission for UIC program approval is complete the Administrator shall issue public notice of the submission in the FEDERAL REGISTER and in accordance with paragraph (a)(1) of this section. Such notice shall:

(1) Indicate that a public hearing will be held by EPA no earlier than 30 days after notice of the hearing. The notice may require persons wishing to present testimony to file a request with the Regional Administrator, who may cancel the public hearing if sufficient public interest in a hearing is not expressed;

(2) Afford the public 30 days after the notice to comment on the State's submission; and

(3) Note the availability of the State submission for inspection and copying by the public.

(d) The Administrator shall approve State programs which conform to the applicable requirements of this part.

(e) Within 90 days of the receipt of a complete submission (as provided in §145.22) or material amendment thereto, the Administrator shall by rule either fully approve, disapprove, or approve in part the State's UIC program taking into account any comments submitted. The Administrator shall give notice of this rule in the FEDERAL REGISTER and in accordance with paragraph (a)(1) of this section. If the Administrator determines not to approve the State program or to approve it only in part, the notice shall include a concise statement of the reasons for this determination. A responsiveness summary shall be prepared by the Regional Office which identifies the public participation activities conducted, describes the matters presented to the public, summarizes significant comments received, and explains the Agency's response to these comments. The responsiveness summary shall be sent to those who testified at the public hearing, and to others upon request.

§145.32 Procedures for revision of State programs.

(a) Either EPA or the approved State may initiate program revision. Program revision may be necessary when the controlling Federal or State statutory or regulatory authority is modified or supplemented. The state shall keep EPA fully informed of any proposed modifications to its basic statutory or regulatory authority, its forms, procedures, or priorities.

(b) Revision of a State program shall be accomplished as follows:

(1) The State shall submit a modified program description, Attorney General's statement, Memorandum of Agreement, or such other documents as EPA determines to be necessary under the circumstances.

(2) Whenever EPA determines that the proposed program revision is substantial, EPA shall issue public notice and provide an opportunity to comment for a period of at least 30 days. The public notice shall be mailed to interested persons and shall be published in the FEDERAL REGISTER and in enough of the largest newspapers in the State to provide Statewide coverage. The public notice shall summarize the proposed revisions and provide for the opportunity to request a public hearing. Such a hearing will be held is there if significant public interest based on requests received.

(3) The Administrator shall approve or disapprove program revisions based on the requirements of this part and of the Safe Drinking Water Act.

(4) A program revision shall become effective upon the approval of the Administrator. Notice of approval of any substantial revision shall be published in the FEDERAL REGISTER. Notice of approval of non-substantial program revisions may be given by a letter from the Administrator to the State Governor or his designee.

(c) States with approved programs shall notify EPA whenever they propose to transfer all or part of any program from the approved State agency to any other State agency, and shall identify any new division of responsibilities among the agencies involved. The new agency is not authorized to administer the program until approval by the Administrator under paragraph (b) of this section. Organizational charts required under §145.23(b) shall be revised and resubmitted.

(d) Whenever the Administrator has reason to believe that circumstances have changed with respect to a State program, he may request, and the State shall provide, a supplemental Attorney General's statement, program description, or such other documents or information as are necessary.

(e) The State shall submit the information required under paragraph (b)(1) of this section within 270 days of any amendment to this part or 40 CFR part 144, 146, or 124 which revises or adds any requirement respecting an approved UIC program.

§145.33 Criteria for withdrawal of State programs.

(a) The Administrator may withdraw program approval when a State program no longer complies with the requirements of this part, and the State

fails to take corrective action. Such circumstances include the following:

(1) When the State's legal authority no longer meets their requirements of this part, including:

(i) Failure of the State to promulgate or enact new authorities when necessary; or

(ii) Action by a State legislature or court striking down or limiting State authorities.

(2) When the operation of the State program fails to comply with the requirements of this part, including:

(i) Failure to exercise control over activities required to be regulated under this part, including failure to issue permits;

(ii) Repeated issuance of permits which do not conform to the requirements of this part; or

(iii) Failure to comply with the public participation requirements of this part.

(3) When the State's enforcement program fails to comply with the requirements of this part, including:

(i) Failure to act on violations of permits or other program requirements;

(ii) Failure to seek adequate enforcement penalties or to collect administrative fines when imposed; or

(iii) Failure to inspect and monitor activities subject to regulation.

(4) When the State program fails to comply with the terms of the Memorandum of Agreement required under § 145.24.

§ 145.34 Procedures for withdrawal of State programs.

(a) A State with a program approved under this part may voluntarily transfer program responsibilities required by Federal law to EPA by taking the following actions, or in such other manner as may be agreed upon with the Administrator.

(1) The State shall give the Administrator 180 days notice of the proposed transfer and shall submit a plan for the orderly transfer of all relevant program information not in the possession of EPA (such as permits, permit files, compliance files, reports, permit applications) which are necessary for EPA to administer the program.

(2) Within 60 days of receiving the notice and transfer plan, the Administrator shall evaluate the State's transfer plan and shall identify any additional information needed by the Federal government for program administration and/or identify any other deficiencies in the plan.

(3) At least 30 days before the transfer is to occur the Administrator shall publish notice of the transfer in the FEDERAL REGISTER and in enough of the largest newspapers in the State to provide Statewide coverage, and shall mail notice to all permit holders, permit applicants, other regulated persons and other interested persons on appropriate EPA and State mailing lists.

(b) Approval of a State UIC program may be withdrawn and a Federal program established in its place when the Administrator determines, after holding a public hearing, that the State program is not in compliance with the requirements of SDWA and this part.

(1) *Notice to State of public hearing.* If the Administrator has cause to believe that a State is not administering or enforcing its authorized program in compliance with the requirements of SDWA and this part, he or she shall inform the State by registered mail of the specific areas of alleged noncompliance. If the State demonstrates to the Administrator within 30 days of such notification that the State program is in compliance, the Administrator shall take no further action toward withdrawal and shall so notify the State by registered mail.

(2) *Public hearing.* If the State has not demonstrated its compliance to the satisfaction of the Administrator within 30 days after notification, the Administrator shall inform the State Director and schedule a public hearing to discuss withdrawal of the State program. Notice of such public hearing shall be published in the FEDERAL REGISTER and in enough of the largest newspapers in the State to attract statewide attention, and mailed to persons on appropriate State and EPA mailing lists. This hearing shall be convened not less than 60 days nor more than 75 days following the publication of the notice of the hearing. Notice of the hearing shall identify the

Administrator's concerns. All interested persons shall be given opportunity to make written or oral presentation on the State's program at the public hearing.

(3) *Notice to State of findings.* When the Administrator finds after the public hearing that the State is not in compliance, he or she shall notify the State by registered mail of the specific deficiencies in the State program and of necessary remedial actions. Within 90 days of receipt of the above letter, the State shall either carry out the required remedial action or the Administrator shall withdraw program approval. If the State carries out the remedial action or, as a result of the hearing is found to be in compliance, the Administrator shall so notify the State by registered mail and conclude the withdrawal proceedings.

Subpart E—Indian Tribes

SOURCE: 53 FR 37412, Sept. 26, 1988, unless otherwise noted.

§ 145.52 Requirements for Tribal eligibility.

The Administrator is authorized to treat an Indian Tribe as eligible to apply for primary enforcement responsibility for the Underground Injection Control Program if it meets the following criteria:

(a) The Indian Tribe is recognized by the Secretary of the Interior.

(b) The Indian Tribe has a Tribal governing body which is currently ''carrying out substantial governmental duties and powers'' over a defined area, (*i.e.,* is currently performing governmental functions to promote the health, safety, and welfare of the affected population within a defined geographic area).

(c) The Indian Tribe demonstrates that the functions to be performed in regulating the underground injection wells that the applicant intends to regulate are within the area of the Indian Tribal government's jurisdiction.

(d) The Indian Tribe is reasonably expected to be capable, in the Administrator's judgment, of administering (in a manner consistent with the terms and purposes of the Act and all applicable regulations) an effective Underground Injection Control Program.

[53 FR 37412, Sept. 26, 1988, as amended at 59 FR 64345, Dec. 14, 1994]

§ 145.56 Request by an Indian Tribe for a determination of eligibility.

An Indian Tribe may apply to the Administrator for a determination that it meets the criteria of section 1451 of the Act. The application shall be concise and describe how the Indian Tribe will meet each of the requirements of § 145.52. The application shall consist of the following:

(a) A statement that the Tribe is recognized by the Secretary of the Interior.

(b) A descriptive statement demonstrating that the Tribal governing body is currently carrying out substantial governmental duties and powers over a defined area. The statement should:

(1) Describe the form of the Tribal government;

(2) Describe the types of governmental functions currently performed by the Tribal governing body such as, but not limited to, the exercise of police powers affecting (or relating to) the health, safety, and welfare of the affected population; taxation; and the exercise of the power of eminent domain; and

(3) Identify the sources of the Tribal government's authority to carry out the governmental functions currently being performed.

(c) A map or legal description of the area over which the Indian Tribe asserts jurisdiction; a statement by the Tribal Attorney General (or equivalent official) which describes the basis for the Tribe's jurisdictional assertion (including the nature or subject matter of the asserted jurisdiction); a copy of those documents such as Tribal constitutions, by-laws, charters, executive orders, codes, ordinances, and/or resolutions which the Tribe believes are relevant to its assertions regarding jurisdiction; and a description of the locations of the underground injection wells the Tribe proposes to regulate.

(d) A narrative statement describing the capability of the Indian Tribe to administer an effective Underground

Injection Control program which should include:

(1) A description of the Indian Tribe's previous management experience which may include, the administration of programs and services authorized under the Indian Self-Determination and Education Assistance Act (25 U.S.C. 450 *et seq.*), the Indian Mineral Development Act (25 U.S.C. 2101 *et seq.*), or the Indian Sanitation Facilities Construction Activity Act (42 U.S.C. 2004a).

(2) A list of existing environmental or public health programs administered by the Tribal governing body and a copy of related Tribal laws, regulations and policies.

(3) A description of the Indian Tribe's accounting and procurement systems.

(4) A description of the entity (or entities) which exercise the executive, legislative, and judicial functions of the Tribal government.

(5) A description of the existing, or proposed, agency of the Indian Tribe which will assume primary enforcement responsibility, including a description of the relationship between owners/operators of the underground injection wells and the agency.

(6) A description of the technical and administrative capabilities of the staff to administer and manage an effective Underground Injection Control Program or a plan which proposes how the Tribe will acquire additional administrative and/or technical expertise. The plan must address how the Tribe will obtain the funds to acquire the additional administrative and technical expertise.

(e) The Adminstrator may, in his discretion, request further documentation necessary to support a Tribe's eligibility.

(f) If the Administrator has previously determined that a Tribe has met the prerequisites that make it eligible to assume a role similar to that of a State as provided by statute under the Safe Drinking Water Act, the Clean Water Act, or the Clean Air Act, then that Tribe need provide only that information unique to the Underground Injection Control program (§ 145.76(c) and (d)(6)).

[53 FR 37412, Sept. 26, 1988, as amended at 59 FR 64345, Dec. 14, 1994]

§ 145.58 Procedure for processing an Indian Tribe's application.

(a) The Administrator shall process a completed application of an Indian Tribe in a timely manner. He shall promptly notify the Indian Tribe of receipt of the application.

(b) A tribe that meets the requirements of § 145.52 is eligible to apply for development grants and primary enforcement responsibility for an Underground Injection Control program and the associated funding under section 1443(b) of the Act and primary enforcement responsibility for the Underground Injection Control Program under sections 1422 and/or 1425 of the Act.

[53 FR 37412, Sept. 26, 1988, as amended at 59 FR 64345, Dec. 14, 1994]

PART 146—UNDERGROUND INJECTION CONTROL PROGRAM: CRITERIA AND STANDARDS

Subpart A—General Provisions

Sec.
146.1 Applicability and scope.
146.2 Law authorizing these regulations.
146.3 Definitions.
146.4 Criteria for exempted aquifers.
146.5 Classification of injection wells.
146.6 Area of review.
146.7 Corrective action.
146.8 Mechanical integrity.
146.9 Criteria for establishing permitting priorities.
146.10 Plugging and abandoning Class I–III wells.

Subpart B—Criteria and Standards Applicable to Class I Wells

146.11 Criteria and standards applicable to Class I nonhazardous wells.
146.12 Construction requirements.
146.13 Operating, monitoring and reporting requirements.
146.14 Information to be considered by the Director.

Subpart C—Criteria and Standards Applicable to Class II Wells

146.21 Applicability.
146.22 Construction requirements.
146.23 Operating, monitoring, and reporting requirements.
146.24 Information to be considered by the Director.

Subpart D—Criteria and Standards Applicable to Class III Wells

146.31 Applicability.
146.32 Construction requirements.
146.33 Operating, monitoring and reporting requirements.
146.34 Information to be considered by the Director.

Subpart E—Criteria and Standards Applicable to Class IV Injection Wells [Reserved]

Subpart F—Criteria and Standards Applicable to Class V Injection Wells

146.51 Applicability.

Subpart G—Criteria and Standards Applicable to Class I Hazardous Waste Injection Wells

146.61 Applicability.
146.62 Minimum criteria for siting.
146.63 Area of review.
146.64 Corrective action for wells in the area of review.
146.65 Construction requirements.
146.66 Logging, sampling, and testing prior to new well operation.
146.67 Operating requirements.
146.68 Testing and monitoring requirements.
146.69 Reporting requirements.
146.70 Information to be evaluated by the Director.
146.71 Closure.
146.72 Post-closure care.
146.73 Financial responsibility for post-closure care.

AUTHORITY: Safe Drinking Water Act, 42 U.S.C. 300f *et seq.;* Resource Conservation and Recovery Act, 42 U.S.C. 6901 *et seq.*

SOURCE: 45 FR 42500, June 24, 1980, unless otherwise noted.

EDITORIAL NOTE: 1. For a rule-related notice affecting part 146, see 52 FR 26342, July 14, 1987.

2. For a document removing the OMB control number wherever it appeared in part 146, see 58 FR 34370, June 25, 1993.

Subpart A—General Provisions

§146.1 Applicability and scope.

(a) This part sets forth technical criteria and standards for the Underground Injection Control Program. This part should be read in conjunction with 40 CFR parts 124, 144, and 145, which also apply to UIC programs. 40 CFR part 144 defines the regulatory framework of EPA administered permit programs. 40 CFR part 145 describes the elements of an approvable State program and procedures for EPA approval of State participation in the permit programs. 40 CFR part 124 describes the procedures the Agency will use for issuing permits under the covered programs. Certain of these procedures will also apply to State-administered programs as specified in 40 CFR part 145.

(b) Upon the approval, partial approval or promulgation of a State UIC program by the Administrator, any underground injection which is not authorized by the Director by rule or by permit is unlawful.

(Clean Water Act, Safe Drinking Water Act, Clean Air Act, Resource Conservation and Recovery Act: 42 U.S.C. 6905, 6912, 6925, 6927, 6974)

[45 FR 42500, June 24, 1980, as amended at 48 FR 14293, Apr. 1, 1983]

§146.2 Law authorizing these regulations.

The Safe Drinking Water Act, 42 U.S.C. 300f *et seq.* authorizes these regulations and all other UIC program regulations referenced in 40 CFR part 144. Certain regulations relating to the injection of hazardous waste are also authorized by the Resource Conservation and Recovery Act, 42 U.S.C. 6901 *et seq.*

[58 FR 63898, Dec. 3, 1993]

§146.3 Definitions.

The following definitions apply to the underground injection control program.

Abandoned well means a well whose use has been permanently discontinued or which is in a state of disrepair such that it cannot be used for its intended purpose or for observation purposes.

Administrator means the Administrator of the United States Environmental Protection Agency, or an authorized representative.

Application means the EPA standard national forms for applying for a permit, including any additions, revisions or modifications to the forms; or forms approved by EPA for use in approved States, including any approved modifications or revisions. For RCRA, application also includes the information required by the Director under §122.25

(contents of Part B of the RCRA application).

Aquifer means a geological formation, group of formations, or part of a formation that is capable of yielding a significant amount of water to a well or spring.

Area of review means the area surrounding an injection well described according to the criteria set forth in § 146.06 or in the case of an area permit, the project area plus a circumscribing area the width of which is either ¼ of a mile or a number calculated according to the criteria set forth in § 146.06.

Casing means a pipe or tubing of appropriate material, of varying diameter and weight, lowered into a borehole during or after drilling in order to support the sides of the hole and thus prevent the walls from caving, to prevent loss of drilling mud into porous ground, or to prevent water, gas, or other fluid from entering or leaving the hole.

Catastrophic collapse means the sudden and utter failure of overlying ''strata'' caused by removal of underlying materials.

Cementing means the operation whereby a cement slurry is pumped into a drilled hole and/or forced behind the casing.

Confining bed means a body of impermeable or distinctly less permeable material stratigraphically adjacent to one or more aquifers.

Confining zone means a geological formation, group of formations, or part of a formation that is capable of limiting fluid movement above an injection zone.

Contaminant means any physical, chemical, biological, or radiological substance or matter in water.

Conventional mine means an open pit or underground excavation for the production of minerals.

Director means the Regional Administrator, the State director or the Tribal director as the context requires, or an authorized representative. When there is no approved State or Tribal program, and there is an EPA administered program, ''Director'' means the Regional Administrator. When there is an approved State or Tribal program, ''Director'' normally means the State or Tribal director. In some circumstances, however, EPA retains the authority to take certain actions even when there is an approved State or Tribal program. (For example, when EPA has issued an NPDES permit prior to the approval of a State program, EPA may retain jurisdiction over that permit after program approval; see § 123.69). In such cases, the term *Director* means the Regional Administrator and not the State or Tribal director.

Disposal well means a well used for the disposal of waste into a subsurface stratum.

Effective date of a UIC program means the date that a State UIC program is approved or established by the Administrator.

Environmental Protection Agency (''EPA'') means the United States Environmental Protection Agency.

EPA means the United States ''Environmental Protection Agency.''

Exempted aquifer means an aquifer or its portion that meets the criteria in the definition of ''underground source of drinking water'' but which has been exempted according to the procedures of § 144.8(b).

Existing injection well means an ''injection well'' other than a ''new injection well.''

Experimental technology means a technology which has not been proven feasible under the conditions in which it is being tested.

Facility or activity means any ''HWM facility,'' UIC ''injection well,'' NPDES ''point source,'' or State 404 dredge and fill activity, or any other facility or activity (including land or appurtenances thereto) that is subject to regulation under the RCRA, UIC, NPDES, or 404 programs.

Fault means a surface or zone of rock fracture along which there has been displacement.

Flow rate means the volume per time unit given to the flow of gases or other fluid substance which emerges from an orifice, pump, turbine or passes along a conduit or channel.

Fluid means material or substance which flows or moves whether in a semisolid, liquid, sludge, gas, or any other form or state.

Formation means a body of rock characterized by a degree of lithologic homogeneity which is prevailingly, but not necessarily, tabular and is

mappable on the earth's surface or traceable in the subsurface.

Formation fluid means ''fluid'' present in a ''formation'' under natural conditions as opposed to introduced fluids, such as drilling mud.

Generator means any person, by site location, whose act or process produces hazardous waste identified or listed in 40 CFR part 261.

Ground water means water below the land surface in a zone of saturation.

Hazardous waste means a hazardous waste as defined in 40 CFR 261.3.

Hazardous Waste Management facility (''HWM facility'') means all contiguous land, and structures, other appurtenances, and improvements on the land used for treating, storing, or disposing of hazardous waste. A facility may consist of several treatment, storage, or disposal operational units (for example, one or more landfills, surface impoundments, or combination of them).

HWM facility means ''Hazardous Waste Management facility.''

Indian Tribe means any Indian Tribe having a Federally recognized governing body carrying out substantial governmental duties and powers over a defined area.

Injection well means a ''well'' into which ''fluids'' are being injected.

Injection zone means a geological ''formation'', group of formations, or part of a formation receiving fluids through a well.

Lithology means the description of rocks on the basis of their physical and chemical characteristics.

Owner or operator means the owner or operator of any facility or activity subject to regulation under the RCRA, UIC, NPDES, or 404 programs.

Packer means a device lowered into a well to produce a fluid-tight seal.

Permit means an authorization, license, or equivalent control document issued by EPA or an ''approved State'' to implement the requirements of this part and parts 124, 144, and 145. Permit does not include RCRA interim status (§ 122.23), UIC authorization by rule (§§ 144.21 to 144.26 and 144.15), or any permit which has not yet been the subject of final agency action, such as a ''draft permit'' or a ''proposed permit.''

Plugging means the act or process of stopping the flow of water, oil or gas into or out of a formation through a borehole or well penetrating that formation.

Plugging record means a systematic listing of permanent or temporary abandonment of water, oil, gas, test, exploration and waste injection wells, and may contain a well log, description of amounts and types of plugging material used, the method employed for plugging, a description of formations which are sealed and a graphic log of the well showing formation location, formation thickness, and location of plugging structures.

Pressure means the total load or force per unit area acting on a surface.

Project means a group of wells in a single operation.

Radioactive waste means any waste which contains radioactive material in concentrations which exceed those listed in 10 CFR part 20, appendix B, table II column 2.

RCRA means the Solid Waste Disposal Act as amended by the Resource Conservation and Recovery Act of 1976 (Pub. L. 94–580, as amended by Pub. L. 95–609, 42 U.S.C. 6901 *et seq.).*

SDWA means the Safe Drinking Water Act (Pub. L. 95–523, as amended by Pub. L. 95–190, 42 U.S.C. 300(f) *et seq.).*

Site means the land or water area where any facility or activity is physically located or conducted, including adjacent land used in connection with the facility or activity.

Sole or principal source aquifer means an aquifer which has been designated by the Administrator pursuant to section 1424 (a) or (e) of the SDWA.

State Director means the chief administrative officer of any State, interstate, or Tribal agency operating an ''approved program,'' or the delegated representative of the State Director. If the responsibility is divided among two or more State, interstate, or Tribal agencies, ''State Director'' means the chief administrative officer of the State, interstate, or Tribal agency authorized to perform the particular procedure or function to which reference is made.

Stratum (plural *strata*) means a single sedimentary bed or layer, regardless of

thickness, that consists of generally the same kind of rock material.

Subsidence means the lowering of the natural land surface in response to: Earth movements; lowering of fluid pressure; removal of underlying supporting material by mining or solution of solids, either artificially or from natural causes; compaction due to wetting (Hydrocompaction); oxidation of organic matter in soils; or added load on the land surface.

Surface casing means the first string of well casing to be installed in the well.

Total dissolved solids (''TDS'') means the total dissolved (filterable) solids as determined by use of the method specified in 40 CFR part 136.

UIC means the Underground Injection Control program under Part C of the Safe Drinking Water Act, including an ''approved program.''

Underground injection means a ''well injection.''

Underground source of drinking water (USDW) means an aquifer or its portion:

(1)(i) Which supplies any public water system; or

(ii) Which contains a sufficient quantity of ground water to supply a public water system; and

(A) Currently supplies drinking water for human consumption; or

(B) Contains fewer than 10,000 mg/l total dissolved solids; and

(2) Which is not an exempted aquifer.

USDW means ''underground source of drinking water.''

Well means a bored, drilled or driven shaft, or a dug hole, whose depth is greater than the largest surface dimension.

Well injection means the subsurface emplacement of fluids through a bored, drilled or driven well; or through a dug well, where the depth of the dug well is greater than the largest surface dimension.

Well plug means a watertight and gastight seal installed in a borehole or well to prevent movement of fluids.

Well stimulation means several processes used to clean the well bore, enlarge channels, and increase pore space in the interval to be injected thus making it possible for wastewater to move more readily into the formation, and includes (1) surging, (2) jetting, (3) blasting, (4) acidizing, (5) hydraulic fracturing.

Well monitoring means the measurement, by on-site instruments or laboratory methods, of the quality of water in a well.

(Clean Water Act, Safe Drinking Water Act, Clean Air Act, Resource Conservation and Recovery Act: 42 U.S.C. 6905, 6912, 6925, 6927, 6974)

[45 FR 42500, June 24, 1980, as amended at 46 FR 43161, Aug. 27, 1981; 47 FR 4998, Feb. 3, 1982; 48 FR 14293, Apr. 1, 1983; 53 FR 37414, Sept. 26, 1988]

§ 146.4 Criteria for exempted aquifers.

An aquifer or a portion thereof which meets the criteria for an ''underground source of drinking water'' in § 146.3 may be determined under 40 CFR 144.8 to be an ''exempted aquifer'' if it meets the following criteria:

(a) It does not currently serve as a source of drinking water; and

(b) It cannot now and will not in the future serve as a source of drinking water because:

(1) It is mineral, hydrocarbon or geothermal energy producing, or can be demonstrated by a permit applicant as part of a permit application for a Class II or III operation to contain minerals or hydrocarbons that considering their quantity and location are expected to be commercially producible.

(2) It is situated at a depth or location which makes recovery of water for drinking water purposes economically or technologically impractical;

(3) It is so contaminated that it would be economically or technologically impractical to render that water fit for human consumption; or

(4) It is located over a Class III well mining area subject to subsidence or catastrophic collapse; or

(c) The total dissolved solids content of the ground water is more than 3,000 and less than 10,000 mg/l and it is not reasonably expected to supply a public water system.

(Clean Water Act, Safe Drinking Water Act, Clean Air Act, Resource Conservation and Recovery Act: 42 U.S.C. 6905, 6912, 6925, 6927, 6974)

[45 FR 42500, June 24, 1980, as amended at 47 FR 4998, Feb. 3, 1982; 48 FR 14293, Apr. 1, 1983]

§146.5 Classification of injection wells.

Injection wells are classified as follows:

(a) *Class I.* (1) Wells used by generators of hazardous waste or owners or operators of hazardous waste management facilities to inject hazardous waste beneath the lowermost formation containing, within one quarter (¼) mile of the well bore, an underground source of drinking water.

(2) Other industrial and municipal disposal wells which inject fluids beneath the lowermost formation containing, within one quarter mile of the well bore, an underground source of drinking water.

(b) *Class II.* Wells which inject fluids:

(1) Which are brought to the surface in connection with conventional oil or natural gas production and may be commingled with waste waters from gas plants which are an integral part of production operations, unless those waters are classified as a hazardous waste at the time of injection.

(2) For enhanced recovery of oil or natural gas; and

(3) For storage of hydrocarbons which are liquid at standard temperature and pressure.

(c) *Class III.* Wells which inject for extraction of minerals including:

(1) Mining of sulfur by the Frasch process;

(2) In situ production of uranium or other metals. This category includes only in-situ production from ore bodies which have not been conventionally mined. Solution mining of conventional mines such as stopes leaching is included in Class V.

(3) Solution mining of salts or potash.

(d) *Class IV.* (1) Wells used by generators of hazardous waste or of radioactive waste, by owners or operators of hazardous waste management facilities, or by owners or operators of radioactive waste disposal sites to dispose of hazardous waste or radioactive waste into a formation which within one quarter (¼) mile of the well contains an underground source of drinking water.

(2) Wells used by generators of hazardous waste or of radioactive waste, by owners or operators of hazardous waste management facilities, or by owners or operators of radioactive waste disposal sites to dispose of hazardous waste or radioactive waste above a formation which within one quarter (¼) mile of the well contains an underground source of drinking water.

(3) Wells used by generators of hazardous waste or owners or operators of hazardous waste management facilities to dispose of hazardous waste, which cannot be classified under §146.05(a)(1) or §146.05(d) (1) and (2) (e.g., wells used to dispose of hazardous wastes into or above a formation which contains an aquifer which has been exempted pursuant to §146.04).

(e) *Class V.* Injection wells not included in Class I, II, III, or IV. Class V wells include:

(1) Air conditioning return flow wells used to return to the supply aquifer the water used for heating or cooling in a heat pump;

(2) Cesspools including multiple dwelling, community or regional cesspools, or other devices that receive wastes which have an open bottom and sometimes have perforated sides. The UIC requirements do not apply to single family residential cesspools nor to non-residential cesspools which receive solely sanitary wastes and have the capacity to serve fewer than 20 persons a day.

(3) Cooling water return flow wells used to inject water previously used for cooling;

(4) Drainage wells used to drain surface fluid, primarily storm runoff, into a subsurface formation;

(5) Dry wells used for the injection of wastes into a subsurface formation;

(6) Recharge wells used to replenish the water in an aquifer;

(7) Salt water intrusion barrier wells used to inject water into a fresh water aquifer to prevent the intrusion of salt water into the fresh water;

(8) Sand backfill and other backfill wells used to inject a mixture of water and sand, mill tailings or other solids into mined out portions of subsurface mines whether what is injected is a radioactive waste or not.

(9) Septic system wells used to inject the waste or effluent from a multiple

dwelling, business establishment, community or regional business establishment septic tank. The UIC requirements do not apply to single family residential septic system wells, nor to non-residential septic system wells which are used solely for the disposal of sanitary waste and have the capacity to serve fewer than 20 persons a day.

(10) Subsidence control wells (not used for the purpose of oil or natural gas production) used to inject fluids into a non-oil or gas producing zone to reduce or eliminate subsidence associated with the overdraft of fresh water;

(11) Radioactive waste disposal wells other than Class IV;

(12) Injection wells associated with the recovery of geothermal energy for heating, aquaculture and production of electric power.

(13) Wells used for solution mining of conventional mines such as stopes leaching;

(14) Wells used to inject spent brine into the same formation from which it was withdrawn after extraction of halogens or their salts;

(15) Injection wells used in experimental technologies.

(16) Injection wells used for in situ recovery of lignite, coal, tar sands, and oil shale.

[45 FR 42500, June 24, 1980, as amended at 46 FR 43161, Aug. 27, 1981; 47 FR 4999, Feb. 3, 1982]

§ 146.6 Area of review.

The area of review for each injection well or each field, project or area of the State shall be determined according to either paragraph (a) or (b) of this section. The Director may solicit input from the owners or operators of injection wells within the State as to which method is most appropriate for each geographic area or field.

(a) *Zone of endangering influence.* (1) The zone of endangering influence shall be:

(i) In the case of application(s) for well permit(s) under § 122.38 that area the radius of which is the lateral distance in which the pressures in the injection zone may cause the migration of the injection and/or formation fluid into an underground source of drinking water; or

(ii) In the case of an application for an area permit under § 122.39, the project area plus a circumscribing area the width of which is the lateral distance from the perimeter of the project area, in which the pressures in the injection zone may cause the migration of the injection and/or formation fluid into an underground source of drinking water.

(2) Computation of the zone of endangering influence may be based upon the parameters listed below and should be calculated for an injection time period equal to the expected life of the injection well or pattern. The following modified Theis equation illustrates one form which the mathematical model may take.

$$r = \left(\frac{2.25KHt}{S10^{X}} \right)^{1/2}$$

where:

$$X = \frac{4\pi KH(h_w - h_{bo} \times S_p G_b)}{2.3Q}$$

r=Radius of endangering influence from injection well (length)

k=Hydraulic conductivity of the injection zone (length/time)

H=Thickness of the injection zone (length)

t=Time of injection (time)

S=Storage coefficient (dimensionless)

Q=Injection rate (volume/time)

h_{bo}=Observed original hydrostatic head of injection zone (length) measured from the base of the lowermost underground source of drinking water

h_w=Hydrostatic head of underground source of drinking water (length) measured from the base of the lowest underground source of drinking water

S_pG_b=Specific gravity of fluid in the injection zone (dimensionless)

π=3.142 (dimensionless)

The above equation is based on the following assumptions:

(i) The injection zone is homogenous and isotropic;

(ii) The injection zone has infinite area extent;

(iii) The injection well penetrates the entire thickness of the injection zone;

(iv) The well diameter is infinitesimal compared to ''r'' when injection time is longer than a few minutes; and

(v) The emplacement of fluid into the injection zone creates instantaneous increase in pressure.

(b) *Fixed radius.* (1) In the case of application(s) for well permit(s) under §122.38 a fixed radius around the well of not less than one-fourth (¼) mile may be used.

(2) In the case of an application for an area permit under §122.39 a fixed width of not less than one-fourth (¼) mile for the circumscribing area may be used.

In determining the fixed radius, the following factors shall be taken into consideration: Chemistry of injected and formation fluids; hydrogeology; population and ground-water use and dependence; and historical practices in the area.

(c) If the area of review is determined by a mathematical model pursuant to paragraph (a) of this section, the permissible radius is the result of such calculation even if it is less than one-fourth (¼) mile.

[45 FR 42500, June 24, 1980, as amended at 46 FR 43161, Aug. 27, 1981; 47 FR 4999, Feb. 3, 1982]

§146.7 Corrective action.

In determining the adequacy of corrective action proposed by the applicant under 40 CFR 144.55 and in determining the additional steps needed to prevent fluid movement into underground sources of drinking water, the following criteria and factors shall be considered by the Director:

(a) Nature and volume of injected fluid;

(b) Nature of native fluids or by-products of injection;

(c) Potentially affected population;

(d) Geology;

(e) Hydrology;

(f) History of the injection operation;

(g) Completion and plugging records;

(h) Abandonment procedures in effect at the time the well was abandoned; and

(i) Hydraulic connections with underground sources of drinking water.

(Clean Water Act, Safe Drinking Water Act, Clean Air Act, Resource Conservation and Recovery Act: 42 U.S.C. 6905, 6912, 6925, 6927, 6974)

[45 FR 42500, June 24, 1980, as amended at 46 FR 43162, Aug. 27, 1981; 48 FR 14293, Apr. 1, 1983]

§146.8 Mechanical integrity.

(a) An injection well has mechanical integrity if:

(1) There is no significant leak in the casing, tubing or packer; and

(2) There is no significant fluid movement into an underground source of drinking water through vertical channels adjacent to the injection well bore.

(b) One of the following methods must be used to evaluate the absence of significant leaks under paragraph (a)(1) of this section:

(1) Following an initial pressure test, monitoring of the tubing-casing annulus pressure with sufficient frequency to be representative, as determined by the Director, while maintaining an annulus pressure different from atmospheric pressure measured at the surface;

(2) Pressure test with liquid or gas; or

(3) Records of monitoring showing the absence of significant changes in the relationship between injection pressure and injection flow rate for the following Class II enhanced recovery wells:

(i) Existing wells completed without a packer provided that a pressure test has been performed and the data is available and provided further that one pressure test shall be performed at a time when the well is shut down and if the running of such a test will not cause further loss of significant amounts of oil or gas; or

(ii) Existing wells constructed without a long string casing, but with surface casing which terminates at the base of fresh water provided that local geological and hydrological features allow such construction and provided further that the annular space shall be visually inspected. For these wells, the Director shall prescribe a monitoring program which will verify the absence of significant fluid movement from the injection zone into an USDW.

(c) One of the following methods must be used to determine the absence of significant fluid movement under paragraph (a)(2) of this section:

(1) The results of a temperature or noise log; or

(2) For Class II only, cementing records demonstrating the presence of

adequate cement to prevent such migration; or

(3) For Class III wells where the nature of the casing precludes the use of the logging techniques prescribed at paragraph (c)(1) of this section, cementing records demonstrating the presence of adequate cement to prevent such migration;

(4) For Class III wells where the Director elects to rely on cementing records to demonstrate the absence of significant fluid movement, the monitoring program prescribed by § 146.33(b) shall be designed to verify the absence of significant fluid movement.

(d) The Director may allow the use of a test to demonstrate mechanical integrity other than those listed in paragraphs (b) and (c)(2) of this section with the written approval of the Administrator. To obtain approval, the Director shall submit a written request to the Administrator, which shall set forth the proposed test and all technical data supporting its use. The Administrator shall approve the request if it will reliably demonstrate the mechanical integrity of wells for which its use is proposed. Any alternate method approved by the Administrator shall be published in the FEDERAL REGISTER and may be used in all States unless its use is restricted at the time of approval by the Administrator.

(e) In conducting and evaluating the tests enumerated in this section or others to be allowed by the Director, the owner or operator and the Director shall apply methods and standards generally accepted in the industry. When the owner or operator reports the results of mechanical integrity tests to the Director, he shall include a description of the test(s) and the method(s) used. In making his/her evaluation, the Director shall review monitoring and other test data submitted since the previous evaluation.

(f) The Director may require additional or alternative tests if the results presented by the owner or operator under § 146.8(e) are not satisfactory to the Director to demonstrate that there is no movement of fluid into or between USDWs resulting from the injection activity.

[45 FR 42500, June 24, 1980, as amended at 46 FR 43162, Aug. 27, 1981; 47 FR 4999, Feb. 3, 1982; 58 FR 63898, Dec. 3, 1993]

§ 146.9 Criteria for establishing permitting priorities.

In determining priorities for setting times for owners or operators to submit applications for authorization to inject under the procedures of § 144.31 (a), (c), (g) or § 144.22(f), the Director shall base these priorities upon consideration of the following factors:

(a) Injection wells known or suspected to be contaminating underground sources of drinking water;

(b) Injection wells known to be injecting fluids containing hazardous contaminants;

(c) Likelihood of contamination of underground sources of drinking water;

(d) Potentially affected population;

(e) Injection wells violating existing State requirements;

(f) Coordination with the issuance of permits required by other State or Federal permit programs;

(g) Age and depth of the injection well; and

(h) Expiration dates of existing State permits, if any.

(Clean Water Act, Safe Drinking Water Act, Clean Air Act, Resource Conservation and Recovery Act: 42 U.S.C. 6905, 6912, 6925, 6927, 6974)

[45 FR 42500, June 24, 1980, as amended at 48 FR 14293, Apr. 1, 1983]

§ 146.10 Plugging and abandoning Class I–III wells.

(a) Prior to abandoning Class I to III wells the well shall be plugged with cement in a manner which will not allow the movement of fluids either into or between underground sources of drinking water. The Director may allow Class III wells to use other plugging materials if he is satisfied that such materials will prevent movement of fluids into or between underground sources of drinking water.

(b) Placement of the cement plugs shall be accomplished by one of the following:

(1) The Balance method;

(2) The Dump Bailer method;

(3) The Two-Plug method; or

(4) An alternative method approved by the Director, which will reliably provide a comparable level of protection to underground sources of drinking water.

(c) The well to be abandoned shall be in a state of static equilibrium with the mud weight equalized top to bottom, either by circulating the mud in the well at least once or by a comparable method prescribed by the Director, prior to the placement of the cement plug(s).

(d) The plugging and abandonment plan required in 40 CFR 144.52(a)(6) and 144.51(n) shall, in the case of a Class III project which underlies or is in an aquifer which has been exempted under 40 CFR 146.04, also demonstrate adequate protection of USDWs. The Director shall prescribe aquifer cleanup and monitoring where he deems it necessary and feasible to insure adequate protection of USDWs.

(Clean Water Act, Safe Drinking Water Act, Clean Air Act, Resource Conservation and Recovery Act: 42 U.S.C. 6905, 6912, 6925, 6927, 6974)

[45 FR 42500, June 24, 1980, as amended at 47 FR 5000, Feb. 3, 1982; 48 FR 14293, Apr. 1, 1983]

Subpart B—Criteria and Standards Applicable to Class I Wells

§ 146.11 Criteria and standards applicable to Class I nonhazardous wells.

This subpart establishes criteria and standards for underground injection control programs to regulate Class I nonhazardous wells.

[53 FR 28148, July 26, 1988]

§ 146.12 Construction requirements.

(a) All Class I wells shall be sited in such a fashion that they inject into a formation which is beneath the lowermost formation containing, within one quarter mile of the well bore, an underground source of drinking water.

(b) All Class I wells shall be cased and cemented to prevent the movement of fluids into or between underground sources of drinking water. The casing and cement used in the construction of each newly drilled well shall be designed for the life expectancy of the well. In determining and specifying casing and cementing requirements, the following factors shall be considered:

(1) Depth to the injection zone;

(2) Injection pressure, external pressure, internal pressure, and axial loading;

(3) Hole size;

(4) Size and grade of all casing strings (wall thickness, diameter, nominal weight, length, joint specification, and construction material);

(5) Corrosiveness of injected fluid, formation fluids, and temperatures;

(6) Lithology of injection and confining intervals; and

(7) Type or grade of cement.

(c) All Class I injection wells, except those municipal wells injecting noncorrosive wastes, shall inject fluids through tubing with a packer set immediately above the injection zone, or tubing with an approved fluid seal as an alternative. The tubing, packer, and fluid seal shall be designed for the expected service.

(1) The use of other alternatives to a packer may be allowed with the written approval of the Director. To obtain approval, the operator shall submit a written request to the Director, which shall set forth the proposed alternative and all technical data supporting its use. The Director shall approve the request if the alternative method will reliably provide a comparable level of protection to underground sources of drinking water. The Director may approve an alternative method solely for an individual well or for general use.

(2) In determining and specifying requirements for tubing, packer, or alternatives the following factors shall be considered:

(i) Depth of setting;

(ii) Characteristics of injection fluid (chemical content, corrosiveness, and density);

(iii) Injection pressure;

(iv) Annular pressure;

(v) Rate, temperature and volume of injected fluid; and

(vi) Size of casing.

(d) Appropriate logs and other tests shall be conducted during the drilling and construction of new Class I wells. A descriptive report interpreting the results of such logs and tests shall be prepared by a knowledgeable log analyst and submitted to the Director. At

a minimum, such logs and tests shall include:

(1) Deviation checks on all holes constructed by first drilling a pilot hole, and then enlarging the pilot hole by reaming or another method. Such checks shall be at sufficiently frequent intervals to assure that vertical avenues for fluid migration in the form of diverging holes are not created during drilling.

(2) Such other logs and tests as may be needed after taking into account the availability of similar data in the area of the drilling site, the construction plan, and the need for additional information, that may arise from time to time as the construction of the well progresses. In determining which logs and tests shall be required, the following logs shall be considered for use in the following situations:

(i) For surface casing intended to protect underground sources of drinking water:

(A) Resistivity, spontaneous potential, and caliper logs before the casing is installed; and

(B) A cement bond, temperature, or density log after the casing is set and cemented.

(ii) For intermediate and long strings of casing intended to facilitate injection:

(A) Resistivity, spontaneous potential, porosity, and gamma ray logs before the casing is installed;

(B) Fracture finder logs; and

(C) A cement bond, temperature, or density log after the casing is set and cemented.

(e) At a minimum, the following information concerning the injection formation shall be determined or calculated for new Class I wells:

(1) Fluid pressure;

(2) Temperature;

(3) Fracture pressure;

(4) Other physical and chemical characteristics of the injection matrix; and

(5) Physical and chemical characteristics of the formation fluids.

[45 FR 42500, June 24, 1980, as amended at 46 FR 43162, Aug. 27, 1981]

§ 146.13 Operating, monitoring and reporting requirements.

(a) *Operating requirements.* Operating requirements shall at a minimum, specify that:

(1) Except during stimulation injection pressure at the wellhead shall not exceed a maximum which shall be calculated so as to assure that the pressure in the injection zone during injection does not initiate new fractures or propagate existing fractures in the injection zone. In no case shall injection pressure initiate fractures in the confining zone or cause the movement of injection or formation fluids into an underground source of drinking water.

(2) Injection between the outermost casing protecting underground sources of drinking water and the well bore is prohibited.

(3) Unless an alternative to a packer has been approved under § 146.12(c), the annulus between the tubing and the long string of casings shall be filled with a fluid approved by the Director and a pressure, also approved by the Director, shall be maintained on the annulus.

(b) *Monitoring requirements.* Monitoring requirements shall, at a minimum, include:

(1) The analysis of the injected fluids with sufficient frequency to yield representative data of their characteristics;

(2) Installation and use of continuous recording devices to monitor injection pressure, flow rate and volume, and the pressure on the annulus between the tubing and the long string of casing;

(3) A demonstration of mechanical integrity pursuant to § 146.8 at least once every five years during the life of the well; and

(4) The type, number and location of wells within the area of review to be used to monitor any migration of fluids into and pressure in the underground sources of drinking water, the parameters to be measured and the frequency of monitoring.

(c) *Reporting requirements.* Reporting requirements shall, at a minimum, include:

(1) Quarterly reports to the Director on:

(i) The physical, chemical and other relevant characteristics of injection fluids;

(ii) Monthly average, maximum and minimum values for injection pressure, flow rate and volume, and annular pressure; and

(iii) The results of monitoring prescribed under paragraph (b)(4) of this section.

(2) Reporting the results, with the first quarterly report after the completion, of:

(i) Periodic tests of mechanical integrity;

(ii) Any other test of the injection well conducted by the permittee if required by the Director; and

(iii) Any well work over.

(d) *Ambient monitoring.* (1) Based on a site-specific assessment of the potential for fluid movement from the well or injection zone and on the potential value of monitoring wells to detect such movement, the Director shall require the owner or operator to develop a monitoring program. At a minimum, the Director shall require monitoring of the pressure buildup in the injection zone annually, including at a minimum, a shut down of the well for a time sufficient to conduct a valid observation of the pressure fall-off curve.

(2) When prescribing a monitoring system the Director may also require:

(i) Continuous monitoring for pressure changes in the first aquifer overlying the confining zone. When such a well is installed, the owner or operator shall, on a quarterly basis, sample the aquifer and analyze for constituents specified by the Director;

(ii) The use of indirect, geophysical techniques to determine the position of the waste front, the water quality in a formation designated by the Director, or to provide other site specific data;

(iii) Periodic monitoring of the ground water quality in the first aquifer overlying the injection zone;

(iv) Periodic monitoring of the ground water quality in the lowermost USDW; and

(v) Any additional monitoring necessary to determine whether fluids are moving into or between USDWs.

[45 FR 42500, June 24, 1980, as amended at 46 FR 43162, Aug. 27, 1981; 47 FR 32129, July 26, 1982; 53 FR 28148, July 26, 1988]

§146.14 Information to be considered by the Director.

This section sets forth the information which must be considered by the Director in authorizing Class I wells. For an existing or converted new Class I well the Director may rely on the existing permit file for those items of information listed below which are current and accurate in the file. For a newly drilled Class I well, the Director shall require the submission of all the information listed below. For both existing and new Class I wells certain maps, cross-sections, tabulations of wells within the area of review and other data may be included in the application by reference provided they are current, readily available to the Director (for example, in the permitting agency's files) and sufficiently identified to be retrieved. In cases where EPA issues the permit all the information in this section must be submitted to the Administrator.

(a) Prior to the issuance of a permit for an existing Class I well to operate or the construction or conversion of a new Class I well the Director shall consider the following:

(1) Information required in 40 CFR 144.31 and 144.31(g);

(2) A map showing the injection well(s) for which a permit is sought and the applicable area of review. Within the area of review, the map must show the number, or name, and location of all producing wells, dry holes, surface bodies of water, springs, mines (surface and subsurface), quarries, water wells and other pertinent surface features including residences and roads. The map should also show faults, if known or suspected. Only information of public record is required to be included on this map;

(3) A tabulation of data on all wells within the area of review which penetrate into the proposed injection zone. Such data shall include a description of each well's type, construction, date drilled, location, depth, record of plugging and/or completion, and any additional information the Director may require;

(4) Maps and cross sections indicating the general vertical and lateral limits of all underground sources of drinking water within the area of review, their

position relative to the injection formation and the direction of water movement, where known, in each underground source of drinking water which may be affected by the proposed injection;

(5) Maps and cross sections detailing the geologic structure of the local area;

(6) Generalized maps and cross sections illustrating the regional geologic setting;

(7) Proposed operating data:

(i) Average and maximum daily rate and volume of the fluid to be injected;

(ii) Average and maximum injection pressure; and

(iii) Source and an analysis of the chemical, physical, radiological and biological characteristics of injection fluids;

(8) Proposed formation testing program to obtain an analysis of the chemical, physical and radiological characteristics of and other information on the receiving formation;

(9) Proposed stimulation program;

(10) Proposed injection procedure;

(11) Schematic or other appropriate drawings of the surface and subsurface construction details of the well.

(12) Contingency plans to cope with all shut-ins or well failures so as to prevent migration of fluids into any underground source of drinking water;

(13) Plans (including maps) for meeting the monitoring requirements in § 146.13(b);

(14) For wells within the area of review which penetrate the injection zone but are not properly completed or plugged, the corrective action proposed to be taken under 40 CFR 144.55;

(15) Construction procedures including a cementing and casing program, logging procedures, deviation checks, and a drilling, testing, and coring program; and

(16) A certificate that the applicant has assured, through a performance bond or other appropriate means, the resources necessary to close, plug or abandon the well as required by 40 CFR 122.42(g).

(b) Prior to granting approval for the operation of a Class I well the Director shall consider the following information:

(1) All available logging and testing program data on the well;

(2) A demonstration of mechanical integrity pursuant to § 146.8;

(3) The anticipated maximum pressure and flow rate at which the permittee will operate;

(4) The results of the formation testing program;

(5) The actual injection procedure;

(6) The compatibility of injected waste with fluids in the injection zone and minerals in both the injection zone and the confining zone; and

(7) The status of corrective action on defective wells in the area of review.

(c) Prior to granting approval for the plugging and abandonment of a Class I well the Director shall consider the following information:

(1) The type and number of plugs to be used;

(2) The placement of each plug including the elevation of the top and bottom;

(3) The type and grade and quantity of cement to be used;

(4) The method for placement of the plugs; and

(5) The procedure to be used to meet the requirement of § 146.10(c).

(Clean Water Act, Safe Drinking Water Act, Clean Air Act, Resource Conservation and Recovery Act: 42 U.S.C. 6905, 6912, 6925, 6927, 6974)

[45 FR 42500, June 24, 1980, as amended at 46 FR 43162, Aug. 27, 1981; 48 FR 14293, Apr. 1, 1983]

Subpart C—Criteria and Standards Applicable to Class II Wells

§ 146.21 Applicability.

This subpart establishes criteria and standards for underground injection control programs to regulate Class II wells.

§ 146.22 Construction requirements.

(a) All new Class II wells shall be sited in such a fashion that they inject into a formation which is separated from any USDW by a confining zone that is free of known open faults or fractures within the area of review.

(b)(1) All Class II injection wells shall be cased and cemented to prevent movement of fluids into or between underground sources of drinking water.

The casing and cement used in the construction of each newly drilled well shall be designed for the life expectancy of the well. In determining and specifying casing and cementing requirements, the following factors shall be considered:

(i) Depth to the injection zone;

(ii) Depth to the bottom of all USDWs; and

(iii) Estimated maximum and average injection pressures;

(2) In addition the Director may consider information on:

(i) Nature of formation fluids;

(ii) Lithology of injection and confining zones;

(iii) External pressure, internal pressure, and axial loading;

(iv) Hole size;

(v) Size and grade of all casing strings; and

(vi) Class of cement.

(c) The requirements in paragraph (b) of this section need not apply to existing or newly converted Class II wells located in existing fields if:

(1) Regulatory controls for casing and cementing existed for those wells at the time of drilling and those wells are in compliance with those controls; and

(2) Well injection will not result in the movement of fluids into an underground source of drinking water so as to create a significant risk to the health of persons.

(d) The requirements in paragraph (b) of this section need not apply to newly drilled wells in existing fields if;

(1) They meet the requirements of the State for casing and cementing applicable to that field at the time of submission of the State program to the Administrator; and

(2) Well injection will not result in the movement of fluids into an underground source of drinking water so as to create a significant risk to the health of persons.

(e) Where a State did not have regulatory controls for casing and cementing prior to the time of the submission of the State program to the Administrator, the Director need not apply the casing and cementing requirements in paragraph (b) of this section if he submits as a part of his application for primacy, an appropriate plan for casing and cementing of existing, newly converted, and newly drilled wells in existing fields, and the Administrator approves the plan.

(f) Appropriate logs and other tests shall be conducted during the drilling and construction of new Class II wells. A descriptive report interpreting the results of that portion of those logs and tests which specifically relate to (1) an USDW and the confining zone adjacent to it, and (2) the injection and adjacent formations shall be prepared by a knowledgeable log analyst and submitted to the director. At a minimum, these logs and tests shall include:

(1) Deviation checks on all holes constructed by first drilling a pilot hole and then enlarging the pilot hole, by reaming or another method. Such checks shall be at sufficiently frequent intervals to assure that vertical avenues for fluid movement in the form of diverging holes are not created during drilling.

(2) Such other logs and tests as may be needed after taking into account the availability of similar data in the area of the drilling site, the construction plan, and the need for additional information that may arise from time to time as the construction of the well progresses. In determining which logs and tests shall be required the following shall be considered by the Director in setting logging and testing requirements:

(i) For surface casing intended to protect underground sources of drinking water in areas where the lithology has not been determined:

(A) Electric and caliper logs before casing is installed; and

(B) A cement bond, temperature, or density log after the casing is set and cemented.

(ii) for intermediate and long strings of casing intended to facilitate injection:

(A) Electric porosity and gamma ray logs before the casing is installed;

(B) Fracture finder logs; and

(C) A cement bond, temperature, or density log after the casing is set and cemented.

(g) At a minimum, the following information concerning the injection formation shall be determined or calculated for new Class II wells or projects:

(1) Fluid pressure;

(2) Estimated fracture pressure;

(3) Physical and chemical characteristics of the injection zone.

[45 FR 42500, June 24, 1980, as amended at 46 FR 43162, Aug. 27, 1981; 47 FR 5000, Feb. 3, 1982]

§ 146.23 Operating, monitoring, and reporting requirements.

(a) *Operating requirements.* Operating requirements shall, at a minimum, specify that:

(1) Injection pressure at the wellhead shall not exceed a maximum which shall be calculated so as to assure that the pressure during injection does not initiate new fractures or propagate existing fractures in the confining zone adjacent to the USDWs. In no case shall injection pressure cause the movement of injection or formation fluids into an underground source of drinking water

(2) Injection between the outermost casing protecting underground sources of drinking water and the well bore shall be prohibited.

(b) *Monitoring requirements.* Monitoring requirements shall, at a minimum, include:

(1) Monitoring of the nature of injected fluids at time intervals sufficiently frequent to yield data representative of their characteristics;

(2) Observation of injection pressure, flow rate, and cumulative volume at least with the following frequencies:

(i) Weekly for produced fluid disposal operations;

(ii) Monthly for enhanced recovery operations;

(iii) Daily during the injection of liquid hydrocarbons and injection for withdrawal of stored hydrocarbons; and

(iv) Daily during the injection phase of cyclic steam operations

And recording of one observation of injection pressure, flow rate and cumulative volume at reasonable intervals no greater than 30 days.

(3) A demonstration of mechanical integrity pursuant to § 146.8 at least once every five years during the life of the injection well;

(4) Maintenance of the results of all monitoring until the next permit review (see 40 CFR 144.52(a)(5)); and

(5) Hydrocarbon storage and enhanced recovery may be monitored on a field or project basis rather than on an individual well basis by manifold monitoring. Manifold monitoring may be used in cases of facilities consisting of more than one injection well, operating with a common manifold. Separate monitoring systems for each well are not required provided the owner/operator demonstrates that manifold monitoring is comparable to individual well monitoring.

(c) *Reporting requirements.* (1) Reporting requirements shall at a minimum include an annual report to the Director summarizing the results of monitoring required under paragraph (b) of this section. Such summary shall include monthly records of injected fluids, and any major changes in characteristics or sources of injected fluid. Previously submitted information may be included by reference.

(2) Owners or operators of hydrocarbon storage and enhanced recovery projects may report on a field or project basis rather than an individual well basis where manifold monitoring is used.

(Clean Water Act, Safe Drinking Water Act, Clean Air Act, Resource Conservation and Recovery Act; 42 U.S.C. 6905, 6912, 6925, 6927, 6974)

[45 FR 42500, June 24, 1980, as amended at 46 FR 43162, Aug. 27, 1981; 47 FR 5000, Feb. 3, 1982; 48 FR 14293, Apr. 1, 1983; 48 FR 31404, July 8, 1983]

§ 146.24 Information to be considered by the Director.

This section sets forth the information which must be considered by the Director in authorizing Class II wells. Certain maps, cross-sections, tabulations of wells within the area of review, and other data may be included in the application by reference provided they are current, readily available to the Director (for example, in the permitting agency's files) and sufficiently identified to be retrieved. In cases where

EPA issues the permit, all the information in this section is to be submitted to the Administrator.

(a) Prior to the issuance of a permit for an existing Class II well to operate or the construction or conversion of a new Class II well the Director shall consider the following:

(1) Information required in 40 CFR 144.31 and 144.31(g);

(2) A map showing the injection well or project area for which a permit is sought and the applicable area of review. Within the area of review, the map must show the number or name and location of all existing producing wells, injection wells, abandoned wells, dry holes, and water wells. The map may also show surface bodies of waters, mines (surface and subsurface), quarries and other pertinent surface features including residences and roads, and faults if known or suspended. Only information of public record and pertinent information known to the applicant is required to be included on this map. This requirement does not apply to existing Class II wells; and

(3) A tabulation of data reasonably available from public records or otherwise known to the applicant on all wells within the area of review included on the map required under paragraph (a)(2) of this section which penetrate the proposed injection zone or, in the case of Class II wells operating over the fracture pressure of the injection formation, all known wells within the area of review which penetrate formations affected by the increase in pressure. Such data shall include a description of each well's type, construction, date drilled, location, depth, record of plugging and complete, and any additional information the Director may require. In cases where the information would be repetitive and the wells are of similar age, type, and construction the Director may elect to only require data on a representative number of wells. This requirement does not apply to existing Class II wells.

(4) Proposed operating data:

(i) Average and maximum daily rate and volume of fluids to be injected.

(ii) Average and maximum injection pressure; and

(iii) Source and an appropriate analysis of the chemical and physical characteristics of the injection fluid.

(5) Appropriate geological data on the injection zone and confining zone including lithologic description, geological name, thickness and depth;

(6) Geologic name and depth to bottom of all underground sources of drinking water which may be affected by the injection;

(7) Schematic or other appropriate drawings of the surface and subsurface construction details of the well;

(8) In the case of new injection wells the corrective action proposed to be taken by the applicant under 40 CFR 122.44;

(9) A certificate that the applicant has assured through a performance bond or other appropriate means, the resources necessary to close plug or abandon the well as required by 40 CFR 122.42(g);

(b) In addition the Director may consider the following:

(1) Proposed formation testing program to obtain the information required by §146.22(g);

(2) Proposed stimulation program;

(3) Proposed injection procedure;

(4) Proposed contingency plans, if any, to cope with well failures so as to prevent migration of contaminating fluids into an underground source of drinking water;

(5) Plans for meeting the monitoring requirements of §146.23(b).

(c) Prior to granting approval for the operation of a Class II well the Director shall consider the following information:

(1) All available logging and testing program data on the well;

(2) A demonstration of mechanical integrity pursuant to §146.8;

(3) The anticipated maximum pressure and flow rate at which the permittee will operate.

(4) The results of the formation testing program;

(5) The actual injection procedure; and

(6) For new wells the status of corrective action on defective wells in the area of review.

(d) Prior to granting approval for the plugging and abandonment of a Class II

well the Director shall consider the following information:

(1) The type, and number of plugs to be used;

(2) The placement of each plug including the elevation of top and bottom;

(3) The type, grade, and quantity of cement to be used;

(4) The method of placement of the plugs; and

(5) The procedure to be used to meet the requirements of § 146.10(c).

(Clean Water Act, Safe Drinking Water Act, Clean Air Act, Resource Conservation and Recovery Act: 42 U.S.C. 6905, 6912, 6925, 6927, 6974)

[45 FR 42500, June 24, 1980, as amended at 46 FR 43162, Aug. 27, 1981; 47 FR 5000, Feb. 3, 1982; 48 FR 14293, Apr. 1, 1983]

Subpart D—Criteria and Standards Applicable to Class III Wells

§ 146.31 Applicability.

This subpart establishes criteria and standards for underground injection control programs to regulate Class III wells.

§ 146.32 Construction requirements.

(a) All new Class III wells shall be cased and cemented to prevent the migration of fluids into or between underground sources of drinking water. The Director may waive the cementing requirement for new wells in existing projects or portions of existing projects where he has substantial evidence that no contamination of underground sources of drinking water would result. The casing and cement used in the construction of each newly drilled well shall be designed for the life expectancy of the well. In determining and specifying casing and cementing requirements, the following factors shall be considered:

(1) Depth to the injection zone;

(2) Injection pressure, external pressure, internal pressure, axial loading, etc.;

(3) Hole size;

(4) Size and grade of all casing strings (wall thickness, diameter, nominal weight, length, joint specification, and construction material);

(5) Corrosiveness of injected fluids and formation fluids;

(6) Lithology of injection and confining zones; and

(7) Type and grade of cement.

(b) Appropriate logs and other tests shall be conducted during the drilling and construction of new Class III wells. A descriptive report interpreting the results of such logs and tests shall be prepared by a knowledgeable log analyst and submitted to the Director. The logs and tests appropriate to each type of Class III well shall be determined based on the intended function, depth, construction and other characteristics of the well, availability of similar data in the area of the drilling site and the need for additional information that may arise from time to time as the construction of the well progresses. Deviation checks shall be conducted on all holes where pilot holes and reaming are used, unless the hole will be cased and cemented by circulating cement to the surface. Where deviation checks are necessary they shall be conducted at sufficiently frequent intervals to assure that vertical avenues for fluid migration in the form of diverging holes are not created during drillings.

(c) Where the injection zone is a formation which is naturally water-bearing the following information concerning the injection zone shall be determined or calculated for new Class III wells or projects:

(1) Fluid pressure;

(2) Fracture pressure; and

(3) Physical and chemical characteristics of the formation fluids.

(d) Where the injection formation is not a water-bearing formation, the information in paragraph (c)(2) of this section must be submitted.

(e) Where injection is into a formation which contains water with less than 10,000 mg/l TDS monitoring wells shall be completed into the injection zone and into any underground sources of drinking water above the injection zone which could be affected by the mining operation. These wells shall be located in such a fashion as to detect any excursion of injection fluids, process by-products, or formation fluids outside the mining area or zone. If the operation may be affected by subsidence or catastrophic collapse the monitoring wells shall be located so that they will not be physically affected.

(f) Where injection is into a formation which does not contain water with less than 10,000 mg/l TDS, no monitoring wells are necessary in the injection stratum.

(g) Where the injection wells penetrate an USDW in an area subject to subsidence or catastrophic collapse an adequate number of monitoring wells shall be completed into the USDW to detect any movement of injected fluids, process by-products or formation fluids into the USDW. The monitoring wells shall be located outside the physical influence of the subsidence or catastrophic collapse.

(h) In determining the number, location, construction and frequency of monitoring of the monitoring wells the following criteria shall be considered:

(1) The population relying on the USDW affected or potentially affected by the injection operation;

(2) The proximity of the injection operation to points of withdrawal of drinking water;

(3) The local geology and hydrology;

(4) The operating pressures and whether a negative pressure gradient is being maintained;

(5) The nature and volume of the injected fluid, the formation water, and the process by-products; and

(6) The injection well density.

[45 FR 42500, June 24, 1980, as amended at 46 FR 43163, Aug. 27, 1981; 47 FR 5000, Feb. 3, 1982]

§146.33 Operating, monitoring, and reporting requirements.

(a) *Operating requirements.* Operating requirements prescribed shall, at a minimum, specify that:

(1) Except during well stimulation injection pressure at the wellhead shall be calculated so as to assure that the pressure in the injection zone during injection does not initiate new fractures or propagate existing fractures in the injection zone. In no case, shall injection pressure initiate fractures in the confining zone or cause the migration of injection or formation fluids into an underground source of drinking water.

(2) Injection between the outermost casing protecting underground sources of drinking water and the well bore is prohibited.

(b) *Monitoring requirements.* Monitoring requirements shall, at a minimum, specify:

(1) Monitoring of the nature of injected fluids with sufficient frequency to yield representative data on its characteristics. Whenever the injection fluid is modified to the extent that the analysis required by §146.34(a)(7)(iii) is incorrect or incomplete, a new analysis as required by §146.34(a)(7)(iii) shall be provided to the Director.

(2) Monitoring of injection pressure and either flow rate or volume semi-monthly, or metering and daily recording of injected and produced fluid volumes as appropriate.

(3) Demonstration of mechanical integrity pursuant to §146.08 at least once every five years during the life of the well for salt solution mining.

(4) Monitoring of the fluid level in the injection zone semi-monthly, where appropriate and monitoring of the parameters chosen to measure water quality in the monitoring wells required by §146.32(e), semi-monthly.

(5) Quarterly monitoring of wells required by §146.32(g).

(6) All Class III wells may be monitored on a field or project basis rather than an individual well basis by manifold monitoring. Manifold monitoring may be used in cases of facilities consisting of more than one injection well, operating with a common manifold. Separate monitoring systems for each well are not required provided the owner/operator demonstrates that manifold monitoring is comparable to individual well monitoring.

(c) *Reporting requirements.* Reporting requirements shall, at a minimum, include:

(1) Quarterly reporting to the Director on required monitoring;

(2) Results of mechanical integrity and any other periodic test required by the Director reported with the first regular quarterly report after the completion of the test; and

(3) Monitoring may be reported on a project or field basis rather than individual well basis where manifold monitoring is used.

[45 FR 42500, June 24, 1980, as amended at 46 FR 43163, Aug. 27, 1981; 46 FR 5001, Feb. 3, 1982; 48 FR 31404, July 8, 1983]

§ 146.34 Information to be considered by the Director.

This section sets forth the information which must be considered by the Director in authorizing Class III wells. Certain maps, cross sections, tabulations of wells within the area of review, and other data may be included in the application by reference provided they are current, readily available to the Director (for example, in the permitting agency's files) and sufficiently identified to be retrieved. In cases where EPA issues the permit, all the information in this section must be submitted to the Administrator.

(a) Prior to the issuance of a permit for an existing Class III well or area to operate or the construction of a new Class III well the Director shall consider the following:

(1) Information required in 40 CFR 144.31 and 144.31(g);

(2) A map showing the injection well or project area for which a permit is sought and the applicable area of review. Within the area of review, the map must show the number or name and location of all existing producing wells, injection wells, abandoned wells, dry holes, public water systems and water wells. The map may also show surface bodies of waters, mines (surface and subsurface), quarries and other pertinent surface features including residences and roads, and faults if known or suspected. Only information of public record and pertinent information known to the applicant is required to be included on this map.

(3) A tabulation of data reasonably available from public records or otherwise known to the applicant on wells within the area of review included on the map required under paragraph (a)(2) of this section which penetrate the proposed injection zone. Such data shall include a description of each well's type, construction, date drilled, location, depth, record of plugging and completion, and any additional information the Director may require. In cases where the information would be repetitive and the wells are of similar age, type, and construction the Director may elect to only require data on a representative number of wells.

(4) Maps and cross sections indicating the vertical limits of all underground sources of drinking water within the area of review, their position relative to the injection formation, and the direction of water movement, where known, in every underground source of drinking water which may be affected by the proposed injection:

(5) Maps and cross sections detailing the geologic structure of the local area;

(6) Generalized map and cross sections illustrating the regional geologic setting;

(7) Proposed operating data:

(i) Average and maximum daily rate and volume of fluid to be injected;

(ii) Average and maximum injection pressure; and

(iii) Qualitative analysis and ranges in concentrations of all constituents of injected fluids. The applicant may request Federal confidentiality as specified in 40 CFR part 2. If the information is proprietary an applicant may, in lieu of the ranges in concentrations, choose to submit maximum concentrations which shall not be exceeded. In such a case the applicant shall retain records of the undisclosed concentrations and provide them upon request to the Director as part of any enforcement investigation.

(8) Proposed formation testing program to obtain the information required by § 146.32(c).

(9) Proposed stimulation program;

(10) Proposed injection procedure;

(11) Schematic or other appropriate drawings of the surface and subsurface construction details of the well;

(12) Plans (including maps) for meeting the monitoring requirements of § 146.33(b);

(13) Expected changes in pressure, native fluid displacement, direction of movement of injection fluid;

(14) Contingency plans to cope with all shut-ins or well failures so as to prevent the migration of contaminating fluids into underground sources of drinking water;

(15) A certificate that the applicant has assured, through a performance bond, or other appropriate means, the resources necessary to close, plug, or abandon the well as required by 40 CFR 144.52(a)(7) and

(16) The corrective action proposed to be taken under 40 CFR 144.55.

(b) Prior to granting approval for the operation of a Class III well the Director shall consider the following information:

(1) All available logging and testing data on the well;

(2) A satisfactory demonstration of mechanical integrity for all new wells and for all existing salt solution wells pursuant to §146.08;

(3) The anticipated maximum pressure and flow rate at which the permittee will operate;

(4) The results of the formation testing program;

(5) The actual injection procedures; and

(6) The status of corrective action on defective wells in the area of review.

(c) Prior to granting approval for the plugging and abandonment of a Class III well the Director shall consider the following information:

(1) The type and number of plugs to be used;

(2) The placement of each plug including the elevation of the top and bottom;

(3) The type, grade, and quantity of cement to be used;

(4) The method of placement of the plugs; and

(5) The procedure to be used to meet the requirements of §146.10(c).

(Clean Water Act, Safe Drinking Water Act, Clean Air Act, Resource Conservation and Recovery Act: 42 U.S.C. 6905, 6912, 6925, 6927, 6974)

[45 FR 42500, June 24, 1980, as amended at 46 FR 43163, Aug. 27, 1981; 47 FR 5001, Feb. 3, 1982; 48 FR 14293, Apr. 1, 1983]

Subpart E—Criteria and Standards Applicable to Class IV Injection Wells [Reserved]

Subpart F—Criteria and Standards Applicable to Class V Injection Wells

§146.51 Applicability.

This subpart sets forth criteria and standards for underground injection control programs to regulate all injection not regulated in subparts B, C, D, and E.

(a) Generally, wells covered by this subpart inject non-hazardous fluids into or above formations that contain underground sources of drinking water. It includes all wells listed in §146.5(e) but is not limited to those types of injection wells.

(b) It also includes wells not covered in Class IV that inject radioactive material listed in 10 CFR part 20, appendix B, table II, column 2.

[45 FR 42500, June 24, 1980, as amended at 47 FR 5001, Feb. 3, 1982]

Subpart G—Criteria and Standards Applicable to Class I Hazardous Waste Injection Wells

SOURCE: 53 FR 28148, July 26, 1988, unless otherwise noted.

§146.61 Applicability

(a) This subpart establishes criteria and standards for underground injection control programs to regulate Class I hazardous waste injection wells. Unless otherwise noted this subpart supplements the requirements of subpart A and applies instead of subpart B to Class I hazardous waste injection wells.

(b) *Definitions.*

Cone of influence means that area around the well within which increased injection zone pressures caused by injection into the hazardous waste injection well would be sufficient to drive fluids into an underground source of drinking water (USDW).

Existing well means a Class I well which was authorized prior to August 25, 1988, by an approved State program, or an EPA-administered program or a well which has become a Class I well as a result of a change in the definition of the injected waste which would render the waste hazardous under §261.3 of this part.

Injection interval means that part of the injection zone in which the well is screened, or in which the waste is otherwise directly emplaced.

New well means any Class I hazardous waste injection well which is not an existing well.

Transmissive fault or fracture is a fault or fracture that has sufficient permeability and vertical extent to allow fluids to move between formations.

§ 146.62 Minimum criteria for siting.

(a) All Class I hazardous waste injection wells shall be sited such that they inject into a formation that is beneath the lowermost formation containing within one quarter mile of the well bore an underground source of drinking water.

(b) The siting of Class I hazardous waste injection wells shall be limited to areas that are geologically suitable. The Director shall determine geologic suitability based upon:

(1) An analysis of the structural and stratigraphic geology, the hydrogeology, and the seismicity of the region;

(2) An analysis of the local geology and hydrogeology of the well site, including, at a minimum, detailed information regarding stratigraphy, structure and rock properties, aquifer hydrodynamics and mineral resources; and

(3) A determination that the geology of the area can be described confidently and that limits of waste fate and transport can be accurately predicted through the use of models.

(c) Class I hazardous waste injection wells shall be sited such that:

(1) The injection zone has sufficient permeability, porosity, thickness and areal extent to prevent migration of fluids into USDWs.

(2) The confining zone:

(i) Is laterally continuous and free of transecting, transmissive faults or fractures over an area sufficient to prevenet the movement of fluids into a USDW; and

(ii) Contains at least one formation of sufficient thickness and with lithologic and stress characteristics capable of preventing vertical propagation of fractures.

(d) The owner or operator shall demonstrate to the satisfaction of the Director that:

(1) The confining zone is separated from the base of the lowermost USDW by at least one sequence of permeable and less permeable strata that will provide an added layer of protection for the USDW in the event of fluid movement in an unlocated borehole or transmissive fault; or

(2) Within the area of review, the piezometric surface of the fluid in the injection zone is less than the piezometric surface of the lowermost USDW, considering density effects, injection pressures and any significant pumping in the overlying USDW; or

(3) There is no USDW present.

(4) The Director may approve a site which does not meet the requirements in paragraphs (d) (1), (2), or (3) of this section if the owner or operator can demonstrate to the Director that because of the geology, nature of the waste, or other considerations, abandoned boreholes or other conduits would not cause endangerment of USDWs.

§ 146.63 Area of review.

For the purposes of Class I hazardous waste wells, this section shall apply to the exclusion of § 146.6. The area of review for Class I hazardous waste injection wells shall be a 2-mile radius around the well bore. The Director may specify a larger area of review based on the calculated cone of influence of the well.

§ 146.64 Corrective action for wells in the area of review.

For the purposes of Class I hazardous waste wells, this section shall apply to the exclusion of §§ 144.55 and 146.07.

(a) The owner or operator of a Class I hazardous waste well shall as part of the permit application submit a plan to the Director outlining the protocol used to:

(1) Identify all wells penetrating the confining zone or injection zone within the area of review; and

(2) Determine whether wells are adequately completed or plugged.

(b) The owner or operator of a Class I hazardous waste well shall identify the location of all wells within the area of review that penetrate the injection zone or the confining zone and shall submit as required in § 146.70(a):

(1) A tabulation of all wells within the area of review that penetrate the injection zone or the confining zone; and

(2) A description of each well or type of well and any records of its plugging or completion.

(c) For wells that the Director determines are improperly plugged, completed, or abandoned, or for which

plugging or completion information is unavailable, the applicant shall also submit a plan consisting of such steps or modification as are necessary to prevent movement of fluids into or between USDWs. Where the plan is adequate, the Director shall incorporate it into the permit as a condition. Where the Director's review of an application indicates that the permittee's plan is inadequate (based at a minimum on the factors in paragraph (e) of this section), the Director shall:

(1) Require the applicant to revise the plan;

(2) Prescribe a plan for corrective action as a condition of the permit; or

(3) Deny the application.

(d) Requirements:

(1) Existing injection wells. Any permit issued for an existing Class I hazardous waste injection well requiring corrective action other than pressure limitations shall include a compliance schedule requiring any corrective action accepted or prescribed under paragraph (c) of this section. Any such compliance schedule shall provide for compliance no later than 2 years following issuance of the permit and shall require observance of appropriate pressure limitations under paragraph (d)(3) until all other corrective action measures have been implemented.

(2) New injection wells. No owner or operator of a new Class I hazardous waste injection well may begin injection until all corrective actions required under this section have been taken.

(3) The Director may require pressure limitations in lieu of plugging. If pressure limitations are used in lieu of plugging, the Director shall require as a permit condition that injection pressure be so limited that pressure in the injection zone at the site of any improperly completed or abandoned well within the area of review would not be sufficient to drive fluids into or between USDWs. This pressure limitation shall satisfy the corrective action requirement. Alternatively, such injection pressure limitation may be made part of a compliance schedule and may be required to be maintained until all other required corrective actions have been implemented.

(e) In determining the adequacy of corrective action proposed by the applicant under paragraph (c) of this section and in determining the additional steps needed to prevent fluid movement into and between USDWs, the following criteria and factors shall be considered by the Director:

(1) Nature and volume of injected fluid;

(2) Nature of native fluids or byproducts of injection;

(3) Geology;

(4) Hydrology;

(5) History of the injection operation;

(6) Completion and plugging records;

(7) Closure procedures in effect at the time the well was closed;

(8) Hydraulic connections with USDWs;

(9) Reliability of the procedures used to identify abandoned wells; and

(10) Any other factors which might affect the movement of fluids into or between USDWs.

§146.65 Construction requirements.

(a) *General.* All existing and new Class I hazardous waste injection wells shall be constructed and completed to:

(1) Prevent the movement of fluids into or between USDWs or into any unauthorized zones;

(2) Permit the use of appropriate testing devices and workover tools; and

(3) Permit continuous monitoring of injection tubing and long string casing as required pursuant to §146.67(f).

(b) *Compatibility.* All well materials must be compatible with fluids with which the materials may be expected to come into contact. A well shall be deemed to have compatibility as long as the materials used in the construction of the well meet or exceed standards developed for such materials by the American Petroleum Institute, The American Society for Testing Materials, or comparable standards acceptable to the Director.

(c) *Casing and Cementing of New Wells.* (1) Casing and cement used in the construction of each newly drilled well shall be designed for the life expectancy of the well, including the post-closure care period. The casing and cementing program shall be designed to prevent the movement of fluids into or

between USDWs, and to prevent potential leaks of fluids from the well. In determining and specifying casing and cementing requirements, the Director shall consider the following information as required by § 146.70:

(i) Depth to the injection zone;

(ii) Injection pressure, external pressure, internal pressure and axial loading;

(iii) Hole size;

(iv) Size and grade of all casing strings (well thickness, diameter, nominal weight, length, joint specification and construction material);

(v) Corrosiveness of injected fluid, formation fluids and temperature;

(vi) Lithology of injection and confining zones;

(vii) Type or grade of cement; and

(viii) Quantity and chemical composition of the injected fluid.

(2) One surface casing string shall, at a minimum, extend into the confining bed below the lowest formation that contains a USDW and be cemented by circulating cement from the base of the casing to the surface, using a minimum of 120% of the calculated annual volume. The Director may require more than 120% when the geology or other circumstances warrant it.

(3) At least one long string casing, using a sufficient number of centralizers, shall extend to the injection zone and shall be cemented by circulating cement to the surface in one or more stages:

(i) Of sufficient quantity and quality to withstand the maximum operating pressure; and

(ii) In a quantity no less than 120% of the calculated volume necessary to fill the annular space. The Director may require more than 120% when the geology or other circumstances warrant it.

(4) Circulation of cement may be accomplished by staging. The Director may approve an alternative method of cementing in cases where the cement cannot be recirculated to the surface, provided the owner or operator can demonstrate by using logs that the cement is continuous and does not allow fluid movement behind the well bore.

(5) Casings, including any casing connections, must be rated to have sufficient structural strength to withstand, for the design life of the well:

(i) The maximum burst and collapse pressures which may be experienced during the construction, operation and closure of the well; and

(ii) The maximum tensile stress which may be experienced at any point along the length of the casing during the construction, operation, and closure of the well.

(6) At a minimum, cement and cement additivies must be of sufficient quality and quantity to maintain integrity over the design life of the well.

(d) *Tubing and packer.* (1) All Class I hazardous waste injection wells shall inject fluids through tubing with a packer set at a point specified by the Director.

(2) In determining and specifying requirements for tubing and packer, the following factors shall be considered:

(i) Depth of setting;

(ii) Characteristics of injection fluid (chemical content, corrosiveness, temperature and density);

(iii) Injection pressure;

(iv) Annular pressure;

(v) Rate (intermittent or continuous), temperature and volume of injected fluid;

(vi) Size of casing; and

(vii) Tubing tensile, burst, and collapse strengths.

(3) The Director may approve the use of a fluid seal if he determines that the following conditions are met:

(i) The operator demonstrates that the seal will provide a level of protection comparable to a packer;

(ii) The operator demonstrates that the staff is, and will remain, adequately trained to operate and maintain the well and to identify and interpret variations in parameters of concern;

(iii) The permit contains specific limitations on variations in annular pressure and loss of annular fluid;

(iv) The design and construction of the well allows continuous monitoring of the annular pressure and mass balance of annular fluid; and

(v) A secondary system is used to monitor the interface between the annulus fluid and the injection fluid and the permit contains requirements for testing the system every three months and recording the results.

§146.66 Logging, sampling, and testing prior to new well operation.

(a) During the drilling and construction of a new Class I hazardous waste injection well, appropriate logs and tests shall be run to determine or verify the depth, thickness, porosity, permeability, and rock type of, and the salinity of any entrained fluids in, all relevant geologic units to assure conformance with performance standards in §146.65, and to establish accurate baseline data against which future measurements may be compared. A descriptive report interpreting results of such logs and tests shall be prepared by a knowledgeable log analyst and submitted to the Director. At a minimum, such logs and tests shall include:

(1) Deviation checks during drilling on all holes constructed by drilling a pilot hole which are enlarged by reaming or another method. Such checks shall be at sufficiently frequent intervals to determine the location of the borehole and to assure that vertical avenues for fluid movement in the form of diverging holes are not created during drilling; and

(2) Such other logs and tests as may be needed after taking into account the availability of similar data in the area of the drilling site, the construction plan, and the need for additional information that may arise from time to time as the construction of the well progresses. At a minimum, the following logs shall be required in the following situations:

(i) Upon installation of the surface casing:

(A) Resistivity, spontaneous potential, and caliper logs before the casing is installed; and

(B) A cement bond and variable density log, and a temperature log after the casing is set and cemented.

(ii) Upon installation of the long string casing:

(A) Resistivity, spontaneous potential, porosity, caliper, gamma ray, and fracture finder logs before the casing is installed; and

(B) A cement bond and variable density log, and a temperature log after the casing is set and cemented.

(iii) The Director may allow the use of an alternative to the above logs when an alternative will provide equivalent or better information; and

(3) A mechanical integrity test consisting of:

(i) A pressure test with liquid or gas;

(ii) A radioactive tracer survey;

(iii) A temperature or noise log;

(iv) A casing inspection log, if required by the Director; and

(v) Any other test required by the Director.

(b) Whole cores or sidewall cores of the confining and injection zones and formation fluid samples from the injection zone shall be taken. The Director may accept cores from nearby wells if the owner or operator can demonstrate that core retrieval is not possible and that such cores are representative of conditions at the well. The Director may require the owner or operator to core other formations in the borehole.

(c) The fluid temperature, pH, conductivity, pressure and the static fluid level of the injection zone must be recorded.

(d) At a minimum, the following information concerning the injection and confining zones shall be determined or calculated for Class I hazardous waste injection wells:

(1) Fracture pressure;

(2) Other physical and chemical characteristics of the injection and confining zones; and

(3) Physical and chemical characteristics of the formation fluids in the injection zone.

(e) Upon completion, but prior to operation, the owner or operator shall conduct the following tests to verify hydrogeologic characteristics of the injection zone:

(1) A pump test; or

(2) Injectivity tests.

(f) The Director shall have the opportunity to witness all logging and testing by this Subpart. The owner or operator shall submit a schedule of such activities to the Director 30 days prior to conducting the first test.

§146.67 Operating requirements.

(a) Except during stimulation, the owner or operator shall assure that injection pressure at the wellhead does not exceed a maximum which shall be calculated so as to assure that the pressure in the injection zone during

injection does not initiate new fractures or propagate existing fractures in the injection zone. The owner or operator shall assure that the injection pressure does not initiate fractures or propagate existing fractures in the confining zone, nor cause the movement of injection or formation fluids into a USDW.

(b) Injection between the outermost casing protecting USDWs and the well bore is prohibited.

(c) The owner or operator shall maintain an annulus pressure that exceeds the operating injection pressure, unless the Director determines that such a requirement might harm the integrity of the well. The fluid in the annulus shall be noncorrosive, or shall contain a corrosion inhibitor.

(d) The owner or operator shall maintain mechanical integrity of the injection well at all times.

(e) Permit requirements for owners or operators of hazardous waste wells which inject wastes which have the potential to react with the injection formation to generate gases shall include:

(1) Conditions limiting the temperature, pH or acidity of the injected waste; and

(2) Procedures necessary to assure that pressure imbalances which might cause a backflow or blowout do not occur.

(f) The owner or operator shall install and use continuous recording devices to monitor: the injection pressure; the flow rate, volume, and temperature of injected fluids; and the pressure on the annulus between the tubing and the long string casing, and shall install and use:

(1) Automatic alarm and automatic shut-off systems, designed to sound and shut-in the well when pressures and flow rates or other parameters approved by the Director exceed a range and/or gradient specified in the permit; or

(2) Automatic alarms, designed to sound when the pressures and flow rates or other parameters approved by the Director exceed a rate and/or gradient specified in the permit, in cases where the owner or operator certifies that a trained operator will be on-site at all times when the well is operating.

(g) If an automatic alarm or shutdown is triggered, the owner or operator shall immediately investigate and identify as expeditiously as possible the cause of the alarm or shutoff. If, upon such investigation, the well appears to be lacking mechanical integrity, or if monitoring required under paragraph (f) of this section otherwise indicates that the well may be lacking mechanical integrity, the owner or operator shall:

(1) Cease injection of waste fluids unless authorized by the Director to continue or resume injection.

(2) Take all necessary steps to determine the presence or absence of a leak; and

(3) Notify the Director within 24 hours after the alarm or shutdown.

(h) If a loss of mechanical integrity is discovered pursuant to paragraph (g) of this section or during periodic mechanical integrity testing, the owner or operator shall:

(1) Immediately cease injection of waste fluids;

(2) Take all steps reasonably necessary to determine whether there may have been a release of hazardous wastes or hazardous waste constituents into any unauthorized zone;

(3) Notify the Director within 24 hours after loss of mechanical integrity is discovered;

(4) Notify the Director when injection can be expected to resume; and

(5) Restore and demonstrate mechanical integrity to the satisfaction of the Director prior to resuming injection of waste fluids.

(i) Whenever the owner or operator obtains evidence that there may have been a release of injected wastes into an unauthorized zone:

(1) The owner or operator shall immediately case injection of waste fluids, and:

(i) Notify the Director within 24 hours of obtaining such evidence;

(ii) Take all necessary steps to identify and characterize the extent of any release;

(iii) Comply with any remediation plan specified by the Director;

(iv) Implement any remediation plan approved by the Director; and

(v) Where such release is into a USDW currently serving as a water

supply, place a notice in a newspaper of general circulation.

(2) The Director may allow the operator to resume injection prior to completing cleanup action if the owner or operator demonstrates that the injection operation will not endanger USDWs.

(j) The owner or operator shall notify the Director and obtain his approval prior to conducting any well workover.

§146.68 Testing and monitoring requirements.

Testing and monitoring requirements shall at a minimum include:

(a) Monitoring of the injected wastes. (1) The owner or operator shall develop and follow an approved written waste analysis plan that describes the procedures to be carried out to obtain a detailed chemical and physical analysis of a representative sample of the waste, including the quality assurance procedures used. At a minimum, the plan shall specify:

(i) The paramenters for which the waste will be analyzed and the rationale for the selection of these parameters;

(ii) The test methods that will be used to test for these parameters; and

(iii) The sampling method that will be used to obtain a representative sample of the waste to be analyzed.

(2) The owner or operator shall repeat the analysis of the injected wastes as described in the waste analysis plan at frequencies specified in the waste analysis plan and when process or operating changes occur that may significantly alter the characteristics of the waste stream.

(3) The owner or operator shall conduct continuous or periodic monitoring of selected parameters as required by the Director.

(4) The owner or operator shall assure that the plan remains accurate and the analyses remain representative.

(b) Hydrogeologic compatibility determination. The owner or operator shall submit information demonstrating to the satisfaction of the Director that the waste stream and its anticipated reaction products will not alter the permeability, thickness or other relevant characteristics of the confining or injection zones such that they would no longer meet the requirements specified in §146.62.

(c) Compatibility of well materials. (1) The owner or operator shall demonstrate that the waste stream will be compatible with the well materials with which the waste is expected to come into contact, and submit to the Director a description of the methodology used to make that determination. Compatibility for purposes of this requirement is established if contact with injected fluids will not cause the well materials to fail to satisfy any design requirement imposed under §146.65(b).

(2) The Director shall require continuous corrosion monitoring of the construction materials used in the well for wells injecting corrosive waste, and may require such monitoring for other waste, by:

(i) Placing coupons of the well construction materials in contact with the waste stream; or

(ii) Routing the waste stream through a loop constructed with the material used in the well; or

(iii) Using an alternative method approved by the Director.

(3) If a corrosion monitoring program is required:

(i) The test shall use materials identical to those used in the construction of the well, and such materials must be continuously exposed to the operating pressures and temperatures (measured at the well head) and flow rates of the injection operation; and

(ii) The owner or operator shall monitor the materials for loss of mass, thickness, cracking, pitting and other signs of corrosion on a quarterly basis to ensure that the well components meet the minimum standards for material strength and performance set forth in §146.65(b).

(d) *Periodic mechanical integrity testing.* In fulfilling the requirements of §146.8, the owner or operator of a Class I hazardous waste injection well shall conduct the mechanical integrity testing as follows:

(1) The long string casing, injection tube, and annular seal shall be tested by means of an approved pressure test with a liquid or gas annually and whenever there has been a well workover;

(2) The bottom-hole cement shall be tested by means of an approved radioactive tracer survey annually;

(3) An approved temperature, noise, or other approved log shall be run at least once every five years to test for movement of fluid along the borehole. The Director may require such tests whenever the well is worked over;

(4) Casing inspection logs shall be run whenever the owner or operator conducts a workover in which the injection string is pulled, unless the Director waives this requirement due to well construction or other factors which limit the test's reliability, or based upon the satisfactory results of a casing inspection log run within the previous five years. The Director may require that a casing inspection log be run every five years, if he has reason to believe that the integrity of the long string casing of the well may be adversely affected by naturally-occurring or man-made events;

(5) Any other test approved by the Director in accordance with the procedures in § 146.8(d) may also be used.

(e) *Ambient monitoring.* (1) Based on a site-specific assessment of the potential for fluid movement from the well or injection zone, and on the potential value of monitoring wells to detect such movement, the Director shall require the owner or operator to develop a monitoring program. At a minimum, the Director shall require monitoring of the pressure buildup in the injection zone annually, including at a minimum, a shut down of the well for a time sufficient to conduct a valid observation of the pressure fall-off curve.

(2) When prescribing a monitoring system the Director may also require:

(i) Continuous monitoring for pressure changes in the first aquifer overlying the confining zone. When such a well is installed, the owner or operator shall, on a quarterly basis, sample the aquifer and analyze for constituents specified by the Director;

(ii) The use of indirect, geophysical techniques to determine the position of the waste front, the water quality in a formation designated by the Director, or to provide other site specific data;

(iii) Periodic monitoring of the ground water quality in the first aquifer overlying the injection zone;

(iv) Periodic monitoring of the ground water quality in the lowermost USDW; and

(v) Any additional monitoring necessary to determine whether fluids are moving into or between USDWs.

(f) The Director may require seismicity monitoring when he has reason to believe that the injection activity may have the capacity to cause seismic disturbances.

[53 FR 28148, July 26, 1988, as amended at 57 FR 46294, Oct. 7, 1992]

§ 146.69 Reporting requirements.

Reporting requirements shall, at a minimum, include:

(a) Quarterly reports to the Director containing:

(1) The maximum injection pressure;

(2) A description of any event that exceeds operating parameters for annulus pressure or injection pressure as specified in the permit;

(3) A description of any event which triggers an alarm or shutdown device required pursuant to § 146.67(f) and the response taken;

(4) The total volume of fluid injected;

(5) Any change in the annular fluid volume;

(6) The physical, chemical and other relevant characteristics of injected fluids; and

(7) The results of monitoring prescribed under § 146.68.

(b) Reporting, within 30 days or with the next quarterly report whichever comes later, the results of:

(1) Periodic tests of mechanical integrity;

(2) Any other test of the injection well conducted by the permittee if required by the Director; and

(3) Any well workover.

§ 146.70 Information to be evaluated by the Director.

This section sets forth the information which must be evaluated by the Director in authorizing Class I hazardous waste injection wells. For a new Class I hazardous waste injection well, the owner or operator shall submit all the information listed below as part of the permit application. For an existing or converted Class I hazardous waste injection well, the owner or operator shall submit all information listed

below as part of the permit application except for those items of information which are current, accurate, and available in the existing permit file. For both existing and new Class I hazardous waste injection wells, certain maps, cross-sections, tabulations of wells within the area of review and other data may be included in the application by reference provided they are current and readily available to the Director (for example, in the permitting agency's files) and sufficiently identifiable to be retrieved. In cases where EPA issues the permit, all the information in this section must be submitted to the Administrator or his designee.

(a) Prior to the issuance of a permit for an existing Class I hazardous waste injection well to operate or the construction or conversion of a new Class I hazardous waste injection well, the Director shall review the following to assure that the requirements of this part and part 144 are met:

(1) Information required in § 144.31;

(2) A map showing the injection well for which a permit is sought and the applicable area of review. Within the area of review, the map must show the number or name and location of all producing wells, injection wells, abandoned wells, dry holes, surface bodies of water, springs, mines (surface and subsurface), quarries, water wells and other pertinent surface features, including residences and roads. The map should also show faults, if known or suspected;

(3) A tabulation of all wells within the area of review which penetrate the proposed injection zone or confining zone. Such data shall include a description of each well's type, construction, date drilled, location, depth, record of plugging and/or completion and any additional information the Director may require;

(4) The protocol followed to identify, locate and ascertain the condition of abandoned wells within the area of review which penetrate the injection or the confining zones;

(5) Maps and cross-sections indicating the general vertical and lateral limits of all underground sources of drinking water within the area of review, their position relative to the injection formation and the direction of water movement, where known, in each underground source of drinking water which may be affected by the proposed injection;

(6) Maps and cross-sections detailing the geologic structure of the local area;

(7) Maps and cross-sections illustrating the regional geologic setting;

(8) Proposed operating data;

(i) Average and maximum daily rate and volume of the fluid to be injected; and

(ii) Average and maximum injection pressure;

(9) Proposed formation testing program to obtain an analysis of the chemical, physical and radiological characteristics of and other information on the injection formation and the confining zone;

(10) Proposed stimulation program;

(11) Proposed injection procedure;

(12) Schematic or other appropriate drawings of the surface and subsurface construction details of the well;

(13) Contingency plans to cope with all shut-ins or well failures so as to prevent migration of fluids into any USDW;

(14) Plans (including maps) for meeting monitoring requirements of § 146.68;

(15) For wells within the area of review which penetrate the injection zone or the confining zone but are not properly completed or plugged, the corrective action to be taken under § 146.64;

(16) Construction procedures including a cementing and casing program, well materials specifications and their life expectancy, logging procedures, deviation checks, and a drilling, testing and coring program; and

(17) A demonstration pursuant to part 144, subpart F, that the applicant has the resources necessary to close, plug or abandon the well and for post-closure care.

(b) Prior to the Director's granting approval for the operation of a Class I hazardous waste injection well, the owner or operator shall submit and the Director shall review the following information, which shall be included in the completion report:

(1) All available logging and testing program data on the well;

(2) A demonstration of mechanical integrity pursuant to § 146.68;

(3) The anticipated maximum pressure and flow rate at which the permittee will operate;

(4) The results of the injection zone and confining zone testing program as required in § 146.70(a)(9);

(5) The actual injection procedure;

(6) The compatibility of injected waste with fluids in the injection zone and minerals in both the injection zone and the confining zone and with the materials used to construct the well;

(7) The calculated area of review based on data obtained during logging and testing of the well and the formation, and where necessary revisions to the information submitted under § 146.70(a) (2) and (3).

(8) The status of corrective action on wells identified in § 146.70(a)(15).

(c) Prior to granting approval for the plugging and abandonment (i.e., closure) of a Class I hazardous waste injection well, the Director shall review the information required in §§ 146.71(a)(4) and 146.72(a).

(d) Any permit issued for a Class I hazardous waste injection well for disposal on the premises where the waste is generated shall contain a certification by the owner or operator that:

(1) The generator of the hazardous waste has a program to reduce the volume or quantity and toxicity of such waste to the degree determined by the generator to be economically practicable; and

(2) Injection of the waste is that practicable method of disposal currently available to the generator which minimizes the present and future threat to human health and the environment.

§ 146.71 Closure.

(a) *Closure Plan*. The owner or operator of a Class I hazardous waste injection well shall prepare, maintain, and comply with a plan for closure of the well that meets the requirements of paragraph (d) of this section and is acceptable to the Director. The obligation to implement the closure plan survives the termination of a permit or the cessation of injection activities. The requirement to maintain and implement an approved plan is directly enforceable regardless of whether the requirement is a condition of the permit.

(1) The owner or operator shall submit the plan as a part of the permit application and, upon approval by the Director, such plan shall be a condition of any permit issued.

(2) The owner or operator shall submit any proposed significant revision to the method of closure reflected in the plan for approval by the Director no later than the date on which notice of closure is required to be submitted to the Director under paragraph (b) of this section.

(3) The plan shall assure financial responsibility as required in § 144.52(a)(7).

(4) The plan shall include the following information:

(i) The type and number of plugs to be used;

(ii) The placement of each plug including the elevation of the top and bottom of each plug;

(iii) The type and grade and quantity of material to be used in plugging;

(iv) The method of placement of the plugs;

(v) Any proposed test or measure to be made;

(vi) The amount, size, and location (by depth) of casing and any other materials to be left in the well;

(vii) The method and location where casing is to be parted, if applicable;

(viii) The procedure to be used to meet the requirements of paragraph (d)(5) of this section;

(ix) The estimated cost of closure; and

(x) Any proposed test or measure to be made.

(5) The Director may modify a closure plan following the procedures of § 124.5.

(6) An owner or operator of a Class I hazardous waste injection well who ceases injection temporarily, may keep the well open provided he:

(i) Has received authorization from the Director; and

(ii) Has described actions or procedures, satisfactory to the Director, that the owner or operator will take to ensure that the well will not endanger USDWs during the period of temporary disuse. These actions and procedures

shall include compliance with the technical requirements applicable to active injection wells unless waived by the Director.

(7) The owner or operator of a well that has ceased operations for more than two years shall notify the Director 30 days prior to resuming operation of the well.

(b) *Notice of intent to close.* The owner or operator shall notify the Director at least 60 days before closure of a well. At the discretion of the Director, a shorter notice period may be allowed.

(c) *Closure report.* Within 60 days after closure or at the time of the next quarterly report (whichever is less) the owner or operator shall submit a closure report to the Director. If the quarterly report is due less than 15 days after completion of closure, then the report shall be submitted within 60 days after closure. The report shall be certified as accurate by the owner or operator and by the person who performed the closure operation (if other than the owner or operator). Such report shall consist of either:

(1) A statement that the well was closed in accordance with the closure plan previously submitted and approved by the Director; or

(2) Where actual closure differed from the plan previously submitted, a written statement specifying the differences between the previous plan and the actual closure.

(d) *Standards for well closure.* (1) Prior to closing the well, the owner or operator shall observe and record the pressure decay for a time specified by the Director. The Director shall analyze the pressure decay and the transient pressure observations conducted pursuant to §146.68(e)(1)(i) and determine whether the injection activity has conformed with predicted values.

(2) Prior to well closure, appropriate mechanical integrity testing shall be conducted to ensure the integrity of that portion of the long string casing and cement that will be left in the ground after closure. Testing methods may include:

(i) Pressure tests with liquid or gas;

(ii) Radioactive tracer surveys;

(iii) Noise, temperature, pipe evaluation, or cement bond logs; and

(iv) Any other test required by the Director.

(3) Prior to well closure, the well shall be flushed with a buffer fluid.

(4) Upon closure, a Class I hazardous waste well shall be plugged with cement in a manner that will not allow the movement of fluids into or between USDWs.

(5) Placement of the cement plugs shall be accomplished by one of the following:

(i) The Balance Method;

(ii) The Dump Bailer Method;

(iii) The Two-Plug Method; or

(iv) An alternate method, approved by the Director, that will reliably provide a comparable level of protection.

(6) Each plug used shall be appropriately tagged and tested for seal and stability before closure is completed.

(7) The well to be closed shall be in a state of static equilibrium with the mud weight equalized top to bottom, either by circulating the mud in the well at least once or by a comparable method prescribed by the Director, prior to the placement of the cement plug(s).

§146.72 Post-closure care.

(a) The owner or operator of a Class I hazardous waste well shall prepare, maintain, and comply with a plan for post-closure care that meets the requirements of paragraph (b) of this section and is acceptable to the Director. The obligation to implement the post-closure plan survives the termination of a permit or the cessation of injection activities. The requirement to maintain an approved plan is directly enforceable regardless of whether the requirement is a condition of the permit.

(1) The owner or operator shall submit the plan as a part of the permit application and, upon approval by the Director, such plan shall be a condition of any permit issued.

(2) The owner or operator shall submit any proposed significant revision to the plan as appropriate over the life of the well, but no later than the date of the closure report required under §146.71(c).

(3) The plan shall assure financial responsibility as required in §146.73.

(4) The plan shall include the following information:

(i) The pressure in the injection zone before injection began;

(ii) The anticipated pressure in the injection zone at the time of closure;

(iii) The predicted time until pressure in the injection zone decays to the point that the well's cone of influence no longer intersects the base of the lowermost USDW;

(iv) Predicted position of the waste front at closure;

(v) The status of any cleanups required under § 146.64; and

(vi) The estimated cost of proposed post-closure care.

(5) At the request of the owner or operator, or on his own initiative, the Director may modify the post-closure plan after submission of the closure report following the procedures in § 124.5.

(b) The owner or operator shall:

(1) Continue and complete any cleanup action required under § 146.64, if applicable;

(2) Continue to conduct any groundwater monitoring required under the permit until pressure in the injection zone decays to the point that the well's cone of influence no longer intersects the base of the lowermost USDW. The Director may extend the period of post-closure monitoring if he determines that the well may endanger a USDW.

(3) Submit a survey plat to the local zoning authority designated by the Director. The plat shall indicate the location of the well relative to permanently surveyed benchmarks. A copy of the plat shall be submitted to the Regional Administrator of the appropriate EPA Regional Office.

(4) Provide appropriate notification and information to such State and local authorities as have cognizance over drilling activities to enable such State and local authorities to impose appropriate conditions on subsequent drilling activities that may penetrate the well's confining or injection zone.

(5) Retain, for a period of three years following well closure, records reflecting the nature, composition and volume of all injected fluids. The Director shall require the owner or operator to deliver the records to the Director at the conclusion of the retention period, and the records shall thereafter be retained at a location designated by the Director for that purpose.

(c) Each owner of a Class I hazardous waste injection well, and the owner of the surface or subsurface property on or in which a Class I hazardous waste injection well is located, must record a notation on the deed to the facility property or on some other instrument which is normally examined during title search that will in perpetuity provide any potential purchaser of the property the following information:

(1) The fact that land has been used to manage hazardous waste;

(2) The name of the State agency or local authority with which the plat was filed, as well as the address of the Regional Environmental Protection Agency Office to which it was submitted;

(3) The type and volume of waste injected, the injection interval or intervals into which it was injected, and the period over which injection occurred.

§ 146.73 Financial responsibility for post-closure care.

The owner or operator shall demonstrate and maintain financial responsibility for post-closure by using a trust fund, surety bond, letter of credit, financial test, insurance or corporate guarantee that meets the specifications for the mechanisms and instruments revised as appropriate to cover closure and post-closure care in 40 CFR part 144, subpart F. The amount of the funds available shall be no less than the amount identified in § 146.72(a)(4)(vi). The obligation to maintain financial responsibility for post-closure care survives the termination of a permit or the cessation of injection. The requirement to maintain financial responsibility is enforceable regardless of whether the requirement is a condition of the permit.

PART 147—STATE UNDERGROUND INJECTION CONTROL PROGRAMS

Subpart A—General Provisions

Sec.

147.1 Purpose and scope.
147.2 Severability of provisions.

Subpart B—Alabama

147.50 State-administered program—Class II wells.
147.51 State-administered program—Class I, III, IV, and V wells.
147.60 EPA-administered program—Indian lands.

Subpart C—Alaska

147.100 State-administered program—Class II wells.
147.101 EPA-administered program.
147.102 Aquifer exemptions.
147.103 Existing Class I, II (except enhanced recovery and hydrocarbon storage) and III wells authorized by rule.
147.104 Existing Class II enhanced recovery and hydrocarbon storage wells authorized by rule.

Subpart D—Arizona

147.150 State-administered program. [Reserved]
147.151 EPA-administered program.
147.152 Aquifer exemptions. [Reserved]

Subpart E—Arkansas

147.200 State-administered program—Class I, III, IV, and V wells.
147.201 State-administered program—Class II wells. [Reserved]
147.205 EPA-administered program—Indian lands.

Subpart F—California

147.250 State-administered program—Class II wells.
147.251 EPA-administered program—Class I, III, IV, and V wells and Indian lands.
147.252 Aquifer exemptions. [Reserved]
147.253 Existing Class I, II (except enhanced recovery and hydrocarbon storage) and III wells authorized by rule.

Subpart G—Colorado

147.300 State-administered program—Class II wells.
147.301 EPA-administered program—Class I, III, IV, V wells and Indian lands.
147.302 Aquifer exemptions.
147.303 Existing Class I, II (except enhanced recovery and hydrocarbon storage) and III wells authorized by rule.
147.304 Existing Class II enhanced recovery and hydrocarbon storage wells authorized by rule.
147.305 Requirements for all wells.

Subpart H—Connecticut

147.350 State–administered program.
147.351—147.352 [Reserved]
147.353 EPA-administered program—Indian lands.
147.354—147.359 [Reserved]

Subpart I—Delaware

147.400 State-administered program.
147.401—147.402 [Reserved]
147.403 EPA-administered program—Indian lands.
147.404—147.449 [Reserved]

Subpart J—District of Columbia

147.450 State-administered program. [Reserved]
147.451 EPA-administered program.
147.452 Aquifer exemptions. [Reserved]

Subpart K—Florida

147.500 State-administered program—Class I, III, IV, and V wells.
147.501 EPA-administered program—Class II wells and Indian lands.
147.502 Aquifer exemptions. [Reserved]
147.503 Existing Class II (except enhanced recovery and hydrocarbon storage) wells authorized by rule.
147.504 Existing Class II enhanced recovery and hydrocarbon storage wells authorized by rule.

Subpart L—Georgia

147.550 State-administered program.
147.551—147.552 [Reserved]
147.553 EPA-administered program—Indian lands.
147.554—147.559 [Reserved]

Subpart M—Hawaii

147.600 State-administered program. [Reserved]
147.601 EPA-administered program.

Subpart N—Idaho

147.650 State-administrative program—Class I, II, III, IV, and V wells.
147.651 EPA-administered program—Indian lands.
147.652 Aquifer exemptions. [Reserved]

Subpart O—Illinois

147.700 State-administered program—Class I, III, IV, and V wells.
147.701 State-administered program—Class II wells.
147.703 EPA-administered program—Indian lands.

Subpart P—Indiana

147.750 State-administered program—Class II wells.
147.751 EPA-administered program.

147.752 Aquifer exemptions. [Reserved]
147.753 Existing Class I and III wells authorized by rule.

Subpart Q—Iowa

147.800 State-administered program. [Reserved]
147.801 EPA-administered program.
147.802 Aquifer exemptions. [Reserved]

Subpart R—Kansas

147.850 State-administered program—Class I, III, IV and V wells.
147.851 State-administered program—Class II wells.
147.852—147.859 [Reserved]
147.860 EPA-administered program—Indian lands.

Subpart S—Kentucky

147.900 State-administered program. [Reserved]
147.901 EPA-administered program.
147.902 Aquifer exemptions. [Reserved]
147.903 Existing Class I, II (except enhanced recovery and hydrocarbon storage) and III wells authorized by rule.
147.904 Existing Class II enhanced recovery and hydrocarbon storage wells authorized by rule.
147.905 Requirements for all wells—area of review.

Subpart T—Louisiana

147.950 State-administered program.
147.951 EPA-administered program—Indian lands.

Subpart U—Maine

147.1000 State-administered program.
147.1001 EPA-administered program—Indian lands.

Subpart V—Maryland

147.1050 State-administered program—Class I, II, III, IV, and V wells.
147.1051—147.1052 [Reserved]
147.1053 EPA-administered program—Indian lands.
147.1054—147.1099 [Reserved]

Subpart W—Massachusetts

147.1100 State-administered program.
147.1101 EPA-administered program—Indian lands.

Subpart X—Michigan

147.1150 State-administered program. [Reserved]
147.1151 EPA-administered program.
147.1152 Aquifer exemptions. [Reserved]
147.1153 Existing Class I, II (except enhanced recovery and hydrocarbon storage) and III wells authorized by rule.
147.1154 Existing Class II enhanced recovery and hydrocarbon storage wells authorized by rule.
147.1155 Requirements for all wells.

Subpart Y—Minnesota

147.1200 State-administered program. [Reserved]
147.1201 EPA-administered program.
147.1202 Aquifer exemptions. [Reserved]
147.1210 Requirements for Indian lands.

Subpart Z—Mississippi

147.1250 State-administered program—Class I, III, IV, and V wells.
147.1251 State-administered program—Class II wells.
147.1252 EPA-administered program—Indian lands.

Subpart AA—Missouri

147.1300 State-administered program.
147.1301 State-administered program—Class I, III, IV, and V wells.
147.1302 Aquifer exemptions. [Reserved]
147.1303 EPA-administered program—Indian lands.

Subpart BB—Montana

147.1350 State-administered programs—Class II wells
147.1351 EPA-administered program.
147.1352 Aquifer exemptions.
147.1353 Existing Class I, II (except enhanced recovery and hydrocarbon storage) and III wells authorized by rule.
147.1354 Existing Class II enhanced recovery and hydrocarbon storage wells authorized by rule.
147.1355 Requirements for all wells.
APPENDIX A TO SUBPART BB OF PART 147—STATE REQUIREMENTS INCORPORATED BY REFERENCE IN SUBPART BB OF PART 147 OF THE CODE OF FEDERAL REGULATIONS.

Subpart CC—Nebraska

147.1400 State-administered program—Class II wells.
147.1401 State-administered program—Class I, III, IV, and V wells.
147.1402 Aquifer exemptions. [Reserved]
147.1403 EPA-administered program—Indian lands.

Subpart DD—Nevada

147.1450 State-administered program.
147.1451 EPA-administered program—Indian lands.
147.1452 Aquifer exemptions. [Reserved]
147.1453 Existing Class I, II (except enhanced recovery and hydrocarbon storage) and III wells authorized by rule.

147.1454 Existing Class II enhanced recovery and hydrocarbon storage wells authorized by rule.

Subpart EE—New Hampshire

147.1500 State-administered program.
147.1501 EPA-administered program—Indian lands.

Subpart FF—New Jersey

147.1550 State-administered program.
147.1551 EPA-administered program—Indian lands.

Subpart GG—New Mexico

147.1600 State-administered program—Class II wells.
147.1601 State-administered program—Class I, III, IV and V wells.
147.1603 EPA-administered program—Indian lands.

Subpart HH—New York

147.1650 State-administered program. [Reserved]
147.1651 EPA-administered program.
147.1652 Aquifer exemptions.
147.1653 Existing Class I, II (except enhanced recovery and hydrocarbon storage) and III wells authorized by rule.
147.1654 Existing Class II enhanced recovery and hydrocarbon storage wells authorized by rule.
147.1655 Requirements for wells authorized by permit.

Subpart II—North Carolina

147.1700 State-administered program.
147.1701—147.1702 [Reserved]
147.1703 EPA-administered program—Indian lands.
147.1704—147.1749 [Reserved]

Subpart JJ—North Dakota

147.1750 State-administered program—Class II wells.
147.1751 State-administered program—Class I, III, IV and V wells.
147.1752 EPA-administered program—Indian lands.

Subpart KK—Ohio

147.1800 State-administered program—Class II wells.
147.1801 State-administered program—Class I, III, IV and V wells.
147.1802 Aquifer exemptions. [Reserved]
147.1803 Existing Class I and III wells authorized by rule—maximum injection pressure.
147.1805 EPA-administered program—Indian lands.

Subpart LL—Oklahoma

147.1850 State-administered program—Class I, III, IV and V wells.
147.1851 State-administered program—Class II wells.
147.1852 EPA-administered program—Indian lands.

Subpart MM—Oregon

147.1900 State-administered program.
147.1901 EPA-administered program—Indian lands.

Subpart NN—Pennsylvania

147.1950 State-administered program. [Reserved]
147.1951 EPA-administered program.
147.1952 Aquifer exemptions.
147.1953 Existing Class I, II (except enhanced recovery and hydrocarbon storage) and III wells authorized by rule.
147.1954 Existing Class II enhanced recovery and hydrocarbon storage wells authorized by rule.
147.1955 Requirements for wells authorized by permit.

Subpart OO—Rhode Island

147.2000 State-administered program—Class I, II, III, IV, and V wells.
147.2001 EPA-administered program—Indian lands.

Subpart PP—South Carolina

147.2050 State-administered program.
147.2051 EPA-administered program—Indian lands.

Subpart QQ—South Dakota

147.2100 State-administered program—Class II wells.
147.2101 EPA-administered program—Class I, III, IV and V wells and all wells on Indian lands.
147.2102 Aquifer exemptions.
147.2103 Existing Class II enhanced recovery and hydrocarbon storage wells authorized by rule.
147.2104 Requirements for all wells.

Subpart RR—Tennessee

147.2150 State-administered program. [Reserved]
147.2151 EPA-administered program.
147.2152 Aquifer exemptions. [Reserved]
147.2153 Existing Class I, II (except enhanced recovery and hydrocarbon storage) and III wells authorized by rule.

147.2154 Existing Class II enhanced recovery and hydrocarbon storage wells authorized by rule.
147.2155 Requirements for all wells—area of review.

Subpart SS—Texas

147.2200 State-administered program—Class I, III, IV, and V wells.
147.2201 State-administered program—Class II wells.
147.2205 EPA-administered program—Indian lands.

Subpart TT—Utah

147.2250 State-administered program—Class I, III, IV, and V wells.
147.2251 State-administered program—Class II wells.
147.2253 EPA-administered program—Indian lands.

Subpart UU—Vermont

147.2300 State-administered program.
147.2301—147.2302 [Reserved]
147.2303 EPA-administered program—Indian lands.

Subpart VV—Virginia

147.2350 State-administered program. [Reserved]
147.2351 EPA-administered program.
147.2352 Aquifer exemptions. [Reserved]

Subpart WW—Washington

147.2400 State-administered program—Class I, II, III, IV, and V wells.
147.2403 EPA-administered program—Indian lands.
147.2404 EPA-administered program—Colville Reservation.

Subpart XX—West Virginia

147.2450—147.2452 [Reserved]
147.2453 EPA-administered program—Indian lands.
147.2454—147.2499 [Reserved]

Subpart YY—Wisconsin

147.2500 State-administered program.
147.2510 EPA-administered program—Indian lands.

Subpart ZZ—Wyoming

147.2550 State-administered program—Class I, III, IV, and V wells.
147.2551 State-administered program—Class II wells.
147.2553 EPA-administered program—Indian lands.
147.2554 Aquifer exemptions.

Subpart AAA—Guam

147.2600 State-administered program.
147.2601 EPA-administered program—Indian lands.

Subpart BBB—Puerto Rico

147.2650 State-administered program—Class I, II, III, IV, and V wells.
147.2651 EPA-administered program—Indian lands.

Subpart CCC—Virgin Islands

147.2700 State-administered program. [Reserved]
147.2701 EPA-administered program.

Subpart DDD—American Samoa

147.2750 State administered program. [Reserved]
147.2751 EPA-administered program.
147.2752 Aquifer exemptions. [Reserved]

Subpart EEE—Commonwealth of the Northern Mariana Islands

147.2800 State-administered program—Class I, II, III, IV, and V wells.
147.2801 EPA-administered program.
147.2802 Aquifer exemptions. [Reserved]

Subpart FFF—Trust Territory of the Pacific Islands

147.2850 State-administered program. [Reserved]
147.2851 EPA-administered program.
147.2852 Aquifer exemptions. [Reserved]

Subpart GGG—Osage Mineral Reserve—Class II Wells

147.2901 Applicability and scope.
147.2902 Definitions.
147.2903 Prohibition of unauthorized injection.
147.2904 Area of review.
147.2905 Plugging and abandonment.
147.2906 Emergency permits.
147.2907 Confidentiality of information.
147.2908 Aquifer exemptions.
147.2909 Authorization of existing wells by rule.
147.2910 Duration of authorization by rule.
147.2911 Construction requirements for wells authorized by rule.
147.2912 Operating requirements for wells authorized by rule.
147.2913 Monitoring and reporting requirements for wells authorized by rule.
147.2914 Corrective action for wells authorized by rule.
147.2915 Requiring a permit for wells authorized by rule.

147.2916 Coverage of permitting requirements.
147.2917 Duration of permits.
147.2918 Permit application information.
147.2919 Construction requirements for wells authorized by permit.
147.2920 Operating requirements for wells authorized by permit.
147.2921 Schedule of compliance.
147.2922 Monitoring and reporting requirements for wells authorized by permit.
147.2923 Corrective action for wells authorized by permit.
147.2924 Area permits.
147.2925 Standard permit conditions.
147.2926 Permit transfers.
147.2927 Permit modification.
147.2928 Permit termination.
147.2929 Administrative permitting procedures.

Subpart HHH—Lands of the Navajo, Ute Mountain Ute, and All Other New Mexico Tribes

147.3000 EPA-administered program.
147.3001 Definition.
147.3002 Public notice of permit actions.
147.3003 Aquifer exemptions.
147.3004 Duration of rule authorization for existing Class I and III wells.
147.3005 Radioactive waste injection wells.
147.3006 Injection pressure for existing Class II wells authorized by rule.
147.3007 Application for a permit.
147.3008 Criteria for aquifer exemptions.
147.3009 Area of review.
147.3010 Mechanical integrity tests.
147.3011 Plugging and abandonment of Class III wells.
147.3012 Construction requirements for Class I wells.
147.3013 Information to be considered for Class I wells.
147.3014 Construction requirements for Class III wells.
147.3015 Information to be considered for Class III wells.
147.3016 Criteria and standards applicable to Class V wells.
APPENDIX A TO SUBPART HHH—EXEMPTED AQUIFERS IN NEW MEXICO.

Subpart III—Lands of Certain Oklahoma Indian Tribes

147.3100 EPA-administered program.
147.3101 Public notice of permit actions.
147.3102 Plugging and abandonment plans.
147.3103 Fluid seals.
147.3104 Notice of abandonment.
147.3105 Plugging and abandonment report.
147.3106 Area of review.
147.3107 Mechanical integrity.
147.3108 Plugging Class I, II, and III wells.
147.3109 Timing of mechanical integrity test.

AUTHORITY: 42 U.S.C. 300h; and 42 U.S.C. 6901 *et seq.*

SOURCE: 49 FR 20197, May 11, 1984, unless otherwise noted.

Subpart A—General Provisions

§147.1 Purpose and scope.

(a) This part sets forth the applicable Underground Injection Control (UIC) programs for each of the states, territories, and possessions identified pursuant to the Safe Drinking Water Act (SDWA) as needing a UIC program.

(b) The applicable UIC program for a State is either a State-administered program approved by EPA, or a federally-administered program promulgated by EPA. In some cases, the UIC program may consist of a State-administered program applicable to some classes of wells and a federally-administered program applicable to other classes of wells. Approval of a State program is based upon a determination by the Administrator that the program meets the requirements of section 1422 or section 1425 of the Safe Drinking Water Act and the applicable provisions of parts 124, 144, and 146 of this chapter. A federally-administered program is promulgated in those instances where the state has failed to submit a program for approval or where the submitted program does not meet the minimum statutory and regulatory requirements.

(c) In the case of State programs approved by EPA pursuant to section 1422 of the SDWA, each State subpart describes the major elements of such programs, including State statutes and regulations, Statement of Legal Authority, Memorandum of Agreement, and Program Description. State statutes and regulations that contain standards, requirements, and procedures applicable to owners or operators have been incorporated by reference pursuant to regulations of the Office of the Federal Register. Material incorporated by reference is available for inspection in the appropriate EPA Regional Office, in EPA Headquarters, and at the Office of the Federal Register Information Center, Room 8301, 800 North Capitol Street, NW., suite

700, Washington, DC. Other State statutes and regulations containing standards and procedures that constitute elements of the State program but do not apply directly to owners or operators have been listed but have not been incorporated by reference.

(d) In the case of State programs promulgated under section 1422 that are to be administered by EPA, the State subpart makes applicable the provisions of parts 124, 144, and 146, and provides additional requirements pertinent to the specific State program.

(e) Regulatory provisions incorporated by reference (in the case of approved State programs) or promulgated by EPA (in the case of EPA-administered programs), and all permit conditions or permit denials issued pursuant to such regulations, are enforceable by the Administrator pursuant to section 1423 of the SDWA.

(f) The information requirements located in the following sections have been cleared by the Office of Management and Budget: Sections 147.104, 147.304, 147.754, 147.904, 147.1154, 147.1354, 147.1454, 147.1654, 147.1954, and 147.2154.

The OMB clearance number is No. 2040–0042.

§ 147.2 Severability of provisions.

The provisions in this part and the various applications thereof are distinct and severable. If any provision of this part or the application thereof to any person or circumstances is held invalid, such invalidity shall not affect other provisions or application of such provision to other persons or circumstances which can be given effect without the invalid provision or application.

Subpart B—Alabama

§ 147.50 State-administered program—Class II wells.

The UIC program for Class II wells in the State of Alabama, except those on Indian lands, is the program administered by the State Oil and Gas Board of Alabama, approved by EPA pursuant to section 1425 of the SDWA. Notice of this approval was published in the FEDERAL REGISTER on August 2, 1982 (47 FR 33268); the effective date of this program is August 2, 1982. This program consists of the following elements, as submitted to EPA in the State's program application:

(a) *Incorporation by reference.* The requirements set forth in the State statutes and regulations cited in this paragraph are hereby incorporated by reference and made a part of the applicable UIC program under the SDWA for the State of Alabama. This incorporation by reference was approved by the Director of the Federal Register on June 25, 1984.

(1) Code of Alabama Sections 9–17–1 through 9–17–109 (Cumm. Supp. 1989);

(2) State Oil and Gas Board of Alabama Administrative Code, Oil and Gas Report 1 (supplemented through May 1989), Rules and Regulations Governing the Conservation of Oil and Gas in Alabama, and Oil and Gas Statutes of Alabama with Oil and Gas Board Forms, § 400–1–2 and § 400–1–5–.04.

(b) The Memorandum of Agreement between EPA Region IV and the Alabama Oil and Gas Board, signed by the EPA Regional Administrator on June 15, 1982.

(c) *Statement of legal authority.* ''State Oil and Gas Board has Authority to Carry Out Underground Injection Control Program Relating to Class II Wells as Described in Federal Safe Drinking Water Act—Opinion by Assistant Attorney General,'' May 28, 1982.

(d) The Program Description and any other materials submitted as part of the application or as supplements thereto.

[49 FR 20197, May 11, 1984, as amended at 53 FR 43086, Oct. 25, 1988; 56 FR 9411, Mar. 6, 1991]

§ 147.51 State-administered program—Class I, III, IV, and V wells.

The UIC program for Class I, III, IV and V wells in the State of Alabama, except those on Indian lands, is the program administered by the Alabama Department of Environmental Management, approved by EPA pursuant to section 1422 of the SDWA. Notice of this approval was published in the FEDERAL REGISTER on August 25, 1983 (48 FR 38640); the effective date of this program is August 25, 1983. This program consists of the following elements, as submitted to EPA in the State's program application:

(a) *Incorporation by reference.* The requirements set forth in the State statutes and regulations cited in this paragraph are hereby incorporated by reference and made a part of the applicable UIC program under the SDWA for the State of Alabama. This incorporation by reference was approved by the Director of the Federal Register on June 25, 1984.

(1) Alabama Water Pollution Control Act, Code of Alabama 1975, sections 22–22–1 through 22–22–14 (1980 and Supp. 1983);

(2) Regulations, Policies and Procedures of the Alabama Water Improvement Commission, Title I (Regulations) (Rev. December 1980), as amended May 17, 1982, to add Chapter 9, Underground Injection Control Regulations (effective June 10, 1982), as amended April 6, 1983 (effective May 11, 1983).

(b) The Memorandum of Agreement between EPA Region IV and the Alabama Department of Environment Management, signed by the EPA Regional Administrator on May 24, 1983.

(c) *Statement of legal authority.* (1) ''Water Pollution—Public Health—State has Authority to Carry Out Underground Injection Control Program Described in Federal Safe Drinking Water Act—Opinion by Legal Counsel for the Water Improvement Commission,'' June 25, 1982;

(2) Letter from Attorney, Alabama Water Improvement Commission, to Regional Administrator, EPA Region IV, ''Re: AWIC Response to Phillip Tate's (U.S. EPA, Washington) Comments on AWIC's Final Application for Class I, III, IV, and V UIC Program,'' September 21, 1982;

(3) Letter from Alabama Chief Assistant Attorney General to Regional Counsel, EPA Region IV, ''Re: Status of Independent Legal Counsel in Alabama Water Improvement Commission's Underground Injection Control Program,'' September 14, 1982.

(d) The Program Description and any other materials submitted as part of the application or as supplements thereto.

[49 FR 20197, May 11, 1984, as amended at 53 FR 43086, Oct. 25, 1988]

§ 147.60 EPA-administered program—Indian lands.

(a) *Contents.* The UIC program for all classes of wells on Indian lands in Alabama is administered by EPA. This program consists of the UIC program requirements of 40 CFR parts 124, 144, 146, 148 and any additional requirements set forth in the remainder of this subpart. Injection well owners and operators, and EPA shall comply with these requirements.

(b) *Effective date.* The effective date of the UIC program for Indian lands in Alabama is November 25, 1988.

[53 FR 43086, Oct. 25, 1988, as amended at 56 FR 9411, Mar. 6, 1991]

Subpart C—Alaska

§ 147.100 State-administered program—Class II wells.

The UIC program for Class II wells in the State of Alaska, other than those on Indian lands, is the program administered by the Alaska Oil and Gas Conservation Commission approved by EPA pursuant to section 1425 of the SDWA. Notice of this approval was published in the FEDERAL REGISTER [May 6, 1986]; the effective date of this program is June 19, 1986. This program consists of the following elements, as submitted to EPA in the State's program application.

(a) *Incorporation by reference.* The requirements set forth in the State statutes and regulations cited in this paragraph are hereby incorporated by reference and made a part of the applicable UIC program under the SDWA for the State of Alaska. This incorporation by reference was approved by the Director of the FEDERAL REGISTER effective June 19, 1986.

(1) Alaska Statutes, Alaska Oil and Gas Conservation Act, Title 31, §§ 31.05.005 through 31.30.010 (1979 and Cum. Supp. 1984);

(2) Alaska Statutes, Administrative Procedures Act, Title 44, §§ 44.62.010 through 44.62.650 (1984);

(3) Alaska Administrative Code, Alaska Oil and Gas Conservation Commission, 20 AAC 25.005 through 20 AAC 25.570 (Supp. 1986).

(b) *Memorandum of Agreement.* The Memorandum of Agreement between

EPA Region 10, and the Alaska Oil and Gas Conservation Commission, signed by the EPA Regional Administrator on January 29, 1986, as amended on June 21, 1988.

(c) *Statement of Legal Authority.* Statement from the Attorney General of the State of Alaska, signed by the Assistant Attorney General on December 10, 1985.

(d) The Program Description and any other materials submitted as part of the original application or as supplements thereto.

[51 FR 16684, May 6, 1986, as amended at 56 FR 9411, Mar. 6, 1991]

§ 147.101 EPA-administered program.

(a) *Contents.* The UIC program in the State of Alaska for Class I, III, IV, and V wells, and for all classes of wells on Indian lands, is administered by EPA. This program consists of the UIC program requirements of 40 CFR parts 124, 144, 146, 148, and any additional requirements set forth in the remainder of this subpart. Injection well owners and operators, and EPA shall comply with these requirements.

(b) *Effective dates.* The effective date of the UIC program for all non-Class II wells in Alaska and for all wells on Indian lands, is June 25, 1984.

[52 FR 17680, May 11, 1987, as amended at 56 FR 9412, Mar. 6, 1991]

§ 147.102 Aquifer exemptions.

(a) This section identifies any aquifers or their portions exempted in accordance with §§ 144.7(b) and 146.4 of this chapter at the time of program promulgation. EPA may in the future exempt other aquifers or portions, according to applicable procedures, without codifying such exemptions in this section. An updated list of exemptions will be maintained in the Regional office.

(b) The following aquifers are exempted in accordance with the provisions of §§ 144.7(b) and 146.4 of this chapter for Class II injection activities only:

(1) The portions of aquifers in the Kenai Peninsula, greater than the indicated depths below the ground surface, and described by a ¼ mile area beyond and lying directly below the following oil and gas producing fields:

(i) Swanson River Field—1700 feet.

(ii) Beaver Creek Field—1650 feet.

(iii) Kenai Gas Field—1300 feet.

(2) The portion of aquifers beneath Cook Inlet described by a ¼ mile area beyond and lying directly below the following oil and gas producing fields:

(i) Granite Point.

(ii) McArthur River Field.

(iii) Middle Ground Shoal Field.

(iv) Trading Bay Field.

(3) The portions of aquifers on the North Slope described by a ¼ mile area beyond and lying directly below the Kuparuk River Unit oil and gas producing field.

§ 147.103 Existing Class I, II (except enhanced recovery and hydrocarbon storage) and III wells authorized by rule.

Maximum injection pressure. The owner or operator shall limit injection pressure to the lesser of:

(a) A value which will not exceed the operating requirements of § 144.28(f)(3)(i) or (ii) as applicable; or

(b) A value for well head pressure calculated by using the following formula:

$$Pm=(0.733–0.433\ Sg)d$$

where

Pm=injection pressure at the well head in pounds per square inch

Sg=specific gravity of inject fluid (unitless)

d=injection depth in feet.

§ 147.104 Existing Class II enhanced recovery and hydrocarbon storage wells authorized by rule.

(a) *Maximum injection pressure.* (1) To meet the operating requirements of § 144.28(f)(3)(ii) (A) and (B) of this chapter, the owner or operator:

(i) Shall use an injection pressure no greater than the pressure established by the Regional Administrator for the field or formation in which the well is located. The Regional Administrator shall establish maximum injection pressures after notice, opportunity for comment, and opportunity for a public hearing, according to the provisions of part 124, subpart A of this chapter, and will inform owners and operators in writing of the applicable maximum pressure; or

(ii) May inject at pressures greater than those specified in paragraph (a)(1)(i) of this section for the field or formation in which he is operating provided he submits a request in writing to the Regional Administrator, and demonstrates to the satisfaction of the Regional Administrator that such injection pressure will not violate the requirement of § 144.28(f)(3)(ii) (A) and (B). The Regional Administrator may grant such a request after notice, opportunity for comment, and opportunity for a public hearing, according to the provisions of part 124, subpart A of this chapter.

(2) Prior to such time as the Regional Administrator establishes rules for maximum injection pressure based on data provided pursuant to paragraph (a)(2)(ii) of this section the owner or operator shall:

(i) Limit injection pressure to a value which will not exceed the operating requirements of § 144.28(f)(3)(ii); and

(ii) Submit data acceptable to the Regional Administrator which defines the fracture pressure of the formation in which injection is taking place. A single test may be submitted on behalf of two or more operators conducting operations in the same formation, if the Regional Administrator approves such submission. The data shall be submitted to the Regional Administrator within 1 year of the effective date of this program.

(b) *Casing and cementing.* Where the Regional Administrator determines that the owner or operator of an existing enhanced recovery or hydrocarbon storage well may not be in compliance with the requirements of §§ 144.28(e) and 146.22, the owner or operator shall comply with paragraphs (b) (1) through (4) of this section, when required by the Regional Administrator:

(1) Protect USDWs by:

(i) Cementing surface casing by recirculating the cement to the surface from a point 50 feet below the lowermost USDW; or

(ii) Isolating all USDWs by placing cement between the outermost casing and the well bore; and

(2) Isolate any injection zones by placing sufficient cement to fill the calculated space between the casing and the well bore to a point 250 feet above the injection zone; and

(3) Use cement:

(i) Of sufficient quantity and quality to withstand the maximum operating pressure;

(ii) Which is resistant to deterioration from formation and injection fluids; and

(iii) In a quantity no less than 120% of the calculated volume necessary to cement off a zone.

(4) The Regional Administrator may specify other requirements in addition to or in lieu of the requirements set forth in paragraphs (b) (1) through (3) as needed to protect USDWs.

Subpart D—Arizona

§ 147.150 State-administered program. [Reserved]

§ 147.151 EPA-administered program.

(a) *Contents.* The UIC program that applies to all injection activities in Arizona, including those on Indian lands, is administered by EPA. The UIC program for Navajo Indian lands consists of the requirements contained in subpart HHH of this part. The program for all injection activity except that on Navajo Indian lands consists of the UIC program requirements of 40 CFR parts 124, 144, 146, 148, and any additional requirements set forth in the remainder of this subpart. Injection well owners and operators, and EPA shall comply with these requirements.

(b) *Effective dates.* The effective date for the UIC program in Arizona, except for the lands of the Navajo Indians, is June 25, 1984. The effective date for the UIC program on the lands of the Navajo is November 25, 1988.

[53 FR 43086, Oct. 25, 1988, as amended at 56 FR 9412, Mar. 6, 1991]

§ 147.152 Aquifer exemptions. [Reserved]

Subpart E—Arkansas

§ 147.200 State-administered program—Class I, III, IV, and V wells.

The UIC program for Class I, III, IV and V wells in the State of Arkansas, except those wells on Indian lands, is

the program administered by the Arkansas Department of Pollution Control and Ecology approved by EPA pursuant to section 1422 of the SDWA. Notice of this approval was published in the FEDERAL REGISTER on July 6, 1982 (47 FR 29236); the effective date of this program is July 6, 1982. This program consists of the following elements, as submitted to EPA in the State's program application.

(a) *Incorporation by reference.* The requirements set forth in the State statutes and regulations cited in this paragraph are hereby incorporated by reference and made a part of the applicable UIC program under the SDWA for the State of Arkansas. This incorporation by reference was approved by the Director of the Federal Register on June 25, 1984.

(1) Arkansas Water and Air Pollution Control Act, Act 472 of 1949 as amended, Arkansas Statutes Annotated sections 82–1901 through 82–1943 (1976);

(2) Act 105 of 1939, Arkansas Statutes Annotated sections 53–101 through 53–130 (1971 and Supp. 1981); Act 937 of 1979, Arkansas Statutes Annotated sections 53–1301 through 53–1320 (Supp. 1981); Act 523 of 1981;

(3) Arkansas Underground Injection Control Code, Department of Pollution Control and Ecology, promulgated January 22, 1982;

(4) General Rule and Regulations, Arkansas Oil and Gas Commission (Order No. 2–39, revised July 1972);

(5) Arkansas Hazardous Waste Management Code, Department of Pollution Control and Ecology, promulgated August 21, 1981.

(b) The Memorandum of Agreement and Addendum No. 1 to the Memorandum of Agreement, between EPA Region VI and the Arkansas Department of Pollution Control and Ecology and the Arkansas Oil and Gas Commission, signed by the EPA Regional Administrator on May 25, 1982.

(c) *Statement of legal authority.* (1) Letter from Chief Attorney, Arkansas Department of Pollution Control and Ecology, to Acting Regional Administrator, EPA Region VI, ''Re: Legal Authority of the Department of Pollution Control and Ecology of the State of Arkansas to Administer an Underground Injection Control Program,'' July 29, 1981;

(2) Letter from Chief Attorney, Arkansas Department of Pollution Control and Ecology, to Acting Regional Counsel, EPA Region VI, ''Re: Addendum to Legal Statement—Underground Injection Control Program,'' October 13, 1981;

(3) Letter from General Counsel, Arkansas Oil and Gas Commission, to Acting Regional Counsel, EPA Region VI, ''Re: Supplemental Addendum to Legal Statement—Underground Injection Control Program,'' October 20, 1981;

(4) Letter from Chief Attorney, Arkansas Department of Pollution Control and Ecology, to Attorney, Office of Regional Counsel, EPA Region VI (re: status as independent legal counsel), December 31, 1981;

(5) Letter from General Counsel, Arkansas Oil and Gas Commission, to Acting Regional Counsel, EPA Region VI, ''Re: Supplemental Addendum to Legal Statement—Underground Injection Control Program,'' January 13, 1982;

(6) Letter from Chief Counsel, Arkansas Department of Pollution Control and Ecology, to Acting Regional Counsel, EPA Region VI, ''Re: Addendum to Legal Statement—Underground Injection Control Program,'' February 15, 1982;

(7) Letter from Chief Counsel, Arkansas Department of Pollution Control and Ecology, to Acting Regional Counsel, EPA Region VI, ''Re: Addendum to Legal Statement—Underground Injection Control Program,'' May 13, 1982.

(d) The Program Description and any other materials submitted as part of the application or as supplements thereto.

[49 FR 20197, May 11, 1984, as amended at 53 FR 43086, Oct. 25, 1988]

§ 147.201 State-administered program—Class II wells. [Reserved]

§ 147.205 EPA-administered program—Indian lands.

(a) *Contents.* The UIC program for all classes of wells on Indian lands in Arkansas is administered by EPA. This program consists of the UIC program requirements of 40 CFR parts 124, 144,

146, 148 and any additional requirements set forth in this subpart. Injection well owners and operators, and EPA shall comply with these requirements.

(b) *Effective date.* The effective date of the UIC program for Indian lands in Arkansas is November 25, 1988.

[53 FR 43086, Oct. 25, 1988, as amended at 56 FR 9412, Mar. 6, 1991]

Subpart F—California

§ 147.250 State-administered program—Class II wells.

The UIC program for Class II wells in the State of California, except those on Indian lands, is the program administered by the California Division of Oil and Gas, approved by EPA pursuant to SDWA section 1425.

(a) *Incorporation by reference.* The requirements set forth in the State statutes and regulations cited in this paragraph are hereby incorporated by reference and made a part of the applicable UIC program under the SDWA for the State of California. This incorporation by reference was approved by the Director of the Federal Register on June 25, 1984.

(1) California Laws for Conservation of Petroleum and Gas, California Public Resources Code Div. 3, Chapt. 1, §§ 3000–3359 (1989);

(2) California Administrative Code, title 14, §§ 1710 to 1724.10 (May 28, 1988).

(b) The Memorandum of Agreement between EPA Region IX and the California Division of Oil and Gas, signed by the EPA Regional Administrator on September 29, 1982.

(c) *Statement of legal authority.* (1) Letter from California Deputy Attorney General to the Administrator of EPA, ''Re: Legal Authority of California Division of Oil and Gas to Carry Out Class II Injection Well Program,'' April 1, 1981;

(2) Letter from California Deputy Attorney General to Chief of California Branch, EPA Region IX, ''Re: California Application for Primacy, Class II UIC Program,'' December 3, 1982.

(d) The Program Description and any other materials submitted as part of the application or as supplements thereto.

[49 FR 20197, May 11, 1984, as amended at 52 FR 17681, May 11, 1987; 56 FR 9412, Mar. 6, 1991]

§ 147.251 EPA-administered program—Class I, III, IV and V wells and Indian lands.

(a) *Contents.* The UIC program in the State of California for Class I, III, IV and V wells, and for all classes of wells on Indian lands, is administered by EPA. The program consists of the UIC program requirements of 40 CFR parts 124, 144, 146, 148, and any additional requirements set forth in the remainder of this subpart. Injection well owners and operators, and EPA shall comply with these requirements.

(b) *Effective dates.* The effective date for the UIC program for all lands in California, including Indian lands, is June 25, 1984.

[52 FR 17681, May 11, 1987, as amended at 56 FR 9412, Mar. 6, 1991]

§ 147.252 Aquifer exemptions. [Reserved]

§ 147.253 Existing Class I, II (except enhanced recovery and hydrocarbon storage) and III wells authorized by rule.

Maximum injection pressure. The owner or operator shall limit injection pressure to the lesser of:

(a) A value which will not exceed the operating requirements of § 144.28(f)(3)(i) or (ii) as applicable; or

(b) A value for well head pressure calculated by using the following formula:

$$Pm=(0.733-0.433\ Sg)d$$

where

Pm=injection pressure at the well head in pounds per square inch

Sg=specific gravity of inject fluid (unitless)

d=injection depth in feet.

Subpart G—Colorado

§ 147.300 State-administered program—Class II wells.

The UIC program for Class II wells in the State of Colorado, except those wells on Indian Lands, is the program administered by the Colorado Oil and

Gas Commission approved by EPA pursuant to section 1425 of the SDWA. Notice of this approval was published in the FR on April 2, 1984 (49 FR 13040); the effective date of this program is April 2, 1984. This program consists of the following elements, as submitted to EPA in the State's program application:

(a) *Incorporation by reference.* The requirements set forth in the State statutes and regulations cited in this paragraph are hereby incorporated by reference and made a part of the applicable UIC program under the SDWA for the State of Colorado. This incorporation by reference was approved by the Director of the OFR in accordance with 5 U.S.C. 552(a) and 1 CFR part 51. Copies may be obtained at the State of Colorado Oil and Gas Conservation Commission, Department of Natural Resources, Suite 380 Logan Tower Building, 1580 Logan Street, Denver, Colorado, 80203. Copies may be inspected at the Environmental Protection Agency, Region VIII, 999 18th Street, Suite 500, Denver, Colorado, 80202–2405, or at the Office of the Federal Register, 800 North Capitol Street NW., suite 700, Washington, DC.

(1) Colorado Revised Statutes, 1989 replacement volume, Section 34–60–101 through 34–60–123;

(2) Colorado Revised Statutes, 1989 replacement volume, Section 25–8–101 through 25–8–612;

(3) Rules and Regulations, Rules of Practice and Procedure, and Oil and Gas Conservation Act (As Amended) Department of Natural Resources, Oil and Gas Conservation Commission of the State of Colorado (revised July 1989);

(4) Oil and Gas Conservation Commission Revised Rules and Regulations in the 300, 400, 500, and 600 series, effective March 20, 1989.

(b) *Memorandum of agreement.* The Memorandum of Agreement between EPA Region VIII and the Colorado Oil and Gas Conservation Commission, signed by the EPA Regional Administrator on March 3, 1984 and amended on August 30, 1989.

(c) *Statement of legal authority.* (1) Letter from Colorado Assistant Attorney General to the Acting Regional Counsel, EPA Region VIII, ''Re: Class II Well Underground Injection Control Program of Colorado Oil and Gas Conservation Commission'', March 15, 1983;

(2) Letter from Colorado Assistant Attorney General to the Acting Regional Counsel, EPA Region VIII, ''Re: Class II Well Injection Control Program of Colorado Oil and Gas Conservation Commission'', April 29, 1983;

(3) Letter from Colorado Assistant Attorney General to the Acting Regional Counsel, EPA Region VIII, ''Re: Class II Underground Injection Control Program of Colorado Oil and Gas Conservation Commission, interpretation of C.R.S. 1973, 34–60–110'', July 11, 1983;

(4) Letter from Colorado Assistant Attorney General to the Acting Regional Counsel, EPA Region VIII, ''Re: Class II Well Underground Injection Control Program of Colorado Oil and Gas Conservation Commission'', February 17, 1984;

(5) Memorandum from Colorado Assistant Attorney General to the Acting Regional Counsel, EPA Region VIII, ''Re: Authority to set and enforce maximum pressure for injecting fluids into Class II wells with existing permits'', March 7, 1984.

(d) *Program description.* The Program Description and any other materials submitted as part of the application or as supplements thereto:

(1) Application and accompanying materials for approval of Colorado's UIC program for Class II wells submitted by the Director of the Colorado Oil and Gas Conservation Commission to the Regional Administrator, May 3, 1983;

(2) Supplemental amendment to Colorado's application for primacy for the UIC program for Class II wells describing the process through which the State will ensure enforceable limits for maximum injection pressure, describing the Commission's plan of administration for Class II wells, and describing Mechanical Integrity Test procedures for Class II wells, March 7, 1984;

(3) Official correspondence concerning various program issues between the Colorado Oil and Gas Conservation Commission and EPA Region VIII, for the period from March 7, 1984 to May 8, 1989.

[56 FR 9412, Mar. 6, 1991]

§ 147.301 EPA-administered program—Class I, III, IV, V wells and Indian lands.

(a) *Contents.* The UIC program for Class I, III, IV and V wells on all lands in Colorado, including Indian lands, and for Class II wells on Indian lands, is administered by EPA. The program for all EPA-administered wells in Colorado other than Class II wells on the lands of the Ute Mountain Ute consists of the UIC program requirements of 40 CFR parts 124, 144, 146, 148, and any additional requirements set forth in the remainder of this subpart. Injection well owners and operators, and EPA shall comply with these requirements.

(b) *Effective dates.* The effective date for the UIC program on all lands in Colorado, including Indian lands, except for Class II wells on lands of the Ute Mountain Ute, is June 25, 1984.

[52 FR 17681, May 11, 1987, as amended at 56 FR 9413, Mar. 6, 1991]

§ 147.302 Aquifer exemptions.

(a) This section identifies any aquifers of their portions exempted in accordance with §§ 144.7(b) and 146.4 of this chapter at the time of program promulgation. EPA may in the future exempt other aquifers or portions according to applicable procedures without codifying such exemptions in this section. An updated list of exemptions will be maintained in the Regional office.

(b) For all aquifers into which existing Class II wells are injecting, those portions within a ¼ mile radius of the well are exempted for the purpose of Class II injection activities only.

§ 147.303 Existing Class I, II (except enhanced recovery and hydrocarbon storage) and III wells authorized by rule.

Maximum injection pressure. The owner or operator shall limit injection pressure to the lesser of:

(a) A value which will not exceed the operating requirements of § 144.28(f)(3)(i) or (ii) as applicable; or

(b) A value for wellhead pressure calculated by using the following formula;

$$Pm=(0.733–0.433\ Sg)d$$

where:

Pm=injection pressure at the wellhead in pounds per square inch

Sg=specific gravity of injected fluid (unitless)

d=injection depth in feet.

§ 147.304 Existing Class II enhanced recovery and hydrocarbon storage wells authorized by rule.

(a) *Maximum injection pressure.* (1) To meet the operating requirements of § 144.28(f)(3)(ii) (A) and (B) of this chapter, the owner or operator:

(i) Shall use an injection pressure no greater than the pressure established by the Regional Administrator for the field or formation in which the well is located. The Regional Administrator shall establish such a maximum pressure after notice, opportunity for comment, and opportunity for a public hearing, according to the provisions of part 124, subpart A of this chapter, and will inform owners and operators in writing of the applicable maximum pressure; or

(ii) May inject at pressures greater than those specified in paragraph (a)(1)(i) of this section for the field or formation in which he is operating provided he submits a request in writing to the Regional Administrator and demonstrates to the satisfaction of the Regional Administrator that such injection pressure will not violate the requirements of § 144.28(f)(3)(ii) (A) and (B). The Regional Administrator may grant such a request after notice, opportunity for comment, and opportunity for a public hearing, according to the provisions of part 124, subpart A of this chapter.

(2) Prior to such time as the Regional Administrator establishes rules for maximum injection pressures based on data provided pursuant to paragraph (a)(2)(ii) of this section the owner or operator shall:

(i) Limit injection pressure to a value which will not exceed the operating requirements of § 144.28(f)(3)(ii); and

(ii) Submit data acceptable to the Regional Administrator which defines the fracture pressure of the formation in which injection is taking place. A single test may be submitted on behalf of two or more operators conducting operations in the same formation, if the Regional Administrator approves

such submission. The data shall be submitted to the Regional Administrator within one year of the effective date of this program.

(b) *Casing and cementing.* Where the Regional Administrator determines that the owner or operator of an existing enhanced recovery or hydrocarbon storage well may not be in compliance with the requirements of §§ 144.28(e) and 146.22, the owner or operator shall comply with paragraphs (b) (1) through (4) of this section, when required by the Regional Administrator:

(1) Protect USDWs by:

(i) Cementing surface casing by recirculating the cement to the surface from a point 50 feet below the lowermost USDW; or

(ii) Isolating all USDWs by placing cement between the outermost casing and the well bore; and

(2) Isolate any injection zones by placing sufficient cement to fill the calculated space between the casing and the well bore to a point 250 feet above the injection zone; and

(3) Use cement:

(i) Of sufficient quantity and quality to withstand the maximum operating pressure;

(ii) Which is resistent to deterioration from formation and injection fluids; and

(iii) In quantity no less than 120% of the calculated volume necessary to cement off a zone.

(4) The Regional Administrator may specify other requirements in addition to or in lieu of the requirements set forth in paragraphs (b) (1) through (3) as needed to protect USDWs.

§ 147.305 Requirements for all wells.

(a) The owner or operator converting an existing well to an injection well shall check the condition of the casing with one of the following logging tools:

(1) A Pipe analysis log; or

(2) A Caliper log.

(b) The owner or operator of a new injection well cased with plastic (PVC, ABS, and others) casings shall:

(1) Not construct a well deeper than 500 feet;

(2) Use cement and additives compatible with such casing material;

(3) Cement the annular space above the injection interval from the bottom of the blank casing to the surface.

(c) The owner or operator of a newly drilled well shall install centralizers as directed by the Regional Administrator.

(d) The owner or operator shall as required by the Regional Administrator:

(1) Protect USDWs by:

(i) Setting surface casing 50 feet below the base of the lowermost USDW;

(ii) Cementing surface casing by recirculating the cement to the surface from a point 50 feet below the lowermost USDW; or

(iii) Isolating all USDWs by placing cement between the outermost casing and the well bore; and

(2) Isolate any injection zones by placing sufficient cement to fill the calculated space between the casing and the well bore to a point 250 feet above the injection zone; and

(3) Use cement:

(i) Of sufficient quantity and quality to withstand the maximum operating pressure;

(ii) Which is resistant to deterioration from formation and injection fluids; and

(iii) In a quantity no less than 120% of the calculated volume necessary to cement off a zone.

(4) The Regional Administrator may approve alternate casing and cementing practices provided that the owner or operator demonstrates that such practices will adequately protect USDWs.

(e) *Area of review.* Notwithstanding the alternatives presented in § 146.6 of this chapter, the area of review shall be a fixed radius as described in § 146.6(b) of this chapter.

(f) The applicant must give separate notice of intent to apply for a permit to each owner or tenant of the land within one-quarter mile of the site. The addresses of those to whom notice is given, and a description of how notice is given, shall be submitted with the permit application. The notice shall include:

(1) Name and address of applicant;

(2) A brief description of the planned injection activities, including well location, name and depth of the injection

zone, maximum injection pressure and volume, and fluid to be injected;

(3) EPA contact person; and

(4) A statement that opportunity to comment will be announced after EPA prepares a draft permit. This requirement may be waived by the Regional Administrator when he determines that individual notice to all land owners and tenants would be impractical.

Subpart H—Connecticut

§ 147.350 State-administered program.

The UIC program for all classes of wells in the State of Connecticut, except those wells on Indian lands, is the program administered by the Connecticut Department of Environmental Protection approved by EPA pursuant to section 1422 of the SDWA. Notice of this approval was published in the FR on March 26, 1984 (49 FR 11179); the effective date of this program is March 26, 1984. This program consists of the following elements, as submitted to EPA in the State's program application:

(a) *Incorporation by reference.* The requirements set forth in the State statutes and regulations cited in this paragraph are hereby incorporated by reference and made part of the applicable UIC program under the SDWA for the State of Connecticut. This incorporation by reference was approved by the Director of the OFR in accordance with 5 U.S.C. 552(a) and CFR part 51. Copies may be obtained at the State of Connecticut, Department of Environmental Protection, State Office Building, 165 Capitol Avenue, Hartford, Connecticut, 06106. Copies may be inspected at the Environmental Protection Agency, Region I, John F. Kennedy Federal Building, room 2203, Boston, Massachusetts, 02203, or at the Office of the Federal Register, 800 North Capitol Street NW., suite 700, Washington, DC.

(1) Connecticut General Statutes Annotated, title 22a (Environmental Protection), chapter 439, sections 22a–1 through 22a–27 (1985 and Cumm. Supp. 1990);

(2) Connecticut General Statutes Annotated, Title 22a (Environmental Protection), Chapter 446K (1985 and Cumm. Supp. 1990).

(b) *Memorandum of Agreement.* The Memorandum of Agreement between EPA Region I and the Connecticut Department of Environmental Protection, signed by the EPA Regional Administrator on August 9, 1983.

(c) *Statement of legal authority.* (1) Statement from the Attorney General of the State of Connecticut, signed by the Attorney General on May 8, 1981;

(2) Addendum to the Statement from the Attorney General of the State of Connecticut, signed by the Attorney General on May 10, 1983.

(d) *Program Description.* The Program Description and any other materials submitted as part of the application or as supplements thereto.

[56 FR 9413, Mar. 6, 1991]

§§ 147.351—147.352 [Reserved]

§ 147.353 EPA-administered program—Indian lands.

(a) *Contents.* The UIC program for all classes of wells on Indian lands in Connecticut is administered by EPA. This program consists of the UIC program requirements of 40 CFR parts 124, 144, 146, 148, and any additional requirements set forth in the remainder of this subpart. Injection well owners and operators, and EPA shall comply with these requirements.

(b) *Effective date.* The effective date of the UIC program for Indian lands in Connecticut is November 25, 1988.

[53 FR 43086, Oct. 25, 1988, as amended at 56 FR 9413, Mar. 6, 1991]

§§ 147.354—147.359 [Reserved]

Subpart I—Delaware

§ 147.400 State-administered program.

The UIC program for all classes of wells in the State of Delaware, except those wells on Indian lands, is the program administered by the Delaware Department of Natural Resources and Environmental Control approved by EPA pursuant to section 1422 of the SDWA. Notice of this approval was published in the FR on April 5, 1984 (49 FR 13525); the effective date of this program is May 7, 1984. This program consists of the following elements, as submitted to EPA in the State's program application:

(a) *Incorporation by reference.* The requirements set forth in the State statutes and regulations cited in this paragraph are hereby incorporated by reference and made a part of the applicable UIC program under the SDWA for the State of Delaware. This incorporation by reference was approved by the Director of the OFR in accordance with 5 U.S.C. 552(a) and 1 CFR part 5l. Copies may be obtained at the Delaware Department of Natural Resources and Environmental Control, 89 Kings Highway, P.O. Box 1401, Dover, Delaware, 19903. Copies may be inspected at the Environmental Protection Agency, Region III, 841 Chestnut Street, Philadelphia, Pennsylvania, 19107, or at the Office of the Federal Register, 800 North Capitol Street NW., suite 700, Washington, DC.

(1) Delaware Environmental Protection Act, (Environmental Control) 7 Delaware Code Annotated, Chapter 60, Sections 6001–6060 (Revised 1974 and Cumm. Supp. 1988);

(2) State of Delaware Regulations Governing Underground Injection Control, parts 122, 124 and 146 (Department of Natural Resources and Environmental Control), effective August 15, 1983.

(b) *Memorandum of agreement.* The Memorandum of Agreement between EPA Region III and the Delaware Department of Natural Resources and Environmental Control, signed by the EPA Regional Administrator on March 28, 1984.

(c) *Statement of legal authority.* Statement of the Delaware Attorney General for the Underground Injection Control Program, signed by the Attorney General on January 26, 1984.

(d) *Program Description.* The Program Description and any other materials submitted as part of the application (August 10, 1983), or as supplements thereto (October 14, 1983).

[56 FR 9413, Mar. 6, 1991]

§§ 147.401—147.402 [Reserved]

§ 147.403 EPA-administered program—Indian lands.

(a) *Contents.* The UIC program for all classes of wells on Indian lands in Delaware is administered by EPA. This program consists of the UIC program requirements of 40 CFR parts 124, 144, 146, 148, and any additional requirements set forth in the remainder of this subpart. Injection well owners and operators and EPA shall comply with these requirements.

(b) *Effective date.* The effective date of the UIC program for Indian lands in Delaware is November 25, 1988.

[53 FR 43086, Oct. 25, 1988, as amended at 56 FR 9413, Mar. 6, 1991]

§§ 147.404—147.449 [Reserved]

Subpart J—District of Columbia

§ 147.450 State-administered program. [Reserved]

§ 147.451 EPA-administered program.

(a) *Contents.* The UIC program for the District of Columbia, including any Indian lands in the District, is administered by EPA. This program consists of the UIC program requirements of 40 CFR parts 124, 144, 146, 148, and any additional requirements set forth in the remainder of this subpart. Injection well owners and operators, and EPA shall comply with these requirements.

(b) *Effective date.* The effective date of the UIC program for Indian lands in the District of Columbia is November 25, 1988. The effective date for the UIC program in the rest of the District is June 25, 1984.

[53 FR 43087, Oct. 25, 1988, as amended at 56 FR 9413, Mar. 6, 1991]

§ 147.452 Aquifer exemptions. [Reserved]

Subpart K—Florida

§ 147.500 State-administered program—Class I, III, IV, and V wells.

The UIC program for Class I, III, IV, and V wells in the State of Florida, except for those on Indian lands is administered by the Florida Department of Environmental Regulations, approved by EPA pursuant to section 1422 of the SDWA. Notice of this approval was published in the FEDERAL REGISTER on February 7, 1983 (48 FR 5556); the effective date of this program is March 9, 1983. This program consists of the following elements, as submitted to EPA in the State's program application:

(a) *Incorporation by reference.* The requirements set forth in the State statutes and regulations cited in this paragraph are hereby incorporated by reference and made a part of the applicable UIC program under the SDWA for the State of Florida. This incorporation by reference was approved by the Director of the Federal Register on June 25, 1984.

(1) Florida Air and Water Pollution Control Act, Florida Statutes Annotated sections 403.011 through 403.90 (1973 and Supp. 1983);

(2) Chapter 17–28, Underground Injection Control, Florida Administrative Code (April 27, 1989).

(b) *Other laws.* The following statutes and regulations although not incorporated by reference, also are part of the approved State-administered program:

(1) Administrative Procedures Act, Florida Statutes Chapter 120;

(2) Florida Administrative Code, Chapter 17–1 (1982) (Administrative Procedures Act);

(3) Florida Administrative Code, Chapter 17–3 (1982) (Water Quality Standards);

(4) Florida Administrative Code, Chapter 17–4 (1982) (Permits);

(5) Florida Administrative Code, Chapter 28–5 (1982) (Decisions Determining Substantial Interests);

(6) Florida Administrative Code, Chapter 28–6 (1982) (Licensing);

(c) The Memorandum of Agreement between EPA Region IV and the Florida Department of Environmental Regulation, signed by the EPA Regional Administrator on March 31, 1983.

(d) *Statement of legal authority.* (1) ''Statement of Legal Authority for Implementation of Underground Injection Control Program'' and accompanying certifications, signed by General Counsel for the Florida Department of Environmental Regulation, January 14, 1982;

(2) ''Addendum to Statement of Legal Authority for Implementation of Underground Injection Control Program'' and accompanying certifications, signed by Acting General Counsel for the Florida Department of Environmental Regulation, September 20, 1982.

(e) The Program Description and any other materials submitted as part of the original application or as supplements thereto.

[49 FR 20197, May 11, 1984, as amended at 53 FR 43087, Oct. 25, 1988; 56 FR 9414, Mar. 6, 1991]

§ 147.501 EPA-administered program—Class II wells and Indian lands.

(a) *Contents.* The UIC program for all classes of wells on Indian lands and for Class II wells on non-Indian lands in the State of Florida is administered by EPA. This program consists of the UIC program requirements of 40 CFR parts 124, 144, 146, 148, and any additional requirements set forth in the remainder of this subpart. Injection well owners and operators, and EPA shall comply with these requirements.

(b) *Effective dates.* The effective date of the UIC program for Indian lands in Florida is November 25, 1988. The effective date for Class II wells on non-Indian lands is December 30, 1984.

[53 FR 43087, Oct. 25, 1988, as amended at 56 FR 9414, Mar. 6, 1991]

§ 147.502 Aquifer exemptions. [Reserved]

§ 147.503 Existing Class II (except enhanced recovery and hydrocarbon storage) wells authorized by rule.

Maximum injection pressure. To meet the operating requirements of § 144.28(f)(3)(i) of this chapter, the owner or operator shall use an injection pressure at the well head no greater than the pressure calculated using the following formula:

$$Pm=(0.733-0.433\ Sg)d$$

where

Pm=injection pressure at the well head in pounds per square inch

Sg=specific gravity of injected fluid (unitless)

d=injection depth in feet.

[49 FR 45306, Nov. 15, 1984]

§ 147.504 Existing Class II enhanced recovery and hydrocarbon storage wells authorized by rule.

(a) *Maximum injection pressure.* (1) To meet the operating requirements of § 144.28(f)(3)(ii) (A) and (B) of this chapter, the owner or operator:

(i) Shall use an injection pressure no greater than the pressure established by the Regional Administrator for the

field or formation in which the well is located. The Regional Administrator shall establish such a maximum pressure after notice, opportunity for comment and opportunity for a public hearing, according to the provisions of part 124, subpart A of this chapter, and will inform owners and operators in writing of the applicable maximum pressure; or

(ii) May inject at pressure greater than those specified in paragraph (a)(1)(i) of this section for the field or formation in which he is operating provided he submits a request in writing to the Regional Administrator, and demonstrates to the satisfaction of the Regional Administrator that such injection pressure will not violate the requirement of § 144.28(f)(3)(ii) (A) and (B). The Regional Administrator may grant such a request after notice, opportunity for comment, and opportunity for a public hearing, according to the provisions of part 124, subpart A of this chapter.

(2) Prior to such time as the Regional Administrator establishes rules for maximum injection pressure based on data provided pursuant to paragraph (a)(2)(ii) of this section the owner or operator shall:

(i) Limit injection pressure to a value which will not exceed the operating requirements of § 144.28(f)(3)(ii); and

(ii) Submit data acceptable to the Regional Administrator which defines the fracture pressure of the formation in which injection is taking place. A single test may be submitted on behalf of two or more operators conducting operations in the same formation, if the Regional Administrator approves such submission. The data shall be submitted to the Regional Administrator within 1 year of the effective date of this program.

(b) *Casing and cementing.* Where the Regional Administrator determines that the owner or operator of an existing enhanced recovery or hydrocarbon storage well may not be in compliance with the requirements of §§ 144.28(e) and 146.22, the owner or operator shall, when required by the Regional Administrator:

(1) Protect USDWs by:

(i) Cementing surface casing by recirculating the cement to the surface from a point 50 feet below the lowermost USDW; or

(ii) Isolating all USDWs by placing cement between the outermost casing and the well bore; and

(2) Isolate any injection zones by placing sufficient cement to fill the calculated space between the casing and the well bore to a point 250 feet above the injection zone; and

(3) Use cement:

(i) Of sufficient quantity and quality to withstand the maximum operating pressure;

(ii) Which is resistant to deterioration from formation and injection fluids; and

(iii) In a quantity no less than 120% of the calculated volume necessary to cement off a zone.

(4) Comply with other requirements which the Regional Administrator may specify either in addition to or in lieu of the requirements set forth in paragraphs (b)(1) through (3) of this section as needed to protect USDWs.

(c) *Area of review.* Notwithstanding the alternatives presented in § 146.06 of this chapter, the area of review shall be a minimum fixed radius as described in § 146.06(b) of this chapter.

(The information collection requirements contained in paragraph (a)(2)(ii) were appoved by the Office of Management and Budget under control number 2040–0042)

[49 FR 45306, Nov. 15, 1984]

Subpart L—Georgia

§ 147.550 State-administered program.

The UIC program for all classes of wells in the State of Georgia, except those wells on Indian lands, is the program administered by the Georgia Department of Natural Resources, Environmental Protection Division approved by EPA pursuant to section 1422 of the SDWA. Notice of this approval was published in the FEDERAL REGISTER on April 19, 1984 (49 FR 15553); the effective date of this program is May 21, 1984. This program consists of the following elements, as submitted to EPA in the State's program application:

(a) *Incorporation by reference.* The requirements set forth in the State statutes and regulations cited in this paragraph are hereby incorporated by reference and made a part of the applicable UIC program under the SDWA for the State of Georgia. This incorporation by reference was approved by the Director of the OFR in accordance with 5 U.S.C. 552(a) and 1 CFR part 51. Copies may be obtained at the Georgia Department of Natural Resources, Environmental Protection Division, 270 Washington Street, SW., Atlanta, Georgia, 30334. Copies may be inspected at the Environmental Protection Agency, Region IV, 345 Courtland Street, NE., Atlanta, Georgia, 30365, or at the Office of the Federal Register, 800 North Capitol Street NW., suite 700, Washington, DC.

(1) Oil and Gas and Deep Drilling Act of 1975, Official Code of Georgia Annotated (O.C.G.A.) §§ 12–4–40 through 12–4–53 (1988);

(2) Ground Water Use Act of 1972, O.C.G.A. §§ 12–5–90 through 12–5–107 (1988);

(3) Water Well Standards Act of 1985, O.C.G.A. §§ 12–5–120, through 12–5–138 (1988);

(4) Georgia Administrative Procedure Act, O.C.G.A. §§ 50–13–1 through 50–13–22 (Reprinted from the 0.C.G.A. and 1988 Cumm. Supp.);

(5) Georgia Water Quality Control Act, O.C.G.A. §§ 12–5–20 through 12–5–53 (1988);

(6) Georgia Hazardous Waste Management Act, O.C.G.A. §§ 12–8–60 through 12–8–83 (1988);

(7) Georgia Safe Drinking Water Act of 1977, O.C.G.A. §§ 12–5–170 through 12–5–193 (1988);

(8) Rules of Georgia Department of Natural Resources, Environmental Protection Division, Water Quality Control, GA. COMP. R. & REGS. Chapter 391–3–6–.13 (Revised July 28, 1988).

(b) *Memorandum of Agreement.* The Memorandum of Agreement between EPA Region IV and the State of Georgia, signed March 1, 1984.

(c) *Statement of legal authority.* (1) Unofficial Opinion of the Georgia Attorney General, Op. Atty. Gen. 080–24, June 12, 1980;

(2) Underground Injection Control Program, Attorney General's Statement, February 4, 1982;

(3) Amended Attorney General's Statement Relating to Authority of the State of Georgia to Implement an Underground Injection Control Program, April 22, 1983;

(4) Letter to EPA Office of General Counsel from Senior Assistant Attorney General ''Re: State UIC Program'', July 13, 1983.

(d) *Program Description.* The Program Description and any other materials submitted as part of the application or as supplements thereto.

[56 FR 9414, Mar. 6, 1991; 56 FR 14150, Apr. 5, 1991]

§§ 147.551—147.552 [Reserved]

§ 147.553 EPA-administered program—Indian lands.

(a) *Contents.* The UIC program for all classes of wells on Indian lands in the State of Georgia is administered by EPA. This program consists of the UIC program requirements of 40 CFR parts 124, 144, 146, 148, and any additional requirements set forth in the remainder of this subpart. Injection well owners and operators, and EPA shall comply with these requirements.

(b) *Effective date.* The effective date of the UIC program for Indian lands in Georgia is November 25, 1988.

[53 FR 43087, Oct. 25, 1988, as amended at 56 FR 9414, Mar. 6, 1991]

§§ 147.554—147.559 [Reserved]

Subpart M—Hawaii

§ 147.600 State-administered program. [Reserved]

§ 147.601 EPA-administered program.

(a) *Contents.* The UIC program for the State of Hawaii, including all Indian lands, is administered by EPA. This program consists of the UIC program requirements of 40 CFR parts 124, 144, 146, 148, and any additional requirements set forth in the remainder of this subpart. Injection well owners and operators, and EPA shall comply with these requirements.

(b) *Effective date.* The effective date of the UIC program for Indian lands in

Hawaii is November 25, 1988. The effective date for the UIC program for all other lands in Hawaii is December 30, 1984.

[53 FR 43087, Oct. 25, 1988, as amended at 56 FR 9414, Mar. 6, 1991]

Subpart N—Idaho

§ 147.650 State-administrative program—Class I, II, III, IV, and V wells.

The UIC program for Class I, II, III, IV, and V wells in the State of Idaho, other than those on Indian lands, is the program administered by the Idaho Department of Water Resources, approved by EPA pursuant to section 1422 of the SDWA. Notice of this approval was published in the FEDERAL REGISTER on June 7, 1985; the effective date of this program is July 22, 1985. This program consists of the following elements, as submitted to EPA in the State's program application.

(a) *Incorporation by reference.* The requirements set forth in the State statutes and regulations cited in this paragraph are hereby incorporated by reference and made a part of the applicable UIC program under the SDWA for the State of Idaho. This incorporation by reference was approved by the Director of the FEDERAL REGISTER effective July 22, 1985.

(1) Public Writings, Title 9, Chapter 3, Idaho Code, sections 9–301 through 9–302 (Bobbs-Merrill 1979);

(2) Crimes and Punishments, Title 18, Chapter 1, Idaho Code, sections 18–113 through 18–114 (Bobbs-Merrill 1979 and Supp. 1984);

(3) Department of Health and Welfare, Title 39, Chapter 1, Idaho Code, Chapter 39–108 (Bobbs-Merrill 1977);

(4) Drainage-Water Rights and Reclamation, Title 42, Chapter 2, Idaho Code sections 42–237(e); section 42–238 (Bobbs-Merrill 1977 and Supp. 1984);

(5) Department of Water Resources-Water Resources Board, Title 42, Chapter 17, Idaho Code, sections 42–1701, 42–1703, 42–1735 (Bobbs-Merrill 1977, section 42–1701A (Supp. 1984);

(6) Director of Department of Water Resources, Title 42, Chapter 18, Idaho Code, sections 42–1801 through 42–1805 (Bobbs-Merrill 1977);

(7) Waste Disposal and Injection Wells, Title 42, Chapter 39, Idaho Code, sections 42–3901 through 42–3914 (Bobbs-Merrill 1977), sections 42–3915 through 42–3919 (Supp. 1984);

(8) Idaho Trade Secrets Act, Title 48, Chapter 8, Idaho Code, sections 48–801 through 48–807 (Bobbs-Merrill 1977 and Supp. 1984);

(9) Administrative Procedure, Title 67, Chapter 52, Idaho Code, sections 67–5201 through 67–5218 (Bobbs-Merrill 1980 and Supp. 1984);

(10) Idaho Radiation Control Regulations (IRCR section 1–9002.70; sections 1–9100 through 1–9110, Department of Health and Welfare (May 1981);

(11) Rules and Regulations: Construction and Use of Injection Wells, Idaho Department of Water Resources, Rules 1 through 14 (August 1984);

(12) Rules and Regulations: Practice and Procedures, Idaho Department of Water Resources, Rules 1 through 14 (October 1983).

(b) The Memorandum of Agreement between EPA and Region X and the Idaho Department of Water Resources signed by the EPA Regional Administrator on February 11, 1985.

(c) *Statement of legal authority.* (1) The Idaho Attorney General's Statement for the Underground Injection Control Program, October 31, 1984.

(2) Letter from David J. Barber, Deputy Attorney General, Idaho Department of Water Resources to Harold Scott, EPA, Region 10, revising the Attorney General's Statement, February 14, 1985.

(d) The Program Description and any other materials submitted as part of the application or as supplements thereto.

[50 FR 23957, June 7, 1985]

§ 147.651 EPA-administered program—Indian lands.

(a) *Contents.* The UIC program for all classes of wells on Indian lands in the State of Idaho is administered by EPA. This program consists of the UIC program requirements of 40 CFR parts 124, 144, 146, 148, and any additional requirements set forth in the remainder of this subpart. Injection well owners and operators, and EPA shall comply with these requirements.

(b) *Effective dates.* The effective date of the UIC program for Indian lands in Idaho is June 11, 1984.

[52 FR 17681, May 11, 1987, as amended at 56 FR 9414, Mar. 6, 1991]

§ 147.652 Aquifer exemptions. [Reserved]

Subpart O—Illinois

§ 147.700 State-administered program—Class I, III, IV, and V wells.

The UIC program for Class I, III, IV and V wells in the State of Illinois, except those on Indian lands, is the program administered by the Illinois Environmental Protection Agency, approved by EPA pursuant to section 1422 of the SDWA. Notice of the approval was published in the FEDERAL REGISTER on February 1, 1984 (49 FR 3991); the effective date of this program is March 3, 1984. This program consists of the following elements, as submitted to EPA in the State's program application:

(a) *Incorporation by reference.* The requirements set forth in the state statutes and regulations cited in this paragraph are hereby incorporated by reference and made a part of the applicable UIC program under the SDWA for the State of Illinois. This incorporation by reference was approved by the Director of the Federal Register on June 25, 1984.

(1) Illinois Environmental Protection Act, Illinois ch. 111½, sections 1001 to 1051 (Smith-Hurd 1977 Revised Statutes and Supp. 1983), as amended by Public Act No. 83–431, 1983 Illinois Legislative Service, pages 2910 to 2916 (West);

(2) Illinois Pollution Control Board Rules and Regulations at Title 35, Illinois Administrative Code, Chapter I, Part 700, Outline of Waste Disposal Regulations; Part 702, RCRA and UIC Permit Programs; Part 704, UIC Permit Program; Part 705, Procedures for Permit Issuance and Part 730, Underground Injection Control Operating Requirements as amended by IPCB Order No. R–83039 on December 15, 1983.

(b) The Memorandum of Agreement between EPA Region V and the Illinois Environmental Protection Agency, signed by the EPA Regional Administrator on March 22, 1984.

(c) *Statement of legal authority.* Letter from Illinois Attorney General to Regional Administrator, EPA Region V, and attached statement, December 16, 1982.

(d) The Program Description and any other materials submitted as part of the application or as supplements thereto.

[49 FR 20197, May 11, 1984, as amended at 53 FR 43087, Oct. 25, 1988]

§ 147.701 State-administered program—Class II wells.

The UIC program for Class II wells in the State of Illinois, except those on Indian lands, is the program administered by the Illinois Environmental Protection Agency, approved by EPA pursuant to section 1425 of the SDWA. Notice of the approval was published in the FEDERAL REGISTER on February 1, 1984 (49 FR 3990); the effective date of this program is March 3, 1984. This program consists of the following elements, as submitted to EPA in the state's program application:

(a) *Incorporation by reference.* The requirements set forth in the State Statutes and regulations cited in this paragraph are hereby incorporated by reference and made a part of the applicable UIC program under the SDWA for the State of Illinois. This incorporation by reference was approved by the Director of the FEDERAL REGISTER on June 25, 1984.

(1) Conservation of Oil and Gas, etc., Illinois Revised Statutes ch. 96½, sections 5401 to 5457 (Smith-Hurd 1979 and Supp. 1983), as amended by Public Act No. 83–1074 1983 Illinois Legislative Service pages 7183 to 7185 (West);

(2) Illinois Environmental Protection Act, Illinois Revised Statutes ch. 111½, sections 1001–1051 (Smith-Hurd 1977 and Supp. 1983), as amended by Public Act No. 83–431, 1983 Illinois Legislative Services pages 2910 to 2916 (West);

(3) Illinois Revised Statutes ch. 100½, section 26 (Smith-Hurd Supp. 1983);

(4) Illinois Department of Mines and Minerals Regulations for the Oil and Gas Division, Rules I, II, IIA, III, V, VII, and IX (1981).

(b) The Memorandum of Agreement between EPA Region V and the Illinois Department of Mines and Minerals,

signed by the EPA Regional Administrator on March 22, 1984.

(c) *Statement of legal authority.* ''Certification of Legal Authority,'' signed by State Attorney, Richland County, Illinois, May 5, 1982.

(d) The Program Description and any other materials submitted as part of the application or as supplements thereto.

[49 FR 20197, May 11, 1984, as amended at 53 FR 43087, Oct. 25, 1988]

§ 147.703 EPA-administered program—Indian lands.

(a) *Contents.* The UIC program for all classes of wells on Indian lands in the State of Illinois is administered by EPA. This program consists of the UIC program requirements of 40 CFR parts 124, 144, 146, 148, and any additional requirements set forth in the remainder of this subpart. Injection well owners and operators, and EPA shall comply with these requirements.

(b) *Effective dates.* The effective date for the UIC program for Indian lands is November 25, 1988.

[53 FR 43087, Oct. 25, 1988, as amended at 56 FR 9414, Mar. 6, 1991]

Subpart P—Indiana

§ 147.750 State-administered program—Class II wells.

The UIC program for Class II injection wells in the State of Indiana on non-Indian lands is the program administered by the Indiana Department of Natural Resources (INDR) approved by the EPA pursuant to section 1425 of the SDWA. Notice of this approval was published in the FR on August 19, 1991; the effective date of this program is August 19, 1991. This program consists of the following elements, as submitted to EPA in the State's program application:

(a) *Incorporation by reference.* The requirements set forth in the State statutes and regulations cited in this paragraph are hereby incorporated by reference and made a part of the applicable UIC program under the SDWA for the State of Indiana. This incorporation by reference was approved by the Director of the FR in accordance with 5 U.S.C. 552(a) and 1 CFR part 51. Copies may be obtained at the Indiana Department of Natural Resources, Division of Oil and Gas, 402 West Washington Street, room 293, Indianapolis, Indiana, 46204. Copies may be inspected at the Environmental Protection Agency, Region V, 77 West Jackson Boulevard, Chicago, Illinois, 60604, or at the Office of the Federal Register, 800 North Capitol Street NW., suite 700, Washington, DC.

(1) Indiana Code, title 4, article 21.5, chapters 1 through 6 (1988).

(2) West's Annotated Indiana Code, title 13, article 8, chapters 1 through 15 (1990 and Cumm. Supp. 1990).

(3) Indiana Administrative Code, title 310, article 7, rules 1 through 3 (Cumm. Supp. 1991).

(b) *Memorandum of agreement.* The Memorandum of Agreement between EPA Region V and the Indiana Department of Natural Resources signed by the EPA Regional Administrator on February 18, 1991.

(c) *Statement of legal authority.* Statement and Amendment to the Statement from the Attorney General of the State of Indiana, signed on July 12, 1990, and December 13, 1990, respectively.

(d) The Program Description and any other materials submitted as part of the original application or as supplements thereto.

[56 FR 41072, Aug. 19, 1991, as amended at 62 FR 1834, Jan. 14, 1997]

§ 147.751 EPA-administered program.

(a) *Contents.* The UIC program for all classes of wells on Indian lands, and for Class I, III, IV, and V wells on non-Indian lands in the State of Indiana is administered by the EPA. The program consists of the UIC program requirements of 40 CFR parts 124, 144, 146, and 148 and the additional requirements set forth in the remainder of this subpart. Injection well owners and operators, and EPA shall comply with these requirements.

(b) *Effective dates.* The effective date for the UIC program on Indian lands is November 25, 1988. The effective date of the UIC program for the rest of Indiana is June 25, 1984.

[53 FR 43087, Oct. 25, 1988, as amended at 56 FR 9414, Mar. 6, 1991; 56 FR 41072, Aug. 19, 1991]

§ 147.752 Aquifer exemptions. [Reserved]

§ 147.753 Existing Class I and III wells authorized by rule.

Maximum injection pressure. The owner or operator shall limit injection pressure to the lessor of:

(a) A value which will not exceed the operating requirements of § 144.28(f)(3)(i) or (ii) as applicable; or

(b) A value for well head pressure calculated by using the following formula:

$$Pm=(0.800–0.433\ Sg)d$$

where:

Pm=injection pressure at the wellhead in pounds per square inch
Sg=specific gravity of injected fluid (unitless)
d=injection depth in feet.

[49 FR 20197, May 11, 1984, as amended at 56 FR 41072, Aug. 19, 1991]

Subpart Q—Iowa

§ 147.800 State-administered program. [Reserved]

§ 147.801 EPA-administered program.

(a) *Contents.* The UIC program for the State of Iowa, including all Indian lands, is administered by EPA. This program consists of the UIC program requirements of 40 CFR parts 124, 144, 146, 148, and any additional requirements set forth in the remainder of this subpart. Injection well owners and operators, and EPA shall comply with these requirements.

(b) *Effective dates.* The effective date for the UIC program for all lands in Iowa, including Indian lands, is June 25, 1984.

[52 FR 17681, May 11, 1987, as amended at 56 FR 9415, Mar. 6, 1991]

§ 147.802 Aquifer exemptions. [Reserved]

Subpart R—Kansas

§ 147.850 State-administered program—Class I, III, IV and V wells.

The UIC program for Class I, III, IV and V wells in the State of Kansas, except those on Indian lands as described in § 147.860, is the program administered by the Kansas Department of Health and Environment, approved by EPA pursuant to section 1422 of the SDWA. Notice of this approval was published in the FEDERAL REGISTER on December 2, 1983 (48 FR 54350); the effective date of this program is December 2, 1983. This program consists of the following elements, as submitted to EPA in the State's program application.

(a) *Incorporation by reference.* The requirements set forth in the State statutes and regulations cited in this paragraph are hereby incorporated by reference and made a part of the applicable UIC program under the SDWA for the State of Kansas. This incorporation by reference was approved by the Director of the OFR in accordance with 5 U.S.C. 552(a) and 1 CFR part 51. Copies may be obtained at the Kansas Department of Health and Environment, Forbes Field, Building 740, Topeka, Kansas, 66620. Copies may be inspected at the Environmental Protection Agency, Region VII, 726 Minnesota Avenue, Kansas City, Kansas, 66101, or at the Office of the FEDERAL REGISTER, 800 North Capitol Street, NW., suite 700, Washington, DC.

(1) Chapter 28, Article 46, Underground Injection Control Regulations, Kansas Administrative Regulations §§ 28–46–1 through 28–46–42 (1986 and Supp. 1987);

(2) Chapter 28, Article 43, Construction, operation, monitoring and abandonment of salt solution mining wells, Kansas Administrative Regulations §§ 28–43–1 through 28–43–10 (1986);

(3) Kansas Statutes Annotated §§ 65–161, 65–164 through 65–166a, 65–171d (1980 and Cumm. Supp. 1989).

(b) *Other laws.* The following statutes and regulations, although not incorporated by reference except for the select sections identified in paragraph (a) of this section, are also part of the approved State-administered program: Kansas Statutes Annotated §§ 65–161 through 65–171(w), (1980 and Supp. 1983).

(c) *Memorandum of Agreement.* (1) The Memorandum of Agreement between EPA Region VII and the Kansas Department of Health and Environment, signed by the EPA Regional Administrator on July 29, 1983;

(2) Addendum No. 1 of the Memorandum of Agreement, signed by the EPA

Regional Administrator on August 29, 1983.

(d) *Statement of legal authority.* (1) ''Statement of Attorney General'', signed by the Attorney General of the State of Kansas, November 25, 1981;

(2) ''Supplemental Statement of Attorney General'', signed by the Attorney General of the State of Kansas, undated (one page).

(e) *Program description.* The program description and any other materials submitted as part of the application or supplements thereto.

[49 FR 45306, Nov. 15, 1984, as amended at 56 FR 9415, Mar. 6, 1991]

§ 147.851 State-administered program—Class II wells.

The UIC program for Class II wells in the State of Kansas, except those on Indian lands as described in § 147.860, is the program administered by the Kansas Corporation Commission and the Kansas Department of Health and Environment, approved by EPA pursuant to section 1425 of the SDWA. Notice of this approval was published in the FEDERAL REGISTER on February 8, 1984 (49 FR 4735); the effective date of this program is February 8, 1984. This program consists of the following elements, as submitted to EPA in the State's program application.

[49 FR 45306, Nov. 15, 1984]

§§ 147.852—147.859 [Reserved]

§ 147.860 EPA-administered program—Indian lands.

(a) *Contents.* The UIC program for all classes of wells on Indian lands in the State of Kansas is administered by EPA. This program consists of the UIC program requirements of 40 CFR parts 124, 144, 146, 148, and any additional requirements set forth in the remainder of this subpart. Injection well owners and operators, and EPA shall comply with these requirements.

(b) *Effective date.* The effective date of the UIC program for Indian lands in Kansas is December 30, 1984.

[49 FR 45307, Nov. 15, 1984, as amended at 56 FR 9415, Mar. 6, 1991]

Subpart S—Kentucky

§ 147.900 State-administered program. [Reserved]

§ 147.901 EPA-administered program.

(a) *Contents.* The UIC program for the Commonwealth of Kentucky, including all Indian lands, is administered by EPA. This program consists of the UIC program requirements of 40 CFR parts 124, 144, 146, 148, and any additional requirements set forth in the remainder of this subpart. Injection well owners and operators, and EPA shall comply with these requirements.

(b) *Effective dates.* The effective date for the UIC program on Indian lands is November 25, 1988. The effective date for the UIC program in the remainder of Kentucky is June 25, 1984.

[53 FR 43087, Oct. 25, 1988, as amended at 56 FR 9415, Mar. 6, 1991]

§ 147.902 Aquifer exemptions. [Reserved]

§ 147.903 Existing Class I, II (except enhanced recovery and hydrocarbon storage) and III wells authorized by rule.

Maximum injection pressure. The owner or operator shall limit injection pressure to the lesser of:

(a) A value which will not exceed the operating requirements of § 144.28(f)(3)(i) or (ii) as applicable or;

(b) A value for well head pressure calculated by using the following formula:

$$Pm=(0.733–0.433\ Sg)d$$

where

Pm=injection pressure at the well head in pounds per square inch

Sg=specific gravity of inject fluid (unitless)

d=injection depth in feet.

§ 147.904 Existing Class II enhanced recovery and hydrocarbon storage wells authorized by rule.

(a) *Maximum injection pressure.* (1) To meet the operating requirements of § 144.28(f)(3)(ii) (A) and (B) of this chapter, the owner or operator:

(i) Shall use an injection pressure no greater than the pressure established by the Regional Administrator for the field or formation in which the well is located. The Regional Administrator

shall establish such a maximum pressure after notice, opportunity for comment, and opportunity for a public hearing, according to the provisions of part 124, subpart A of this chapter, and will inform owners and operators in writing of the applicable maximum pressure; or

(ii) May inject at pressures greater than those specified in paragraph (a)(1)(i) of this section for the field or formation in which he is operating provided he submits a request in writing to the Regional Administrator, and demonstrates to the satisfaction of the Regional Administrator that such injection pressure will not violate the requirement of §144.28(f)(3)(ii) (A) and (B). The Regional Administrator may grant such a request after notice, opportunity for comment, and opportunity for a public hearing, according to the provisions of part 124, subpart A of this chapter.

(2) Prior to such time as the Regional Administrator establishes rules for maximum injection pressure based on data provided pursuant to paragraph (a)(2)(ii) of this section the owner or operator shall:

(i) Limit injection pressure to a value which will not exceed the operating requirements of §144.28(f)(3)(ii); and

(ii) Submit data acceptable to the Regional Administrator which defines the fracture pressure of the formation in which injection is taking place. A single test may be submitted on behalf of two or more operators conducting operations in the same formation, if the Regional Administrator approves such submission. The data shall be submitted to the Regional Administrator within 1 year of the effective date of this program.

(b) *Casing and Cementing.* Where the Regional Administrator determines that the owner or operator of an existing enhanced recovery or hydrocarbon storage well may not be in compliance with the requirements of §§144.28(e) and 146.22, the owner or operator shall comply with paragraphs (b) (1) through (4) of this section, when required by the Regional Administrator:

(1) Protect USDWs by:

(i) Cementing surface casing by recirculating the cement to the surface from a point 50 feet below the lowermost USDW; or

(ii) Isolating all USDWs by placing cement between the outermost casing and the well bore; and

(2) Isolate any injection zones by placing sufficient cement to fill the calculated space between and the casing the well bore to a point 250 feet above the injection zone; and

(3) Use cement:

(i) Of sufficient quantity and quality to withstand the maximum operating pressure;

(ii) Which is resistant to deterioration from formation and injection fluids; and

(iii) In a quantity no less than 120% of the calculated volume necessary to cement off a zone.

(4) The Regional Administrator may specify other requirements in addition to or in lieu of the requirements set forth in paragraphs (b) (1) through (3) of this section, as needed to protect USDWs.

§147.905 Requirements for all wells—area of review.

Notwithstanding the alternatives presented in §146.6 of this chapter, the area of review shall be a minimum fixed radius as described in §146.6(b) of this chapter.

Subpart T—Louisiana

§147.950 State-administered program.

The UIC program for Class I, II, III, IV, and V wells in the State of Louisiana, except those wells on Indian lands, is the program administered by the Louisiana Department of Natural Resources approved by EPA pursuant to sections 1422 and 1425 of the SDWA. Notice of this approval was published in the FEDERAL REGISTER on April 23, 1982 (47 FR 17487); the effective date of this program is March 23, 1982. This program consists of the following elements, as submitted to EPA in the State's program application:

(a) *Incorporation by reference.* The requirements set forth in the State statutes and regulations cited in this paragraph are hereby incorporated by reference and made a part of the applicable UIC program under the SDWA for

the State of Louisiana. This incorporation by reference was approved by the Director of the Federal Register on June 25, 1984.

(1) Louisiana Revised Statutes Annotated sections 30:1–30:24 (1975 and Supp. 1982);

(2) Underground Injection Control Program Regulations for Class I, III, IV, and V wells, Statewide Order No. 29–N–1 (February 20, 1982), as amended June 1, 1985 and January 20, 1986;

(3)(i) Statewide Order Governing the Drilling for and Producing of Oil and Gas in the State of Louisiana, Statewide Order No. 29–B (August 26, 1974) (Composite Order Incorporating Amendments through March 1, 1974);

(ii) Amendments to Statewide Order No. 29–B (Off-site Disposal of Drilling Mud and Salt Water Generated from Drilling and Production of Oil and Gas Wells) (effective July 20, 1980);

(iii) Amendment to Statewide Order No. 29–B (Amendment concerning the use of Tables 5A and 6A, etc.) (December 15, 1980, effective January 1, 1981);

(iv) Amendment to Statewide Order No. 29–B (Amendment concerning the underground injection control of saltwater disposal wells, enhanced recovery injection wells, and liquid hydrocarbon storage wells) (effective February 20, 1982);

(v) Amendment to Statewide Order No. 29–B (Amendment concerning the offsite disposal of drilling mud and saltwater) (effective May 20, 1983);

(vi) Amendment to Statewide Order No. 29–B (Amendment concerning disposal of nonhazardous oilfield waste) (March 20, 1984, effective May 20, 1984);

(vii) Amendment to Statewide Order No. 29–B (Amendment concerning the administrative approval of injectivity tests and pilot projects in order to determine the feasibility of proposed enhanced recovery projects) (June 20, 1985, effective July 1, 1985).

(4) (i) Statewide Order adopting rules and regulations pertaining to the use of salt dome cavities (i.e., storage chambers) for storage of liquid and/or gaseous hydrocarbons, etc., Statewide Order No. 29–M (July 6, 1977, effective July 20, 1977);

(ii) Supplement to Statewide Order No. 29–M (October 2, 1978);

(iii) Second Supplement to Statewide Order No. 29–M (June 8, 1979).

(b)(1) The Memorandum of Agreement (Class I, III, IV, and V wells) between EPA Region VI and the Louisiana Department of Natural Resources, Office of Conservation, signed by the EPA Regional Administrator on March 17, 1982 and amended by Addendum 1 and Addendum 2 on November 3, 1989;

(2) The Memorandum of Agreement (Class II wells) between EPA Region VI and the Louisiana Department of Natural Resources, Office of Conservation, signed by the EPA Regional Administrator on March 17, 1982.

(c) *Statement of legal authority*. (1) Letter from Attorney General of Louisiana to EPA, ''Re: Louisiana Underground Injection Control Program Authorization for State of Louisiana'' (Class I, III, IV and V Wells), January 13, 1982, (10 pages);

(2) Letter from Attorney General of Louisiana to EPA, ''Re: Louisiana Underground Injection Control Program Authorization for State of Louisiana'' (Class II Wells), January 13, 1982 (5 pages).

(3) Letter from Attorney General of Louisiana to EPA, ''Re: Class I Hazardous Waste Injection Well Regulatory Program; Attorney General's Statement, October 9, 1989 (9 pages);

(d) The Program Description and any other materials submitted as part of the application or as supplements thereto.

[49 FR 20197, May 11, 1984, as amended at 56 FR 9415, Mar. 6, 1991]

§ 147.951 EPA-administered program—Indian lands.

(a) *Contents.* The UIC program for all classes of wells on Indian lands in the State of Louisiana is administered by EPA. This program consists of the UIC program requirements of 40 CFR parts 124, 144, 146, 148, and any additional requirements set forth in the remainder of this subpart. Injection well owners and operators, and EPA shall comply with these requirements.

(b) *Effective dates.* The effective date of the UIC program for Indian lands in Louisiana is November 25, 1988.

[53 FR 43087, Oct. 25, 1988, as amended at 56 FR 9415, Mar. 6, 1991]

Subpart U—Maine

§147.1000 State-administered program.

The UIC program for all classes of wells in the State of Maine, except those on Indian lands, is the program administered by the Maine Department of Environmental Protection approved by EPA pursuant to section 1422 of the SDWA. Notice of this approval was published in the FEDERAL REGISTER on August 25, 1983 (48 FR 38641); the effective date of this program is September 26, 1983. This program consists of the following elements, as submitted to EPA in the State's program application.

(a) *Incorporation by reference.* The requirements set forth in the State statutes and regulations cited in this paragraph are hereby incorporated by reference and made part of the applicable UIC program under the SDWA for the State of Maine. This incorporation by reference was approved by the Director of the OFR on June 25, 1984.

(1) Maine Revised Statutes Annotated title 38, sections 361–A, 363–B, 413, 414, 414–A, 420, and 1317–A (1978);

(2) Rules to Control the Subsurface Discharge of Pollutants by Well Injection, Rules of the Department of Environmental Protection, Chapter 543 (adopted June 22, 1983, effective July 4, 1983).

(b) The Memorandum of Agreement between EPA Region I and the Maine Department of Environmental Protection, signed by the EPA Regional Administrator on May 16, 1983.

(c) *Statement of legal authority.* Letter from Attorney General of Maine to EPA Regional Administrator, ''Re: Attorney General's Statement: Maine Underground Injection Control Program Primacy Application,'' June 30, 1983.

(d) The Program Description and any other materials submitted as part of the application or as supplements thereto.

[49 FR 20197, May 11, 1984, as amended at 53 FR 43088, Oct. 25, 1988; 56 FR 9415, Mar. 6, 1991]

§147.1001 EPA-administered program—Indian lands.

(a) *Contents.* The UIC program for all classes of wells on Indian lands in the State of Maine is administered by EPA. This program consists of the UIC program requirements of 40 CFR parts 124, 144, 146, 148, and any additional requirements set forth in the remainder of this subpart. Injection well owners and operators and EPA shall comply with these requirements.

(b) *Effective dates.* The effective date of the UIC program for Indian lands in Maine is November 25, 1988.

[53 FR 43088, Oct. 25, 1988, as amended at 56 FR 9416, Mar. 6, 1991]

Subpart V—Maryland

§147.1050 State-administered program—Class I, II, III, IV, and V wells.

The UIC program for Class I, II, III, IV, and V wells in the State of Maryland, except those wells on Indian lands, is the program administered by the Maryland Department of the Environment approved by EPA pursuant to section 1422 of the SDWA. Notice of this approval was published in the FR on April 19, 1984 (49 FR 15553); the effective date of this program is June 4, 1984. This program consists of the following elements, as submitted to EPA in the State's program application:

(a) *Incorporation by reference.* The requirements set forth in the State statutes and regulations cited in this paragraph are hereby incorporated by reference and made a part of the applicable UIC program under the SDWA for the State of Maryland. This incorporation by reference was approved by the Director of the OFR in accordance with 5 U.S.C. 552(a) and 1 CFR part 51. Copies may be obtained at the Maryland Department of the Environment, 2500 Broening Highway, Baltimore, Maryland, 21224. Copies may be inspected at the Environmental Protection Agency, Region III, 841 Chestnut Street, Philadelphia, Pennsylvania, 19107, or at the Office of the Federal Register, 800 North Capitol Street, NW., suite 700, Washington, DC.

(1) Code of Maryland Regulations, Title 26, Subtitle 08, Chapter 07 promulgated and effective as of March 1, 1989;

(2) Code of Maryland Regulations, Title 26, Subtitle 08, Chapter 01, promulgated and effective as of March 1, 1989;

(3) Code of Maryland Regulations, Title 26, Subtitle 08, Chapter 02, promulgated and effective as of March 1, 1989;

(4) Code of Maryland Regulations, Title 26, Subtitle 08, Chapter 03, promulgated and effective as of March 1, 1989;

(5) Code of Maryland Regulations, Title 26, Subtitle 08, Chapter 04, promulgated and effective as of March 1, 1989;

(6) Code of Maryland Regulations, Title 26, Subtitle 13, Chapter 05, section .19, promulgated and effective as of August 1, 1989;

(7) Code of Maryland Regulations, Title 26, Subtitle 01, Chapter 02, promulgated and effective as of March 1, 1989;

(8) Code of Maryland Regulations, Title 26, Subtitle 01, Chapter 04, promulgated and effective as of March 1, 1989.

(b) *Memorandum of Agreement*. The Memorandum of Agreement between EPA Region III and the Maryland Department of the Environment, as submitted on August 2, 1983, and revised on February 16, 1984.

(c) *Statement of legal authority*. Statement from the Maryland Attorney General on the Underground Injection Control Program, as submitted on August 2, 1983, and revised on February 16, 1984.

(d) *Program Description*. The Program Description and other materials submitted as part of the application or as supplements thereto.

[56 FR 9416, Mar. 6, 1991]

§§ 147.1051—147.1052 [Reserved]

§ 147.1053 EPA-administered program—Indian lands.

(a) *Contents.* The UIC program for all classes of wells on Indian lands in the State of Maryland is administered by EPA. This program consists of the UIC program requirements of 40 CFR parts 124, 144, 146, 148, and any additional requirements set forth in the remainder of this subpart. Injection well owners and operators, and EPA shall comply with these requirements.

(b) *Effective date.* The effective date of the UIC program for Indian lands in Maryland is November 25, 1988.

[53 FR 43088, Oct. 25, 1988, as amended at 56 FR 9416, Mar. 6, 1991]

§§ 147.1054—147.1099 [Reserved]

Subpart W—Massachusetts

§ 147.1100 State-administered program.

The UIC program for all classes of wells in the State of Massachusetts, except those on Indian lands, is the program administered by the Massachusetts Department of Environmental Protection, approved by EPA pursuant to section 1422 of the SDWA. Notice of this approval was published in the FEDERAL REGISTER on November 23, 1982 (47 FR 52705); the effective date of this program is December 23, 1982. This program consists of the following elements, as submitted to EPA in the State's program application:

(a) *Incorporation by reference.* The requirements set forth in the State statutes and regulations cited in this paragraph are hereby incorporated by reference and made a part of the applicable UIC program under the SDWA for the State of Massachusetts. This incorporation by reference was approved by the Director of the Federal Register on June 25, 1984.

(1) Massachusetts General Laws Annotated chapter 21, sections 27, 43, and 44 (West 1981);

(2) Code of Massachusetts Regulations, title 310, sections 23.01–23.11 as amended April 26, 1982.

(b) The Memorandum of Agreement between EPA Region I and the Massachusetts Department of Environmental Quality Engineering, signed by the EPA Regional Administrator on August 18, 1982.

(c) *Statement of legal authority*. ''Underground Injection Control Program—Attorney General's Statement for Class I, II, III, IV and V Injection Wells,'' signed by Assistant Attorney General for Attorney General of Massachusetts, May 13, 1982.

(d) The Program Description and any other materials submitted as part of

the application or as supplements thereto.

[49 FR 20197, May 11, 1984, as amended at 53 FR 43088, Oct. 25, 1988]

§147.1101 EPA-administered program—Indian lands.

(a) *Contents.* The UIC program for all classes of wells on Indian lands in the State of Massachusetts is administered by EPA. This program consists of the UIC program requirements of 40 CFR parts 124, 144, 146, 148, and any additional requirements set forth in the remainder of this subpart. Injection well owners and operators, and EPA shall comply with these requirements.

(b) *Effective date.* The effective date of the UIC program for Indian lands in Massachusetts is November 25, 1988.

[53 FR 43088, Oct. 25, 1988, as amended at 56 FR 9416, Mar. 6, 1991]

Subpart X—Michigan

§147.1150 State-administered program. [Reserved]

§147.1151 EPA-administered program.

(a) *Contents.* The UIC program for the State of Michigan, including all Indian lands, is administered by EPA. This program consists of the UIC program requirements of 40 CFR parts 124, 144, 146, 148, and any additional requirements set forth in the remainder of this subpart. Injection well owners and operators, and EPA shall comply with these requirements.

(b) *Effective dates.* The effective date for the UIC program for all lands in Michigan, including Indian lands, is June 25, 1984.

[52 FR 17681, May 11, 1987, as amended at 56 FR 9416, Mar. 6, 1991]

§147.1152 Aquifer exemptions. [Reserved]

§147.1153 Existing Class I, II (except enhanced recovery and hydrocarbon storage) and III wells authorized by rule.

Maximum injection pressure. The owner or operator shall limit injection pressure to the lesser of:

(a) A value which will not exceed the operating requirements of §144.28(f)(3)(i) or (ii) as applicable; or

(b) A value for well head pressure calculated by using the following formula:

$$Pm=(0.800-0.433\ Sg)d$$

where

Pm=injection pressure at the well head in pounds per square inch

Sg=specific gravity of injected fluid (unitless)

d=injection depth in feet.

§147.1154 Existing Class II enhanced recovery and hydrocarbon storage wells authorized by rule.

(a) *Maximum injection pressure.* (1) To meet the operating requirements of §144.28(f)(3)(ii) (A) and (B) of this chapter, the owner or operator:

(i) Shall use an injection pressure no greater than the pressure established by the Regional Administrator for the field or formation in which the well is located. The Regional Administrator shall establish such a maximum pressure after notice, opportunity for comment, and opportunity for a public hearing, according to the provisions of part 124, subpart A of this chapter, and will inform owners and operators in writing of the applicable maximum pressure; or

(ii) May inject at pressures greater than those specified in paragraph (a)(1)(i) of this section for the field or formation in which he is operating provided he submits a request in writing to the Regional Administrator, and demonstrates to the satisfaction of the Regonal Administrator that such injection pressure will not violate the requirement of §144.28(f)(3)(ii) (A) and (B). The Regional Administrator may grant such a request after notice, opportunity for comment, and opportunity for a public hearing, according to the provisions of part 124, subpart A of this chapter.

(2) Prior to such time as the Regional Administrator establishes field rules for maximum injection pressure based on data provided pursuant to paragraph (a)(2)(ii) of this section the owner or operator shall:

(i) Limit injection pressure to a value which will not exceed the operating requirements of §144.28(f)(3)(ii); and

(ii) Submit data acceptable to the Regional Administrator which defines the fracture pressure of the formation in which injection is taking place. A

single test may be submitted on behalf of two or more operators conducting operations in the same formation, if the Regional Administrator approves such submission. The data shall be submitted to the Regional Administrator within 1 year following the effective date of this program.

(b) *Casing and cementing.* Where the Regional Administrator determines that the owner or operator of an existing enhanced recovery or hydrocarbon storage will may not be in compliance with the requirements of §§ 144.28(e) and 146.22, the owner or operator shall comply with paragraphs (b) (1) through (4) of this section, when required by the regional Administrator:

(1) Protect USDWs by:

(i) Cementing surface casing by recirculating the cement to the surface from a point 50 feet below the lowermost USDW; or

(ii) Isolating all USDWs by placing cement between the outermost casing and the well bore; and

(2) Isolate any injection zones by placing sufficient cement to fill the calculated space between the casing and the well bore to a point 250 feet above the injection zone; and

(3) Use cement:

(i) Of sufficient quantity and quality to withstand the maximum operating pressure;

(ii) Which is resistant to deterioration from formation and injection fluids; and

(iii) In a quantity no less than 120% of the calculated volume necessary to cement off a zone.

(4) The Regional Administrator may specify other requirements in addition to or in lieu of the requirements set forth in paragraphs (b) (1) through (3) of this section, as needed to protect USDWs.

§ 147.1155 Requirements for all wells.

(a) *Area of review.* Notwithstanding the alternatives presented in § 146.6 of this chapter, the area of review for Class II wells shall be a fixed radius as described in § 146.6(b) of this chapter.

(b) *Tubing and packer.* The owner or operator of an injection well injecting salt water for disposal shall inject through tubing and packer. The owner of an existing well must comply with this requirement within one year of the effective date of this program.

Supbart Y—Minnesota

§ 147.1200 State-administered program. [Reserved]

§ 147.1201 EPA-administered program.

(a) *Contents.* The UIC program for the State of Minnesota is administered by EPA. This program consists of the UIC program requirements of 40 CFR parts 124, 144, 146, 148, and any additional requirements set forth in the remainder of this subpart. Injection well owners and operators, and EPA shall comply with these requirements.

(b) *Effective date.* The effective date of the UIC program for Minnesota is: June 11, 1984.

[49 FR 20197, May 11, 1984, as amended at 56 FR 9416, Mar. 6, 1991]

§ 147.1202 Aquifer exemptions. [Reserved]

§ 147.1210 Requirements for Indian lands.

(a) *Purpose and scope.* This section sets forth additional requirements that apply to injection activities on Indian lands in Minnesota.

(b) *Requirements.* Notwithstanding the other requirements of this subpart, for Indian lands described in paragraph (a) of this section, no owner or operator shall construct, operate, maintain, or convert any Class I, II, III, or IV well. The UIC program for Class V wells on such Indian Lands is administered by EPA, and consists of the applicable requirements of 40 CFR parts 124, 144, and 146. In addition, no owner or operator shall abandon a well without the approval of the Regional Administrator.

(c) *Effective date.* The effective date of the UIC program requirements for Indian lands in Minnesota is December 30, 1984.

[49 FR 45307, Nov. 15, 1984]

Subpart Z—Mississippi

§147.1250 State-administered program—Class I, III, IV, and V wells.

The UIC program for Class I, III, IV and V wells in the State of Mississippi, except those on Indian lands, is the program administered by the Mississippi Department of Natural Resources approved by EPA pursuant to section 1422 of the SDWA. Notice of this approval was published in the FEDERAL REGISTER on August 25, 1983 (48 FR 38641); the effective date of this program is September 26, 1983. This program consists of the following elements, as submitted to EPA in the State's program application:

(a) *Incorporation by reference.* The requirements set forth in the State statutes and regulations cited in this paragraph are hereby incorporated by reference and made a part of the applicable UIC program under the SDWA for the State of Mississippi. This incorporation by reference was approved by the Director of the Federal Register on June 25, 1984.

(1) Mississippi Air and Water Pollution Control Law, Mississippi Code Annotated sections 49–17–1 through 49–17–29 (1972) and Supp. 1983);

(2) Mississippi Department of Natural Resources, Bureau of Pollution Control, Underground Injection Control Program Regulations (adopted February 11, 1982);

(3) Mississippi Department of Natural Resources, Bureau of Pollution Control, State of Mississippi Wastewater Permit Regulations for National Pollutant Discharge Elimination System (NPDES), Underground Injection Control (UIC), and State Operating Permits (adopted May 1, 1974; amended February 11, 1982).

(b) The Memorandum of Agreement between EPA Region IV and the Mississippi Department of Natural Resources, signed by the EPA Regional Administrator on February 8, 1983.

(c) *Statement of legal authority.* (1) Letter from Attorney General of Mississippi (by Special Assistant Attorney General) to Executive Director, Mississippi Department of Natural Resources, ''Re: Mississippi Department of Natural Resources, Bureau of Pollution Control, State Underground Injection Control (UIC) Program; Statement of the Attorney General of the State of Mississippi,'' December 3, 1981;

(2) Letter from Attorney General of Mississippi (by Special Assistant Attorney General) to Executive Director, Mississippi Department of Natural Resources, ''Re: Authority to Regulate and Take Samples from Underground Injection Systems,'' October 18, 1982;

(3) Letter from Attorney General of Mississippi (by Special Assistant Attorney General) to Regional Administrator, EPA Region IV, ''Re: Public Participation in State Enforcement Actions, UIC Program,'' June 10, 1983.

(d) The Program Description and any other materials submitted as part of the application or supplements thereto.

[49 FR 20197, May 11, 1984, as amended at 53 FR 43088, Oct. 25, 1988]

§147.1251 State-administered program—Class II wells.

The UIC program for Class II wells in the State of Mississippi, other than those on Indian lands, is the program administered by the State Oil and Gas Board of Mississippi approved by EPA pursuant to section 1425 of the SDWA. Notice of this approval was published in the FEDERAL REGISTER on March 2, 1989; the effective date of this program is March 2, 1989. This program consists of the following elements, as submitted to EPA in the State's program application:

(a) Incorporation by reference. The requirements set forth in the State statutes and regulations cited in this paragraph are hereby incorporated by reference and made a part of the applicable UIC program under the SDWA for the State of Mississippi. This incorporation by reference was approved by the Director of the Federal Register in accordance with 5 U.S.C. 552(a).

(1) Mississippi Code Annotated, section 5–9–9 (Supp. 1988).

(2) Mississippi Code Annotated, sections 53–1–1 through 53–1–47, inclusive and sections 53–1–71 through 53–1–77, inclusive (1972 and Supp. 1988).

(3) Mississippi Code Annotated, sections 53–3–1 through 53–3–165, inclusive (1972 and Supp. 1988).

(4) State Oil and Gas Board Statewide Rules and Regulations, Rules 1

through 65, inclusive (Aug. 1, 1987, as amended, Sept. 17, 1987).

(b) The Memorandum of Agreement between EPA Region IV and the State Oil and Gas Board of Mississippi signed by the Regional Administrator on October 31, 1988.

(c) Statement of legal authority. Statement from the Attorney General signed on October 1, 1987 with amendments to the Statement signed August 5, 1988 and September 15, 1988 by the Special Assistant Attorney General.

(d) The Program Description and any other materials submitted as part of the original application or as supplements thereto.

[54 FR 8735, Mar. 2, 1989]

§ 147.1252 EPA-administered program—Indian lands.

(a) *Contents.* The UIC program for all classes of wells on Indian lands in the State of Mississippi is administered by EPA. This program consists of the UIC program requirements of 40 CFR parts 124, 144, 146, 148, and any additional requirements set forth in the remainder of this subpart. Injection well owners and operators, and EPA shall comply with these requirements.

(b) *Effective date.* The effective date of the UIC program on Indian lands is November 25, 1988.

[53 FR 8735, Mar. 2, 1989, as amended at 56 FR 9416, Mar. 6, 1991]

Subpart AA—Missouri

147.1300 State-administered program.

The UIC program for all classes of wells in the State of Missouri, except those on Indian lands, is administered by the Missouri Department of Natural Resources, approved by EPA pursuant to section 1422 and 1425 of the SDWA. Notice of this approval was published in the FEDERAL REGISTER on December 2, 1983 (48 FR 54349); the effective date of this program is December 2, 1983). This program consists of the following elements, as submitted to EPA in the State's program application.

(a) *Incorporation by reference.* The requirements set forth in the State statutes and regulations cited in this paragraph are hereby incorporated by reference and made a part of the applicable UIC program under the SDWA for the State of Missouri. This incorporation by reference was approved by the Director of the Federal Register on June 25, 1984.

(1) Vernon's Annotated Missouri Statutes sections 259.010 to 259.240 (Supp. 1984);

(2) Missouri Code of State Regulations, title 10, division 50, chapters 1 and 2 (June 1984);

(3) Vernon's Annotated Missouri Statutes chapter 204, §§ 204.006 through 204.470 (1983 and Cumm. Supp. 1990).

(b) The Memorandum of Agreement between EPA Region VII and the Missouri Department of Oil and Gas, signed by the EPA Regional Administrator on December 3, 1982.

(c) *Statement of legal authority.* (1) Opinion Letter No. 63 and attached Memorandum Opinion, signed by Attorney General of Missouri, March 16, 1982;

(2) Addendum to Opinion Letter No. 63 (1982), signed by Attorney General of Missouri, October 28, 1982.

(3) Opinion No. 127–83, signed by Attorney General of Missouri, July 11, 1983.

(d) The Program Description and any other materials submitted as part of the application or as supplements thereto.

[49 FR 20197, May 11, 1984, as amended at 53 FR 43088, Oct. 25, 1988; 56 FR 9416, Mar. 6, 1991]

§ 147.1301 State-administered program—Class I, III, IV, and V wells.

The UIC program for Class I, III, IV, and V wells in the State of Missouri, other than those on Indian lands, is the program administered by the Missouri Department of Natural Resources, approved by EPA pursuant to section 1422 of the SDWA. Notice of this approval was published in the FEDERAL REGISTER on November 2, 1984; the effective date of this program is July 31, 1985. This program consists of the following elements, as submitted to EPA in the State's program application.

(a) *Incorporation by reference.* The requirements set forth in the State statutes and regulations cited in this paragraph are hereby incorporated by reference and made a part of the applicable UIC program under the SDWA for the State of Missouri. This incorporation by reference was approved by the Director of the Federal Register effective July 31, 1985.

(1) Revised Statutes of the State of Missouri, Volume 2, sections 204.016, 204.026, 204.051, 204.056 and Volume V, section 577.155 (1978 and Cumm. Supp. 1984);

(2) Missouri Code of State Regulations, title 10, division 20, Chapter 6, sections 20–6.010, 20–6.020, 20–6.070, 20–6.080, 20–6.090, and title 10, division 20, Chapter 7, section 20–7.031 (1977, amended 1984).

(b) *Other laws.* The following statutes and regulations, although not incorporated by reference except for select sections identified in paragraph (a) of this section, are also part of the approved State-administered program.

(1) Revised Statutes of the State of Missouri, chapters 204, 260, 536, 557, 558 and 560; sections 640.130.1 and 1.020 (1978 and Cumm. Supp. 1984);

(2) Rule 52.12 Vernon's Annotated Missouri Rules (1978);

(3) Missouri Code of State Regulations, title 10, division 20, Chapters 1 through 7 (1977, amended 1984).

(c) The Memorandum of Agreement between EPA Region VII and the Missouri Department of Natural Resources, signed by the EPA Regional Administrator on October 10, 1984.

(d) *Statement of Legal Authority.* Opinion No. 123–84, signed by Attorney General of Missouri, September 24, 1984. Amended April 2, 1985.

(e) The Program Description and any other materials submitted as part of the application or as supplements thereto.

[50 FR 28942, July 17, 1985]

§147.1302 Aquifer exemptions. [Reserved]

§147.1303 EPA-administered program—Indian lands.

(a) *Contents.* The UIC program for all classes of wells on Indian lands in the State of Missouri is administered by EPA. This program consists of the UIC program requirements of 40 CFR parts 124, 144, 145, 146, 148, and any additional requirements set forth in the remainder of this subpart. Injection well owners and operators, and EPA shall comply with these requirements.

(b) *Effective date.* The effective date for the UIC program for Indian lands is November 25, 1988.

[53 FR 43088, Oct. 25, 1988, as amended at 56 FR 9417, Mar. 6, 1991]

Subpart BB—Montana

§147.1350 State-administered programs—Class II wells.

The UIC program for Class II injection wells in the State of Montana, except for those in Indian Country, is the program administered by the Montana Board of Oil and Gas Conservation (MBOGC) approved by the EPA pursuant to Section 1425 of the SDWA. Notice of this approval was published in the FEDERAL REGISTER on November 19, 1996; the effective date of this program is November 19, 1996. This program consists of the following elements as submitted to EPA in the State's program application:

(a) *Incorporation by reference.* The requirements set forth in the State statutes and regulations cited in this paragraph are hereby incorporated by reference and made part of the applicable UIC program under the SDWA for the State of Montana. This incorporation by reference was approved by the Director of the FR in accordance with 5 U.S.C. 552(a) and 1 CFR part 51. Copies may be obtained at the Montana Board of Oil and Gas Conservation, 2535 St. Johns Avenue, Billings, Montana, 59102. Copies may be inspected at the Environmental Protection Agency, Region VIII, 999 18th Street, Suite 500, Denver, Colorado, 80202–2466, or at the Office of the Federal Register, 800 North Capitol Street, NW, Suite 700, Washington, D.C.

(1) Montana Statutory Requirements Applicable to the Underground Injection Control Program, August, 1996.

(2) Montana Regulatory Requirements Applicable to the Underground Injection Control Program, August, 1996.

(b) *Memorandum of Agreement (MOA).* (1) The MOA between EPA Region VIII and the MBOGC signed by the Acting EPA Regional Administrator on June 9, 1996.

(2) Letter dated May 24, 1996, from the Administrator of the MBOGC and the attached addendum (Addendum No. 1–96) to the MOA between MBOGC and EPA Region VIII, signed by the Acting EPA Regional Administrator on August 14, 1996.

(c) *Statement of legal authority.* (1) Letter from the Montana Attorney General to the Regional Administrator dated August 1, 1995.

(2) MBOGC independent counsel's certification of Montana's UIC program for Class II wells dated July 24, 1995.

(3) Letter dated March 8,1996, from MBOGC independent counsel to USEPA, Region VIII; ''Re: EPA comments of November 29, 1995, on Montana Class II primacy application.''

(4) Letter dated March 8, 1996, from the Administrator of the MBOGC and the attached proposed replacement language for the MOA; ''Re: Responses to EPA comments on Montana Class II Primacy Application.''

(d) *Program Description.* The Program Description and any other materials submitted as part of the application or as supplemented thereto:

(1) Application and accompanying materials for approval of Montana's UIC program for Class II wells submitted by the Governor of Montana, August 3, 1995.

(2) [Reserved]

[61 FR 58933, Nov. 19, 1996]

§ 147.1351 EPA-administered program.

(a) *Contents.* The UIC program in the State of Montana for Class I, III, IV, and V wells, and for all Classes of wells in Indian Country is administered by EPA. This program consists of the UIC program requirements of 40 CFR parts 124, 144, 146, 148, and any additional requirements set forth in the remainder of this subpart. Injection well owners and operators, and EPA shall comply with these requirements.

(b) *Effective dates.* The effective date for the UIC program on all lands in Montana, including all Indian lands, is June 25, 1984.

[52 FR 17681, May 11, 1987, as amended at 56 FR 9417, Mar. 6, 1991; 61 FR 58933, Nov. 19, 1996]

§ 147.1352 Aquifer exemptions.

Those portions of aquifers within one-quarter mile of existing Class II wells are exempted for the purpose of Class II injection activities only.

NOTE: A complete listing of the exemptions and their location is available for review in the EPA Regional Office, 1860 Lincoln Street, Denver, Colorado. An updated list of exemptions will be maintained in the Regional Office.

§ 147.1353 Existing Class I, II (except enhanced recovery hydrocarbon storage) and III wells authorized by rule.

Maximum injection pressure. The owner or operator shall limit injection pressure to the lesser of:

(a) A value which will not exceed the operating requirements of § 144.28(f)(3)(i) or (ii) as applicable or

(b) A value for well head pressure calculated by using the following formula:

$$Pm=(0.733 - 0.433\ Sg)d$$

where

Pm=injection pressure at the well head in pounds per square inch

Sg=specific gravity of inject fluid (unitless)

d=injection depth in feet.

§ 147.1354 Existing Class II enhanced recovery and hydrocarbon storage wells authorized by rule.

(a) *Maximum injection pressure.* (1) To meet the operating requirements of § 144.28(f)(3)(ii) (A) and (B) of this chapter, the owner or operator:

(i) Shall use an injection pressure no greater than the pressure established by the Regional Administrator for the field or formation in which the well is located. The Regional Administrator shall establish such a maximum pressure after notice, opportunity for comment, and opportunity for a public hearing, according to the provisions of part 124, subpart A of this chapter, and will inform owners and operators in writing of the applicable maximum pressure; or

(ii) May inject at pressures greater than those specified in paragraph

(a)(1)(i) of this section for the field or formation in which he is operating provided he submits a request in writing to the Regional Administrator, and demonstrates to the satisfaction of the Regional Administrator that such injection pressure will not violate the requirement of §144.28(f)(3)(ii) (A) and (B). The Regional Administrator may grant such a request after notice, opportunity for comment, and opportunity for a public hearing, according to the provisions of part 124, subpart A of this chapter.

(2) Prior to such time as the Regional Administrator established rules for maximum injection pressure based on data provided pursuant to paragraph (ii) below the owner or operator shall:

(i) Limit injection pressure to a value which will not exceed the operating requirements of §144.28(f)(3)(ii); and

(ii) Submit data acceptable to the Regional Administrator which defines the fracture pressure of the formation in which injection is taking place. A single test may be submitted on behalf of two or more operators conducting operations in the same formation, if the Regional Administrator approves such submission. The data shall be submitted to the Regional Administrator within 1 year of the effective date of this program.

(b) *Casing and cementing.* Where the Regional Administrator determines that the owner or operator of an existing enhanced recovery or hydrocarbon storage well may not be in compliance with the requirements of §§144.28(e) and 146.22, the owner or operator shall when required by the Regional Administrator:

(1) Isolate all USDWs by placing cement between the outermost casing and the well bore as follows:

(i) If the injection well is east of the 108th meridian, cement the outermost casing from a point 50 feet into a major shale formation underlying the uppermost USDW to the surface. For the purpose of this paragraph, major shale formations are defined as the Bearpaw, Clagget, and Colorado formations.

(ii) If the injection well is west of the 108th meridian, cement the outermost casing to a depth of 1,000 feet, or to the base of the lowermost USDW in use as a source of drinking water whichever is deeper. The Regional Administrator may allow an owner or operator to cement to a lesser depth if he can demonstrate to the satisfaction of the Regional Administrator that no USDW will be affected by the injection facilities.

(2) Isolate any injection zones by placing sufficient cement to fill the calculated space between the casing and the well bore to a point 250 feet above the injection zone; and

(3) Use cement:

(i) Of sufficient quantity and quality to withstand the maximum operating pressure;

(ii) Which is resistant to deteriortion from formation and injection fluids; and

(iii) In a quantity no less than 120% of the calculated volume necessary to cement off a zone.

(4) The Regional Administrator may specify other requirements in addition to or in lieu of the requirements set forth in paragraphs (b) (1) through (3) of this section, as needed to protect USDWs.

§147.1355 Requirements for all wells.

(a) *Area of review.* Notwithstanding the alternatives presented in §146.6 of this chapter, the area of review shall be a fixed radius as described in §146.06(b) of this chapter.

(b) The applicant must give separate notice of intent to apply for a permit to each owner or tenant of the land within one-quarter mile of the site. This requirement may be waived by the Regional Administrator where individual notice to all land owners and tenants would be impractical. The addresses of those to whom notice is given, and a description of how notice was given, shall be submitted with the permit application. The notice shall include:

(1) Name and address of applicant;

(2) A brief description of the planned injection activities, including well location, name and depth of the injection zone, maximum injection pressure and volume, and fluid to be injected;

(3) EPA contact person; and

(4) A statement that opportunity to comment will be announced after EPA prepares a draft permit.

(c) Owners and operators on or within one-half mile of Indian lands shall provide notice as specified in paragraph (b) of this section, except that such notice shall be provided within a one-half mile radius of the site.

APPENDIX A TO SUBPART BB OF PART 147—STATE REQUIREMENTS INCORPORATED BY REFERENCE IN SUBPART BB OF PART 147 OF THE CODE OF FEDERAL REGULATIONS

The following is an informational listing of state requirements incorporated by reference in Subpart BB of part 147 of the Code of Federal Regulations:

Subpart BB—Montana

(a) The statutory provisions include:

(1) Montana Code annotated, 1995, Title 2, Chapter 15:

Section 2–15–121. Allocation for administrative purposes only.

Section 2–15–124. Quasi-judicial boards.

Section 2–15–3303. Board of oil and gas conservation-composition—allocation—quasi-judicial.

(2) Montana Code annotated, 1995, Title 82, Chapter 10:

Section 82–10–101. Action for accounting for royalty.

Section 82–10–102. Remedy not exclusive.

Section 82–10–103. Obligation to pay royalties as essence of contract-interest.

Section 82–10–104. Payment of royalties-form of record required.

Section 82–10–105 through 82–10–109 reserved.

Section 82–10–110. Division order-definition-effect.

Section 82–10–201. Authorization for lease and terms-land not subject to leasing.

Section 82–10–202. Acreage pooling.

Section 82–10–203. Interference with normal use of land prohibited.

Section 82–10–204. Lease of acquired oil and gas interests.

Section 82–10–301. Definitions.

Section 82–10–302. Policy.

Section 82–10–303. Use of eminent domain to acquire underground reservoirs.

Section 82–10–304. Certificate of board required prior to use of eminent domain.

Section 82–10–305. Proceedings.

Section 82–10–401. Notice required before abandonment of well-owner's option.

Section 82–10–402. Inventory of abandoned wells and seismic operations-reclamation procedures.

Section 82–10–501. Purpose-legislative findings.

Section 82–10–502. Definitions.

Section 82–10–503. Notice of drilling operations.

Section 82–10–504. Surface damage and disruption payments-penalty for late payment.

Section 82–10–505. Liability for damages to property.

Section 82–10–506. Notification of injury.

Section 82–10–0507. Agreement—offer of settlement.

Section 82–10–508. Rejection—legal action.

Section 82–10–509 and 82–10–510. Reserved.

Section 82–10–511. Remedies cumulative.

(3) Montana Code annotated, 1995, Title 82, Chapter 11:

Section 82–11–101. Definitions.

Section 82–11–102. Oil or gas wells not public utilities.

Section 82–11–103. Lands subject to law.

Section 82–11–104. Construction-no conflict with board of land commissioners' authority.

Section 82–11–105 through 82–11–110 reserved.

Section 82–11–111. Powers and duties of board.

Section 82–11–112. Intergovernmental cooperation.

Section 82–11–113. Role of board in implementation of national gas policy.

Section 82–11–114. Appointment of examiners.

Section 82–11–115. Procedure to make determinations.

Section 82–11–116. Public access.

Section 82–11–117. Confidentiality of records.

Section 82–11–118. Fees for processing applications.

Section 82–11–119 through 82–11–120 reserved.

Section 82–11–121. Oil and gas waste prohibited.

Section 82–11–122. Notice of intention to drill or conduct seismic operations-notice to surface owner.

Section 82–11–123. Requirements for oil and gas operations.

Section 82–11–124. Requirement relating to waste prevention.

Section 82–11–125. Availability of cores or chips, cuttings, and bottom-hole temperatures to board.

Section 82–11–126. Availability of facilities to bureau of mines.

Section 82–11–127. Prohibited activity.

Section 82–11–128 through 82–11–130 reserved.

Section 82–11–131. Privilege and license tax.

Section 82–11–132. Statements to treasurer and payment of tax.

Section 82–11–133. Penalty for late payment.

Section 82–11–134. Permit fees.

Section 82–11–135. Money earmarked for board expenses.

Section 82–11–136. Expenditure of funds from bonds for plugging wells.

Section 82–11–137. Class II injection well operating fee.

Section 82–11–138 through 82–11–140 reserved.

Section 82–11–141. Administrative procedure.

Section 82–11–142. Subpoena power-civil actions.

Section 82–11–143. Rehearing.

Section 82–11–144. Court review.

Section 82–11–145. Injunction or restraining order.

Section 82–11–146. Appeal.

Section 82–11–147. Violations.

Section 82–11–148. Criminal penalties.

Section 82–11–149. Civil penalties.

Section 82–11–150. Legal assistance.

Section 82–11–151. Emergencies-notice and hearing.

Section 82–11–152 through 82–11–160 reserved.

Section 82–11–161. Oil and gas production damage mitigation account-statutory appropriation.

Section 82–11–162. Release of producing oil or gas well from drilling bond-fee.

Section 82–11–163. Landowner's bond on noncommercial well.

Section 82–11–164. Lien created.

Section 82–11–165 through 82–11–170 reserved.

Section 82–11–171. Terminated.

Section 82–11–201. Establishment of well spacing units.

Section 82–11–202. Pooling of interest within spacing unit.

Section 82–11–203. Pooling agreements not in violation of antitrust laws.

Section 82–11–204. Hearing on operation of pool as unit.

Section 82–11–205. Board order for unit operation-criteria.

Section 82–11–206. Terms and conditions of plan for unit operations.

Section 82–11–207. Approval of plan for unit operations by persons paying costs.

Section 82–11–208. Board orders-amendment.

Section 82–11–209. Units established by previous order.

Section 82–11–210. Unit operations-less than whole of pool.

Section 82–11–211. Operations considered as done by all owners in unit.

Section 82–11–212. Property rights and operator's lien.

Section 82–11–213. Contract not terminated by board order.

Section 82–11–214. Title to oil and gas rights not affected by board order.

Section 82–11–215. Unit operation not restraint of trade.

Section 82–11–216. No creation of relationship between parties in unit.

Section 82–11–301. Authorization to join interstate compact for conservation of oil and gas.

Section 82–11–302. Interstate oil and gas compact.

Section 82–11–303. Extension of expiration date.

Section 82–11–304. Governor as member of Interstate Oil Compact Commission.

Section 82–11–305. Limitation on power of representative.

Section 82–11–306. Expenses of representative.

(b) The regulatory provisions include: Administrative Rules of Montana Board of Oil and Gas Conservation, Chapter 22, revised March 1996:

Rule 36.22.101. Organizational Rule.

Rule 36.22.201. Procedural Rules.

Rule 36.22.202. Environmental Policy Act Procedural Rules.

Rule 36.22.301. Effective Scope of Rules.
Rule 36.22.302. Definitions.
Rule 36.22.303. Classification of Wildcat or Exploratory Wells.
Rule 36 22.304. Inspection of Record, Properties, and Wells.
Rule 36.22.305. Naming of Pools.
Rule 36.22.306. Organization of Reports.
Rule 36.22.307. Adoption of Forms.
Rule 36.22.308. Seal of Board.
Rule 36.22.309. Referral of Administrative Decisions.
Rule 36.22.401. Office and Duties of Petroleum Engineer.
Rule 36.22.402. Office and Duties of Administrator.
Rule 36.22.403. Office and Duties of Geologist.
Rule 36.22.501. Shot Location Limitations.
Rule 36.22.502. Plugging and Abandonment.
Rule 36.22.503. Notification.
Rule 36.22.504. Identification.
Rule 36.22.601. Notice of Intention and Permit to Drill.
Rule 36.22.602. Notice of Intention to Drill and Application for Permit to Drill.
Rule 36.22.603. Permit Fees.
Rule 36.22.604. Permit Issuance - Expiration - Extension.
Rule 36.22.605. Transfer of Permits.
Rule 36.22.606. Notice and Eligibility Statement for Drilling or Recompletion in Unit Operations.
Rule 36.22.607. Drilling Permits Pending Special Field Rules.
Rule 36.22.701. Spacing Units - General.
Rule 36.22.702. Spacing of Wells.
Rule 36.22.703. Horizontal Wells.
Rule 36.22.1001. Rotary Drilling Procedure.
Rule 36.22.1002. Cable Drilling Procedure.
Rule 36.22.1003. Vertical Drilling Required Deviation.
Rule 36.22.1004. Dual Completion of Wells.
Rule 36.22.1005. Drilling Waste Disposal and Surface Restoration.
Rules 36.22.1006 through 36.22.1010. Reserved.
Rule 36.22.1011. Well Completion and Recompletion Reports.
Rule 36.22.1012. Samples of Cores and Cuttings.
Rule 36.22.1013. Filing of Completion Reports, Well Logs, Analyses, Reports, and Surveys.
Rule 36.22.1014. Blowout Prevention and Well Control Equipment.
Rule 36.22.1101. Fire Hazard Prevention.
Rule 36.22.1102. Fire Walls Required.
Rule 36.22.1103. Notification and Report of Emergencies and Undesirable Incidents.
Rule 36.22.1104. Control and Cleanup.
Rule 36.22.1105. Solid Waste.
Rule 36.22.1201. Surface Equipment.
Rule 36.22.1202. Identification.
Rule 36.22.1203. Chokes Required.
Rule 36.22.1204. Separators Required.
Rule 36.22.1205. Vacuum Pumps Prohibited.
Rule 36.22.1206. Tubing Required.
Rule 36.22.1207. Earthen Pits and Open Vessels.
Rule 36.22.1208. Producing from Different Pools Through the Same Casing.
Rules 36.22.1209 through 36.22.1212. Reserved.
Rule 36.22.1213. Reservoir or Pool Surveys.
Rule 36.22.1214. Subsurface Pressure Tests.
Rule 36.22.1215. Stabilized Production Test.
Rule 36.22.1216. Gas Oil Ratio Tests.
Rule 36.22.1217. Water Production Report.
Rule 36.22.1218. Gas to be Metered.
Rule 36.22.1219. Gas Waste Prohibited.
Rule 36.22.1220. Associated Gas Flaring Limitation—Application to exceed—Board Review and Action.
Rule 36.22.1221. Burning of Waste Gas Required.
Rule 36.22.1222. Hydrogen Sulfide Gas.
Rule 36.22.1223. Fencing, Screening, and Netting of Pits.
Rules 36.22.1224 and 36.22.1425. Reserved.
Rule 36.22.1226. Disposal of Water.
Rule 36.22.1227. Earthen Pits and Ponds.
Rule 36.22.1228. Disposal by Injection.
Rule 36.22.1229. Water Injection and Gas Repressuring.
Rule 36.22.1230. Application Contents and Requirements.
Rule 36.22.1231. Notice of Application Objections.
Rule 36.22.1232. Board Authorization.

Rule 36.22.1233. Notice of Commencement or Discontinuance—Plugging of Abandoned Wells.
Rule 36.22.1234. Record Required.
Rules 36.22.1235 through 36.22.1239. Reserved.
Rule 36.22.1240. Report of Well Status Change.
Rule 36.22.1241. Service Company Reports.
Rule 36.22.1242. Reports by Producers.
Rule 36.22.1243. Reports from Transporters, Refiners, and Gasoline or Extraction Plants.
Rule 36.22.1244. Producer's Certificate of Compliance.
Rule 36.22.1245. Illegal Production.
Rule 36.22.1301. Notice and Approval of Intention to Abandon Report.
Rule 36.22.1302. Notice of Abandonment.
Rule 36.22.1303. Well Plugging Requirement.
Rule 36.22.1304. Plugging Methods and Procedure.
Rule 36.22.1305. Exception for Fresh Water Wells.
Rule 36.22.1306. Approval for Pulling Casing and Reentering Wells.
Rule 36.22.1307. Restoration of Surface.
Rule 36.22.1308. Plugging and Restoration Bond.
Rule 36.22.1309. Subsequent Report of Abandonment.
Rule 36.22.1401. Definitions.
Rule 36.22.1402. Underground Injection.
Rule 36.22.1403. Application Contents and Requirements Rules.
Rule 36.22.1404 and 36.22.1405. Reserved.
Rule 36.22.1406. Corrective Action.
Rule 36.22.1407. Signing the Application.
Rule 36.22.1408. Financial Responsibility.
Rule 36.22.1409. Hearings.
Rule 36.22.1410. Notice of Application.
Rule 36.22.1411. Board Authorization.
Rules 36.22.1412 and 36.22.1413. Reserved.
Rule 36.22.1414. Notice of Commencement or Discontinuance—Plugging of Abandoned Wells.
Rule 36.22.1415. Records Required.
Rule 36.22.1416. Mechanical Integrity.
Rule 36.22.1417. Notification of Tests—Reporting Results.
Rule 36.22.1418. Exempt Aquifers.
Rule 36.22.1419. Tubingless Completions.
Rules 36.22.1420 and 36.22.1421. Reserved.
Rule 36.22.1422. Permit Conditions.
Rule 36.22.1423. Injection Fee—Well Classification.
Rule 36.22.1601. Who May Apply for Determination.
Rule 36.22.1602. Application Requirements and Contents.
Rule 36.22.1603. Documents and Technical Data Supporting Application.
Rule 36.22.1604. Docket Number.
Rule 36.22.1605. List of Applications—Public Access.
Rule 36.22.1606. Objections to Applications.
Rule 36.22.1607. Deadlines for Action Determinations.
Rule 36.22.1608. Deficient Applications.
Rule 36.22.1609. Board Action on Applications.
Rule 36.22.1610. Special Findings and Determinations New Onshore Production Wells Under Section 103.
Rule 36.22.1611. Special Findings and Determinations Stripper Well Production.

[61 FR 58934, Nov. 19, 1996]

Subpart CC—Nebraska

§147.1400 State-administered program—Class II wells.

The UIC program for Class II wells in the State of Nebraska, except those on Indian lands, is the program administered by the Nebraska Oil and Gas Conservation Commission, approved by EPA pursuant to section 1425 of the SDWA.

(a) *Incorporation by reference.* The requirements set forth in the State statutes and regulations cited in this paragraph are hereby incorporated by reference and made a part of the applicable UIC program under the SDWA for the State of Nebraska. This incorporation by reference was approved by the Director of the Federal Register on June 25, 1984.

(1) Rules and Regulations of the Nebraska Oil and Gas Conservation Commission, Rules 1 through 6 (as published by the Commission, May 1981);

(2) Revised Statutes of Nebraska, sections 57–903 and 57–906 (Reissue 1988).

(b) *Other laws.* The following statutes and regulations, although not incorporated by reference except for select sections identified in paragraph (a) of this section, are also part of the approved state-administered program:

(1) Chapter 57, Oil and Gas Conservation, Revised Statutes of Nebraska sections 57–901 through 57–922 (Reissue 1985).

(c) The Memorandum of Agreement between EPA Region VII and the Nebraska Oil and Gas Conservation Commission, signed by the EPA Regional Administrator on July 12, 1982.

(d) *Statement of legal authority.* (1) ''Nebraska Underground Injection Control Program, Attorney General's Statement for Class II Wells,'' signed by Assistant Attorney General for Attorney General of Nebraska, as submitted with ''State of Nebraska Request for Administration of UIC Program,'' January 23, 1982;

(2) ''Re: Nebraska Underground Injection Control Program, Addendum to Attorney General's Statement for Class II Wells,'' signed by Assistant Attorney General for Attorney General of Nebraska,'' undated.

(e) The Program Description and any other materials submitted as part of the application or as supplements thereto.

[49 FR 20197, May 11, 1984, as amended at 52 FR 17681, May 11, 1987; 56 FR 9417, Mar. 6, 1991]

§ 147.1401 State administered program—Class I, III, IV and V wells.

The UIC program for Class I, III, IV, and V wells in the State of Nebraska, except those on Indian lands, is the program administered by the Nebraska Department of Environmental Control, approved by EPA pursuant to section 1422 of the SDWA.

(a) *Incorporation by reference.* The requirements set forth in the State statutes and regulations cited in this paragraph are hereby incorporated by reference and made a part of the applicable UIC program under the SDWA for the State of Nebraska. This incorporation by reference was approved by the Director of the Federal Register effective June 26, 1984.

(1) Nebraska Environmental Protection Act, Revised Statutes of Nebraska sections 81–1502, 81–1506, 81–1519, and 81–1520 (Reissue 1987);

(2) Nebraska Department of Environmental Control, Title 122—Rules and Regulations for Underground Injection and Mineral Production Wells, Effective Date: February 16, 1982, Amended Dates: November 12, 1983, March 22, 1984; as amended by amendment approved by the Governor on January 2, 1989.

(b) *Other laws.* The following statutes and regulations although not incorporated by reference, also are part of the approved State-administered program:

(1) Nebraska Environmental Protection Act, Nebraska Revised Statutes sections 81–1502, 81–1506, 81–1519, and 81–1520 (Reissue 1987 and Cumm. Supp. 1988);

(c)(1) The Memorandum of Agreement between EPA Region VII and the Nebraska Department of Environmental Control, signed by the EPA Regional Administrator on July 12, 1982.

(2) Addendum to Underground Injection Control Memorandum of Agreement signed by the EPA Regional Administrator on July 12, 1982.

(3) Amendments to the Memorandum of Agreement signed by the EPA Regional Administrator on November 22, 1983.

(d) *Statement of legal authority.* (1) ''Nebraska Underground Injection Control Program, Attorney General's Statement for Class I, III, IV, and V Wells'', signed by Assistant Attorney General for Attorney General of Nebraska, as submitted with ''State of Nebraska Request for Administration of UIC Program, January 28, 1982;

(2) Letter from Attorney General (of Nebraska), by Assistant Attorney General, to Director, (Nebraska) Department of Environmental Control, August 7, 1981;

(3) Letter from Attorney General (of Nebraska), by Assistant Attorney General, to Director, (Nebraska) Department of Environmental Control, April 29, 1982;

(4) Letter from Attorney General (of Nebraska), by Assistant Attorney General, to Legal Counsel, (Nebraska) Department of Environmental Control, October 18, 1983.

(e) The Program Description and any other materials submitted as part of the original application or as supplements thereto.

(42 U.S.C. 1422)

[49 FR 24134, June 12, 1984, as amended at 52 FR 17681, May 11, 1987; 56 FR 9417, Mar. 6, 1991]

§ 147.1402 Aquifer exemptions. [Reserved]

§ 147.1403 EPA-administered program—Indian lands.

(a) *Contents.* The UIC program for all classes of wells on Indian lands in the State of Nebraska is administered by EPA. This program consists of the UIC program requirements of 40 CFR parts 124, 144, 146, 148, and any additional requirements set forth in the remainder of this subpart. Injection well owners and operators, and EPA shall comply with these requirements.

(b) *Effective date.* The effective date of the UIC program for Indian Lands in Nebraska is June 25, 1984.

[52 FR 17681, May 11, 1987, as amended at 56 FR 9417, Mar. 6, 1991]

Subpart DD—Nevada

§ 147.1450 State-administered program.

The UIC program for all classes of underground injection wells in the State of Nevada, other than those on Indian lands, is the program administered by the Nevada Division of Environmental Protection approved by EPA pursuant to section 1422 of the SDWA. Notice of this approval was published in the FEDERAL REGISTER on February 18, 1988; the effective date of this program is October 5, 1988. This program consists of the following elements, as submitted to EPA in the State's program application.

(a) *Incorporation by reference.* The requirements set forth in the State statutes and regulations cited in this paragraph are hereby incorporated by reference and made a part of the applicable UIC program under the SDWA for the State of Nevada. This incorporation by reference was approved by the Director of the Federal Register in accordance with 5 U.S.C. 552(a) and 1 CFR part 51. Copies may be obtained at the Nevada Department of Conservation and Natural Resources, Division of Environmental Protection, 201 South Fall Street, Carson City, Nevada 89710.

Copies may be inspected at the Environmental Protection Agency, Region IX, 215 Fremont Street, San Francisco, California 99105, or at the Office of the Federal Register, 800 North Capitol Street NW., suite 700, Washington, DC.

(1) Nevada Revised Statutes [NRS], Volume 25, Chapters 445.131 through 445.354, Inclusive. 1987.

(2) Nevada Revised Statutes [NRS], Volume 29, Chapters 534A.010 through 534A.090, Inclusive. 1987.

(3) Nevada Revised Statutes [NRS], Volume 28, Chapters 522.010 through 522.190, Inclusive. 1987.

(4) Nevada Administrative Code [NAC], Underground Injection Control Regulations, Sections 1 through 96.1, Inclusive. July 22, 1987, revised September 3, 1987 (amending NAC Chapter 445).

(5) Nevada Administrative Code [NAC], Regulations and Rules of Practice and Procedure adopted Pursuant to NRS 534A, Sections 1 through 69, Inclusive. November 12, 1985 (amending NAC Chapter 534A).

(6) Nevada Administrative Code [NAC], Regulations and Rules of Practice and Procedure adopted Pursuant to NRS 522.010 through 522.625, Inclusive. July 22, 1987 (amending NAC Chapter 522).

(b) The Memorandum of Agreement between EPA Region 9 and the Nevada Department of Conservation and Natural Resources signed by the EPA Regional Administrator on April 6, 1988.

(c) *Statement of Legal Authority.* Statement and Amendment to the Statement from the Attorney General of the State of Nevada, signed on July 22, 1987 and November 6, 1987 respectively, by the Deputy Attorney General.

(d) The Program Description and any other materials submitted as part of the original application or as supplements thereto.

[53 FR 39089, Oct. 5, 1988]

§ 147.1451 EPA administered program—Indian lands.

(a) *Contents.* The UIC program for all classes of wells on Indian lands in the State of Nevada is administered by EPA. This program consists of the UIC program requirements of 40 CFR parts 124, 144, 146, 148, and any additional requirements set forth in the remainder of this subpart. Injection well owners and operators, and EPA shall comply with these requirements.

(b) *Effective dates.* The effective date of the UIC program for Indian lands in Nevada is June 25, 1984.

[53 FR 43088, Oct. 25, 1988, as amended at 56 FR 9417, Mar. 6, 1991]

§ 147.1452 Aquifer exemptions. [Reserved]

§ 147.1453 Existing Class I, II (except enhanced recovery and hydrocarbon storage) and III wells authorized by rule.

Maximum injection pressure. The owner or operator shall limit injection pressure to the lesser of:

(a) A value which will not exceed the operating requirements of § 144.28(f)(3)(i) or (ii) as applicable; or

(b) A value for well head pressure calculated by using the formula:

$$Pm=(0.733–0.433\ Sg)d$$

where

Pm=injection pressure at the wellhead in pounds per square inch

Sg=specific gravity of injected fluid (unitless)

d=injection depth in feet.

§ 147.1454 Existing Class II enhanced recovery and hydrocarbon storage wells authorized by rule.

(a) *Maximum injection pressure.* (1) To meet the operating requirements of § 144.28(f)(3)(ii) (A) and (B) of this chapter, the owner or operator:

(i) Shall use an injection pressure no greater than the pressure established by the Regional Administrator for the field or formation in which the well is located. The Regional Administrator shall establish such a maximum pressure after notice, opportunity for comment, and opportunity for a public hearing, according to the provisions of part 124, subpart A of this chapter, and will inform owners and operators in writing of the applicable maximum pressure; or

(ii) May inject at pressures greater than those specified in paragraph (a)(1)(i) of this section for the field or formation in which he is operating provided he submits a request in writing to the Regional Administrator, and demonstrates to the satisfaction of the Regional Administrator that such injection pressure will not violate the requirement of § 144.28(f)(3)(ii) (A) and (B). The Regional Administrator may grant such a request after notice, opportunity for comment, and opportunity for public hearing, according to the provisions of part 124, subpart A of this chapter.

(2) Prior to such time as the Regional Administrator establishes field rules for maximum injection pressure based on data provided pursuant to paragraph (a)(2)(ii) of this section the owner or operator shall:

(i) Limit injection pressure to a value which will not exceed the operating requirements of § 144.28(f)(3)(ii); and

(ii) Submit data acceptable to the Regional Administrator which defines the fracture pressure of the formation in which injection is taking place. A single test may be submitted on behalf of two or more operators conducting operations in the same formation, if the Regional Administrator approves such submission. The data shall be submitted to the Regional Administrator within one year following the effective date of this program.

(b) *Casing and cementing.* Where the Regional Administrator determines that the owner or operator of an existing enhanced recovery or hydrocarbon storage well may not be in compliance with the requirements of §§ 144.28(e) and 146.22, the owner or operator shall comply with paragraphs (b) (1) through (4) of this section, when required by the Regional Administrator:

(1) Protect USDWs by:

(i) Cementing surface casing by recirculating the cement to the surface from a point 50 feet below the lowermost USDW; or

(ii) Isolating all USDWs by placing cement between the outermost casing and the well bore; and

(2) Isolate any injection zones by placing sufficient cement to fill the

calculated space between the casing and the well bore to a point 250 feet above the injection zone; and

(3) Use cement:

(i) Of sufficient quantity and quality to withstand the maximum operating pressure;

(ii) Which is resistant to deterioration from formation and injection fluids; and

(iii) In a quantity no less than 120% of the calculated volume necessary to cement off a zone.

(4) The Regional Administrator may specify other requirements in addition to or in lieu of the requirements set forth in paragraphs (b) (1) through (3) of this section, as needed to protect USDWs.

Subpart EE—New Hampshire

§147.1500 State-administered program.

The UIC program for all classes of wells in the State of New Hampshire, except those wells on Indian lands, is the program administered by the New Hampshire Department of Environmental Services, approved by the EPA pursuant to section 1422 of the SDWA. Notice of this approval was published in the FR on September 21, 1982 (47 FR 41561); the effective date of this program is October 21, 1982. This program consists of the following elements:

(a) *Incorporation by reference.* The requirements set forth in the State statutes and regulations cited in this paragraph are hereby incorporated by reference and made a part of the applicable UIC program under the SDWA for the State of New Hampshire. This incorporation by reference was approved by the Director of the Federal Register on June 25, 1984.

(1) New Hampshire Revised Statutes Annotated section 149:8 III(a) (1978);

(2) New Hampshire Code of Administrative Rules, Part Wc 410 (Protection of Groundwaters of the State, sections Ws 410.1 through Ws 410.16) (Issue Ws 3–82).

(b)(1) The Memorandum of Agreement between EPA Region I and the New Hampshire Water Supply and Pollution Control Commission, signed by the EPA Regional Administrator on August 23, 1982;

(2) Amendment No. 1 to the Memorandum of Agreement, signed by the EPA Regional Administrator on July 16, 1982.

(c) *Statement of legal authority.* (1) Letter from Attorney General of New Hampshire to Regional Administrator, EPA Region I, ''Re: Attorney General's Statement—Underground Injection Control Program,'' March 23, 1982;

(2) Letter from Attorney General of New Hampshire to Regional Administrator, EPA Region I, ''Re: Attorney General's Statement—Underground Injection Control Program,'' July 1, 1982.

(d) The Program Description and any other materials submitted as part of the application or as supplements thereto.

[49 FR 20197, May 11, 1984, as amended at 53 FR 43088, Oct. 25, 1988; 56 FR 9417, Mar. 6, 1991]

§147.1501 EPA-administered program—Indian lands.

(a) *Contents.* The UIC program for all classes of wells on Indian lands in the State of New Hampshire is administered by EPA. This program consists of the UIC program requirements of 40 CFR parts 124, 144, 146, 148, and any additional requirements set forth in the remainder of this subpart. Injection well owners and operators, and EPA shall comply with these requirements.

(b) *Effective date.* The effective date of the UIC program for Indian lands in New Hampshire is November 25, 1988.

[53 FR 43088, Oct. 25, 1988, as amended at 56 FR 9417, Mar. 6, 1991]

Subpart FF—New Jersey

§147.1550 State-administered program.

The UIC program for all classes of wells in the State of New Jersey, except those on Indian lands, is the program administered by the New Jersey Department of Environmental Protection, approved by EPA pursuant to section 1422 of the SDWA. Notice of this approval was published in the Federal Register on July 15, 1983 (48 FR 32343); the effective date of this program is August 15, 1983. This program consists of the following elements, as submitted

to EPA in the State's program application.

(a) *Incorporation by reference.* The requirements set forth in the State statutes and regulations cited in this paragraph are hereby incorporated by reference and made a part of the applicable UIC program under the SDWA for the State of New Jersey. This incorporation by reference was approved by the Director of the Federal Register on June 25, 1984.

(1) Water Pollution Control Act, New Jersey Statutes Annotated sections 58:10A–1 through 58:10A–20 (West 1982 and Supp. 1990);

(2) New Jersey Administrative Code, sections 7:14A–1.1 through 1.9 (subchapter 1), 7:14A–2.1 through 2.15 (subchapter 2), 7:14A–5.1 through 5.17, (subchapter 5) (amended March 1988).

(b)(1) The Memorandum Agreement between EPA Region II and the New Jersey Department of Environmental Protection, signed by the EPA Regional Administrator on September 9, 1982;

(2) Letter from Commissioner, New Jersey Department of Environmental Protection, to Regional Administrator, EPA Region II, March 21, 1983.

(c) *Statement of legal authority.* (1) Letter from Attorney General of New Jersey (by Deputy Attorney General) to Commissioner, Department of Environmental Protection, ''Re: New Jersey Pollutant Discharge Elimination System—Underground Injection Control,'' February 9, 1982;

(2) Letter from Attorney General of New Jersey (by Deputy Attorney General) to Commissioner, Department of Environmental Protection, ''Re: New Jersey Pollutant Discharge Elimination System—Underground Injection Control,'' April 15, 1983 (six pages);

(3) Letter from Attorney General of New Jersey (by Assistant Attorney General) to Commissioner, Department of Environmental Protection, ''Re: New Jersey Pollutant Discharge Elimination System—Underground Injection Control,'' April 15, 1983 (two pages).

(d) The Program Description and any other materials submitted as part of the application or as supplements thereto.

[49 FR 20197, May 11, 1984, as amended at 53 FR 43089, Oct. 25, 1988; 56 FR 9417, Mar. 6, 1991]

§ 147.1551 EPA-administered program—Indian lands.

(a) *Contents.* The UIC program for all classes of wells on Indian lands in the State of New Jersey is administered by EPA. This program consists of the UIC program requirements of 40 CFR parts 124, 144, 146, 148, and any additional requirements set forth in the remainder of this subpart. Injection well owners and operators, and EPA shall comply with these requirements.

(b) *Effective date.* The effective date of the UIC program for Indian lands in New Jersey is November 25, 1988.

[53 FR 43089, Oct. 25, 1988, as amended at 56 FR 9417, Mar. 6, 1991]

Subpart GG—New Mexico

§ 147.1600 State-administered program—Class II wells.

The UIC program for Class II wells in the State of New Mexico, except for those on Indian lands, is the program administered by the New Mexico Energy and Minerals Department, Oil Conservation Division, approved by EPA pursuant to section 1425 of the SDWA. Notice of this approval was published in the FEDERAL REGISTER on February 5, 1982 (47 FR 5412); the effective date of this program is March 7, 1982. This program consists of the following elements as submitted to EPA in the State's program application:

(a) *Incorporation by reference.* The requirements set forth in the State statutes and regulations cited in this paragraph are hereby incorporated by reference and made a part of the applicable UIC program under the SDWA for the State of New Mexico. This incorporation by reference was approved by the Director of the Federal Register on June 25, 1984.

(1) Oil and Gas Act, New Mexico Statutes Annotated sections 70–2–1 through –36 (1978);

(2) *State of New Mexico Energy and Mineral Department, Oil Conservation Division—Rules and Regulations* (dated 10–1–78), sections B–3, I–701 through I–708, M–1100 through M–1121.

(b)(1) The Memorandum of Agreement between EPA Region VI and the New Mexico Energy and Minerals Department, Oil Conservation Division, signed by the EPA Regional Administrator on December 10, 1981;

(2) Addendum No. 1 to the Memorandum of Agreement, signed by the EPA Regional Administrator on June 28, 1982;

(3) Addendum No. 2 to the Memorandum of Agreement, signed by the EPA Regional Administrator on November 18, 1982;

(4) Letter from Director, Oil Conservation Division, New Mexico Energy and Minerals Department, and Assistant Attorney General of New Mexico, to Regional Administrator, EPA Region VI, November 6, 1981.

(c) *Statement of legal authority.* ''Statement of Legal Authority of the State of New Mexico by and through its Oil Conservation Division of the Energy and Mines Department to conduct an Underground Injection Control Program,'' signed by Assistant Attorney General and General Counsel to the Oil Conservation Division.

(d) The Program Description and any other materials submitted as part of the application or as supplements thereto.

[49 FR 20197, May 11, 1984, as amended at 53 FR 43089, Oct. 25, 1988]

§ 147.1601 State-administered program—Class I, III, IV and V wells.

The UIC program for Class I, III, IV and V injection wells in the State of New Mexico, except for those on Indian lands, is the program administered by the New Mexico Water Quality Control Commission, the Environmental Improvement Division, and the Oil Conservation Division, approved by EPA pursuant to section 1422 of the SDWA. Notice of this approval was published in the FEDERAL REGISTER on July 11, 1983 (48 FR 31640); the effective date of this program is August 10, 1983. This program consists of the following elements, as submitted to EPA in the State's program application:

(a) *Incorporation by reference.* The requirements set forth in the State statutes and regulations cited in this paragraph are hereby incorporated by reference and made a part of the applicable UIC program under the SDWA for the State of New Mexico. This incorporation by reference was approved by the Director of the Federal Register on June 25, 1984.

(1) New Mexico Water Quality Control Commission Regulations (WQCC 82–1) sections 1–100 through 5–300 (September 20, 1982).

(b) *Other laws.* The following statutes and regulations, although not incorporated by reference, are also part of the approved State-administered UIC program:

(1) Water Quality Act, New Mexico Statutes Annotated sections 74–6–1 through 74–6–13 (1978 and Supp. 1982);

(2) Geothermal Resources Conservation Act, New Mexico Statutes Annotated sections 71–5–1 through 71–5–24 (1978 and Supp. 1982);

(3) Surface Mining Act, New Mexico Statutes Annotated sections 69–25A–1 through 69–25A–35 (1978 and Supp. 1980).

(c)(1) The Memorandum of Agreement between EPA Region VI and the New Mexico Water Quality Control Commission, the Environmental Improvement Division, and the Oil Conservation Division, signed by the EPA Regional Administrator on April 13, 1983;

(2) Letter from the Director, Environmental Improvement Division and the Director, Oil Conservation Division, to Regional Administrator, EPA Region IV, ''Re: New Mexico Underground Injection Control Program—Clarification,'' February 10, 1983.

(d) *Statement of legal authority.* ''Attorney General's Statement,'' signed by the Assistant Attorney General for the Environmental Improvement Division, the Assistant Attorney General for Oil Conservation Division, and the Deputy Attorney General, Civil Division, Counsel for the Mining and Minerals Division, undated, submitted December 8, 1982.

(e) The Program Description and any other materials submitted as part of

the application or as supplements thereto.

[49 FR 20197, May 11, 1984, as amended at 53 FR 43089, Oct. 25, 1988]

§ 147.1603 EPA-administered program—Indian lands.

(a) *Contents.* The UIC program for all classes of wells on Indian lands in New Mexico is administered by EPA. The program consists of the requirements set forth at Subpart HHH of this part. Injection well owners and operators and EPA shall comply with these requirements.

(b) *Effective date.* The effective date for the UIC program on Indian lands in New Mexico is November 25, 1988.

[53 FR 43089, Oct. 25, 1988]

Subpart HH—New York

§ 147.1650 State-administered program. [Reserved]

§ 147.1651 EPA-administered program.

(a) *Contents.* The UIC program for the State of New York, including all Indian lands, is administered by EPA. The program consists of the UIC program requirements of 40 CFR parts 124, 144, 146, 148, and any additional requirements set forth in the remainder of this subpart. Injection well owners and operators, and EPA shall comply with these requirements.

(b) *Effective dates.* The effective date of the UIC program for New York for all injection activities except those on lands of the Seneca Indian Tribe is June 25, 1984. The effective date for the UIC program for the lands of the Seneca Indian Tribe is November 25, 1988.

[53 FR 43089, Oct. 25, 1988; 54 FR 10616, Mar. 14, 1989, as amended at 56 FR 9417, Mar. 6, 1991]

§ 147.1652 Aquifer exemptions.

(a) This section identifies any aquifer or their portions exempted in accordance with §§ 144.7(b) and 146.4 of this chapter at the time of program promulgation. EPA may in the future exempt other aquifers or portions, according to applicable procedures, without codifying such exemptions in this section. An updated list of exemptions will be maintained in the Regional office.

(b) The following portions of aquifers are exempted in accordance with the provisions of §§ 144.7(b) and 146.4 of this chapter for Class II injection activities only:

(1) The Bradford First, Second, and Third Sand Members and the Kane Sand Member in the Bradford Field in Cattaraugus County.

(2) The Chipmunk Oil field in Cattaraugus County.

§ 147.1653 Existing Class I, II (except enhanced recovery and hydrocarbon storage) and III wells authorized by rule.

Maximum injection pressure. The owner or operator shall limit injection pressure to the lesser of:

(a) A value which will not exceed the operating requirements of § 144.28(f)(3)(i) or (ii) as applicable; or

(b) A value for well head pressure calculated by using the following formula:

$$Pm=(0.733-0.433\ Sg)d$$

where

Pm=injection pressure at the well head in pounds per square inch

Sg=specific gravity of inject fluid (unitless)

d=injection depth in feet.

§ 147.1654 Existing Class II enhanced recovery and hydrocarbon storage wells authorized by rule.

(a) *Maximum injection pressure.* (1) To meet the operating requirements of § 144.28(f)(3)(ii) (A) and (B) of this chapter, the owner or operator:

(i) Shall use an injection pressure no greater than the pressure established by the Regional Administrator for the field or formation in which the well is located. The Regional Administrator shall establish such a maximum pressure after notice, opportunity for comment, and opportunity for a public hearing, according to the provisions of part 124, subpart A of this chapter, and will inform owners and operators in writing of the applicable maximum pressure, or

(ii) May inject at pressures greater than those specified in paragraph (a)(1)(i) of this section for the field or formation in which he is operating provided he submits a request in writing to the Regional Administrator, and demonstrates to the satisfaction of the

Regional Administrator that such injection pressure will not violate the requirement of §144.28(f)(3)(ii) (A) and (B). The Regional Administrator may grant such a request after notice, opportunity for comment, and opportunity for a public hearing, according to the provisions of part 124, subpart A of this chapter.

(2) Prior to such time as the Regional Administrator establishes rules for maximum injection pressure based on data provided pursuant to paragraph (a)(2)(ii) of this section the owner or operator shall:

(i) Limit injection pressure to a value which will not exceed the operating requirements of §144.28(f)(3)(ii); and

(ii) Submit data acceptable to the Regional Administrator which defines the fracture pressure of the formation in which injection is taking place. A single test may be submitted on behalf of two or more operators conducting operations in the same formation, if the Regional Administrator approves such submission. The data shall be submitted to the Regional Administrator within one year of the effective date of this program.

(b) *Casing and cementing.* Where the Regional Administrator determines that the owner or operator of an existing enhanced recovery or hydrocarbon storage well may not be in compliance with the requirements of §§144.28(e) and 146.22, the owner or operator shall comply with paragraphs (b) (1) through (4) of this section, when required by the Regional Administrator:

(1) Protect USDWs by:

(i) Cementing surface casing by recirculating the cement to the surface from a point 50 feet below the lowermost USDW; or

(ii) Isolating all USDWs by placing cement between the outermost casing and the well bore; and

(iii) For wells as described in §146.8(b)(3)(ii), installing a smaller diameter pipe inside the existing injection tubing and setting it on an appropriate packer; and

(2) Isolate any injection zones by placing sufficient cement to fill the calculated space between the casing and the well bore to a point 50 feet above the injection zone; and

(3) Use cement:

(i) Of sufficient quantity and quality to withstand the maximum operating pressure;

(ii) Which is resistant to deterioration from formation and injection fluids; and

(iii) In a quantity no less than 120% of the calculated volume necessary to cement off a zone.

(4) The Regional Administrator may specify other requirements in addition to or in lieu of the requirements set forth in paragraphs (b) (1) through (3) of this section as needed to protect USDWs.

§147.1655 Requirements for wells authorized by permit.

(a) The owner or operator of a Class I well authorized by permit shall install or shall ensure that the well has:

(1) Surface casing present;

(i) Extending from the surface to a depth at least 50 feet below the base of the lowermost USDW; and

(ii) Cemented back to the surface by recirculating the cement; and

(2) Long string casing and tubing;

(i) Extending to the injection zone; and

(ii) Cemented back to 50 feet above the base of the next largest casing string.

(b) The owner or operator of a new Class II well authorized by permit shall:

(1) Install surface casing from the surface to at least 50 feet below the base of the lowermost USDW.

(2) Cement the casing by recirculating to the surface or by using no less than 120% of the calculated annular volume.

(3) For new enhanced recovery wells, install tubing or long string casing extending to the injection zone.

(4) For new salt water disposal wells, install long string casing and tubing extending to the injection zone.

(5) Isolate any injection zone by placing sufficient cement to fill the calculated volume to a point 50 feet above the injection zone.

(c) The Regional Administrator may specify casing and cementing requirements other than those listed in paragraphs (a) and (b) of this section on a case by case basis as conditions of the permit.

Subpart II—North Carolina

§ 147.1700 State-administered program.

The UIC program for all classes of wells in the State of North Carolina, except those wells on Indian lands, is the program administered by the North Carolina Department of Environment, Health and Natural Resources approved by EPA pursuant to section 1422 of the SDWA. Notice of this approval was published in the FEDERAL REGISTER on April 19, 1984 (49 FR 15553); the effective date of this program is April 19, 1984. This program consists of the following elements, as submitted to EPA in the State's program application:

(a) *Incorporation by reference.* The requirements set forth in the State statutes and regulations cited in this paragraph are hereby incorporated by reference and made a part of the applicable UIC program under the SDWA for the State of North Carolina. This incorporation by reference was approved by the Director of the OFR in accordance with 5 U.S.C. 552(a) and 1 CFR part 51. Copies may be obtained at the North Carolina Department of Environment, Health and Natural Resources, P.O. Box 27687, Raleigh, North Carolina 27611. Copies may be inspected at the Environmental Protection Agency, Region IV, 345 Courtland Street, NE., Atlanta, Georgia 30365, or at the Office of the Federal Register, 800 North Capitol Street, NW., suite 700, Washington, DC.

(1) Administrative Procedure Act, N.C. GEN. STAT. 150B–1 through 150B–64 (1987 and Cumm. Supp. 1989);

(2) North Carolina Well Construction Act, N.C. GEN. STAT. §§ 87–83 through 87–99 (1989 and Cumm. Supp. 1989);

(3) Water and Air Resources, N.C. GEN. STAT. §§ 143–211 through 143–215.10 (1987 and Cumm. Supp. 1989);

(4) Solid Waste Management, N.C. GEN. STAT. §§ 130A–290 through 130A–309.03 (1989);

(5) North Carolina Drinking Water Act, N.C. GEN. STAT. §§ 130A–311 through 130A–332 (1989);

(6) Sanitary Sewage Systems, N.C. GEN. STAT. §§ 130A–333 through 130A–335 (1989).

(b) *Other laws.* The following rules and regulations, although not incorporated by reference, are also part of the approved State-administered program:

(1) N.C. ADMIN. CODE, Title 15, r. 02L.0100 *et seq.* Groundwater Classification and Standards: General Considerations (September 22, 1988);

(2) N.C. ADMIN. CODE, Title 15, r. 02L.0100 *et seq.* Criteria and Standards Applicable to Injection Wells (September 22, 1988).

(c) *Memorandum of Agreement.* The Memorandum of Agreement between the State of North Carolina and EPA Region IV, signed March 1, 1984.

(d) *Statement of legal authority.* (1) Underground Injection Control Program, Attorney General's Statement (June 15, 1982);

(2) Amendment to Underground Injection Control Program, Attorney General's Statement (February 9, 1984).

(e) *Program Description.* The Program Description and other materials submitted as part of the application or as supplements thereto.

[56 FR 9417, Mar. 6, 1991]

§§ 147.1701—147.1702 [Reserved]

§ 147.1703 EPA-administered program—Indian lands.

(a) *Contents.* The UIC program for all classes of wells on Indian lands in the State of North Carolina is administered by EPA. This program consists of the UIC program requirements of 40 CFR parts 124, 144, 146, 148, and any additional requirements set forth in the remainder of this subpart. Injection well owners and operators, and EPA shall comply with these requirements.

(b) *Effective date.* The effective date of the UIC program for Indian lands in North Carolina is November 25, 1988.

[53 FR 43089, Oct. 25, 1988, as amended at 56 FR 9418, Mar. 6, 1991]

§§ 147.1704—147.1749 [Reserved]

Subpart JJ—North Dakota

§ 147.1750 State-administered program—Class II wells.

The UIC program for Class II wells in the State of North Dakota, except those on Indian lands, is the program administered by the North Dakota Industrial Commission, approved by EPA pursuant to section 1425 of the SDWA.

Notice of this approval was published in the FEDERAL REGISTER on August 23, 1983 (48 FR 38237); the effective date of this program is September 24, 1983. This program consists of the following elements, as submitted to EPA in the State's program application.

(a) *Incorporation by reference.* The requirements set forth in the State statutes and regulations cited in this paragraph are hereby incorporated by reference and made a part of the applicable UIC program under the SDWA for the State of North Dakota. This incorporation by reference was approved by the Director of the Federal Register on June 25, 1984.

(1) North Dakota Century Code, Chapter 38–08 (Control of Gas and Oil Resources, 1987 and Supp. 1989);

(2) North Dakota Administrative Code, Chapter 43–02–05 (Underground Injection Control, as published in *Statutes and Rules for the Conservation of Oil and Gas,* North Dakota Industrial Commission, revised effective November 1, 1987);

(3) North Dakota Administrative Code, Chapter 43–02–03 (General Rules, as published in *Statutes and Rules for the Conservation of Oil and Gas,* North Dakota Industrial Commission, revised effective November 1, 1987).

(b) The Memorandum of Agreement between EPA Region VIII and the North Dakota Industrial Commission, Oil and Gas Division, signed by the EPA Regional Administrator on June 16, 1983, as amended September 7, 1989.

(c) *Statement of legal authority.* ''Underground Injection Control Program Attorney General's Statement,'' as submitted with the North Dakota Underground Injection Control Program Primacy Application for Class II Injection Wells, transmitted by the Governor on July 15, 1982 (16 pages).

(d) The Program Description and other materials submitted as part of the application or as supplements thereto.

[49 FR 20197, May 11, 1984, as amended at 53 FR 43089, Oct. 25, 1988; 56 FR 9418, Mar. 6, 1991]

§ 147.1751 State-administered program—Class I, III, IV and V wells.

The UIC program for Class I, III, IV, and V wells in the State of North Dakota, except those on Indian lands, is the program administered by the North Dakota Department of Health, approved by EPA pursuant to section 1422 of the SDWA. Notice of this approval was published in the FEDERAL REGISTER on September 21, 1984; the effective date of this program is October 5, 1984. This program consists of the following elements, as submitted to EPA in the State's program application.

(a) *Incorporation by reference.* The requirements set forth in the State statutes and regulations cited in this paragraph are hereby incorporated by reference and made a part of the applicable UIC program under the SDWA for the State of North Dakota. This incorporation by reference was approved by the Director of the Federal Register effective October 5, 1984.

(1) North Dakota Century Code Sections 38–12–01, 38–12–03 (1980);

(2) North Dakota Century Code, Sections 61–28–02 and 61–28–06 (1989);

(3) North Dakota Administrative Code Sections 33–25–01–01 through 33–25–01–18 (North Dakota State Health Department Underground Control Program) (1983);

(4) North Dakota Administrative Code, Chapter 43–02–02 (Subsurface Mineral Exploration and Development) (August 1986), and Chapter 43–02–02.1 (Underground Injection Control Program) (March 1, 1984);

(5) North Dakota Administrative Code Sections 43–02–02–1–01 through 43–02–02–1–18 (North Dakota Geological Survey—Undergound Injection Control Program) (1984);

(b) *Other laws.* The following statutes and regulations, although not incorporated by reference, also are part of the approved State-administered program;

(1) North Dakota Environmental Law Enforcement Act of 1975, North Dakota Century Code Sections 32–40–01 to 32–40–11 (1976);

(2) North Dakota Century Code, Ch. 38–12 (Regulation, Development, and Production of Subsurface Minerals) (1979);

(3) North Dakota Century Code Chapter 61–28 (Control, Prevention and Abatement of Pollution of Surface Waters) (1989);

(4) North Dakota Administrative Code Article 33–22 (Practice and Procedure) (1983).

(c) The Memorandum of Agreement between EPA Region VIII and the North Dakota Department of Health, signed by the EPA Regional Administrator on May 18, 1984.

(d) The Program Description and any other materials submitted as part of the original application or as supplements thereto.

[49 FR 37066, Sept. 21, 1984, as amended at 56 FR 9418, Mar. 6, 1991]

§ 147.1752 EPA-administered program—Indian lands.

(a) *Contents.* The UIC program for all classes of wells on Indian lands in the State of North Dakota is administered by EPA. This program consists of the UIC program requirements of 40 CFR parts 124, 144, 146, 148, and any additional requirements set forth in the remainder of this subpart. Injection well owners and operators, and EPA shall comply with these requirements.

(b) *Effective date.* The effective date of the UIC program for Indian lands in North Dakota is November 25, 1988.

[53 FR 43089, Oct. 25, 1988, as amended at 56 FR 9418, Mar. 6, 1991]

Subpart KK—Ohio

§ 147.1800 State-administered program—Class II wells.

The UIC program for Class II wells in the State of Ohio, except for those on Indian lands, is the program administered by the Ohio Department of Natural Resources, approved by EPA pursuant to section 1425 of the SDWA. Notice of this approval was published in the FEDERAL REGISTER on August 23, 1983 (48 FR 38238); the effective date of this program is September 22, 1983. This program consists of the following elements, as submitted to EPA in the State's program application:

(a) *Incorporation by reference.* The requirements set forth in the State statutes and regulations cited in this paragraph are hereby incorporated by reference and made a part of the applicable UIC program under the SDWA for the State of Ohio. This incorporation by reference was approved by the Director of the Federal Register on June 25, 1984.

(1) Ohio Revised Code Annotated, sections 1509.01 through 1509.22 (Page 1978 and Supp. 1982);

(2) Rules of the Division of Oil and Gas, Ohio Administrative Code sections 1501:91–01, through 1501: 9–11–13 (1983).

(b) The Memorandum of Agreement between EPA Region V and the Ohio Department of Natural Resources.

(c) *Statement of legal authority.* ''Underground Injection Control Program—Attorney General's Statement,'' signed by the Assistant Attorney General, Chief, Environmental Law Section, for the Attorney General of Ohio, September 30, 1982.

(d) The Program Description and any other materials submitted as part of the application or as supplements thereto.

[49 FR 20197, May 11, 1984, as amended at 53 FR 43089, Oct. 25, 1988]

§ 147.1801 State-administered program—Class I, III, IV and V wells.

The UIC program for Class I, III, IV, and V wells in the State of Ohio, other than those on Indian lands, is the program administered by the Ohio Department of Natural Resources and the Ohio Environmental Protection Agency, approved by EPA pursuant to section 1422 of the SDWA. Notice of this approval was published in the FEDERAL REGISTER on November 29, 1984; the effective date of this program is January 14, 1985. This program consists of the following elements, as submitted to EPA in the State's program application.

(a) *Incorporation by reference.* The requirements set forth in the State statutes and regulations cited in this paragraph are hereby incorporated by reference and made a part of the applicable UIC program under the SDWA for the State of Ohio. This incorporation by reference was approved by the Director of the FEDERAL REGISTER effective January 14, 1985.

(1) Ohio Revised Code Annotated, sections 1509.01, 1509.03, 1509.221 (Supp. 1983);

(2) Rules of the Division of Oil and Gas, Ohio Administrative Code, sections 1501:9–7–01 through 7–14 (1984);

(3) Ohio Revised Code Annotated, sections 6111.04, 6111.043, 6111.044 (Supp. 1983);

(4) Rules of the Ohio Environmental Protection Agency, Ohio Administrative Code, sections 3745–34–01 through 34–41; 3745–9–01 through 9–11 (Director Ohio EPA Order, June 18, 1984).

(b) *Other laws.* The following statutes and regulations, although not incorporated by reference, also are part of the approved State-administered program:

(1) Ohio Revised Code, Chapter 119 (1978 Replacement Part);

(2) Ohio Code Supplement, sections 6111.041, 6111.042, 6111.045 (Supp. 1982).

(c) (1) The Memorandum of Agreement between EPA Region V and the Ohio Department of Natural Resources, signed by the EPA Regional Administrator on March 30, 1984;

(2) Memorandum of Agreement between the Ohio Department of Natural Resources and the Ohio Environmental Protection Agency, Related to the Underground Injection Control Program for the State of Ohio, signed August 1, 1984.

(d) *Statement of legal authority.* Statement from Attorney General of the State of Ohio, by Senior Assistant Attorney General, ''Underground Injection Control Program—Attorney General's Statement,'' July 25, 1984.

(e) The Program Description and any other materials submitted as part of the original application or as supplements thereto.

[49 FR 46897, Nov. 29, 1984]

§147.1802 Aquifer exemptions. [Reserved]

§147.1803 Existing Class I and III wells authorized by rule—maximum injection pressure.

The owner or operator shall limit injection pressure to the lesser of:

(a) A value which will not exceed the operating requirements of §144.28(f)(3)(i); or

(b) A value for well head pressure calculated by using the following formula:

$$Pm = (0.8—0.433\ Sg)\ d$$

where

Pm = injection pressure at the well head in pounds per square inch

Sg = specific gravity of injected fluid (unitless)

d = injection depth in feet.

[49 FR 45308, Nov. 15, 1984]

§147.1805 EPA-administered program—Indian lands.

(a) *Contents.* The UIC program for all classes of wells on Indian lands in the State of Ohio is administered by EPA. This program consists of the UIC program requirements of 40 CFR parts 124, 144, 146, 148, and any additional requirements set forth in the remainder of this subpart. Injection well owners and operators, and EPA shall comply with these requirements.

(b) *Effective date.* The effective date of the UIC program for Indian lands in Ohio is November 25, 1988.

[53 FR 43089, Oct. 25, 1988, as amended at 56 FR 9418, Mar. 6, 1991]

Subpart LL—Oklahoma

§147.1850 State-administered program—Class I, III, IV and V wells.

The UIC program for Class I, III, IV, and V wells in the State of Oklahoma, except those on Indian lands, is the program administered by the Oklahoma State Department of Health, approved by EPA pursuant to SDWA section 1422. Notice of this approval was published in the FEDERAL REGISTER on June 24, 1982 (47 FR 27273). The effective date of this program is July 24, 1982. This program consists of the following elements, as submitted to EPA in the State's program application:

(a) *Incorporation by reference.* The requirements set forth in the State statutes and regulations cited in this paragraph are hereby incorporated by reference and made a part of the applicable UIC program under the SDWA for the State of Oklahoma. This incorporation by reference was approved by the Director of the Federal Register on June 25, 1984.

(1) Oklahoma Statutes title 63 sections 1–901, 1–903 (1981);

(2) Oklahoma Controlled Industrial Waste Disposal Act, Oklahoma Statute Annotated title 63 sections 1–2002, 1–2014 (West Supp. 1983–1984);

(3) *Regulations.* [Reserved]

(b) *Other laws.* The following statutes and regulations, although not incorporated by reference except for select sections identified in paragraph (a) of this section, are also part of the approved State-administered UIC program:

(1) Oklahoma Open Meeting Act, Oklahoma Statutes title 25 sections 301 through 314 (Supp. 1978);

(2) Oklahoma Statutes Annotated title 63 sections 1–101 to 1–114, 1–901 to 1–911, 1–1601 *et seq.*, 1–1701, 1–2001 to 1–2014 (West 1973 and Supp. 1982);

(3) Oklahoma Statutes Annotated title 75 sections 301 to 327 (West 1976 and Supp. 1982).

(c) (1) The Memorandum of Agreement between EPA Region VI and the Oklahoma State Department of Health, signed by the EPA Regional Administrator on April 13, 1982;

(2) Memorandum of Understanding between the Oklahoma State Department of Health and the Oklahoma Corporation Commission (OCC), signed by members of the OCC on February 12, 1982;

(3) Memorandum of Understanding between the Oklahoma State Department of Health and the Oklahoma Department of Mines (ODM), signed by the Deputy Chief Mine Inspector, ODM, on February 15, 1982.

(d) *Statement of legal authority.* Letter from Attorney General of Oklahoma to Commissioner of Health, Oklahoma State Department of Health, ''Re: Statement and Memorandum of Law Concerning the Authority for the Oklahoma State Department of Health's Underground Injection Control Program,'' February 12, 1982.

(e) The Program Description and any other materials submitted as part of the application or as supplements thereto.

[49 FR 20197, May 11, 1984, as amended at 53 FR 43090, Oct. 25, 1988]

§ 147.1851 State-administered program—Class II wells.

The UIC program for Class II wells in the State of Oklahoma, including the lands of the Five Civilized Tribes, but not including those on other Indian lands, is the program administered by the Oklahoma Corporation Commission approved by EPA pursuant to SDWA section 1425. Notice of this approval was published in the FEDERAL REGISTER on December 2, 1981 (46 FR 58588). This program consists of the following elements, as submitted to EPA in the State's program application:

(a) *Incorporation by reference.* [Reserved]

(b) *Other laws.* The following statutes and regulations, although not incorporated by reference, are also part of the approved State-administered UIC program:

(1) Oklahoma Statutes, title 17 sections 51–53; title 52 sections 86.1–86.5, 139–153, 243, 307–318.1 (1971).

(2) OCC–OGR Rules No. 1–101–3–303.

(c) (1) The Memorandum of Agreement between EPA Region VI and the Oklahoma Corporation Commission, signed by the EPA Regional Administrator on April 13, 1981;

(2) Letter from the Manager, Underground Injection Control, Oklahoma Corporation Commission, to EPA, June 18, 1981.

(d) *Statement of legal authority.* ''Statement of Legal Authority of the Oklahoma Corporation Commission to Conduct an Underground Injection Control Program,'' (Part IV, pages 30–41 of ''State of Oklahoma Primacy Application for Authority to Regulate Class II Injection Wells,'' submitted April 14, 1981), signed by the Conservation Attorney, Counsel to the Director and the Oklahoma Corporation Commission.

(e) The Program Description and any other materials submitted as part of the application or as supplements thereto.

[49 FR 20197, May 11, 1984, as amended at 53 FR 43090, Oct. 25, 1988]

§ 147.1852 EPA-administered program—Indian lands.

(a) *Contents.* The UIC program for all wells on Indian lands in Oklahoma, except Class II wells on the lands of the Five Civilized Tribes, is administered by EPA. The UIC program for Class II wells on the Osage Mineral Reserve consists of the requirements set forth in subpart GGG of this part. The UIC program for all other wells on Indian lands consists of the requirements set forth in subpart III of this part. Injection well owners and operators and

EPA shall comply with these requirements.

(b) *Effective date.* The effective date for UIC program for Class II wells on the Osage Mineral Reserve is December 30, 1984. The effective date for the UIC program for all other wells on Indian lands is November 25, 1988.

[53 FR 43090, Oct. 25, 1988]

Subpart MM—Oregon

§ 147.1900 State-administered program.

The UIC program for all classes of wells in the State of Oregon, except those on Indian lands, is administered by the Oregon Department of Environmental Quality, approved by EPA pursuant to section 1422 and section 1425 of the SDWA. Notice of this approval was published in the FEDERAL REGISTER on September 25, 1984; the effective date of this program is October 9, 1984. This program consists of the following elements, as submitted to EPA in the State's program application.

(a) *Incorporation by reference.* The requirements set forth in the State statutes and regulations cited in this paragraph are hereby incorporated by reference and made a part of the applicable UIC program under the SDWA for the State of Oregon. This incorporation by reference was approved by the Director of the FEDERAL REGISTER effective October 9, 1984.

(1) Oregon Revised Statutes, Title 16, chapter 164, section 164.785; Title 36, chapter 468, sections 468.005, 468.065 to 468.070, 468.700 to 468.815; Title 43, chapter 520 sections 520.005, 520.095, 520.155—520.330 (1983);

(2) Oregon Administrative Rules, Chapter 340, Division 44, sections 340–44–005 through 340–44–055 (October 1983); Chapter 340, Division 45, sections 340–45–005 through 340–45–075 (January 1990); Chapter 632, Division 10, sections 632–10–002 through 632–10–235 (May 1986); Chapter 632, Division 20, sections 632–20–005 through 632–20–180 (May 1984).

(b) *Other laws.* The following statutes and regulations, although not incorporated by reference, also are part of the approved State-administered program:

(1) Oregon Revised Statutes, Chapter 183 (1987); 192.420, 192.500, 459.460(3), 468.005 through 468.605, and 468.780 through 468.997; Chapters 516 and 522 (1983);

(2) Oregon Administrative Rules, chapter 137, Div. 3 (July 1982); chapter 340, Div. 11 (April 1988); chapter 340, Div. 12 (March 1989); chapter 340, Div. 14 (November 1983); chapter 340, Div. 52 (November 1983); chapter 632, Div. 1 (June 1980); chapter 632, Div. 20 (January 1981).

(c)(1) The Memorandum of Agreement between EPA Region X and the Oregon Department of Environmental Quality, signed by the EPA Regional Administrator on May 3, 1984.

(d) *Statement of legal authority.* (1) ''Underground Injection Control Program Legal Counsel's Statement,'' October 1983, signed by the Assistant Attorney General, Oregon;

(2) Opinion of the Attorney General, Oregon, 35 Op. Attorney General 1042 (1972).

(e) The Program Description and any other materials submitted as part of the original application or as supplements thereto.

[49 FR 37594, Sept. 25, 1984, as amended at 53 FR 43090, Oct. 25, 1988; 56 FR 9418, Mar. 6, 1991]

§ 147.1901 EPA-administered program—Indian lands.

(a) *Contents.* The UIC program for all classes of wells on Indian lands in the State of Oregon is administered by EPA. This program consists of the UIC program requirements of 40 CFR parts 124, 144, 146, 148, and any additional requirements set forth in the remainder of this subpart. Injection well owners and operators, and EPA shall comply with these requirements.

(b) *Effective date.* The effective date of the UIC program for Indian lands in Oregon is November 25, 1988.

[53 FR 43090, Oct. 25, 1988, as amended at 56 FR 9419, Mar. 6, 1991]

Subpart NN—Pennsylvania

§ 147.1950 State-administered program. [Reserved]

§ 147.1951 EPA-administered program.

(a) *Contents.* The UIC program for the State of Pennsylvania, including all Indian lands, is administered by EPA.

This program consists of the UIC program requirements of 40 CFR parts 124, 144, 146, 148, and any additional requirements set forth in the remainder of this subpart. Injection well owners and operators, and EPA shall comply with these requirements.

(b) *Effective dates.* The effective date for the UIC program on Indian lands is November 25, 1988. The effective date for the UIC program for the rest of Pennsylvania is June 25, 1984.

[53 FR 43090, Oct. 25, 1988, as amended at 56 FR 9419, Mar. 6, 1991]

§ 147.1952 Aquifer exemptions.

(a) This section identifies any aquifers or their portions exempted in accordance with §§ 144.7(b) and 146.4 of this chapter at the time of program promulgation. EPA may in the future exempt other aquifers or portions, according to applicable procedures, without codifying such exemptions in this section. An updated list of exemptions will be maintained in the Regional office.

(b) Those portions of the following oil bearing aquifers, which would otherwise meet the definition of a USDW, are exempted in accordance with the provisions of §§ 144.7(b) and 146.4 of this chapter for Class II enhanced recovery injection activities only.

(1) The Sugar Run and Bradford series of oil producing sands of the Bradford Field, in McKean County; including the Bradford, West Branch, Stack, Bennett Brook, Marilla Brook, Brooder Hollow, Cyclone, Minard Run, Minard Run School, and Sugar Run (or Watsonville) Pools.

(2) The Bradford Third oil producing sand of the Guffey Field in McKean County.

(3) The Bradford series of oil producing sands of the Lewis Run Field in McKean County.

(4) The Bradford series of oil producing sands of the Windfall Field and Kings Run Pool in McKean County.

(5) The Red Valley member of the Second Sand formation of the Venango Group of oil producing sands in the Foster-Reno Field in Venango County; including the Foster, Bully Hill, Victory, Bredinsburg, Egypt Corners, Reno, Monarch Park and Seneca Pools.

(6) The Glade and Clarendon oil producing sands of the Morrison Run Field and Elk Run Pool in Warren County.

(7) The Clarendon and Glade oil producing sands of the Clarendon Field in Warren County.

(8) The Bradford Third oil producing sand in the Shinglehouse Field, including the Kings Run, Janders Run and Ceres Pools in Potter and McKean Counties.

§ 147.1953 Existing Class I, II (except enhanced recovery and hydrocarbon storage) and III wells authorized by rule.

Maximum injection pressure. The owner or operator shall limit injection pressure to the lesser of:

(a) A value which will not exceed the operating requirements of § 144.28(f)(3)(i) or (ii) as applicable or

(b) A value for well head pressure calculated by using the following formula:

$$Pm=(0.733—0.433\ Sg)d$$

where

Pm=injection pressure at the well head in pounds per square inch

Sg=specific gravity of injection fluid (unitless)

d=injection depth in feet.

§ 147.1954 Existing Class II enhanced recovery and hydrocarbon storage wells authorized by rule.

(a) *Maximum injection pressure.* (1) To meet the operating requirements of § 144.28(f)(3)(ii) (A) and (B) of this chapter, the owner or operator:

(i) Shall use an injection pressure no greater than the pressure established by the Regional Administrator for the field or formation in which the well is located. The Regional Administrator shall establish such a maximum pressure after notice, opportunity for comment, and opportunity for a public hearing, according to the provisions of part 124, subpart A of this chapter, and will inform owners and operators in writing of the applicable maximum pressure; or

(ii) May inject at pressures greater than those specified in paragraph (a)(1)(i) of this section for the field or formation in which he is operating provided he submits a request in writing to the Regional Administrator, and demonstrates to the satisfaction of the

Regional Administrator that such injection pressure will not violate the requirement of §144.28(f)(3)(ii) (A) and (B). The Regional Administrator may grant such a request after notice, opportunity for comment, and opportunity for a public hearing, according to the provisions of part 124, subpart A of this chapter.

(2) Prior to such time as the Regional Administrator establishes rules for maximum injection pressure based on data provided pursuant to paragraph (a)(2)(ii) of this section the owner or operator shall:

(i) Limit injection pressure to a value which will not exceed the operating requirements of §144.28(f)(3)(ii); and

(ii) Submit data acceptable to the Regional Administrator which defines the fracture pressure of the formation in which injection is taking place. A single test may be submitted on behalf of two or more operators conducting operations in the same formation, if the Regional Administrator approves such submission. The information shall be submitted to the Regional Administrator within one year of the effective date of this regulation.

(b) *Casing and cementing.* Where the Regional Administrator determines that the owner or operator of an existing enhanced recovery or hydrocarbon storage well may not be in compliance with the requirements of §§144.28(e) and 146.22, the owner or operator shall comply with paragraphs (b) (1) through (4) of this section, when required by the Regional Administrator:

(1) Protect USDWs by:

(i) Cementing surface casing by recirculating the cement to the surface from a point 50 feet below the lowermost USDW; or

(ii) Isolating all USDWs by placing cement between the outermost casing and the well bore; and

(iii) For wells as described in §146.8(b)(3)(ii), installing a smaller diameter pipe inside the existing injection tubing and setting it on an appropriate packer; and

(2) Isolate any injection zones by placing sufficient cement to fill the calculated space between the casing and the well bore to a point 50 feet above the injection zone; and

(3) Use cement:

(i) Of sufficient quantity and quality to withstand the maximum operating pressure;

(ii) Which is resistant to deterioration from formation and injection fluids; and

(iii) In a quantity no less than 120% of the calculated volume necessary to cement off a zone.

(4) The Regional Administrator may specify other requirements in addition to or in lieu of the requirements set forth in paragraphs (b) (1) through (3) of this section as needed to protect USDWs.

§147.1955 Requirements for wells authorized by permit.

(a) The owner or operator of a Class I well authorized by permit shall install or shall ensure that the well has:

(1) Surface casing present;

(i) Extending from the surface to a depth at least 50 feet below the base of the lowermost USDW; and

(ii) Cemented back to the surface by recirculating the cement; and

(2) Long string casing and tubing;

(i) Extending to the injection zone; and

(ii) Cemented back to 50 feet above the base of the next largest casing string.

(b) The owner or operator of a new Class II well authorized by permit shall:

(1) Install surface casing from the surface to at least 50 feet below the base of the lowermost USDW.

(2) Cement the casing by recirculating to the surface or by using no less than 120% of the calculated annular volume.

(3) For new enhanced recovery wells, install tubing or long string casing extending to the injection zone.

(4) For new salt water disposal wells, install long string casing and tubing extending to the injection zone.

(5) Isolate any injection zone by placing sufficient cement to fill the calculated volume to a point 50 feet above the injection zone.

(c) The Regional Administrator may specify casing and cementing requirements other than those listed in paragraphs (a) and (b) of this section on a case by case basis as conditions of the permit.

Subpart OO—Rhode Island

§ 147.2000 State-administered program—Class I, II, III, IV, and V wells.

The UIC program for all classes of wells in Rhode Island, except those on Indian lands, is the program administered by the Rhode Island Department of Environmental Management, approved by EPA pursuant to section 1422 of the SDWA. Notice of this approval was published in the FEDERAL REGISTER on August 1, 1984; the effective date of this program is August 15, 1984. This program consists of the following elements, as submitted to EPA in the State's program application.

(a) *Incorporation by reference.* The requirements set forth in the State statutes and regulations cited in this paragraph are hereby incorporated by reference and made a part of the applicable UIC program under the SDWA for the State of Rhode Island. This incorporation by reference was approved by the Director of the Federal Register effective August 15, 1984.

(1) Rhode Island Gen. Laws sections 46–12–1, 46–12–5, and 46–12–28 (Supp. 1983);

(2) ''Underground Injection Control Program Rules and Regulations.'' State of Rhode Island and Providence Plantations Department of Environmental Management. Division of Water Resources (as received by the Secretary of State, May 21, 1984).

(b) *Other laws.* The following statutes and regulations although not incorporated by reference, also are part of the approved State-administered program:

(1) Rhode Island General Laws, Section 10–20–1 *et seq.,* entitled ''State Environmental Rights'';

(2) Rhode Island General Laws, Section 23–19.1–1 *et seq.,* entitled ''Hazardous Waste Management'';

(3) Rhode Island General Laws, Section 42–17.1 *et seq.,* entitled ''Department of Environmental Management'';

(4) Rhode Island General Laws, Section 42–35–1 *et seq.,* entitled ''Administrative Procedures'';

(5) Rhode Island General Laws, Section 46–12–1 *et seq.,* entitled ''Water Pollution'';

(6) Hazardous Waste Management Facility Operating Permit Rules and Regulations—Landfills, at last amended November 2, 1981 (hereinafter referred to as the ''Hazardous Waste Regulation'');

(7) Water Quality Regulations for Water Pollution Control, effective November 19, 1981; and

(8) Administrative Rules of Practices and Procedure for Department of Environmental Management, effective November 12, 1980.

(c) (1) The Memorandum of Agreement between EPA Region I and the Rhode Island Department of Environmental Management, signed by the EPA Regional Administrator on March 29, 1984;

(2) Letter from Director, Rhode Island Department of Environmental Management, to Regional Administrator, EPA Region I, amending Section III, C of the Memorandum of Agreement, April 25, 1984.

(d) *Statement of legal authority.* Letter from Attorney General, State of Rhode Island and Providence Plantations, to Regional Administrator, EPA Region 1, ''Re: Attorney General's Statement, Underground Injection Control Program,'' January 17, 1984.

(e) The Program Description and any other materials submitted as part of the original application or as supplements thereto.

[49 FR 30699, Aug. 1, 1984, as amended at 53 FR 43090, Oct. 25, 1988]

§ 147.2001 EPA-administered program—Indian lands.

(a) *Contents.* The UIC program for all classes of wells on Indian lands in the State of Rhode Island is administered by EPA. This program consists of the UIC program requirements of 40 CFR parts 124, 144, 146, 148, and any additional requirements set forth in the remainder of this subpart. Injection well owners and operators, and EPA shall comply with these requirements.

(b) *Effective date.* The effective date of the UIC program for Indian lands in Rhode Island is November 25, 1988.

[53 FR 43090, Oct. 25, 1988, as amended at 56 FR 9419, Mar. 6, 1991]

Subpart PP—South Carolina

§147.2050 State-administered program.

The UIC program for all classes of wells in the State of South Carolina, except for those on Indian lands, is the program administered by the South Carolina Department of Health and Environmental Control, approved by EPA pursuant to section 1422 of the SDWA. Notice of this approval was published in the FEDERAL REGISTER on July 10, 1984; the effective date of this program is July 24, 1984. This program consists of the following elements, as submitted to EPA in the State's program application.

(a) *Incorporation by reference.* The requirements set forth in the State statutes and regulations cited in this paragraph are hereby incorporated by reference and made a part of the applicable UIC program under the SDWA for the State of South Carolina. This incorporation by reference was approved by the Director of the Federal Register effective July 24, 1984.

(1) Pollution Control Act, S.C. Code Ann. Sections 48–1–10, 48–1–90, 48–1–100, 48–1–110 (Law. Co-op. 1976 and Supp. 1983).

(2) South Carolina Department of Health and Environmental Control, Ground-Water Protection Division, Underground Injection Control Regulations, R–61–87, Effective Date: June 24, 1983 Published in South Carolina State Register, Volume 7, Issue 6; Amended Date: March 23, 1984, as amended by notice in South Carolina State Register, Volume 8, Issue 3.

(b) *Other laws.* The following statutes and regulations although not incorporated by reference, also are part of the approved State-Administered program:

(1) Pollution Control Act, S.C. Code Ann. Sections 48–1–10 to 48–1–350 (Law. Co-op. 1976 and Supp. 1983).

(2) State Safe Drinking Water Act, S.C. Code Ann. Sections 44–55–10 to 44–55–100 (Law. Co-op. 1976 and Supp. 1983).

(3) Administrative Procedures Act, S.C. Code Ann. Sections 1–23–10 et seq., and 1–23–310 to 1–23–400 (Law. Co-op. 1976 and Supp. 1983).

(4) S.C. Code Ann. Sections 15–5–20, 15–5–200 (Law. Co-op. 1976 and Supp. 1983).

(c)(1) The Memorandum of Agreement between EPA Region IV and the South Carolina Department of Health and Environmental Control signed by the EPA Regional Administrator on May 29, 1984.

(d) *Statement of legal authority.* (1) ''Underground Injection Control Program, Attorney General's Statement for Class I, II, III, IV and VA and VB Wells,'' signed by the Attorney General of South Carolina on April 27, 1984.

(e) The Program Description and any other materials submitted as part of the original application or as supplements thereto.

[49 FR 28058, July 10, 1984, as amended at 53 FR 43090, Oct. 25, 1988]

§147.2051 EPA-administered program—Indian lands.

(a) *Contents.* The UIC program for all classes of wells on Indian lands in the State of Rhode Island is administered by EPA. This program consists of the UIC program requirements of 40 CFR parts 124, 144, 146, 148, and any additional requirements set forth in the remainder of this subpart. Injection well owners and operators, and EPA shall comply with these requirements.

(b) *Effective date.* The effective date of the UIC program for Indian lands in South Carolina is November 25, 1988.

[53 FR 43090, Oct. 25, 1988, as amended at 56 FR 9419, Mar. 6, 1991]

Subpart QQ—South Dakota

§147.2100 State-administered program—Class II wells.

The UIC program for Class II wells in the State of South Dakota, except those on Indian lands, is the program administered by the South Dakota Department of Water and Natural Resources, approved by EPA pursuant to section 1425 of the SDWA. Notice of this approval was published in the FEDERAL REGISTER on October 24, 1984; the effective date of this program is December 7, 1984. This program consists of the following elements, as submitted to EPA in the State's program application.

(a) *Incorporation by reference.* The requirements set forth in the State statutes and regulations cited in this paragraph are hereby incorporated by reference and made a part of the applicable UIC program under the SDWA for the State of South Dakota. This incorporation by reference was approved by the Director of the Federal Register effective December 7, 1984.

(1) South Dakota Codified Laws, sections 45–9–2, 45–9–4, 45–9–11, 45–9–13, 45–9–14, 45–9–15 (1983).

(2) Administrative Rules of South Dakota, sections 74:10:02 through 74:10:07, 74:10:09, and 74:10:11 published by the South Dakota Code Commission, as revised through October 4, 1987.

(b) *Other laws.* The following statutes and regulations, although not incorporated by reference, also are part of the approved State-administered program:

(1) South Dakota Codified Laws, Chapter 45–9 (sections not cited above) (1983); 1–26 (1981).

(c)(1) The Memorandum of Agreement between EPA Region VIII and the South Dakota Department of Water and Natural Resources, signed by the EPA Regional Administrator on July 18, 1984.

(d) *Statement of legal authority.* (1) ''Underground Injection Control Program for Class II Wells: Attorney General's Statement,'' signed by Mark V. Meierhery, Attorney General, South Dakota, on January 16, 1984.

(e) The Program Description and any other materials submitted as part of the original application or as supplements thereto.

[50 FR 7061, Feb. 20, 1985, as amended at 56 FR 9419, Mar. 6, 1991]

§ 147.2101 EPA-administered program—Class I, III, IV and V wells and all wells on Indian lands.

(a) *Contents.* The UIC program for all Class I, III, IV, and V wells, including those on Indian lands, and for Class II wells on Indian lands in the state of South Dakota is administered by EPA. This program consists of the UIC program requirements of 40 CFR parts 124, 144, 146, 148, and any additional requirements set forth in the remainder of this subpart. Injection well owners and operators, and EPA shall comply with these requirements.

(b) *Effective date.* The effective date of the UIC program for Class I, III, IV and V wells on all lands in South Dakota, including Indian lands, and for Class II wells on Indian lands only, is December 30, 1984.

[52 FR 17682, May 11, 1987, as amended at 56 FR 9419, Mar. 6, 1991]

§ 147.2102 Aquifer exemptions.

(a) This section identifies any aquifers or their portions exempted in accordance with §§ 144.7(b) and 146.4 of this chapter at the time of program promulgation. EPA may in the future exempt other aquifers or their portions, according to applicable procedures, without codifying such exemptions in this section. An updated list of exemptions will be maintained in the Regional office.

(b) Those portions of all aquifers located on Indian Lands, which meet the definition of USDW and into which existing Class II wells are injecting, are exempted within a ¼ mile radius of the well for the purpose of Class II injection activities only.

[49 FR 45308, Nov. 15, 1984]

§ 147.2103 Existing Class II enhanced recovery and hydrocarbon storage wells authorized by rule.

(a) *Maximum injection pressure.* (1) To meet the operating requirements of § 144.28(f)(3)(ii) (A) and (B) of this chapter, the owner or operator:

(i) Shall use an injection pressure no greater than the pressure established by the Regional Administrator for the field or formation in which the well is located. The Regional Administrator shall establish such a maximum pressure after notice, opportunity for comments, and opportunity for a public hearings, according to the provisions of part 124, subpart A of this chapter, and will inform owners and operators in writing of the applicable maximum pressure; or

(ii) May inject at a pressure greater than those specified in paragraph (a)(1)(i) of this section for the field or formation in which he is operating provided he submits a request in writing to the Regional Administrator, and

demonstrates to the satisfaction of the Regional Administrator that such injection pressure will not violate the requirement of §144.28(f)(3)(ii)(A) and (B). The Regional Administrator may grant such a request after notice, opportunity for comment, and opportunity for a public hearing, according to the provisions of part 124, subpart A of this chapter.

(2) Prior to such time as the Regional Administrator establishes field rules for maximum injection pressure based on data provided pursuant to paragraph (a)(2)(ii) of this section the owner or operator shall:

(i) Limit injection pressure to a value which will not exceed the operating requirements of §144.28(f)(3)(ii); and

(ii) Submit to the Regional Administrator data acceptable to the Regional administrator which defines the fracture pressure of the formation in which injection is taking place. A single test may be submitted on behalf of two or more operators conducting operations in the same formation, if the Regional Administrator approves such submission.

(b) *Casing and cementing.* Where the Regional Administrator determines that the owner or operator of an existing enhanced recovery or hydrocarbon storage well may not be in compliance with the requirement of §§144.28(e) and 146.22, the owner or operator shall when required by the Regional Administrator:

(1) Protect USDWs by:

(i) Cementing surface casing by recirculating the cement to the surface from a point 50 feet below the lowermost USDW; or

(ii) Isolating all USDWs by placing cement between the outermost casing and the well bore; and

(2) Isolate any injection zones by placing sufficient cement to fill the calculated space between the casing and the well bore to a point 250 feet above the injection zone; and

(3) Use cement:

(i) Of sufficient quantity and quality to withstand the maximum operation pressure;

(ii) Which is resistant to deterioration from formation and injection fluids; and

(iii) In a quantity no less than 120% of the calculated volume necessary to cement off a zone; and/or

(4) Comply with other requirements which the Regional Administrator may specify in addition to or in lieu of the requirements set forth in paragraphs (b) (1) through (3) of this section as needed to protect USDWs.

[49 FR 45308, Nov. 15, 1984]

§147.2104 Requirements for all wells.

(a) The owner or operator converting an existing well to an injection well shall check the condition of the casing with one of the following logging tools;

(1) A pipe analysis log; or

(2) A caliper log.

(b) The owner or operator of a new injection well cased with plastic (PVC, ABS, or others) casings shall:

(1) Not construct a well deeper than 500 feet;

(2) Use cement and additives compatible with such casing material; and

(3) Cement the annular space above the injection intermal from the bottom of the blank casing to the surface.

(c) The owner or operator of a newly drilled well shall install centralizers as directed by the Regional Administrator.

(d) The owner or operator shall as required by the Regional Administrator:

(1) Protect USDWs by:

(i) Setting surface casing 50 feet below the lowermost USDW;

(ii) Cementing surface casing by recirculating the cement to the surface from a point 50 feet below the lowermost USDW; or

(iii) Isolating all USDWs by placing cement between the outermost casing and the well bore; and

(2) Isolate any injection zones by placing sufficient cement to fill the calculated space between the casing and the well bore to a point 250 feet above the injection zone; and

(3) Use cement:

(i) Of sufficient quantity and quality to withstand the maximum operating pressure; and

(ii) Which is resistant to deterioration from formation and injection fluids; and

(iii) In a quantity no less than 120% of the calculated volume necessary to cement off a zone.

(4) The Regional Administrator may approve alternate casing and cementing practices provided that the owner or operator demonstrates that such practices will adequately protect USDWs.

(e) *Area of review*. Notwithstanding the alternatives presented in § 146.6 of this chapter, the area of review shall be a fixed radius as described in § 146.6(b) of this chapter.

(f) The applicant must give separate notice of intent to apply for a permit to each owner of record of the land within one-quarter mile of the site. The addresses of those to whom notice is given and the description of how notice was given shall be submitted with the permit application. The notice shall include:

(1) The name and address of applicant;

(2) A brief description of the planned injection activities, including well location, name and depth of the injection zone, maximum injection pressure and volume, and fluid to be injected;

(3) The EPA contact person; and

(4) A statement that opportunity to comment will be announced after EPA prepares a draft permit.

This requirement may be waived by the Regional Administrator if he determines that individual notice to all land owners of record would be impractical.

[49 FR 45308, Nov. 15, 1984]

Subpart RR—Tennessee

§ 147.2150 State-administered program. [Reserved]

§ 147.2151 EPA-administered program.

(a) *Contents*. The UIC program for the State of Tennessee, including all Indian lands, is administered by EPA. This program consists of the UIC program requirements of 40 CFR parts 124, 144, 146, 148, and any additional requirements set forth in the remainder of this subpart. Injection well owners and operators, and EPA shall comply with these requirements.

(b) *Effective dates*. Effective date for the UIC program on Indian lands is November 25, 1988. The effective date for the UIC program for the rest of Tennessee is June 25, 1984.

[53 FR 43090, Oct. 25, 1988, as amended at 56 FR 9419, Mar. 6, 1991]

§ 147.2152 Aquifer exemptions. [Reserved]

§ 147.2153 Existing Class I, II (except enhanced recovery and hydrocarbon storage) and III wells authorized by rule.

Maximum injection pressure. The owner or operator shall limit injection pressure to the lesser of:

(a) A value which will not exceed the operating requirements of § 144.28(f)(3)(i) or (ii) as applicable or

(b) A value for well head pressure calculated by using the following formula:

$$Pm=(0.600-0.433\ Sg)d$$

where

Pm=injection pressure at the well head in pounds per square inch

Sg=specific gravity of inject fluid (unitless)

d=injection depth in feet.

§ 147.2154 Existing Class II enhanced recovery and hydrocarbon storage wells authorized by rule.

(a) *Maximum injection pressure*. (1) To meet the operating requirements of § 144.28(f)(3)(ii) (A) and (B) of this chapter, the owner or operator:

(i) Shall use an injection pressure no greater than the pressure established by the Regional Administrator for the field or formation in which the well is located. The Regional Administrator shall establish such a maximum pressure after notice, opportunity for comment, and opportunity for a public hearing, according to the provisions of part 124, subpart A of this chapter, and will inform owners and operators in writing of the applicable maximum pressure; or

(ii) May inject at pressures greater than those specified in paragraph (a)(1)(i) of this section for the field or formation in which he is operating, provided he submits a request in writing to the Regional Administrator and demonstrates to the satisfaction of the Regional Administrator that such injection pressure will not violate the requirement of § 144.28(f)(3)(ii) (A) and (B). The Regional Administrator may

grant such a request after notice, opportunity for comment, and opportunity for a public hearing, according to the provisions of part 124, subpart A of this chapter.

(2) Prior to such time as the Regional Administrator establishes rules for maximum injection pressure based on data provided pursuant to paragraph (a)(2)(ii) of this section the owner or operator shall:

(i) Limit injection pressure to a value which will not exceed the operating requirements of §144.28(f)(3)(ii); and

(ii) Submit data acceptable to the Regional Administrator which defines the fracture pressure of the formation in which injection is taking place. A single test may be submitted on behalf of two or more operators conducting operations in the same formation, if the Regional Administrator approves such submission. The data shall be submitted to the Regional Administrator within one year of the effective date of this regulation.

(b) *Casing and cementing.* Where the Regional Administrator determines that the owner or operator of an existing enhanced recovery or hydrocarbon storage well may not be in compliance with the requirements of §§144.28(e) and 146.22, the owner or operator shall comply with paragraphs (b) (1) through (4) of this section, when required by the Regional Administrator:

(1) Protect USDWs by:

(i) Cementing surface casing by recirculating the cement to the surface from a point 50 feet below the lowermost USDW; or

(ii) Isolating all USDWs by placing cement between the outermost casing and the well bore; and

(2) Isolate any injection zones by placing sufficient cement to fill the calculated space between the casing and the well bore to a point 250 feet above the injection zone; and

(3) Use cement:

(i) Of sufficient quantity and quality to withstand the maximum operating pressure;

(ii) Which is resistant to deterioration from formation and injection fluids; and

(iii) In a quantity no less than 120% of the calculated volume necessary to cement off a zone.

(4) The Regional Administrator may specify other requirements in addition to or in lieu of the requirements set forth in paragraphs (b) (1) through (3) of this section, as needed to protect USDWs.

§147.2155 Requirements for all wells—area of review.

Notwithstanding the alternatives presented in §146.6 of this chapter, the area of review shall be a minimum fixed radius as described in §146.6(b) of this chapter.

Subpart SS—Texas

§147.2200 State-administered program—Class I, III, IV, and V wells.

Requirements for Class I, III, IV, and V wells. The UIC program for Class I, III, IV, and V wells in the State of Texas, except for those wells on Indian lands, is the State-administered program approved by EPA pursuant to section 1422 of the SDWA. Notice of this approval was published on January 6, 1982 (47 FR 618); the effective date of this program is February 7, 1982. This program consists of the following elements, as submitted to EPA in the State's program application:

(a) *Incorporation by reference.* The requirements set forth in the State statutes and regulations cited in this paragraph are hereby incorporated by reference and made a part of the applicable UIC program under the SDWA for the State of Texas. This incorporation by reference was approved by the Director of the Federal Register on June 25, 1984.

(1) Injection Well Act, Texas Water Code sections 27.002, 27.011 (Vernon Supp. 1984);

(b) *Other laws.* The following statutes and regulations, although not incorporated by reference except for select sections identified in paragraph (a) of this section, are also part of the approved State-administered UIC program:

(1) Texas Water Code Annotated, Chapter 5 (Vernon 1972 and Supp. 1982);

(2) Injection Well Act, Texas Water Code Annotated, Chapter 27 (Vernon 1972 and Supp. 1982);

(3) Rules of Texas Department of Water Resources, Chapter 27; Rules of

Texas Water Development Board, Chapter 22.

(c) The Memorandum of Agreement between EPA Region VI and the Texas Department of Water Resources, signed by the EPA Regional Administrator on October 11, 1981.

(d) *Statement of legal authority.* ''Underground Injection Control Program—Attorney General's Statement for Class I, III, IV, and V Injection Wells,'' signed by the Attorney General of Texas, June 11, 1981.

(e) The Program Description and any other materials submitted as part of the application or as supplements thereto.

(f) Certain Class V wells are under the UIC program of the Texas Railroad Commission approved on April 23, 1982, under the authorities cited in § 147.2201 of this part.

[49 FR 20197, May 11, 1984, as amended at 53 FR 43091, Oct. 25, 1988]

§ 147.2201 State-administered program—Class II wells

The UIC program for Class II wells in the State of Texas, except for those wells on Indian lands, is the program administered by the Railroad Commission of Texas, approved by EPA pursuant to section 1425 of the SDWA. Notice of this approval was published in the FEDERAL REGISTER on April 23, 1982 (47 FR 17488). The effective date of this program was May 23, 1982. This program consists of the following elements, as submitted to EPA in the State's program application:

(a) *Incorporation by reference.* The requirements set forth in the State statutes and regulations cited in this paragraph are hereby incorporated by reference and made a part of the applicable UIC program under the SDWA for the State of Texas. This incorporation by reference was approved by the Director of the Federal Register on June 25, 1984.

(1) Injection Well Act, Texas Water Code Annotated sections 27.031 and 27.033 (Vernon Supp. 1984);

(2) Texas Natural Resources Code Annotated sections 85.041, 85.045, 85.046 and 85.052 (Vernon 1978 and Supp. 1982);

(3) Rules Having Statewide General Application to Oil, Gas, and Geothermal Resource Operations, sections .051.02.02.000 to .051.02.02.080 (Railroad Commission of Texas, Oil and Gas Division, Revised 12–22–81), amended as follows:

(i) Amendment to 16 TAC section 3.9 (section .051.02.02.009) issued December 21, 1981, effective April 1, 1982;

(ii) Amendment to 16 TAC section 3.46 (section .051.02.02.046) issued December 21, 1981, effective April 1, 1982.

(iii) Amendment to 16 TAC section 3.71 (section .051.02.02.074) issued December 21, 1981, effective April 1, 1982.

(b) *Other laws.* The following statutes and regulations, although not incorporated by reference, are also part of the approved State-administered UIC program:

(1) Texas Water Code, Chapters 26, 27 and 29 (Vernon 1972 and Supp. 1982);

(2) Texas Natural Resources Code, Chapters 81, 85–89, 91 and 141 (Vernon 1978 and Supp. 1982);

(3) General Rules of Practice and Procedure, Subchapters A–J (Railroad Commission of Texas, adopted November 24, 1975, revised December 1980).

(c)(1) The Memorandum of Agreement between EPA Region VI and the Railroad Commission of Texas, signed by the EPA Regional Administrator on March 24, 1982.

(2) Letter from Director of Underground Injection Control, Railroad Commission of Texas, to Chief, Ground Water Protection Section, EPA Region VI, ''Re: Letter of Clarification—UIC Program Application,'' March 21, 1982.

(d) *Statement of legal authority.* ''Statement of Legal Authority of the Railroad Commission of Texas to conduct the Underground Injection Control Program,'' signed by Special Counsel, Railroad Commission of Texas, as submitted with ''State of Texas Underground Injection Control Program Application for Primacy Enforcement Authority,'' prepared by the Railroad Commission of Texas, January 15, 1982.

(e) The Program Description and any other materials submitted as part of the application or as supplements thereto.

[49 FR 20197, May 11, 1984, as amended at 53 FR 43091, Oct. 25, 1988]

§ 147.2205 EPA-administered program—Indian lands.

(a) *Contents.* The UIC program for all classes of wells on Indian lands in the State of Texas is administered by EPA. This program consists of the UIC program requirements of 40 CFR parts 124, 144, 146, 148, and any additional requirements set forth in the remainder of this subpart. Injection well owners and operators, and EPA shall comply with these requirements.

(b) *Effective date.* The effective date for the Indian lands program for the State of Texas is November 25, 1988.

[53 FR 43091, Oct. 25, 1988, as amended at 56 FR 9419, Mar. 6, 1991]

Subpart TT—Utah

§ 147.2250 State-administered program—Class I, III, IV, and V wells.

The UIC program for Class I, III, IV, and V wells in the State of Utah, except those on Indian lands, is administered by the Utah Department of Health, Division of Environmental Health, approved by EPA pursuant to Section 1422 of the SDWA. Notice of this approval was published in the FEDERAL REGISTER on January 9, 1983 (47 FR 2321). The effective date of this program is February 10, 1983. Changes to Utah's regulations for Class I wells were made on May 15, 1990, in response to modification of national rules as promulgated by 53 FR 28188, July 26, 1988. Utah's rules were effective July 20, 1990. The revised rules, Program Description, Attorney General's statement, and Memorandum of Agreement were approved as a minor program modification on October 3, 1990. This program consists of the following elements as submitted to EPA:

(a) *Incorporation by reference.* The requirements set forth in the State statutes and regulations cited in this paragraph are hereby incorporated by reference and made a part of the applicable UIC program under the SDWA for the State of Utah. This incorporation by reference was approved by the Director of the Federal Register on June 25, 1984.

(1) Utah Water Pollution Control Act, Utah Code Annotated, Title 26, Chapter 11, Sections 2, 8, and 10 (1989);

(2) Underground Injection Control Regulations; Utah Administrative Code, Section R448–7 (effective as of January 2, 1990);

(3) Underground Injection Control Program (adopted January 20, 1982 and revised effective July 20, 1990) (Officially submitted to EPA by the Executive Secretary of Utah Water Pollution Control Committee on August 16, 1990).

(b) *Other laws.* The following statutes and regulations, although not incorporated by reference except for selected sections identified in paragraph (a) of this section, are also part of the approved State-administered program:

(1) Utah Pollution Control Act, Utah Code Annotated, Sections 26–11–1 through –20 (Supp. 1990);

(c)(1) The revised Memorandum of Agreement between EPA, Region VIII and the Utah Department of Health, Division of Environmental Health, signed by the Regional Administrator on October 3, 1990.

(2) Letter from Director, Utah Department of Health, Division of Environmental Health, Bureau of Water Pollution Control, to EPA Region VIII, Re: Underground Injection Control Program—Utah, March 15, 1982;

(3) Letter from the Executive Secretary of the Utah Water Pollution Control Committee to EPA Region VIII, ''Re: Utah UIC Class I Well Program Changes,'' August 16, 1990;

(d) *Statement of legal authority.* (1) ''Underground Injection Control Program—Attorney General's statement,'' signed by Attorney General, State of Utah, January, 1982;

(2) Letter from Assistant Attorney General of Utah to Chief, Drinking Water Branch, EPA Region VIII, June 18, 1982;

(3) Addendum to Underground Injection Control Program, Attorney General's Statement signed by Attorney General of Utah, August 10, 1990.

(e) The Program Description (revised June 19, 1990) and any other materials submitted as part of the application or supplements thereto.

[56 FR 9419, Mar. 6, 1991]

§ 147.2251 State-administered program—Class II wells.

The UIC program for Class II wells in the State of Utah, except those on Indian lands, is the program administered by the Utah Department of Natural Resources, Division of Oil, Gas, and Mining, approved by EPA pursuant to section 1425 of the SDWA. Notice of this approval was published in the FEDERAL REGISTER on October 8, 1982 (47 FR 44561); the effective date of this program is November 7, 1982. This program consists of the following elements, as submitted to EPA in the State's program application:

(a) *Incorporation by reference.* The requirements set forth in the State statutes and regulations cited in this paragraph are hereby incorporated by reference and made a part of the applicable UIC program under the SDWA for the State of Utah. This incorporation by reference was approved by the Director of the Federal Register on June 25, 1984.

(1) Utah Code Annotated, 1953, section 40–6–1 through 40–6–18, as amended 1988 and Cumm. Supp. 1990;

(2) The Oil and Gas Conservation General Rules, adopted under the authority of the Oil and Gas Conservation Act, 40–6–1 *et seq.*, Utah Code Annotated, as amended 1988 (revised March 1989), rules R615–1 through R615–4, and R615–8 through R615–10.

(b) *Other laws.* [Reserved]

(c)(1) The Memorandum of Agreement between EPA, Region VIII and the Utah Department of Natural Resources, Division of Oil, Gas, and Mining and the Board of Oil, Gas and Mining, signed by the EPA Regional Administrator on July 19, 1983;

(2) Letter from Director, Division of Oil, Gas and Mining, Utah Department of Natural Resources and Energy, to Regional Administrator, EPA Region VIII, ''Re: Aquifer Exemption Process,'' June 16, 1982;

(3) ''Memorandum of Understanding'' between Utah Department of Health and Utah Department of Natural Resources, dated March 5, 1981;

(4) ''Second Addition to Agreement between the Department of Health and the Department of Natural Resources and Energy,'' dated December 15, 1981.

(d) *Statement of legal authority.* (1) Part III of ''Primacy Application—Class II Underground Injection Wells,'' consisting of ''Synopsis of Pertinent Statutes and Regulations,'' ''Statement of Legal Authority,'' and ''Certification by the Attorney General,'' by Assistant Attorney General, Department of Natural Resources and Energy, dated December 18, 1981;

(2) Letter from Assistant Attorney General, State of Utah, to EPA Region VIII, undated, received in the EPA Office of Regional Counsel June 10, 1982.

(3) Memorandum to Director, Division of Oil, Gas and Mining from Assistant Attorney General regarding Underground Injection Control Program, January 8, 1985.

(e) The Program Description and any other materials submitted as part of the application or amendments thereto.

[49 FR 20197, May 11, 1984, as amended at 53 FR 43091, Oct. 25, 1988; 56 FR 9420, Mar. 6, 1991]

§ 147.2253 EPA-administered program—Indian lands.

(a) *Contents.* The UIC program for all classes of wells on Indian lands in the State of Utah is administered by EPA. The program for wells on the lands of the Navajo and Ute Mountain Ute consists of the requirements set forth at subpart HHH of this part. The program for all other wells on Indian lands consists of the UIC program requirements of 40 CFR parts 124, 144, 146, 148, and any additional requirements set forth in the remainder of this subpart. Injection well owners and operators, and EPA shall comply with these requirements.

(b) *Effective date.* The effective date for this program for all other Indian lands in Utah (as well as for the program of the Navajo and Ute Mountain Ute) is November 25, 1988.

[53 FR 40391, Oct. 25, 1988, as amended at 56 FR 9420, Mar. 6, 1991] h

Subpart UU—Vermont

§ 147.2300 State-administered program.

The UIC program for all classes of wells in the State of Vermont, except

those wells on Indian lands, is the program administered by the Vermont Department of Environmental Conservation, approved by EPA pursuant to section 1422 of the SDWA. Notice of this approval was published in the FR on June 22, 1984; the effective date of this program is July 6, 1984. This program consists of the following elements:

(a) *Incorporation by reference.* The requirements set forth in the State statutes and regulations cited in this paragraph are hereby incorporated by reference and made a part of the applicable UIC program under the SDWA for the State of Vermont. This incorporation by reference was approved by the Director of the Federal Register July 6, 1984.

(1) Vt. Stat. Ann. tit. 10, sections 1251, 1259, 1263 (1973 and Supp. 1981), Effective date: July 1, 1982.

(2) Vermont Department of Water Resources and Environmental Engineering, Chapter 13 Water Pollution Control Regulations, Subchapter 13.UIC—Underground Injection Control, Discharges to Injection Wells, Effective Date: June 21, 1984.

(b) *Other laws.* The following statutes and regulations although not incorporated by reference, also are part of the approved State-administered program:

(1) Vt. Stat. Ann. tit. 10, sections 1251 through 1283 (1973 and Supp. 1981).

(2) Vt. Stat. Ann. tit. 10, sections 901 through 911 (1973 and Supp. 1981).

(3) Vt. Stat. Ann. tit. 3, sections 801 through 847 (1973 and Supp. 1981).

(c)(1) The Memorandum of Agreement between EPA Region I and the Vermont Agency of Environmental Conservation signed by the EPA Regional Administrator on January 16, 1984.

(d) *Statement of legal authority.* (1) ''Vermont Attorney General's Statement for Classes I, II, III, IV and V Injection Wells,'' signed by Attorney General John J. Easton, Jr., as submitted with Vermont Application for Primary Enforcement Responsibility to Administer the Underground Water Source Protection Program Pursuant to the Safe Drinking Water Act and 40 CFR 145.21 through 145.24 (December 20, 1983).

(e) The Program Description and any other materials submitted as part of the original application or as supplements thereto.

(42 U.S.C. 300)

[49 FR 25634, June 22, 1984, as amended at 53 FR 43091, Oct. 25, 1988; 56 FR 9420, Mar. 6, 1991]

§§ 147.2301—147.2302 [Reserved]

§ 147.2303 EPA-administered program—Indian lands.

(a) *Contents.* The UIC program for all classes of wells on Indian lands in the State of Vermont is administered by EPA. This program consists of the UIC program requirements of 40 CFR parts 124, 144, 146, 148, and any additional requirements set forth in the remainder of this subpart. Injection well owners and operators, and EPA shall comply with these requirements.

(b) *Effective date.* The effective date of the UIC program for Indian lands in Vermont is November 25, 1988.

[53 FR 43091, Oct. 25, 1988, as amended at 56 FR 9420, Mar. 6, 1991]

§§ 147.2304—147.2349 [Reserved]

Subpart VV—Virginia

§ 147.2350 State-administered program. [Reserved]

§ 147.2351 EPA-administered program.

(a) *Contents.* The UIC program for the State of Virginia, including all Indian lands, is administered by EPA. This program consists of the UIC program requirements of 40 CFR parts 124, 144, 146, 148, and any additional requirements set forth in the remainder of this subpart. Injection well owners and operators, and EPA shall comply with these requirements.

(b) *Effective dates.* The effective date for the UIC program on Indian lands is November 25, 1988. The effective date for the UIC program for the remainder of Virginia is June 25, 1984. (53 FR 43091, October 25, 1988).

[56 FR 9420, Mar. 6, 1991]

§ 147.2352 Aquifer exemptions. [Reserved]

Subpart WW—Washington

§ 147.2400 State-administered program—Class I, II, III, IV, and V wells.

The UIC program for Class I, II, III, IV, and V wells in the State of Washington other than those on Indian lands, is the program administered by the Washington Department of Ecology, approved by EPA pursuant to section 1422 of the SDWA. Notice of this approval was published in the FEDERAL REGISTER on August 9, 1984; the effective date of this program is September 24, 1984. This program consists of the following elements, as submitted to EPA in the State's program application.

(a) *Incorporation by reference.* The requirements set forth in the State statutes and regulations cited in this paragraph are hereby incorporated by reference and made a part of the applicable UIC program under the SDWA for the State of Washington. This incorporation by reference was approved by the Director of the Federal Register effective September 24, 1984.

(1) Revised Code of Washington section 90.48.020, 90.48.080, 90.48.160, and 90.48.162 (Bureau of National Affairs, 1983 Laws);

(2) Washington Administrative Code sections 173–218–010 to 173–218–110 (Bureau of National Affairs, 2/29/84);

(3) Washington Administrative Code sections 344–12–001 to 344–12–262 (1983 Ed.)

(4) Washington Administrative Code Chapter 173–160 (reprinted May 1988).

(b) *Other laws.* The following statutes and regulations although not incorporated by reference, also are part of the approved State-administered program:

(1) Revised Code of Washington, chapter 34.04 (Bureau of National Affairs, 1981 Laws), entitled ''Administrative Procedure act'';

(2) Revised Code of Washington, chapter 43.21A (Bureau of National Affairs, 1980 Laws), entitled ''Department of Ecology,'' as amended by 1983 Washington Laws, Chapter 270;

(3) Revised Code of Washington, chapter 70.105 (Bureau of National Affairs, 1983 Laws), entitled ''Hazardous Waste Disposal'';

(4) Revised Code of Washington, chapter 78.52 (Bureau of National Affairs, 1983 Laws), entitled ''Oil and Gas Conservation'';

(5) Revised Code of Washington, chapter 90.48 (Bureau of National Affairs, 1986 Laws), entitled ''Water Pollution Control.''

(c)(1) The Memorandum of Agreement between EPA Region X and the Washington Department of Ecology, signed by the EPA Regional Administrator on May 14, 1984;

(2) Memorandum of Agreement between the Washington Department of Ecology and Oil and Gas Conservation Committee, Related to the Underground Injection Control Program for the State of Washington, signed March 23, 1984;

(3) Memorandum of Agreement between the Washington Department of Ecology and Washington Department of Natural Resources, Related to the Underground Injection Control Program for the State of Washington, signed March 23, 1984;

(4) Memorandum of Agreement between the Washington Department of Ecology and Department of Social and Health Services, Related to the Underground Injection Control Program for the State of Washington, signed March 23, 1984;

(d) *Statement of legal authority.* Letter from Attorney General of the State of Washington, by Senior Assistant Attorney General, to Director, Washington State Department of Ecology, ''Re: Underground Injection Control Regulatory Program—Attorney General's Statement,'' February 28, 1984.

(e) The Program Description and any other materials submitted as part of the original application or as supplements thereto.

[49 FR 31876, Aug. 9, 1984, as amended at 56 FR 9420, Mar. 6, 1991]

§ 147.2403 EPA-administered program—Indian lands.

(a) *Contents.* The UIC program for all classes of wells on Indian lands in the State of Washington is administered by EPA. This program, for all Indian lands

except those of the Colville Tribe, consists of the UIC program requirements of 40 CFR parts 124, 144, 146, 148, and any additional requirements set forth in the remainder of this subpart. Injection well owners and operators, and EPA shall comply with these requirements.

(b) *Effective date.* The effective date for the UIC program for Indian lands in Washington is November 25, 1988.

[53 FR 43091, Oct. 25, 1988, as amended at 56 FR 9420, Mar. 6, 1991]

§147.2404 EPA-administered program—Colville Reservation.

(a) The UIC program for the Colville Indian Reservation consists of a prohibition of all Class I, II, III and IV injection wells and of a program administered by EPA for Class V wells. This program consists of the UIC program requirements of 40 CFR part 124, 144 and 146 and any additional requirements set forth in the remainder of this subpart. Injection well owners and EPA shall comply with these requirements. The prohibition on Class I–IV wells is effective November 25, 1988. No owner or operator shall construct, operate, maintain, convert, or conduct any other injection activity thereafter using Class I–IV wells.

(b) Owners and operators of Class I, II, III or IV wells in existence on the effective date of the program shall cease injection immediately. Within 60 days of the effective date of the program, the owner or operator shall submit a plan and schedule for plugging and abandoning the well for the Director's approval. The owner or operator shall plug and abandon the well according to the approved plan and schedule.

[53 FR 43091, Oct. 25, 1988]

Subpart XX—West Virginia

§§147.2450—147.2452 [Reserved]

§147.2453 EPA-administered program—Indian lands.

(a) *Contents.* The UIC program for all classes of wells on Indian lands in the State of West Virginia is administered by EPA. This program consists of the UIC program requirements of 40 CFR parts 124, 144, 146, 148, and any additional requirements set forth in the remainder of this subpart. Injection well owners and operators, and EPA shall comply with these requirements.

(b) *Effective date.* The effective date for the UIC program on Indian lands in West Virginia is November 25, 1988.

[53 FR 43092, Oct. 25, 1988, as amended at 56 FR 9420, Mar. 6, 1991]

§§147.2454—147.2499 [Reserved]

Subpart YY—Wisconsin

§147.2500 State-administered program.

The UIC program for Class I, II, III, IV, and V wells in the State of Wisconsin, other than those on Indian lands as described in §147.2510, is the program administered by the Wisconsin Department of Natural Resources, approved by EPA pursuant to SDWA section 1422. Notice of this approval was published in the FEDERAL REGISTER on September 30, 1983 (48 FR 44783); the effective date of this program is November 30, 1983. This program consists of a prohibition of all injection wells except heat pump return flow injection wells and may be found in the following elements, as submitted to EPA in the State's program application.

(a) *Incorporation by reference.* The requirements set forth in the State statutes and regulations cited in this paragraph are hereby incorporated by reference and made a part of the applicable UIC program under the SDWA for the State of Wisconsin. This incorporation by reference was approved by the Director of the OFR in accordance with 5 U.S.C. 552(a) and 1 CFR part 51. Copies may be obtained at the Wisconsin Department of Natural Resources, Box 7921, Madison, Wisconsin, 53707. Copies may be inspected at the Environmental Protection Agency, Region V, 77 West Jackson Boulevard, Chicago, Illinois, 60604, or at the Office of the Federal Register, 800 North Capitol Street, NW., suite 700, Washington, DC.

(1) Wisconsin Statutes Annotated §§147.015, 147.02 and 147.04 (West 1974 and Supp. 1983);

(2) Chapter NR 112, Well Construction and Pump Installation, Wisconsin Administrative Code §§NR 112.03 and 112.20 (October 1981), as amended by

Natural Resources Board Order No. WQ–25–82, approved by the Natural Resources Board on August 25, 1982;

(3) Chapter NR 113, Servicing Septic Tanks, Seepage Pits, Grease Traps or Privies, Wisconsin Administrative Code §§ NR 113.07–113.08 (1979), as amended by Natural Resources Board Order No. WQ–25–82, approved by the Wisconsin Natural Resources Board on August 25, 1982;

(4) Chapter NR 181, Hazardous Waste Management, Wisconsin Administrative Code §§ NR 181.04–181.415 (1981), as amended June 1985;

(5) Chapter NR 210, Sewage Treatment Works, Wisconsin Administrative Code § 210.05 Natural Resources Board Order No. WQ–25–82, approved by the Wisconsin Natural Resources Board on August 25, 1982;

(6) Chapter NR 214, Land Application and Disposal of Liquid Industrial Wastes and By-Products, Wisconsin Administrative Code §§ 214.03 and 214.08 (1983).

(b) *Other laws.* The following statutes and regulations, although not incorporated by reference except for select sections identified in paragraph (a) of this section, are also part of the approved State-administered program:

(1) Chapter 144, Water, Sewage, Refuse, Mining and Air Pollution, Wisconsin Statutes Annotated (West 1974 and Supp. 1983);

(2) Chapter 147, Pollution Discharge Elimination, Wisconsin Statutes Annotated (West 1974 and Supp. 1983);

(3) Chapter 162, Pure Drinking Water, Wisconsin Statutes Annotated (West 1974 and Supp. 1983);

(4) Laws of 1981, Chapter 20, § 2038 (Re: heat pump injection);

(5) Wisconsin Statutes 803.09(1) (West 1977) (intervention as of right in civil actions).

(c) *Memorandum of Agreement.* The Memorandum of Agreement between EPA Region V and the Wisconsin Department of Natural Resources, signed by the Regional Administrator on December 6, 1983.

(d) *Statement of legal authority.* (1) ''Attorney General's Statement,'' signed by Attorney General, State of Wisconsin;

(2) Letter from Assistant Attorney General, State of Wisconsin, to EPA Region, ''Re: Amendments to Attorney General's Statement-UIC,'' June 30, 1983.

(e) *Program Description.* The Program Description and other materials submitted as part of the application or as supplements thereto.

[49 FR 45309, Nov. 15, 1984, as amended at 56 FR 9420, Mar. 6, 1991; 56 FR 14150, Apr. 5, 1991; 62 FR 1834, Jan. 14, 1997]

§ 147.2510 EPA-administered program—Indian lands.

(a) *Contents.* The UIC program for Indian lands in the State of Wisconsin is administered by EPA. This program consists of 40 CFR parts 144 and 146 and additional requirements set forth in this section. Injection well owners and operators, and EPA, shall comply with these requirements.

(b) *Requirements.* Notwithstanding the requirements of paragraph (a) of this section for Indian lands in Wisconsin no owner or operator shall construct, operate, maintain, or convert any Class I, II, III, IV or V injection well.

(c) *Effective date.* The effective date of the UIC program requirements for Indian lands in Wisconsin is December 30, 1984.

[49 FR 45309, Nov. 15, 1984]

Subpart ZZ—Wyoming

§ 147.2550 State-administered program—Class I, III, IV and V wells.

The UIC program for Class I, III, IV and V wells in the State of Wyoming, except those on Indian lands is the program administered by the Wyoming Department of Environmental Quality approved by EPA pursuant to section 1422 of the SDWA. Notice of this approval was published in the FEDERAL REGISTER on July 15, 1983 (48 FR 32344); the effective date of this program is August 17, 1983. The program consists of the following elements as submitted to EPA in the State's program application:

(a) *Incorporation by reference.* The requirements set forth in the State statutes and regulations cited in this paragraph are hereby incorporated by reference and made a part of the applicable UIC program under the SDWA for

the State of Wyoming. This incorporation by reference was approved by the Director of the Federal Register on June 25, 1984.

(1) Wyoming Environmental Quality Act, Wyoming Statutes sections 35–11–101 through 35–11–115, and 35–11–301 through 35–11–305 (1977 Republished Edition and 1989 Cumm. Supp.);

(2) Water Quality Rules and Regulations, Wyoming Department of Environmental Quality, Chapter III: Regulations for Permit to Construct, Install or Modify Public Facilities Capable or, (sic) Causing or Contributing to Pollution (certified copy, signed December 21, 1983);

(3) Water Quality Rules and Regulations, Wyoming Department of Environmental Quality, Chapter VIII: Quality Standards for Groundwaters of Wyoming (certified copy, signed April 9, 1980);

(4) Water Quality Rules and Regulations, Wyoming Department of Environmental Quality, Chapter IX: Wyoming Groundwater Pollution Control Permit (certified copy, signed April 9, 1980);

(5) Water Quality Rules and Regulations, Wyoming Department of Environmental Quality, Chapter XIII: Prohibitions of Permits for New Hazardous Waste Injection Wells (certified copy, signed August 25, 1989);

(6) Land Quality Rules and Regulations, Wyoming Department of Environmental Quality, Chapter XXI: In Situ Mining (effective March 26, 1981).

(b) *Other laws.* The following statutes and regulations, although not incorporated by reference except for select sections identified in paragraph (a) of this section, are also part of the approved State-administered program:

(1) Article 9, Underground Water, Wyoming Statutes sections 41–3–901 through 41–3–938 (September 1982);

(2) Wyoming Administrative Procedure Act, Wyoming Statutes sections 9–4–101 through 9–4–115 (1988);

(3) Department of Environmental Quality Rules of Practice and Procedure (1982).

(c)(1) The Memorandum of Agreement between EPA, Region VIII and the Wyoming Department of Environmental Quality, signed by the EPA Regional Administrator on April 26, 1983.

(2) Letter from Regional Administrator, EPA Region VIII, to Governor of Wyoming, May 21, 1982, with Attachment (regarding aquifer exemptions);

(3) Letter from Governor of Wyoming to Regional Administrator, EPA Region VIII, ''Re: Underground Injection Control (UIC) Program—Aquifer Exemption Issues,'' June 7, 1982;

(4) Letter from Regional Administrator, EPA Region VIII to Governor of Wyoming, ''Re: Underground Injection Control (UIC) Program—Aquifer Exemption Issues,'' June 25, 1982;

(5) Letter from Director, Wyoming Department of Environmental Quality, to Acting Director, Water Management Division, EPA Region VIII, December 1, 1982.

(d) *Statement of legal authority.* (1) ''Attorney General's Statement—Wyoming Statutory and Regulatory Authority for Assumption of the Underground Injection Control Program Pursuant to the Federal Safe Drinking Water Act,'' signed by Attorney General and Assistant Attorney General for the State of Wyoming, September 22, 1982;

(2) Letter from Attorney General for the State of Wyoming to Acting Regional Counsel, EPA Region VIII, ''Re: Wyoming Assumption of the UIC Program—$36, Chapter IX, Wyoming Water Quality Rules and Regulations,'' November 24, 1982.

(e) The Program Description and any other materials submitted as part of the application or amendment thereto.

[49 FR 20197, May 11, 1984, as amended at 53 FR 43092, Oct. 25, 1988; 56 FR 9421, Mar. 6, 1991]

§ 147.2551 State-administered program—Class II wells.

The UIC program for Class II wells in the State of Wyoming, except those on Indian lands, is the program administered by the Wyoming Oil and Gas Conservation Commission approved by EPA pursuant to section 1425 of the SDWA. Notice of this approval was published in the FR on November 23, 1982 (47 FR 52434); the effective date of this program is December 23, 1982. This program consists of the following elements as submitted to EPA in the State's program application:

(a) *Incorporation by reference.* The requirements set forth in the State statutes and regulations cited in this paragraph are hereby incorporated by reference and made a part of the applicable UIC program under the SDWA for the State of Wyoming. This incorporation by reference was approved by the Director of the OFR in accordance with 5 U.S.C. 552(a) and 1 CFR Part 51. Copies may be obtained at the Wyoming Oil and Gas Conservation Commission, Office of the State Oil and Gas Supervisor, P.O. Box 2640, 77 West First Street, Casper, Wyoming, 82602. Copies may be inspected at the Environmental Protection Agency, Region VIII, 999 18th Street, Suite 500, Denver, Colorado, 80202–2405, or at the Office of the Federal Register, 800 North Capitol Street, NW., suite 700, Washington, DC.

(1) Rules and Regulations of the Wyoming Oil and Gas Conservation Commission, including Rules of Practice and Procedure, as published by the Wyoming Oil and Gas Conservation Commission, August 7, 1990;

(2) Title 30, Chapter 5, Wyoming Statutes, sections 30–5–101 through 30–5–126 (June 1983 and Wyoming Statutes Annotated, July 1990 Supp.).

(b) *Memorandum of Agreement.* (1) The initial Memorandum of Agreement between EPA, Region VIII and Wyoming Oil and Gas Conservation Commission, signed by the EPA Regional Administrator and the Oil Field Supervisor of the Commission on June 2, 1982;

(2) Amendment No. 1 to the Memorandum of Agreement, dated December 22, 1982;

(3) Amendment No. 2 to the Memorandum of Agreement, dated January 25, 1990;

(4) Letter from State Oil and Gas Supervisor, Wyoming Oil and Gas Conservation Commission, to the Acting Director, Water Management Division, EPA Region VIII, ''Re: Application for Primacy in the Regulation of Class II Injection Wells,'' March 8, 1982;

(5) Letter from State Oil and Gas Supervisor, Wyoming Oil and Gas Conservation Commission, to EPA Region VIII, ''Re: Regulation of Liquid Hydrocarbon Storage Wells Under the UIC Program,'' July 1, 1982;

(6) Memorandum of Agreement Between the Wyoming State Board of Control, State Engineer, Oil and Gas Conservation Commission, and the Department of Environmental Quality, dated October 14, 1981.

(c) *Statement of legal authority.* (1) ''Statement of Legal Authority'' and ''State Review of Regulations and Statutes Relevant to the UIC Program-Class II Wells,'' signed by Special Assistant Attorney General for the State of Wyoming, as submitted with ''Wyoming Oil and Gas Conservation Commission, Application for Primacy in the Regulation of Class II Injection Wells under Section 1425 of the Safe Drinking Water Act,'' November 1981;

(2) Letter from special Assistant Attorney General for the State of Wyoming to Assistant Regional Counsel, EPA Region VIII, May 13, 1982;

(3) Letter from special Assistant Attorney General for the State of Wyoming to Assistant Regional Counsel, EPA Region VIII, July 1, 1982.

(d) *Program Description.* The Program Description and other material submitted as part of the application or amendments thereto, including the memorandum to the National UIC Branch reporting on Improvement to the Wyoming Oil and Gas 1425 program, dated April 28, 1989.

[56 FR 9421, Mar. 6, 1991]

§ 147.2553 EPA-administered program—Indian lands.

(a) *Contents.* The UIC program for all classes of wells on Indian lands in the State of Wyoming is administered by EPA. This program consists of the UIC program requirements of 40 CFR parts 124, 144, 146, 148, and any additional requirements set forth in the remainder of this subpart. Injection well owners and operators, and EPA shall comply with these requirements.

(b) *Effective date.* The effective date for the UIC program on Indian lands in Wyoming is November 25, 1988.

[53 FR 43092, Oct. 25, 1988, as amended at 56 FR 9422, Mar. 6, 1991]

§ 147.2554 Aquifer exemptions.

In accordance with §§ 144.7(b) and 146.4 of this chapter, those portions of aquifers currently being used for injection in connection with Class II (oil and gas) injection operations on the

Wind River Reservation, which are described below, are hereby exempted for the purpose of Class II injection activity. This exemption applies only to the aquifers tabulated below, and includes those portions of the aquifers defined on the surface by an outer boundary of those quarter-quarter sections dissected by a line drawn parallel to, but one-quarter mile outside, the field boundary, and is restricted to extend no further than one-quarter mile outside the Reservation boundary. Maps showing the exact boundaries of the field may be consulted at the EPA's Region 8 Office, and at the EPA Headquarters in Washington, DC.

AREAS TO BE EXEMPTED FOR THE PURPOSE OF CLASS II INJECTION ON THE WIND RIVER RESERVATION

Formation	Approximate depth	Location
Steamboat Butte Field		
Phosphoria	6,500–7,100	T3N, R1W—W/2 Sec. 4, Sec. 5, E/2 Sec. 6, NE/4 Sec. 8, W/2 Sec. 9. T4N, R1W—W/2 Sec. 29, E/2 Sec. 30, E/2 Sec. 31, Sec. 32.
Tensleep	6,900–7,500	T3N, R1W—W/2 Sec. 4, Sec. 5, E/2 Sec. 6, NE/4 Sec. 8, W/2 Sec. 9. T4N, R1W—W/2 Sec. 29, E/2 Sec. 30, E/2 Sec. 31, Sec. 32.
Winkleman Dome Field		
Tensleep	2,800–3,300	T2N, R1W—SW/4 Sec. 17, Sections 18, 19, 20, 29, NE/4 Sec. 30. T2N, R2W—E/2 Sec. 13, NE/4 Sec. 24.
Phosphoria	2,800–3,600	T2N, R1W—SW/4 Sec. 17, Sections 18, 19, 20, 29, NE/4 Sec. 30. T2N, R2W—E/2 Sec. 13, NE/4 Sec. 24.
Nugget	1,100–1,500	T2N, R1W—SW/4 Sec. 17, Sections 18, 19, 20, 29, NE/4 Sec. 30. T2N, R2W—E/2 Sec. 13, NE/4 Sec. 24.
Lander Field		
Phosphoria	1,100–3,800	T2S, R1E—Sections 12 and 13, E/2 Sec. 24, NE/4 Sec. 25. T2S, R2E—W/2 Sec. 18, W/2 Sec. 19, Sec. 30. T33N, R99W—Sec. 4.
NW Sheldon Field		
Crow Mountain and Cloverly	3,400–3,600	T6N, R3W—SE/4 Sec. 35, SW/4 Sec. 36. T5N, R3W—N/2 Sec. 1.
Circle Ridge Field		
Tensleep	1,500–1,800	T6N, R2W—Sec. 6, N/2 Sec. 7. T7N, R3W—SE/4 Sec. 36. T7N, R2W—SW/4 Sec. 31. T6N, R3W—E/2 Sec. 1.
Phosphoria	800–1,800	T7N, R3W—S/2 Sec. 36. T6N, R3W—NE/4 Sec. 1.
Amsden	700–l,200	T6N, R3W—Sec. 6.
Rolff Lake Field		
Crow Mountain	3,500–3,700	T6N, R3W—SW/4 Sec. 26, NW/4 Sec. 27.

[53 FR 43092, Oct. 25, 1988]

Subpart AAA—Guam

§ 147.2600 State-administered program.

The UIC program for all classes of wells in the territory of Guam, except those on Indian lands, is the program administered by the Guam Environmental Protection Agency, approved by EPA pursuant to SDWA section 1422. Notice of this approval was published in the FEDERAL REGISTER on May 2, 1983 (47 FR 19717); the effective date of this program is June 1, 1983. This program consists of the following elements, as submitted to EPA in the State's program application:

(a) *Incorporation by reference.* The requirements set forth in the State statutes and regulations cited in this paragraph are hereby incorporated by reference and made a part of the applicable UIC program under the SDWA for

the territory of Guam. This incorporation by reference was approved by the Director of the Federal Register on June 25, 1984.

(1) Water Resources Conservation Act, Government Code of Guam sections 57021–57025, Public Law 9–31 (March 9, 1967), as amended by Public Law 9–76 (July 29, 1967), as amended by Public Law 12–191 (December 30, 1974);

(2) Water Pollution Control Act, Government Code of Guam sections 57042 and 57045, Public Law 9–76 (July 29, 1967), as amended by Public Law 9–212 (August 5, 1968), as amended by Public Law 10–31 (March 10, 1969), as amended by Public Law 12–191 (December 30, 1974);

(3) Guam Environmental Protection Agency, Underground Injection Control Regulations, Chapters 1–9, as revised by amendments adopted September 24, 1982;

(4) Guam Environmental Protection Agency, Water Quality Standards, Section I–IV (approved September 25, 1981, effective November 16, 1981).

(b) *Other laws.* The following statutes and regulations, although not incorporated by reference except for specific sections identified in paragraph (a) of this section, are also part of the approved State-administered program:

(1) Government Code of Guam, Title XXV, Chapters I–III (sections 24000–24207);

(2) Government Code of Guam, Title LXI, Chapters I–III (sections 57000–57051);

(3) Government Code of Guam, Title LXI, Chapters VI (sections 57120–57142);

(4) Government Code of Guam, Title LXI, Chapters VIII (sections 57170–57188);

(5) Government Code of Guam, Title LXI, Chapters XII (sections 57285–57299);

(c) The Memorandum of Agreement between EPA, Region IX and the Guam Environmental Protection Agency signed by the Regional Administrator on January 14, 1983.

(d) *Statement of legal authority.* (1) Letter from Attorney General of Guam to Regional Administrator, Region IX, ''Re: Attorney General's Statement for Underground Injection Control Program (UIC), Ground Water Program Guidance #16'' May 12, 1982;

(2) Letter from Attorney General of Guam to Regional Administrator, Region IX, ''Re: Additional comments to be incorporated into the May 12, 1982, Attorney General's Statement for Underground Injection Control Program,'' September 2, 1982.

(e) The Program Description and any other materials submitted as part of the application or amendments thereto.

[49 FR 20197, May 11, 1984, as amended at 53 FR 43092, Oct. 25, 1988]

§ 147.2601 EPA-administered program—Indian lands.

(a) *Contents.* The UIC program for Indian lands in the territory of Guam is administered by EPA. This program consists of the UIC program requirements of 40 CFR parts 124, 144, 146, 148, and any additional requirements set forth in the remainder of this subpart. Injection well owners and operators, and EPA shall comply with these requirements.

(b) *Effective date.* The effective date for the UIC program on Indian lands in the territory of Guam is November 25, 1988.

[53 FR 43093, Oct. 25, 1988, as amended at 56 FR 9422, Mar. 6, 1991]

Subpart BBB—Puerto Rico

§ 147.2650 State-administered program—Class I, II, III, IV, and V wells.

The Underground Injection Control Program for all classes of wells in the Commonwealth of Puerto Rico, other than those on Indian lands, is the program administered by Puerto Rico's Environmental Quality Board (EQB), approved by the EPA pursuant to the Safe Drinking Water Act (SDWA) section 1422. This program consists of the following elements, as submitted to EPA in the Commonwealth's program application.

(a) *Incorporation by reference.* The requirements set forth in the State statutes and regulations cited in this paragraph are hereby incorporated by reference and made a part of the applicable UIC program under the SDWA for the Commonwealth of Puerto Rico.

This incorporation by reference was approved by the Director of the Federal Register in accordance with 5 U.S.C. 552(a) and 1 CFR part 51. Copies may be obtained or inspected at the following locations: EPA, Region II, 26 Federal Plaza, room 845, New York, NY 10278; EPA, Headquarters, 401 M Street, SW., room E1101A, Washington, DC 20460; or the Office of the Federal Register, 800 North Capitol Street NW., suite 700, Washington, DC.

(1) Underground Injection Control Regulations of the Commonwealth of Puerto Rico, Parts I through V and appendices A and B, adopted September 14, 1983 (Amended July 20, 1988).

(2) Puerto Rico Public Policy Environmental Act (PRPPE), Title 12 Laws of Puerto Rico Annotated (LPRA) Chapters 121 and 131, 1977 edition, as amended 1988 edition, and Chapter 122, 1988 edition.

(b) Memorandum of Agreement. The Memorandum of Agreement between EPA Region II and the Commonwealth of Puerto Rico's EQB signed by the Regional Administrator on August 23, 1991.

(c) Statement of legal authority. (1) Attorney General's statement on the Commonwealth of Puerto Rico's Authority to apply for, assume and carry out the UIC Program, dated June 26, 1987. (2) Letter from the Governor of the Commonwealth of Puerto Rico requesting the program, dated July 16, 1987.

(d) Program description. The Description of the Commonwealth of Puerto Rico's Underground Injection Control Program, dated with the effective date October 30, 1986.

[57 FR 33446, July 29, 1992]

§147.2651 EPA-administered program— Indian lands.

(a) Contents. The UIC program for all classes of wells on Indian lands in the Commonwealth of Puerto Rico is administered by EPA. This program consists of the UIC program requirements of 40 CFR parts 124, 144, 146, 148 and any additional requirements set forth in the remainder of this subpart. Injection well owners and operators and EPA shall comply with the requirements.

(b) Effective date. The effective date for the UIC program on Indian Lands in the Commonwealth of Puerto Rico is November 25, 1988.

[57 FR 33446, July 29, 1992]

Subpart CCC—Virgin Islands

§147.2700 State-administered program. [Reserved]

§147.2701 EPA-administered program.

(a) *Contents.* The UIC program for the Virgin Islands, including all Indian lands, is administered by EPA. This program consists of the UIC program requirements of 40 CFR parts 124, 144, 146, 148, and any additional requirements set forth in the remainder of this subpart. Injection well owners and operators, and EPA shall comply with these requirements.

(b) *Effective dates.* The effective date of the UIC program for non-Indian lands in the Virgin Islands is December 30, 1984. The effective date for Indian lands in the Virgin Islands is November 25, 1988.

[53 FR 43093, Oct. 25, 1988, as amended at 56 FR 9422, Mar. 6, 1991]

Subpart DDD—American Samoa

§147.2750 State-administered program. [Reserved]

§147.2751 EPA-administered program.

(a) *Contents.* The UIC program for American Samoa, including all Indian lands, is administered by EPA. This program consists of the UIC program requirements of 40 CFR parts 124, 144, 146, 148, and any additional requirements set forth in the remainder of this subpart. Injection well owners and operators, and EPA shall comply with these requirements.

(b) *Effective dates.* The effective date for the UIC program on non-Indian lands is June 25, 1984. The effective date of the UIC program on Indian lands is November 25, 1988.

[53 FR 43093, Oct. 25, 1988, as amended at 56 FR 9422, Mar. 6, 1991]

§ 147.2752 Aquifer exemptions. [Reserved]

Subpart EEE—Commonwealth of the Northern Mariana Islands

§ 147.2800 State-administered program—Class I, II, III, IV, and V wells.

The UIC program for Class I, II, III, IV, and V wells in the Commonwealth of the Northern Mariana Islands, other than those on Indian lands, is the program administered by the Commonwealth of the Northern Mariana Islands Division of Environmental Quality approved by EPA pursuant to Section 1422 of the SDWA. Notice of this approval was published in the FEDERAL REGISTER on January 18, 1985; the effective date of this program is August 30, 1985. This program consists of the following elements, as submitted to EPA in the State's program application.

(a) *Incorporation by reference.* The requirements set forth in the State statutes and regulations cited in this paragraph are hereby incorporated by reference and made a part of the applicable UIC program under the SDWA for the Commonwealth of the Northern Mariana Islands. This incorporation by reference was approved by the Director of the Federal Register effective July 31, 1985.

(1) CNMI Environmental Protection Act, 2 CMC sections 3101, *et seq.* (1984);

(2) CNMI Coastal Resources Management Act, 2 CMC sections 1501, *et seq.* (1984);

(3) CNMI Drinking Water Regulations, Commonwealth Register, Volume 4, Number 4 (August 15, 1982);

(4) CNMI Underground Injection Control Regulations, Commonwealth Register, Volume 6, Number 5 (May 15, 1984, amended November 15, 1984, January 15, 1985);

(5) CNMI Coastal Resources Management Regulations, Commonwealth Register, Volume 6, Number 12, December 17, 1984.

(b)(1) The Memorandum of Agreement between EPA Region IX and the Commonwealth of the Northern Mariana Islands Division of Environmental Quality, signed by the EPA Regional Administrator on May 3, 1985;

(c) *Statement of legal authority.* Statement from Attorney General Commonwealth of the Northern Mariana Islands, ''Underground Injection Control Program—Attorney General's Statement,'' signed on October 10, 1984.

(d) The Program Description and any other materials submitted as part of the original application or as supplements thereto.

[50 FR 28943, July 17, 1985]

§ 147.2801 EPA-administered program.

(a) *Contents.* The UIC program for Indian lands in the Commonwealth of the Northern Mariana Islands is administered by EPA. This program consists of the UIC program requirements of 40 CFR parts 124, 144, 146, 148, and any additional requirements set forth in the remainder of this subpart. Injection well owners and operators, and EPA shall comply with these requirements.

(b) *Effective date.* The effective date of the UIC program for Indian lands is November 25, 1988.

[53 FR 43093, Oct. 25, 1988, as amended at 56 FR 9422, Mar. 6, 1991]

§ 147.2802 Aquifer exemptions. [Reserved]

Subpart FFF—Trust Territory of the Pacific Islands

§ 147.2850 State-administered program. [Reserved]

§ 147.2851 EPA-administered program.

(a) *Contents.* The UIC program for Trust Territory of the Pacific Islands, including all Indian lands, is administered by EPA. This program consists of the UIC program requirements of 40 CFR parts 124, 144, 146, 148, and any additional requirements set forth in the remainder of this subpart. Injection well owners and operators, and EPA shall comply with these requirements.

(b) *Effective dates.* The effective date of the UIC program for non-Indian lands of the Trust Territory of the Pacific Islands is June 25, 1984. The effective date for the Indian lands is November 25, 1988.

[53 FR 43093, Oct. 25, 1988, as amended at 56 FR 9422, Mar. 6, 1991]

§147.2852 Aquifer exemptions. [Reserved]

Subpart GGG—Osage Mineral Reserve—Class II Wells

AUTHORITY: Safe Drinking Water Act, 42 U.S.C. 300h.

SOURCE: 49 FR 45309, Nov. 15, 1984, unless otherwise noted.

§147.2901 Applicability and scope.

This subpart sets forth the rules and permitting requirements for the Osage Mineral Reserve, Osage County, Oklahoma, Underground Injection Control Program. The regulations apply to owners and operators of Class II injection wells located on the Reserve, and to EPA.

§147.2902 Definitions.

Most of the following terms are defined in §144.3, and have simply been reproduced here for the convenience of the reader. This section also includes definitions of some terms unique to the Osage program. Terms used in this subpart are defined as follows:

Administrator—the Administrator of the United States Environmental Protection Agency, or an authorized representative.

Aquifer—a geologic formation, group of formations, or part of a formation that is capable of yielding a significant amount of water to a well or spring.

BIA—The ''Bureau of Indian Affairs,'' United States Department of Interior.

Casing—a pipe or tubing of varying diameter and weight, lowered into a borehole during or after drilling in order to support the sides of the hole and, thus, prevent the walls from caving, to prevent loss of drilling mud into porous ground, or to prevent water, gas, or other fluid from entering the hole.

Cementing—the operation whereby a cement slurry is pumped into a drilled hole and/or forced behind the casing.

Class II Wells—wells which inject fluids:

(a) Which are brought to the surface in connection with conventional oil or natural gas production and may be commingled with waste waters from gas plants which are an integral part of production operations, unless those waters would be classified as a hazardous waste at the time of injection;

(b) For enhanced recovery of oil or natural gas; and

(c) For storage of hydrocarbons which are liquid at standard temperature and pressure.

Existing Class II Wells—wells that were authorized by BIA and constructed and completed before the effective date of this program.

New Class II Wells—wells constructed or converted after the effective date of this program, or which are under construction on the effective date of this program.

Confining bed—a body of impermeable or distinctly less permeable material stratigraphically adjacent to one or more aquifers.

Confining zone—a geologic formation, group of formations, or part of a formation that is capable of limiting fluid movement above an injection zone.

Contaminant—any physical, chemical, biological, or radiological substance or matter in water.

Disposal well—a well used for the disposal of waste into a subsurface stratum.

EPA—The United States Environmental Protection Agency.

Fault—a surface or zone of rock fracture along which there has been displacement.

Fluid—material or substance which moves or flows whether in a semisolid, liquid, sludge, gas or any other form or state.

Formation—a body of rock characterized by a degree of lithologic homogeneity which is prevailingly, but not necessarily, tabular and is mappable on the earth's surface or traceable in the subsurface.

Freshwater—''Underground source of drinking water.''

Ground water—water below the land surface in a zone of saturation.

Injection well—a well into which fluids are being injected.

Injection zone—a geological formation, group of formations, or part of a formation receiving fluids through a well.

Lithology—the description of rocks on the basis of their physical and chemical characteristics.

Owner/operator—the owner or operator of any facility or activity subject to regulation under the Osage UIC program.

Packer—a device lowered into a well to produce a fluid-tight seal within the casing.

Permit—an authorization issued by EPA to implement UIC program requirements. Permit does not include the UIC authorization by rule or any permit which has not yet been the subject of final Agency action.

Plugging—the act or process of stopping the flow of water, oil or gas into or out of a formation through a borehole or well penetrating that formation.

Pressure—the total load or force per unit area acting on a surface.

Regional Administrator—the Regional Administrator of Region 6 of the United States Environmental Protection Agency, or an authorized representative.

Subsidence—the lowering of the natural land surface in response to: Earth movements; lowering of fluid pressure; removal of underlying supporting material by mining or solution solids, either artificially or from natural causes; compaction due to wetting (hydrocompaction); oxidation of organic matter in soils; or added load on the land surface.

Underground source of drinking water—an aquifer or its portion:

(a)(1) Which supplies any public water system; or

(2) Which contains a sufficient quantity of ground water to supply a public water system; and

(i) Currently supplies drinking water for human consumption; or

(ii) Contains fewer than 10,000 mg/1 total dissolved solids; and

(b) Which is not an exempted aquifer.

USDW—underground source of drinking water.

Well—a bored, drilled, or driven shaft, or a dug hole whose depth is greater than the largest surface dimension.

Well injection—the subsurfac emplacement of fluids through a bored, drilled, or driven well; or through a dug well, where the depth of the dug well is greater than the largest surface dimension.

Well workover—any reentry of an injection well; including, but not limited to, the pulling of tubular goods, cementing or casing repairs; and excluding any routine maintenance (e.g. reseating the packer at the same depth, or repairs to surface equipment).

§ 147.2903 Prohibition of unauthorized injection.

(a) Any underground injection, except as authorized by permit or rule issued under the UIC program, is prohibited. The construction or operation of any well required to have a permit is prohibited until the permit has been issued.

(b) No owner or operator shall construct, operate, maintain, convert, plug, or abandon any injection well, or conduct any other injection activity, in a manner that allows the movement of fluid containing any contaminant into underground sources of drinking water, if the presence of that contaminant may cause the violation of any primary drinking water regulation under 40 CFR part 142 or may otherwise adversely affect the health of persons. The applicant for a permit shall have the burden of showing that the requirements of this paragraph are met.

(c) Injection between the outermost casing protecting underground sources of drinking water and the well bore is prohibited.

§ 147.2904 Area of review.

(a) The area of review for an injection well or project will be a fixed radius of one-forth of a mile from the well, field or project.

(b) The zone of endangering influence is the lateral area around the injection well or project in which the injection zone pressures may cause movement of fluid into an underground source of drinking water (USDW) if there are improperly sealed, completed or abandoned wells present. A zone of endangering influence may be determined by EPA through the use of an appropriate formula that addresses the relevant geologic, hydrologic, engineering and operational features of the well, field, or project.

§147.2905 Plugging and abandonment.

The owner/operator shall notify the Osage UIC office within 30 days of the date injection has terminated. The well must be plugged within 1 year after termination of injection. The Regional Administrator may extend the time to plug, but only if no fluid movement into a USDW will occur, and the operator has presented a viable plan for utilizing the well within a reasonable time.

(a) Until an injection well has been properly plugged and abandoned, annual reports to the Regional Administrator on well status, and mechanical integrity tests as outlined in §§147.2912 and 147.2920 will be required, whether or not injection has ceased.

(b) All wells shall be plugged to prevent movement of fluid into an USDW.

(c) The owner/operator shall notify the Osage UIC office by certified mail at least 5 days prior to the commencement of plugging operations. The Osage UIC office may waive or reduce the 5-day notice requirement when a qualified EPA representative is available to witness the plugging operation. The following information must be submitted as part of the notification:

(1) Type and number of plugs to be used;

(2) Elevation of top and bottom of each plug;

(3) Method of plug placement; and

(4) Type, grade and quantity of cement to be used.

(d) The well shall be kept full of mud as casing is removed. No surface casing shall be removed without written approval from the Regional Administrator.

(e)(1) If surface casing is adequately set and cemented through all freshwater zones (set to at least 50 feet below the base of freshwater), a plug shall be set at least 50 feet below the shoe of the casing and extending at least 50 feet above the shoe of the casing, or

(2) If the surface casing and cementing is inadequate, the well bore shall be filled with cement from a point 50 feet below the base of fresh water to a point 50 feet above the shoe of the surface casing, and any additional plugs as required by the Osage UIC office and/or the Osage Agency.

(3) In all cases, the top 20 feet of the well bore below 3 feet of ground surface shall be filled with cement. Surface casing shall be cut off 3 feet below ground surface and covered with a secure steel cap on top of the surface pipe. The remaining 3 feet shall be filled with dirt.

(f)(1) Except as provided in paragraph (f)(2) of this section, each producing or receiving formation shall be sealed off with a 50-foot cement plug placed at the base of the formation and a 50–foot cement plug placed at the top of the formation.

(2) The requirement in paragraph (f)(1) of this section does not apply if the producing/receiving formation is already sealed off from the well bore with adequate casing and cementing behind casing, and casing is not to be removed, or the only openings from the producing/receiving formation into the well bore are perforations in the casing, and the annulus between the casing and the outer walls of the well is filled with cement for a distance of 50 feet below the base of the formation and 50 feet above the top of the formation. When such conditions exist, a bridge plug capped with 10 feet of cement set at the top of the producing formation may be used.

(g) When specified by the Osage UIC office, any uncased hole below the shoe of any casing to be left in the well shall be filled with cement to a depth of at least 50 feet below the casing shoe, or the bottom of the hole, and the casing above the shoe shall be filled with cement to at least 50 feet above the shoe of the casing. If the well has a screen or liner which is not to be removed, the well bore shall be filled with cement from the base of the screen or liner to at least 50 feet above the top of the screen or liner.

(h) All intervals between cement plugs in the well bore shall be filled with mud.

(i) A report containing copies of the cementing tickets shall be submitted to BIA within 10 days of plugging completion.

(j) A surety bond must be on file with the Bureau of Indian Affairs (BIA), and shall not be released until the well has been properly plugged and the Regional

Administrator has agreed to the release of the bond.

§ 147.2906 Emergency permits.

(a) An emergency permit may be issued if:

(1) There will be an imminent health hazard unless an emergency permit is issued; or

(2) There will be a substantial and irretrievable loss of oil and gas resources, timely application for a permit could not practicably have been made, and injection will not result in movement of fluid into an USDW; or

(3) There will be a substantial delay in oil or gas production, and injection will not result in movement of fluid into an USDW.

(b) *Requirements*—(1) *Permit duration.* (i) Emergency permits issued to avoid an imminent health threat may last no longer than the time necessary to prevent the hazard.

(ii) Emergency permits issued to prevent a substantial and irretrievable loss of oil or gas resources shall be for no longer than 90 days, unless a complete permit application has been submitted during that time; in which case the emergency permit may be extended until a final decision on the permit application has been made.

(iii) Emergency permits to avoid a substantial delay in oil or gas production shall be issued only after a complete permit application has been submitted and shall be effective until a final decision on the permit application is made.

(2) Notice of the emergency permit will be given by the Regional Administrator according to the notice procedure for a draft permit within 10 days after issuance.

(3) An emergency permit may be oral or written. If oral, a written emergency permit must be issued within five calendar days.

§ 147.2907 Confidentiality of information.

(a) The following information cannot be claimed confidential by the submitter:

(1) Name and address of permit applicant or permittee.

(2) Information concerning the existence, absence or level of contaminants in drinking water.

(b) Other information claimed as confidential will be processed in accordance with 40 CFR part 2.

§ 147.2908 Aquifer exemptions.

(a) After notice and opportunity for a public hearing, the Administrator may designate any aquifer or part of an aquifer as an exempted aquifer.

(b) An aquifer or its portion that meets the definition of a USDW may be exempted by EPA from USDW status if the following conditions are met:

(1) It does not currently serve as a source of drinking water, and

(2) It cannot now and will not in the future serve as a source of drinking water because:

(i) It is hydrocarbon producing, or can be demonstrated by a permit applicant as a part of a permit application for a Class II operation to contain hydrocarbons that are expected to be commercially producible (based on historical production or geologic information); or

(ii) It is situated at a depth or location which makes recovery of water for drinking water purposes economically or technologically impractical; or

(iii) It is so contaminated that it would be economically or technologically impractical to render that water fit for human consumption; or

(3) The Total Dissolved Solids content of the groundwater is more than 3,000 and less than 10,000 mg/1 and it is not reasonably expected to supply a public water system.

§ 147.2909 Authorization of existing wells by rule.

All existing Class II injection wells (wells authorized by BIA and constructed or completed on or before the effective date of the Osage UIC program) are hereby authorized. Owners or operators of wells authorized by rule must comply with the provisions of §§ 147.2903, 147.2905, 147.2907, and 147.2910 through 147.2915.

§147.2910 Duration of authorization by rule.

Existing Class II injuction wells are authorized for the life of the well, subject to the obligation to obtain a permit if specifically required by the Regional Administrator pursuant to §147.2915.

§147.2911 Construction requirements for wells authorized by rule.

All Class II wells shall be cased and cemented to prevent movement of fluids into USDWs. The Regional Administrator shall review inventory information, data submitted in permit applications, and other records, to determine the adequacy of construction (completion) or existing injection wells. At the Regional Administrator's discretion, well casing and cementing may be considered adequate if it meets the BIA requirements that were in effect at the time of construction (completion) and will not result in movement of fluid into an USDW. If the Regional Administrator determines that the construction of a well authorized by rule is inadequate, he shall require a permit, or he shall notify the owner/operator and the owner/operator shall correct the problem according to instructions from the Regional Administrator. All corrections must be completed within one year of owner/operator notification of inadequacies.

§147.2912 Operating requirements for wells authorized by rule.

(a) Each well authorized by rule must have mechanical integrity. Mechanical integrity must be demonstrated within five years of program adoption. The Regional Administrator will notify the well owner/operator three months before proof of mechanical integrity must be submitted to EPA. The owner/operator must contact the Osage UIC office at least five days prior to testing. The owner/operator may perform the mechanical integrity test prior to receiving notice from the Regional Administrator, provided the Osage UIC office is notified at least five days in advance. Conditions of both paragraphs (a)(1) and (a)(2) of this section must be met.

(1) There is no significant leak in the casing, tubing or packer. This may be shown by the following:

(i) Performance of a pressure test of the casing/tubing annulus to at least 200 psi, or the pressure specified by the Regional Administrator, to be repeated thereafter, at five year intervals, for the life of the well (pressure tests conducted during well operation shall maintain an injection/annulus pressure differential of at least 100 psi through the tubing length); or

(ii) Maintaining a positive gauge pressure on the casing/tubing annulus (filled with liquid) and monitoring the pressure monthly and reporting of the pressure information annually; or

(iii) Radioactive tracer survey; or

(iv) For enhanced recovery wells, records of monitoring showing the absence of significant changes in the relationship between injection pressure and injection flow rate at the well head, following an initial pressure test as described by paragraph (a)(1)(i) or (v) of this section; or

(v) Testing or monitoring programs approved by the Regional Administrator on a case-by-case basis, and

(2) There is no significant fluid movement into a USDW through vertical channels adjacent to the well bore. This may be shown by any of the following:

(i) Cementing records (need not be reviewed every five years);

(ii) Tracer survey (in appropriate hydrogeologic settings; must be used in conjunction with at least one of the other alternatives);

(iii) Temperature log;

(iv) Noise log; or

(v) Other tests deemed acceptable by the Regional Administrator.

(b) Injection pressure at the wellhead shall be limited so that it does not initiate new fractures or propagate existing fractures in the confining zone adjacent to any UDSW.

(1) For existing Class II salt water disposal wells, The owner/operator shall, except during well stimulation, use an injection pressure at the wellhead no greater than the pressure calculated by using the following formula:

$$Pm=(0.75-0.433Sg)d$$

where:

Pm=injection pressure at the wellhead in pounds per square inch
Sg=specific gravity of injected fluid (unitless)
d=injection depth in feet.

Owner/operator of wells shall comply with the above injection pressure limits no later than one year after the effective date of this regulation.

(2) For existing Class II enhanced recovery wells, the owner or operator:

(i) Shall use an injection pressure no greater than the pressure established by the Regional Administrator for the field or formation in which the well is located. The Regional Administrator shall establish such a maximum pressure after notice, opportunity for comment, and opportunity for a public hearing according to the provisions of part 124, subpart A of this chapter, and will inform owners and operators in writing of the applicable maximum pressure.

(ii) Prior to such time as the Regional Administrator establishes rules for maximum injection pressures based on data provided pursuant to paragraph (b)(2)(ii)(B) of this section the owner/operator shall:

(A) Limit injection pressure at the wellhead to a value which will not initiate new fractures or propagate existing fractures in the confining zone adjacent to any USDW; and

(B) Submit data acceptable to the Regional Administrator which defines the fracture pressure of the formation in which injection is taking place. A single test may be submitted on behalf of two or more operators conducting operations in the same formation, if the Regional Administrator approves such submission. The data shall be submitted to the Regional Administrator within one year of the effective date of this program.

(c) Injection wells or projects which have exhibited failure to confine injected fluids to the authorized injection zone or zones may be subject to restriction of injection volume and pressure, or shut-down, until the failure has been identified and corrected.

(The information collection requirements contained in paragraphs (a)(1) (ii) through (v) and (a)(2) (i) through (v) were approved by the Office of Management and Budget under control number 2040–0042)

§ 147.2913 Monitoring and reporting requirements for wells authorized by rule.

(a) The owner/operator has the duty to submit inventory information to the Regional Administrator upon request. Such request may be a general request to all operators in the County (e.g., public notice, or mailout requesting verification of information).

(b) The operator shall monitor the injection pressure (psi) and rate (bb1/day) at least monthly, with the results reported annually. The annual report shall specify the types of methods used to generate the monitoring data.

(c) The owner/operator shall notify the Osage UIC office within 30 days of any mechanical failure or down-hole problems involving well integrity, well workovers, or any noncompliance. As required, operators must apply for and obtain a workover permit from the Bureau of Indian Affairs Osage Agency before reentering an injection well. If the condition may endanger an USDW, the owner/operator shall notify the Osage UIC office orally within 24 hours, with written notice including plans for testing and/or repair to be submitted within five days. If all the information is not available within five days, a followup report must be submitted within 30 days.

(d) The owner/operator shall determine the nature of injected fluids initially, when the nature of injected fluids is changed or when new constituents are added. The records should reflect the source of character of the new fluid and the date changes were made.

(e) The owner/operator shall retain all monitoring records for three years, unless an enforcement action is pending, and then until three years after the enforcement action has been resolved.

(Approved by the Office of Management and Budget under control number 2040–0042)

§ 147.2914 Corrective action for wells authorized by rule.

Based on the Regional Administrator's discretion, corrective action to prevent movement of fluid into an USDW may be required for improperly sealed, completed or abandoned wells (i.e., wells or well bores which may provide and avenue for fluid migration

into a USDW) within the zone of endangering influence (as defined in §147.2904, Area of Review) of an injection well authorized by rule.

(a) EPA will notify the operator when corrective action is required. Corrective action may include:

(1) Well modifications:

(i) Recementing;

(ii) Workover;

(iii) Reconditioning;

(iv) Plugging or replugging;

(2) Limitations on injection pressure to prevent movement of fluid into an USDW;

(3) A more stringent monitoring program; and/or

(4) Periodic testing of other wells to determine if significant movement of fluid has occurred.

(b) If the monitoring discussed in paragraph (a) (3) or (4) of this section indicate the potential endangerment of an USDW, then action as described in paragraph (a) (1) or (2) of this section must be taken.

§147.2915 Requiring a permit for wells authorized by rule.

(a) The Regional Administrator may require the owner or operator of any well authorized by rule to apply for an individual or area permit. The Regional Administrator shall notify the owner/operator in writing that a permit application is required. The notice shall contain:

(1) Explanation of need for application;

(2) Application form and, if appropriate, a list of additional information to be submitted; and

(3) Deadline for application submission.

(b) Cases in which the Regional Administrator may require a permit include:

(1) The owner or operator is not in compliance with provisions of the rule;

(2) Injection well is no longer within the category of wells authorized by rule;

(3) Protection of USDWs requires that the injection operation be regulated by requirements which are not contained in the rule; or

(4) Discretion of Regional Administrator.

(c) Injection is no longer authorized by rule upon the effective date of a permit or permit denial, or upon failure of the owner/operator to submit an application in a timely manner as specified in the notice described in paragraph (a) of this section.

(d) Any owner/operator authorized by rule may request to be excluded from the coverage of the rules by applying for an individual or area UIC permit.

§147.2916 Coverage of permitting requirements.

The owner or operator of a new Class II injection well or any other Class II well required to have a permit in the Osage Mineral Reserve shall comply with the requirements of §§147.2903, 147.2907, 147.2918, through 147.2928.

§147.2917 Duration of permits.

Unless otherwise specified in the permit, the permits will be in effect until the well is plugged and abandoned or the permit terminated. The Regional Administrator will review each issued permit at least once every five years to determine whether it should be modified or terminated.

§147.2918 Permit application information.

(a) The owner/operator must submit the original and three copies of the permit application, with two complete sets of attachments, to the Osage UIC office. The application should be signed by the owner/operator or a duly authorized representative. The application should also include appropriate forms (i.e., BIA's Application for Operation or Report on Wells and EPA's permit application). The applicant has the burden of proof to show that the proposed injection activities will not endanger USDWs.

(b) The application shall include the information listed below. Information required by paragraphs (b) (5), (7), or (9) of this section that is contained in EPA or BIA files may be included in the application by reference.

(1) Map using township-range sections showing the area of review and identifying all wells of public record penetrating the injection interval.

(2) Tabulation of data on the wells identified in paragraph (b)(1) of this

section, including location, depth, date drilled, and record of plugging and/or completion.

(3) Operating data:

(i) Maximum and average injection rate;

(ii) Maximum and average injection pressure;

(iii) Whether operation is on cyclic or continuous operation basis; and

(iv) Source and appropriate analysis of injected fluids, including total dissolved solids, chlorides, and additives.

(4) Geologic data on the injection and confining zones, including faults, geological name, thickness permeability, depth and lithologic description.

(5) Depth to base of fresh water.

(6) Schematic drawings of the surface and subsurface details of the well, showing:

(i) Total depth or plug-back depth;

(ii) Depth to top and bottom of injection interval;

(iii) Depths to tops and bottoms of casing and cemented intervals, and amount of cement to be used;

(iv) Size of casing and tubing, and depth of packer; and

(v) Hole diameter.

(7) Proof that surety bond has been filed with the BIA Superintendent in accordance with 25 CFR 226.6. A surety bond must be maintained until the well has been properly plugged.

(8) Verification of public notice, consisting of a list showing the names, addresses, and date that notice of permit application was given or sent to:

(i) The surface land owner;

(ii) Tenants on land where injection well is located or proposed to be located; and

(iii) Each operator of a producing lease within one-half mile of the well location.

(9) All available logging and testing data on the well (for existing wells, i.e., wells to be converted or wells previously authorized by rule).

(Approved by the Office of Management and Budget under control number 2040–0042)

§ 147.2919 Construction requirements for wells authorized by permit.

(a) All Class II wells shall be sited so that they inject into a formation that is separated from any USDW by a confining zone free of known open faults or fractures within the area of review.

(b) All Class II wells shall be cased and cemented to prevent movement of fluids into or between USDWs. Requirements shall be based on the depth to base of fresh water, and the depth to the injection zone. Newly drilled Class II wells must have surface casing set and cemented to at least 50 feet below the base of fresh water, or the equivalent (e.g., long string cemented to surface). At the Regional Administrator's discretion, the casing and cementing of wells to be converted may be considered adequate if they meet the BIA requirements that were in effect at the time of construction (completion), and will not result in movement of fluid into a USDW.

(c) Owner/operators shall provide a standard female fitting with cut-off valves, connected to the tubing and the tubing/casing annulus so that the injection pressure and annulus pressure may be measured by an EPA representative by attaching a gauge having a standard male fitting.

(d) No owner or operator may begin construction of a new well until a permit authorizing such construction has been issued, unless such construction is otherwise authorized by an area permit.

§ 147.2920 Operating requirements for wells authorized by permit.

(a) For new Class II wells, injection shall be through adequate tubing and packer. Packer shall be run on the tubing and set inside the casing within 75 feet of the top of the injection interval. For existing Class II, wells, injection shall be through adequate tubing and packer, or according to alternative operating requirements approved by the Regional Administrator, as necessary to prevent the movement of fluid into a USDW.

(b) Each well must have mechanical integrity. Mechanical integrity of the injection well must be shown prior to operation. The owner/operator must notify the Osage UIC office at least five days prior to mechanical integrity testing. Conditions of both paragraphs (b) (1) and (2) of this section must be met.

(1) There is no significant leak in the casing, tubing or packer. This may be shown by the following:

(i) Performance of a pressure test of the casing/tubing annulus to at least 200 psi, or the pressure specified by the Regional Administrator, to be repeated thereafter, at five year intervals, for the life of the well (Pressure tests conducted during well operation shall maintain an injection/annulus pressure differential of at least 100 psi throughout the tubing length); or

(ii) Maintaining a positive gauge pressure on the casing/tubing annulus (filled with liquid) and monitoring the pressure monthly and reporting of the pressure information annually; or

(iii) Radioactive tracer survey; or

(iv) For enhanced recovery wells, record of monitoring showing the absence of significant changes in the relationship between injection pressure and injection flow rate at the wellhead, following an initial pressure test as described by paragraph (b)(1) (i) or (v) of this section; or

(v) Testing or monitoring programs approved by the Administrator on a case-by-case basis, and

(2) There is no significant fluid movement into a USDW through vertical channels adjacent to the well bore. This may be shown by any of the following:

(i) Cementing records (need not be reviewed every five years);

(ii) Tracer survey (in appropriate hydrogelogic settings; must be used in conjunction with at least one of the other alternatives);

(iii) Temperature log;

(iv) Noise log; or

(v) Other tests deemed acceptable by the Administrator.

(c) Injection pressure at the wellhead shall be limited so that it does not initiate new fractures or propagate existing fractures in the confining zone adjacent to any UDSW.

(d) Injection wells or projects which have exhibited failure to confine injected fluids to the authorized injection zone or zones may be subject to restriction of injected volume and pressure or shut-in, until the failure has been identified and corrected.

(e) Operation shall not commence until proof has been submitted to the Regional Administrator, or an EPA representative has witnessed that any corrective action specified in the permit has been completed.

§147.2921 Schedule of compliance.

The permit may, when appropriate, specify a schedule of compliance leading to compliance with the Safe Drinking Water Act and the Osage UIC regulations.

(a) Any schedule of compliance shall require compliance as soon as possible, and in no case later than three years after the effective date of the permit.

(b) If a permit establishes a schedule of compliance which exceeds one year from the date of permit issuance, the schedule shall set forth interim requirements and the dates for their achievement.

(1) The time between interim dates shall not exceed one year.

(2) If the time necessary for completion of any interim requirement is more than 1 year and is not readily divisible into stages for completion, the permit shall specify interim dates for the submission of reports of progress toward completion of the interim requirements and indicate a projected completion date.

(c) The permit shall be written to require that if a schedule of compliance is applicable, progress reports be submitted no later than 30 days following each interim date and the final date of compliance.

§147.2922 Monitoring and reporting requirements for wells authorized by permit.

(a) The owner/operator shall notify the Osage UIC office within 30 days of the date on which injection commenced.

(b) The operator shall monitor the injection pressure (psi) and rate (bbl/day) at least monthly, with the results reported annually. The annual reports shall specify the types or methods used to generate the monitoring data.

(c) The owner/operator shall notify the Osage UIC office within 30 days of any mechanical failure or down-hole problems involving well integrity, well workovers, or any noncompliance. (Operators should note the obligation to apply for and obtain a workover permit

from the Bureau of Indian Affairs Osage Agency before reentering an injection well.) If the condition may endanger an USDW, the owner/operator shall notify the Osage UIC officer orally within 24 hours, with written notice including plans for testing and/or repair to be submitted within five days. If all the information is not available within five days, a followup report must be submitted within 30 days.

(d) The owner/operator shall retain all monitoring records for three years, unless an enforcement action is pending, and then until three years after the enforcement action has been resolved.

(e) The owner/operator shall notify the Osage UIC office in writing of a transfer of ownership at least 10 days prior to such transfer.

(Approved by the Office of Management and Budget under control number 2040–0042)

§ 147.2923 Corrective action for wells authorized by permit.

All improperly sealed, completed or abandoned wells (i.e., wells or well bores which may provide an avenue for movement of fluid into an UDSW) within the zone of endangering influence (as defined in § 147.2904, Area of Review) that penetrate the injection zone of a Class II well, must have corrective action taken to prevent movement of fluid into a USDW.

(a) EPA will review completion and plugging records of wells within the zone of endangering influence that penetrate the injection zone and will notify the operator when corrective action is required. Corrective action may include:

(1) Well modifications, including:

(i) Recementing;

(ii) Workover;

(iii) Reconditioning; and/or

(iv) Plugging or replugging;

(2) Permit conditions to limit injection pressure so as to prevent movement of fluid into a USDW;

(3) A more stringent monitoring program; and/or

(4) Periodic testing of other wells within the area of review to determine if significant movement of fluid has occurred. If the monitoring discussed in paragraph (a)(3) or (a)(4) of this section indicates the potential endangerment of a USDW, then action as described in paragraph (a)(1) or (a)(2) of this section must be taken.

(b) If the Regional Administrator has demonstrable knowledge that wells within the zone of endangering influence will not serve as conduits for fluid movement into a USDW, the permit may be approved without requiring corrective action. However, additional monitoring shall be required to confirm that no significant migration will occur.

§ 147.2924 Area permits.

(a) Area permits may be issued for more than one injection well if the following conditions are met:

(1) All existing wells are described and located in the permit application;

(2) All wells are within the same well field, project, reservoir or similar unit;

(3) All wells are of similar construction; and

(4) All wells are operated by the same owner/operator.

(b) Area permits shall specify:

(1) The area within which injection is authorized; and

(2) The requirements for construction, monitoring, reporting, operation and abandonment for all wells authorized by the permit.

(c) Area permits can authorize the construction and operation of new wells within the permit area, if:

(1) The permittee notifies the Regional Administrator in the annual report of when and where any new wells have or will be drilled;

(2) The new wells meet the criteria outlined in paragraphs (a) and (b) of this section; and

(3) The effects of the new wells were addressed in the permit application and approved by the Regional Administrator.

§ 147.2925 Standard permit conditions.

(a) The permittee must comply with all permit conditions, except as authorized by an emergency permit (described in § 147.2906). Noncompliance is grounds for permit modification, permit termination or enforcement action.

(b) The permittee has a duty to halt or reduce activity in order to maintain compliance with permit conditions.

(c) The permittee shall take all reasonable steps to mitigate any adverse environmental impact resulting from noncompliance.

(d) The permittee shall properly operate and maintain all facilities installed or used to meet permit conditions. Proper operation and maintenance also includes adequate operator staffing and training, adequate funding, and adequate engineering capability available.

(e) This permit may be modified or terminated for cause (see §§147.2927 and 147.2928). The filing of a request by the permittee for a permit modification or termination, or a notification of planned changes or anticipated noncompliance, does not stay any permit condition.

(f) This permit does not convey any property rights, or any exclusive privilege.

(g) The permittee shall furnish, within a reasonable time, information that the Regional Administrator requests, for determination of permit compliance, or if cause exists, for permit modification or termination.

(h) The permittee shall allow EPA representatives, upon presentation of appropriate credentials or other documentation, to:

(1) Enter permittee's premises where a regulated activity is conducted or located, or where records required by this permit are kept;

(2) Have access to and copy records required by this permit;

(3) Inspect any facilities, equipment, practices or operations regulated or required by this permit; and

(4) Sample or monitor any substances or parameters at any location for purpose of assuring compliance with this permit or the SDWA.

(i) Monitoring and records.

(1) Samples and monitoring data shall be representative of injection activity.

(2) Permittee shall retain monitoring records for three years.

(3) Monitoring records shall include:

(i) Date, exact place and time of sampling or measurement;

(ii) Individual(s) who preformed the measurements;

(iii) Date(s) analyses were performed;

(iv) Individual(s) who performed the analyses;

(v) Analytical techniques or methods used, including quality assurance techniques employed to insure the generation of reliable data; and

(vi) Results of analyses.

(j) *Signatory requirements.* All applications, reports or information submitted to the Regional Administrator or the Osage UIC office must be signed by the injection facility owner/operator or his duly authorized representative. The person signing these documents must make the following certification:

"I certify under penalty of law that I have personally examined and am familiar with the information submitted in this document and all attachments and that, based on my inquiry of those individuals immediately responsible for obtaining the information, I believe that the information is true, accurate, and complete. I am aware that there are significant penalties for submitting false information, including the possibility of fine and imprisonment."

(k) *Reporting requirements.* (1) The permittee shall notify the Regional Administrator as soon as possible of any planned changes to the facility.

(2) The permittee shall give advance notice to the Regional Administrator of any planned changes which may result in noncompliance.

(3) This permit is not transferable to any person except after notice to the Regional Administrator in accordance with §147.2926.

(l) A new injection well shall not commence injection until construction is complete and the Regional Administrator has been notified of completion of construction and has given his approval to commence injection.

(The information collection requirements contained in paragraphs (g) and (i) were approved by the Office of Management and Budget under control number 2040–0042)

§147.2926 Permit transfers.

(a) Permits may be transferred to another permittee:

(1) If the current permittee notifies the Regional Administrator at least 10 days before the proposed transfer date; and

(2) If the notice includes a written agreement between the existing and new permittees containing:

(i) A specific date for transfer of permit responsibility, coverage and liability; and

(ii) Assurance that the new permittee has a surety bond on file with BIA; and

(3) If the Regional Administrator does not respond with a notice to the existing permittee that the permit will be modified.

(b) If the conditions in paragraph (a) of this section are met, the transfer is effective on the date specified in paragraph (a)(2)(i) of this section.

§ 147.2927 Permit modification.

(a) Permits may be modified for the following causes only (with the exceptions listed in paragraph (b) of this section regarding minor modifications):

(1) There are substantial changes to the facility or activity which occurred after permit issuance that justify revised or additional permit conditions.

(2) The Regional Administrator has received information (e.g., from monitoring reports, inspections) which warrants a modified permit.

(3) The regulations or standards on which the permit was based have changed.

(4) The Regional Administrator has received notice of a proposed permit transfer.

(5) An interested person requests in writing that a permit be modified, and the Regional Administrator determines that cause for modification exists.

(6) Cause exists for termination under § 147.2928, but the Regional Administrator determines that permit modification is appropriate.

(b) *Minor modifications.* (1) Minor modifications do not require that the procedures listed in paragraph (c) of this section be followed.

(2) Minor modifications consist of:

(i) Correcting typographical errors;

(ii) Requiring more frequent monitoring or reporting;

(iii) Changing ownership or operational control (see § 147.2926, Permit Transfers); or

(iv) Changing quantities or types of injected fluids, provided:

(A) The facility can operate within conditions of permit;

(B) The facility classification would not change.

(c) *Modification procedures.* (1) A draft permit shall be prepared with proposed modifications.

(2) The draft permit shall follow the general permitting procedures (i.e., public comment period, etc.) before a final decision is made.

(3) Only the changed conditions shall be addressed in the draft permit or public review.

§ 147.2928 Permit termination.

(a) Permits may be terminated for the following causes only:

(1) Noncompliance with any permit condition.

(2) Misrepresentation or failure to fully disclose any relevant facts.

(3) Determination that the permitted activity endangers human health or the environment.

(4) Interested person requests in writing that a permit be terminated and the Regional Administrator determines that request is valid.

(b) *Termination procedures.* (1) The Regional Administrator shall issue notice of intent to terminate (which is a type of draft permit).

(2) Notice of intent to terminate shall follow the general permitting procedures (i.e., public comment period, etc.) before a final decision is made.

§ 147.2929 Administrative permitting procedures.

(a) *Completeness review.* (1) The Regional Administrator shall review each permit application for completeness with the application requirements in § 147.2918. The review will be completed in 10 days, and the Regional Administrator shall notify the applicant whether or not the application is complete.

(2) If the application is incomplete, the Regional Administrator shall:

(i) List the additional information needed;

(ii) Specify a date by which the information must be submitted; and

(iii) Notify the applicant when the application is complete.

(3) After an application is determined complete, the Regional Administrator may request additional information to

clarify previously submitted information. The application will still be considered complete.

(4) If an applicant fails or refuses to correct deficiencies in the application, the permit may be denied and appropriate enforcement actions taken.

(b) *Draft permits.* (1) After an application is deemed complete, the Regional Administrator shall either prepare a draft permit or notice of intent to deny the permit (which is a type of draft permit). If the Regional Administrator later decides the tentative decision to deny was wrong, he shall withdraw the notice of intent to deny and prepare a draft permit.

(2) A draft permit shall contain at least the following information:

(i) The standard permit conditions in § 147.2925;

(ii) Any monitoring and reporting requirements;

(iii) The construction and operation requirements; and

(iv) Plugging and abandonment requirements.

(c) *Statement of basis.* (1) The Regional Administrator shall prepare a statement of basis for every draft permit.

(2) The statement of basis shall briefly describe the draft permit conditions and the reasons for them. In the case of a notice of intent to deny or terminate, the statement of basis shall give reasons to support the tentative decision.

(3) The statement of basis shall be sent to the applicant, and to any other person who requests a copy.

(d) *Public notice.* (1)(i) The Regional Administrator shall give public notice when:

(A) A permit application has been tentatively denied;

(B) A draft permit has been prepared;

(C) A hearing has been scheduled; or

(D) An appeal has been granted.

(ii) The applicant shall give public notice that he is submitting a permit application.

(iii) Public notice is not required when a request for permit modification or termination is denied. However, written notice will be given to the permittee and the requester.

(iv) Public notices may include more than one permit or action.

(2)(i) Public notice of a draft permit (including notice of intent to deny) shall allow at least 15 days for public comment.

(ii) Public notice of a hearing shall be given at least 30 days before the hearing.

(3)(i) Public notice given by the Regional Administrator for the reasons listed in paragraph (d)(1)(i) of this section shall be mailed to the applicant, and published in a daily or weekly paper of general circulation in the affected area.

(ii) Notice of application submission required by paragraph (d)(1)(ii) of this section shall be given to the surface landowner, tenants on the land where an injection well is located or is proposed to be located, and to each operator of a producing lease within one-half mile of the well location prior to submitting the application to the Regional Administrator.

(4) The notice of application submission in paragraphs (d)(1)(ii) and (d)(3)(ii) of this section shall contain:

(i) The applicant's name and address;

(ii) The legal location of the injection well;

(iii) Nature of activity;

(iv) A statement that EPA will be preparing a draft permit and that there will be an opportunity for public comment; and

(v) The name and phone number of EPA contact person.

(5) All other notices shall contain:

(i) The name, address, and phone number of the Osage UIC office and contact person for additional information and copies of the draft permit;

(ii) Name and address of permit applicant or permittee;

(iii) Brief description of nature of activity;

(iv) Brief description of comment period and comment procedures;

(v) Location of the information available for public review; and

(vi) In the case of a notice for a hearing the notice shall also include:

(A) Date, time, and location of hearing;

(B) Reference to date of previous notices of the same permit; and

(C) Brief description of the purpose of the hearing, including rules and procedures.

(e) *Public comments.* (1) During the public comment period, any person

may submit written comments on the draft permit, and may request a public hearing. A request for hearing shall be in writing and state the issues proposed to be raised in the hearing.

(2) The Regional Administrator shall consider all comments when making the final decision, and shall respond to comments after the decision is made. The response shall:

(i) Specify if any changes were made from the draft permit to the final permit decision, and why;

(ii) Briefly describe and respond to all significant comments on the draft permit made during the comment period, or hearing, if held; and

(iii) Be made available to the public.

(f) *Public hearings.* (1) The Regional Administrator shall hold a public hearing whenever he finds a significant amount of public interest in a draft permit, based on the requests submitted, or at his discretion.

(2) Any person may submit oral or written statements and data concerning the draft permit. The public comment period shall be automatically extended to the close of any public hearing held, or may be extended by the hearing officer at the hearing.

(3) A tape recording or written transcript of the hearing shall be made available to the public.

(g) *Reopening of the comment period.* (1) If any of the information submitted during the public comment period raises substantial new questions about a permit, the Regional Administrator may:

(i) Prepare a new draft permit;

(ii) Prepare a revised statement of basis; or

(iii) Reopen the comment period.

(2) Comments submitted during a reopened comment period shall be limited to the substantial new questions that caused its reopening.

(3) Public notice about any of the above actions shall be given and shall define the scope of the new questions raised.

(h) *Issuance and effective date of a permit.* (1) After the close of the comment period on a draft permit, the Regional Administrator shall make a final permit decision. The Regional Administrator shall notify the applicant and each person who commented or requested to receive notice. The notice shall include reference to the procedures for appealing a permit decision.

(2) A final permit decision shall become effective 30 days after giving notice of the decision unless:

(i) A later date is specified in the notice;

(ii) Review is requested under § 147.2929(j); or

(iii) No comments requested a change in the draft permit, in which case the permit is effective immediately upon issuance.

(i) *Stays of contested permit conditions.* If a request for review of a final UIC permit § 147.2929(j) is granted, the effect of the contested permit conditions shall be stayed and shall not be subject to judicial review pending final agency action. If the permit involves a new injection well or project, the applicant shall be without a permit for the proposed well pending final agency action. Uncontested provisions which are not severable from those contested provisions shall be stayed with the contested provisions.

(j) *Appeal of permits.* (1) Any person who filed comments on the draft permit or participated in the public hearing may petition the Administrator to review any condition of the permit decision. Any person who failed to file comments or participate in the hearing may petition for administrative review only to the extent of the changes from the preliminary permit to the final permit decision.

(2) A person may request review of a final permit decision within 30 days after a final permit decision has been issued. The 30-day period within which a person may request review begins with the service of notice of the Regional Administrator's final permit decision unless a later date is specified in that notice.

(3) The petition requesting review shall include:

(i) A demonstration that the petition is eligible under the requirements of paragraph (j)(1) of this section; and, when appropriate,

(ii) A showing that the condition in question is based on:

(A) A finding of fact or conclusion of law that is clearly erroneous; or

(B) An exercise of discretion or important policy consideration which the Administrator, in his discretion, should review.

(4) The Administrator may also decide, on his initiative, to review any condition of any UIC permit issued under these requirements. The Administrator must act under this paragraph within 30 days of the date notice was given of the Regional Administrator's action.

(5) Within a reasonable time following the filing of the petition for review, the Administrator shall issue an order either granting or denying the request. To the extent that review is denied, the conditions of the final permit decision become final agency action.

(6) Public notice shall be given by the Regional Administrator of any grant of a review petition by the Administrator. Notice shall be sent to the applicant, the person requesting the review, appropriate persons on the Osage County mailing list and to newspapers of general circulation in the county. Included in the notice shall be a briefing schedule for the appeal and a statement that any interested person may file an amicus brief. Notice of denial of the review petition will be sent only to the person(s) requesting the review.

(7) A petition to the Administrator, under paragraphs (j) (1) and (2) of this section is a prerequisite to the seeking of judicial review of the final agency action. For purposes of judicial review, final agency action occurs when a final UIC permit is issued or denied by the Regional Administrator and agency review procedures are exhausted. A final permit decision shall be issued by the Regional Administrator:

(i) When the Administrator issues notice to the parties involved that review has been denied;

(ii) When the Administrator issues a decision on the merits of the appeal and the decision does not include a remand of the proceedings; or

(iii) Upon the completion of the remand proceedings if the proceedings are remanded, unless the Administrator's remand order specifically provides that the appeal of the remand decision will be required to exhaust the administrative remedies.

Subpart HHH—Lands of the Navajo, Ute Mountain Ute, and All Other New Mexico Tribes

SOURCE: 53 FR 43104, Oct. 25, 1988, unless otherwise noted.

§147.3000 EPA-administered program.

(a) *Contents.* The UIC program for the Indian lands of the Navajo, the Ute Mountain Ute (Class II wells only on Ute Mountain Ute lands in Colorado and all wells on Ute Mountain Ute lands in Utah and New Mexico), and all wells on other Indian lands in New Mexico is administered by EPA. (The term ''Indian lands'' is defined at 40 CFR 144.3.) The Navajo Indian lands are in the States of Arizona, New Mexico, and Utah; and the Ute Mountain Ute lands are in Colorado, New Mexico and Utah. This program consists of the UIC program requirements of 40 CFR parts 124, 144, 146, 148, and additional requirements set forth in the remainder of this subpart. The additions and modifications of this subpart apply only to the Indian lands described above. Injection well owners and operators, and EPA shall comply with these requirements.

(b) *Effective date.* The effective date for the UIC program on these lands is November 25, 1988.

[53 FR 43104, Oct. 25, 1988, as amended at 56 FR 9422, Mar. 6, 1991]

§147.3001 Definition.

Area of review. For the purposes of this subpart, area of review means the area surrounding an injection well or project area described according to the criteria set forth in §147.3009 of this subpart.

§147.3002 Public notice of permit actions.

An applicant shall give public notice of his intention to apply for a permit as follows:

(a) Prior to submitting an application to the Director, the applicant shall give notice to each landowner, tenant, and operator of a producing lease within one-half mile of the well and to the affected Tribal Government. The notice shall include:

(1) Name and address of applicant;

(2) A brief description of the planned injection activities including well location, name and depth of the injection zone, maximum injection pressure and volume, and source and description of the fluid to be injected;

(3) Name, address, and phone number of the EPA contact person; and

(4) A statement that opportunity to comment will be announced to the public after EPA prepares a draft permit.

(b) In addition to the requirements of § 144.31(e) of this chapter, a permit applicant shall submit a description of the way the notice was given and the names and addresses of those to whom it was given.

(c) Upon written request and supporting documentation, the Director may waive the requirement in paragraph (a) of this section to give individual notice of intent to apply for permits in an area where it would be impractical. However, notice to the affected Tribal government shall not be waived.

(d) The Director shall also provide to the affected Tribal government all notices given to State governments under § 124.10(c) of this chapter.

§ 147.3003 Aquifer exemptions.

(a) *Aquifer exemptions in connection with Class II wells.* In accordance with § 144.7(b) and § 146.4 of this chapter, the portions of authorized injection zones into which existing Class II wells are currently injecting which are described in appendix A are hereby exempted. The exempted aquifers are defined by a one-quarter mile radius from the existing injection well. The exemption includes the intended injection zone only and is solely for the purpose of Class II injection.

(b) *Class III wells.* In addition to the requirements of § 144.7(c)(1) of this chapter, an applicant for a uranium mining permit which necessitates an aquifer exemption shall submit a plugging and abandonment plan containing an aquifer cleanup plan, acceptable to the Director, describing the methods or techniques that will be used to meet the standards of § 147.3011. The cleanup plan shall include an analysis of pre-injection water quality for the constituents required by the Director. The Director shall consider the cleanup plan in addition to the other information required for permit applications under §§ 144.31(e) and 146.34 of this chapter.

§ 147.3004 Duration of rule authorization for existing Class I and III wells.

Notwithstanding § 144.21(a)(3)(i)(B) of this chapter, authorization by rule for existing Class I and III wells will expire 90 days after the effective date of this UIC program unless a complete permit application has been submitted to the Director.

§ 147.3005 Radioactive waste injection wells.

Notwithstanding §§ 144.24 and 146.51(b) of this chapter, owners and operators of wells used to dispose of radioactive waste (as defined in 10 CFR part 20, appendix B, table II, but not including high level and transuranic waste and spent nuclear fuel covered by 40 CFR part 191) shall comply with the permitting requirements pertaining to Class I wells in parts 124, 144 and 146 of this chapter, as modified and supplemented by this subpart.

§ 147.3006 Injection pressure for existing Class II wells authorized by rule.

(a) *Rule-authorized Class II saltwater disposal wells.* In addition to the requirements of § 144.28(f)(3)(ii) of this chapter, the owner or operator shall, except during well stimulation, use an injection pressure measured at the wellhead that is not greater than the pressure calculated by using the following formula: $Pm=0.2d$

where:

Pm=injection pressure at the wellhead in pounds per square inch

d=depth in feet to the top of the injection zone.

Owners and operators shall comply with this requirement no later than one year after the effective date of this program.

(b) *Rule-authorized Class II enhanced recovery and hydrocarbon storage wells.* (1) In addition to the requirements of § 144.28(f)(3)(ii) of this chapter, owners and operators shall use an injection pressure no greater than the pressure established by the Director for the field or formation in which the well is located. The Director shall establish

such maximum pressure after notice (including notice to the affected Tribe), opportunity for comment, and opportunity for public hearing according to the provisions of part 124, subpart A, of this chapter, and shall inform owners and operators and the affected Tribe in writing of the applicable maximum pressure; or

(2) An owner or operator may inject at a pressure greater than that specified in paragraph (b)(1) of this section for the field or formation in which he is operating after demonstrating in writing to the satisfaction of the Director that such injection pressure will not violate the requirements of §144.28(f)(3)(ii) of this chapter. The Director may grant such a request after notice (including notice to the affected Tribe), opportunity for comment and opportunity for a public hearing according to the provisions of part 124, subpart A of this chapter.

(3) Prior to the time that the Director establishes rules for maximum injection pressure under paragraph (b)(1) of this section the owner or operator shall:

(i) Limit injection pressure to a value which will not exceed the operating requirements of §144.28(f)(3)(ii); and

(ii) Submit data acceptable to the Director which defines the fracture pressure of the formation in which injection is taking place. A single submission may be made on behalf of two or more operators conducting operations in the same field and formation, if the Director approves. The data shall be submitted to the Director within one year of the effective date of this program.

§147.3007 Application for a permit.

(a) Notwithstanding the requirements of §144.31(c)(1) of this chapter, the owner or operator of an existing Class I or III well shall submit a complete permit application no later than 90 days after the effective date of the program.

(b) The topographic map (or other map if a topographic map is unavailable) required by §144.31(e)(7) of this chapter, shall extend two miles from Class II wells, and 2½ miles from Class I and III wells. These maps will show all the information listed in paragraph 144.31(e)(7) within ½ mile for Class II wells and 2½ miles for Class I and III wells.

§147.3008 Criteria for aquifer exemptions.

The aquifer exemption criterion in §146.4(c) of this chapter shall not be available for this program.

§147.3009 Area of review.

The area of review shall be defined as follows:

(a) *Class II wells.* The area of review for Class II permits and area permits shall be defined by a fixed radius as described in §146.6(b) (1) and (2) of this chapter except that the radius shall be one-half mile.

(b) *Class I and III wells.* The area of review for Class I and III wells are well fields which may be either:

(1) An area defined by a radius two and one-half miles from the well or well field; or

(2) An area one-quarter mile from the well or well field where the well field production at the times exceeds injection to produce a net withdrawal; or

(3) A suitable distance, not less than one-quarter mile, proposed by the owner or operator and approved by the Director based upon a mathematical calculation such as that found in §146.6(a)(2) of this chapter.

§147.3010 Mechanical integrity tests.

The monitoring of annulus pressure listed in §146.8(b)(1) of this chapter will only be acceptable if preceded by a pressure test, using liquid or gas that clearly demonstrates that mechanical integrity exists at the time of the pressure test.

§147.3011 Plugging and abandonment of Class III wells.

To meet the requirements of §146.10(d) of this chapter, owners and operators of Class III uranium projects underlying or in aquifers containing up to 5,000 mg/l TDS which have been exempted under §146.4 of this chapter shall:

(a) Include in the required plugging and abandonment plan a plan for aquifer clean-up and monitoring which demonstrates adequate protection of surrounding USDWs.

(1) The Director shall include in each such permit for a Class III uranium project the concentrations of contaminants to which aquifers must be cleaned up in order to protect surrounding USDWs.

(2) The concentrations will be set as close as is feasible to the original conditions.

(b) When requesting permission to plug a well, owners and operators shall submit for the Director's approval a schedule for the proposed aquifer cleanup, in addition to the information required by § 146.34(c).

(c) Cleanup and monitoring shall be continued until the owner or operator certifies that no constituent listed in the permit exceeds the concentrations required by the permit, and the Director notifies the permittee in writing that cleanup activity may be terminated.

§ 147.3012 Construction requirements for Class I wells.

In addition to the cementing requirement of § 146.12(b) of this chapter, owners and operators of Class I wells shall, through circulation, cement all casing to the surface.

§ 147.3013 Information to be considered for Class I wells.

(a) In addition to the information listed in § 146.14(a) of this chapter, the Director shall consider the following prior to issuing any Class I permit:

(1) Expected pressure changes, native fluid displacement, and direction of movement of the injected fluid; and

(2) Methods to be used for sampling, and for measurement and calculation of flow.

(b) In addition to the information listed in § 146.14(b) of this chapter, the Director shall consider any information required under § 146.14(a) of this chapter (as supplemented by this subpart) that has been gathered during construction.

§ 147.3014 Construction requirements for Class III wells.

(a) In addition to the requirements of § 146.32(c)(3) of this chapter, radiological characteristics of the formation fluids shall be provided to the Director.

(b) In addition to the requirements of § 146.32(e) of this chapter, the Director may require monitoring wells to be completed into USDWs below the injection zone if those USDWs may be affected by mining operations.

§ 147.3015 Information to be considered for Class III wells.

(a) In addition to the requirements of § 146.34(a) of this chapter, the following information shall be considered by the Director:

(1) Proposed construction procedures, including a cementing and casing program, logging procedures, deviation checks, and a drilling, testing and coring program.

(2) Depth to the proposed injection zone, and a chemical, physical and radiological analysis of the ground water in the proposed injection zone sufficient to define pre-injection water quality as required for aquifer cleanup by § 147.3011 of this subpart.

(3) An aquifer cleanup plan if required by § 147.3003(b) of this subpart.

(4) Any additional information that may be necessary to demonstrate that cleanup will reduce the level of contaminants in the surrounding USDWs as close as feasible to the original conditions.

(b) In addition to the requirements of § 146.34(b) of this chapter, the Director shall consider any information required under § 146.34(a) of this chapter (as supplemented by this subpart) that has been gathered during construction.

§ 147.3016 Criteria and standards applicable to Class V wells.

In addition to the criteria and standards applicable to Class V wells set forth in subpart F of part 146 of this chapter, owners and operators of wells that do not fall within the Class IV category but that are used to dispose of radioactive wastes (as defined in 10 CFR part 20, appendix B, table II, col-

umn 2, but not including high level and transuranic wastes and spent nuclear fuel covered by 40 CFR part 191) shall comply with all of the requirements applicable to Class I injection wells in 40 CFR parts 124, 144 and 146 as supplemented by this subpart.

APPENDIX A TO SUBPART HHH—EXEMPTED AQUIFERS IN NEW MEXICO

The areas described by a one-quarter mile radius around the following Class II wells in the listed formations are exempted for the purpose of Class II injection.

Sec.					Well No.
Arco Oil & Gas Co.—Operator/Horseshoe Gallup—Field/Gallup—Formation					
SE/NE	5	T30N	R16W	1650′FNL	330′FEL	134
NW/NW	30	T31N	R16W	660′FNL	703′FWL	8
SE/SW	28	T31N	R16W	790′FSL	2150′FWL	167
NW/SE	33	T31N	R16W	1710′FSL	2310′FEL	199
SE/NW	35	T31N	R16W	2105′FNL	2105′FWL	196
NW/NW	4	T30N	R16W	455′FNL	4435′FEL	219
NW/SW	33	T31N	R16W	1980′FSL	386′FWL	65
NW/SE	27	T31N	R16W	1980′FSL	2080′FEL	164
SE/SE	30	T31N	R16W	660′FSL	660′FEL	5
NW/NW	34	T31N	R16W	730′FNL	515′FWL	180
NW/NE	34	T31N	R16W	813′FNL	2036′FEL	182
NW/NE	2	T30N	R16W	720′FNL	2040′FEL	229
NW/NW	29	T31N	R16W	660′FNL	660′FWL	24
NW/SW	13	T31N	R17W	1975′FSL	670′FWL	77
NW/SE	29	T31N	R16W	1980′FSL	1980′FEL	22
SE/SW	27	T31N	R16W	660′FSL	1980′FWL	171
NW/SW	35	T31N	R16W	1980′FSL	660′FWL	205
SE/NW	30	T31N	R16W	1980′FNL	2061′FWL	7
NW/NE	31	T31N	R16W	660′FNL	1980′FEL	17
NW/NE	4	T30N	R16W	330′FNL	2160′FEL	221
NW/NE	29	T31N	R16W	660′FNL	1980′FEL	26
SE/NE	34	T31N	R16W	1990′FNL	645′FEL	194
SE/SE	31	T31N	R16W	640′FSL	660′FEL	27
NE/SW	14	T31N	R17W	2250′FSL	2630′FWL	94
NE/NW	14	T31N	R17W	625′FNL	1995′FWl,	69
SE/NW	10	T30N	R16W	1900′FNL	2080′FWL	271
SE/SE	29	T31N	R16W	560′FSL		21
SE/NE	30	T31N	R16W	1980′FNL	660′FEL	10
SE/NW	29	T31N	R16W	2080′FNL	1980′FWL	23
NW/SE	25	T31N	R17W	1980′FSL	1980′FEL	122
SE/SW	32	T31N	R16W	660′FSL	1980′FWL	14
NW/SW	30	T31N	R16W	2021′FSL	742′FWL	19
SE/SW	13	T31N	R17W	660′FSL	1980′FWL	82
NW/NW	27	T31N	R16W	520′FNL	660′FWL	150
SE/SE	28	T31N	R16W	660′FSL	660′FEL	169
NW/SW	29	T31N	R16W	1980′FSL	660′FWL	11
SE/NW	34	T31N	R16W	2310′FNL	1650′FWL	192
SE/NW	29	T31N	R16W	660′FSL	1980′FWL	12
NW/SW	27	T31N	R16W	1650′FSL	330′FWL	162
NE/SE	23	T31N	R17W	1880′FSL	340′FEL	96
NW/SW	24	T31N	R17W	2050′FSL	990′FWL	97
SE/NW	4	T30N	R16W	2060′FNL	1710′FWL	232
NW/NW	31	T31N	R16W	620′FNL	701′FWL	30
NW/SE	35	T31N	R16W	1980′FSL	1980′FEL	207
SE/NE	32	T31N	R16W	1980′FNL	417′FEL	20
NE/NW	28	T31N	R16W	1980′FNL	1980′FEL	152

NE/NW	34	T31N	R16W	2140′FSL	735′FWL	201
SE/NW	3	T30N	R16W	2310′FNL	1640′FWL	236
SE/SW	34	T31N	R16W	660′FSL	1980′FWL	213
NW/NE	30	T31N	R16W	660′FNL	1980′FFL	9
SE/SW	26	T31N	R16W	660′FSL	1980′FWL	175
NW/SE	30	T31N	R16W	1980′FSL	1980′FEL	6
SE/NW	9	T30N	R16W	1650′FNL	2131′FWL	264
NW/SW	4	T30N	R16W	2310′FSL	4390′FEL	242
NW/SW	2	T30N	R16W	1980′FSL	660′FWL	250
NW/NW	33	T31N	R16W	660′FNL	386′FWL	66
NE/NE	15	T31N	R17W	660′FNL	660′FEL	67
NW/NE	33	T31N	R16W	660′FNL	1980′FEL	178
NW/SE	24	T31N	R17W	1875′FSL	1900′FEL	99
NW/NE	28	T31N	R16W	660′FNL	1980′FEL	148
NW/NW	19	T31N	R16W	680′FNL	682′FWL	89
NW/SE	4	T30N	R16W	1820′FSL	2130′FEL	244
SE/SW	20	T31N	R16W	660′FSL	1980′FWL	115
NW/NE	25	T31N	R17W	660′FNL	1980′FEL	118
SE/SW	4	T30N	R16W	660′FSL	3300′FEL	253
NW/SW	19	T31N	R16W	1980′FSL	706′FWL	101
NW/SE	32	T31N	R16W	1950′FSL	1980′FEL	22
NW/NW	35	T31N	R16W	605′FNL	690′FWL	184
SE/NE	29	T31N	R16W	1980′FNL	417′FEL	25
SE/NW	19	T31N	R16W	1980′FNL	2023′FWL	95
NW/NW	32	T31N	R16W	660′FNL	660′FWL	4
SE/SW	24	T31N	R17W	660′FSL	3300′FEL	107
SE/NE	28	T31N	R16W	2105′FNL	940′FEL	154
NW/NE	35	T31N	R16W	610′FNL	2000′FEL	186
SE/SW	5	T31N	R16W	990′FSL	2310′FWL	139
NW/SE	28	T31N	R16W	1980′FSL	1980′FEL	160
SE/SE	33	T31N	R16W	330′FSL	990′FEL	211
NW/NE	5	T30N	R16W	330′FNL	1650′FEL	128
SE/NW	27	T31N	R16W	1900′FNL	2050′FWL	156
SE/SW	35	T31N	R16W	660′FSL	1980′FWL	217
NW/NW	10	T30N	R16W	526′FNL	330′FWL	265
NE/SW	21	T31N	R16W	1880′FSL	1980′FWL	143
NW/NE	24	T31N	R17W	409′FNL	1914′FEL	87
NW/SW	32	T31N	R16W	1980′FSL	660′FWL	15
SE/SE	34	T31N	R16W	960′FSL	910′FEL	215
SW/SE	21	T31N	R16W	820′FSL	1820′FEL	145
SE/SE	27	T31N	R16W	610′FSL	640′FEL	173
NW/SW	3	T30N	R16W	1920′FSL	350′FWL	246
SE/SW	19	T31N	R16W	601′FSL	2002′FWL	111
SW/SE	14	T31N	R17W	330′FSL	1900′FEL	79
NW/NW	27	T31N	R16W	520′FNL	660′FWL	150
SE/NW	31	T31N	R16W	1724′FNL	2067′FWL	29
NW/NE	32	T31N	R16W	660′FNL	1980′FEL	13
SE/NE	24	T31N	R17W	1998′FNL	702′FEL	93
NW/NW	5	T30N	R16W	660′FNL	660′FWL	126
NW/SW	28	T31N	R16W	1740′FSL	590′FWL	158

	Sec.					Well No.
SE/NE	31	T31N	R16W	1980′FNL	660′FEL	16
NW/NW	24	T31N	R17W	660′FNL	760′FWL	85
Energy Reserve Backup Inc.—Operator/Horseshoe Gallup—Field/Gallup—Formation						
SE/SE	5	T31N	R17W	660′FSL	660′FEL	4
NE/SW	10	T30N	R16W	1970′FSL	2210′FWL	31
SE/NW	11	T30N	R16W	2090′FNL	2190′FWI	29
SE/SE	10	T30N	R16W	700′FSL	500′FEL	37
Solar Petroelum Inc.—Operator/Horseshoe—Field/Gallup—Formation						
SW/SE	11	T31N	R17W	736′FSL	2045′FEL	205
SE/NE	9	T31N	R17W	1980′FNL	660′FEL	122
NW/SE	4	T31N	R17W	1980′FSL	1980′FFL	127
NE/NE	10	T31N	R17W	660′FNL	660′FEL	136
SE/SW	4	T31N	R17W	660′FSL	1980′FWL	125
SW/NW	11	T31N	R17W	2300′FNL	660′FWL	206
NW/SW	4	T31N	R17W	1980′FSL	660′FWL	103
SE/NW	4	T31N	R17W	1989′FNL	1980′FWL	128
NW/NW	4	T31N	R17W	660′FNL	660′FWL	101
SW/NE	10	T31N	R17W	1980′FNL	1980′FEL	117
SW/NW	10	T31N	R17W	1980′FNL	660′FWL	108
SW/SW	10	T31N	R17W	660′FSL	660′FWL	114
SW/SE	3	T31N	R17W	330′FSL	2310′FEL	143
SE/NE	5	T31N	R17W	1980′FNL	660′FEL	302
NE/NE	5	T31N	R17W	1950′FNL	1050′FEL	307
SE/SE	9	T31N	R17W	990′FSL	850′FEL	140
NE/NW	10	T31N	R17W	660′FNL	1980′FWL	118
SW/SW	11	T31N	R17W	660′FSL	660′FWL	204
NW/SE	9	T31N	R17W	1980′FSL	1980′FEL	115
SW/SE	10	T31N	R17W	990′FSL	1980′FEL	144
NW/NE	9	T31N	R17W	660′FNL	1980′FEL	123
NE/SW	10	T31N	R17W	1980′FSL	1980′FWL	109
NE/SW	11	T31N	R17W	1980′FSL	1980′FWL	203
SE/NW	9	T31N	R17W	1980′FNL	1980′FWL	134
NW/SW	3	T31N	R17W	1980′FSL	660′FWL	132
SW/SW	3	T31N	R17W	560′FSL	660′FWL	110
NW/NW	9	T31N	R16W	660′FNL	660′FWL	133
SE/SE	4	T31N	R17W	660′FSL	660′FEL	124
WTR Oil Co.—Operator/Horseshoe Gallup—Field/Gallup—Formation						
NE/SW	33	T32N	R17W	1980′FSL	1989′FWL	2

Arco Oil & Gas Co.—Operator/Many Rocks Gallup—Field/Gallup—Formation						
NW/NW	7	T31N	R16W	898′FNL	500′FWL	2
SW/NE	17	T31N	R16W	1673′FNL	1789′FEL	21
NW/SE	17	T31N	R16W	1890′FSL	2150′FEL	23
SW/NE	7	T31N	R16W	2310′FNL	2310′FEL	6
NE/SW	8	T31N	R16W	1650′FSL	1650′FWL	12
NE/NW	17	T31N	R16W	660′FNL	2030′FWL	18
NE/NE	18	T31N	R16W	360′FNL	855′FEL	16
SE/SW	7	T31N	R16W	716′FSL	2185′FWL	13
SE/SE	17	T31N	R16W	660′FSL	660′FEL	26
NE/SW	17	T31N	R16W	2040′FSL	2070′FWL	22
SW/SW	6	T31N	R16W	330′FSL	330′FWL	1
SW/NW	17	T31N	R16W	2073′FNL	641′FWL	19
NW/SW	17	T31N	R16W	1967′FSL	981′FWL	8
James P. Woosley—Operator/Many Rocks Gallup—Field/Gallup—Formation						
NW/NE	20	T32N	R17W	330′FNL	2310′FEL	13
SW/SW	27	T32N	R17W	660′FSL	990′FWL	1
SW/NW	17	T32N	R17W	2310′FWL	330′FWL	4
SW/NW	27	T32N	R17W	260′FWL	1360′FNL	11
NE/SW	27	T32N	R17W	1980′FSL	1980′FWL	6
NE/SE	18	T32N	R17W	2474′FSL	133′FEL	18
SW/SE	27	T32N	R17W	625′FNL	2000′FEL	3
NE/SE	28	T32N	R17W	1980′FSL	330′FEL	12
Solar Petroleum Inc.—Operator/Many Rocks Gallup—Field/Gallup—Formation						
SE/NW	1	T31N	R17W	1980′FNL	1980′FWL	216
NW/NE	2	T31N	R17W	805′FNL	940′FEL	215
SE/NE	2	T31N	R17W	1980′FNL	660′FEL	218
NW/SW	1	T31N	R17W	2310′FSL	990′FNL	223
SE/NE	12	T31N	R17W	1820′FNL	500′FEL	217
WTR Oil Co.—Operator/Many Rocks Gallup—Field/Gallup—Formation						
NW/NW	35	T32N	R17W	810′FNL	510′FWL	11
SE/SE	35	T32N	R17W	660′FSL	660′FEL	6
SE/NE	34	T32N	R17W	775′FEL	1980′FNL	8
SE/NW	35	T32N	R16W	1980′FNL	1980′FWL	9
NW/SE	35	T32N	R17W	1980′FSL	1980′FEL	7
Chaco Oil Co.—Operator/Red Mtn Meseverde—Field/Menefee—Formation						
NE/NE	29	T20N	R9W	395′FNL	1265′FEL	6
SE/SW	20	T20N	R9W	442′FSL	2430′FWL	17

	Sec.					Well No.
Geo Engineering Inc.—Operator/Red Mtn Meseverde—Field/Menefee—Formation						
NW/NE	29	T20N	R9W	160′FNL	2135′FEL	35
NE/NE	29	T20N	R9W	225′FNL	1265′FEL	7
SE/NW	29	T20N	R9W	1344′FNL	2555′FWL	20
NW/NE	29	T20N	R9W	615′FNL	1920′FEL	5
NE/NW	29	T20N	R9W	834′FNL	2113′FWL	21
SW/SE	20	T20N	R9W	265′FSL	2150′FEL	36
NE/NE	29	T20N	R9W	5′FNL	1130′FEL	8
SE/SE	20	T20N	R9W	450′FSL	1145′FEL	24
SE/SE	20	T20N	R9W	990′FSL	1280′FEL	10
NW/NE	29	T20N	R9W	1115′FNL	2325′FEL	22
SE/SE	20	T20N	R9W	1085′FSL	860′FEL	12
Tesoro Petroleum Co.—Operator/S. Hospah Lower Sand—Field/Hospah—Formation						
NW/SE	6	T17N	R8W	2310′FSL	2310′FEL	28
SW/SE	6	T17N	R8W	990′FSL	2310′FFL	34
SW/SW	6	T17N	R8W	5′FSL	20′FWL	18
SE/SW	6	T17N	R8W	5′FSL	2635′FWL	20

Subpart III—Lands of Certain Oklahoma Indian Tribes

SOURCE: 53 FR 43109, Oct. 25, 1988, unless otherwise noted.

§147.3100 EPA-administered program.

(a) *Contents.* The UIC program for the Indian lands in Oklahoma, except for that covering the Class II wells of the Five Civilized Tribes, is administered by EPA. The UIC program for all wells on Indian lands in Oklahoma, except Class II wells on the Osage Mineral Reserve (found at 40 CFR part 147, Subpart GGG) and the Class II program for the Five Civilized Tribes, consists of the UIC program requirements of 40 CFR parts 124, 144, 146, 148, and additional requirements set forth in the remainder of this subpart. Injection well owners and operators, and EPA shall comply with these requirements.

(b) *Effective date.* The effective date for the UIC program for all wells on Indian lands except Class II wells on the Osage Mineral Reserve and Class II wells on the lands of the Five Civilized Tribes is November 25, 1988.

[53 FR 43109, Oct. 25, 1988, as amended at 56 FR 9422, Mar. 6, 1991]

§147.3101 Public notice of permit actions.

(a) In addition to the notice requirements of §124.10 of this chapter, the Director shall provide to the affected Tribal government all notices given to an affected State government under §124.10(c) of this chapter.

(b) *Class I and III wells.* In addition to the notice requirements of §124.10 of this chapter:

(1) Owners and operators of Class I and III wells shall notify the affected Tribal government prior to submitting an application for a permit, shall publish such notice in at least two newspapers of general circulation in the area of the proposed well, and shall broadcast notice over at least one local radio station.

(2) The Director shall publish a notice of availability of a draft permit in at least two newspapers of general circulation in the area of the proposed well, and broadcast notice over at least one local radio station. The public notice shall allow at least 45 days for public comment.

(c) *Class II wells.* In addition to the notice requirements of §124.10 of this chapter:

(1) Owners and operators of Class II wells shall give notice of application for a permit to the affected Tribal government prior to submitting the application to the Director.

(2) In addition to the public notice required for each action listed in §124.10(a) of this chapter, the Director shall also publish notice in a daily or weekly newspaper of general circulation in the affected area for actions concerning Class II wells.

§147.3102 Plugging and abandonment plans.

In lieu of the requirements of §144.28(c)(1) and (2) (i)–(iii) of this chapter, owners and operators of Class II wells shall comply with the plugging and abandonment provisions of §147.3108 of this subpart.

§147.3103 Fluid seals.

Notwithstanding §§144.28(f)(2) and 146.12(c) of this chapter, owners and operators shall not use a fluid seal as an alternative to a packer.

§147.3104 Notice of abandonment.

(a) In addition to the notice required by §144.28(j)(2) of this chapter, the owner or operator shall at the same time submit plugging information in conformance with §147.3108 of this subpart including:

(1) Type and number of plugs;

(2) Elevation of top and bottom of each plug;

(3) Method of plug placement; and

(4) Type, grade and quantity of cement to be used.

(b) In addition to the permit conditions specified in §§144.51 and 144.52 of this chapter, each owner and operator shall submit and each permit shall contain the following information (in conformance with §146.3108 of this subpart):

(1) Type and number of plugs;

(2) Elevation of top and bottom of each plug;

(3) Method of plug placement; and

(4) Type, grade and quantity of cement to be used.

§ 147.3105 Plugging and abandonment report.

(a) In lieu of the time periods for submitting a plugging report in § 144.28(k) of this chapter, owners and operators of Class I and III wells shall submit the report within 15 days of plugging the well and owners or operators of Class II wells within 30 days of plugging, or at the time of the next required operational report (whichever is less.) If the required operational report is due less than 15 days following completion of plugging, then the plugging report shall be submitted within 30 days for Class II wells and 15 days for Class I and III wells.

(b) In addition to the requirement of § 144.28(k)(1) of this chapter, owners and operators of Class II wells shall include a statement that the well was plugged in accordance with § 146.10 of this chapter and § 147.3109 of this subpart, and, if the actual plugging differed, specify the actual procedures used.

(c) The schedule upon which reports of plugging must be submitted are changed from those in § 144.51(o) to those specified in paragraph (a) of this section.

§ 147.3106 Area of review.

(a) When determining the area of review under § 146.6(b) of this chapter, the fixed radius shall be no less than one mile for Class I wells and one-half mile for Class II and III wells. In the case of an application for an area permit, determination of the area of review under § 146.6(b) shall be a fixed width of not less than one mile for the circumscribing area of Class I projects and one-half mile for the circumscribing area of Class II and III projects.

(b) However, in lieu of § 146.6(c) of this chapter, if the area of review is determined by a mathematical model pursuant to paragraph § 146.6(a) of this chapter, the permissible radius is the result of such calculation even if it is less than one mile for Class I wells and one-half for Class II and III wells.

§ 147.3107 Mechanical integrity.

(a) Monitoring of annulus pressure conducted pursuant to § 146.8(b)(1) shall be preceded by an initial pressure test. A positive gauge pressure on the casing/tubing annulus (filled with liquid) shall be maintained continuously. The pressure shall be monitored monthly.

(b) Pressure tests conducted pursuant to § 146.8(b)(2) of this chapter shall be performed with a pressure on the casing/tubing annulus of at least 200 p.s.i. unless otherwise specified by the Director. In addition, pressure tests conducted during well operation shall maintain an injection/annulus pressure differential of at least 100 p.s.i. throughout the tubing length.

(c) Monitoring of enhanced recovery wells conducted pursuant to § 146.8(b)(3), must be preceded by an initial pressure test that was conducted no more than 90 days prior to the commencement of monitoring.

§ 147.3108 Plugging Class I, II, and III wells.

In addition to the requirements of § 146.10 of this chapter, owners and operators shall comply with the following when plugging a well:

(a) For Class I and III wells:

(1) The well shall be filled with mud from the bottom of the well to a point one hundred (100) feet below the top of the highest disposal or injection zone and then with a cement plug from there to at least one hundred (100) feet above the top of the disposal or injection zone.

(2) A cement plug shall also be set from a point at least fifty (50) feet below the shoe of the surface casing to a point at least five (5) feet above the top of the lowest USDW.

(3) A final cement plug shall extend from a point at least thirty feet below the ground surface to a point five (5) feet below the ground surface.

(4) All intervals between plugs shall be filled with mud.

(5) The top plug shall clearly show by permanent markings inscribed in the cement or on a steel plate embedded in the cement the well permit number and date of plugging.

(b) For Class II wells:

(1) The well shall be kept full of mud as casing is removed. No surface casing shall be removed without written approval from the Director.

(2) If surface casing is adequately set and cemented through all USDWs (set to at least 50 feet below the base of the USDW), a plug shall be set at least 50

feet below the shoe of the casing and extending at least 50 feet above the shoe of the casing; or

(3) If the surface casing and cementing is inadequate, the well bore shall be filled with cement from a point at least 50 feet below the base of the USDW to a point at least 50 feet above the shoe of the surface casing, and any additional plugs as required by the Director.

(4) In all cases, the top 20 feet of the well bore below 3 feet of ground surface shall be filled with cement. Surface casing shall be cut off 3 feet below ground surface and covered with a secure steel cap on top of the surface pipe. The remaining 3 feet shall be filled with dirt.

(5) Except as provided in sub-paragraph (b)(6) of this section, each producing or receiving formation shall be sealed off with at least a 50-foot cement plug placed at the base of the formation and at least a 50-foot cement plug placed at the top of the formation.

(6) The requirement in sub-paragraph (b)(5) of this section does not apply if the producing/receiving formation is already sealed off from the well bore with adequate casing and cementing behind casing, and casing is not to be removed, or the only openings from the producing/receiving formation into the well bore are perforations in the casing, and the annulus between the casing and the outer walls of the well is filled with cement for a distance of 50 feet above the top of the formation. When such conditions exist, a bridge plug capped with at least 10 feet of cement set at the top of the producing formation may be used.

(7) When specified by the Director, any uncased hole below the shoe of any casing to be left in the well shall be filled with cement to a depth of at least 50 feet below the casing shoe, or the bottom of the hole, and the casing above the shoe shall be filled with cement to at least 50 feet above the shoe of the casing. If the well has a screen or liner which is not to be removed, the well bore shall be filled with cement from the base of the screen or liner to at least 50 feet above the top of the screen or liner.

(8) All intervals between cement plugs in the well bore must be filled with mud.

(c) For the purposes of this section mud shall be defined as: mud of not less than thirty-six (36) viscosity (API Full Funnel Method) and a weight of not less than nine (9) pounds per gallon.

§147.3109 Timing of mechanical integrity test.

The demonstrations of mechanical integrity required by §146.14(b)(2) of this chapter prior to approval for the operation of a Class I well shall, for an existing well, be conducted no more than 90 days prior to application for the permit and the results included in the permit application. The owner or operator shall notify the Director at least seven days in advance of the time and date of the test so that EPA observers may be present.

PART 148—HAZARDOUS WASTE INJECTION RESTRICTIONS

Subpart A—General

Sec.
148.1 Purpose, scope and applicability.
148.2 Definitions.
148.3 Dilution prohibited as a substitute for treatment.
148.4 Procedures for case-by-case extensions to an effective date.
148.5 Waste analysis.

Subpart B—Prohibitions on Injection

148.10 Waste specific prohibitions—solvent wastes.
148.11 Waste specific prohibitions—dioxin-containing wastes.
148.12 Waste specific prohibitions—California list wastes.
148.14 Waste specific prohibitions—first third wastes.
148.15 Waste specific prohibitions—second third wastes.
148.16 Waste specific prohibitions—third third wastes.
148.17 Waste specific prohibitions; newly listed wastes.
148.18 Waste specific prohibitions–newly listed and identified wastes.

Subpart C—Petition Standards and Procedures

148.20 Petitions to allow injection of a waste prohibited under subpart B.

148.21 Information to be submitted in support of petitions.
148.22 Requirements for petition submission, review and approval or denial.
148.23 Review of exemptions granted pursuant to a petition.
148.24 Termination of approved petition.

AUTHORITY: Secs. 3004, Resource Conservation and Recovery Act, 42 U.S.C. 6901 *et seq.*

SOURCE: 53 FR 28154, July 26, 1988, unless otherwise noted.

Subpart A—General

§ 148.1 Purpose, scope and applicability.

(a) This part identifies hazardous wastes that are restricted from disposal into Class I hazarous waste injection wells and defines those circumstances under which a waste, otherwise prohibited from injection, may be injected.

(b) The requirements of this part apply to owners or operators of Class I hazardous waste injection wells used to inject hazardous waste.

(c) Wastes otherwise prohibited from injection may continue to be injected:

(1) If an extension from the effective date of a prohibition has been granted pursuant to §148.4 with respect to such wastes; or

(2) If an exemption from a prohibition has been granted in response to a petition filed under §148.20 to allow injection of restricted wastes with respect to those wastes and wells covered by the exemption; or

(3) If the waste is generated by a conditionally exempt small quantity generator, as defined in §261.5; or

(d) Wastes that are hazardous only because they exhibit a hazardous characteristic, and which are otherwise prohibited under this part, or part 268 of this chapter, are not prohibited if the wastes:

(1) Are disposed into a nonhazardous or hazardous injection well as defined under 40 CFR §146.6(a); and

(2) Do not exhibit any prohibited characteristic of hazardous waste identified in 40 CFR part 261, subpart C at the point of injection.

[53 FR 28154, July 26, 1988, as amended at 55 FR 22683, June 1, 1990; 57 FR 8088, Mar. 6, 1992; 57 FR 31763, July 20, 1992; 60 FR 33932, June 29, 1995; 61 FR 15596, Apr. 8, 1996; 61 FR 33682, June 28, 1996]

EFFECTIVE DATE NOTE: At 61 FR 15596, Apr. 8, 1996, §148.1 was amended by revising paragraph (a), effective Apr. 8, 1998. For the convenience of the user, the revised text is set forth as follow:

§ 148.1 Purpose, scope and applicability.

(a) This part identifies wastes that are restricted from disposal into Class I wells and defines those circumstances under which a waste, otherwise prohibited from injection, may be injected.

* * * * *

§ 148.2 Definitions.

Injection interval means that part of the injection zone in which the well is screened, or in which the waste is otherwise directly emplaced.

Transmissive fault or fracture is a fault or fracture that has sufficient permeability and vertical extent to allow fluids to move between formations.

§ 148.3 Dilution prohibited as a substitute for treatment.

The prohibition of §268.3 shall apply to owners or operators of Class I hazardous waste injection wells.

§ 148.4 Procedures for case-by-case extensions to an effective date.

The owner or operator of a Class I hazardous waste injection well may submit an application to the Administrator for an extension of the effective date of any applicable prohibition established under subpart B of this part according to the procedures of §268.5.

§ 148.5 Waste analysis.

Generators of hazardous wastes that are disposed of into Class I injection wells must comply with the applicable requirements of §268.7 (a) and (b). Owners or operators of Class I hazardous waste injection wells must comply with the applicable requirements of §268.7(c).

Subpart B—Prohibitions on Injection

§ 148.10 Waste specific prohibitions—solvent wastes.

(a) Effective August 8, 1988, the spent solvent wastes specified in §261.31 as EPA Hazardous Waste Nos. F001, F002, F003, F004, and F005 are prohibited

from underground injection unless the solvent waste is a solvent-water mixture or solvent-containing sludge containing less than 1 percent total F001–F005 solvent constituents listed in Table A of this section.

(b) Effective August 8, 1990, all spent F001–F005 solvent wastes containing less than 1 percent total F001–F005 solvent constituents listed in Table A of this section are prohibited from injection.

(c) Effective August 8, 1990, all spent F002 and F005 wastes containing solvent constituents listed in Table B of this section are prohibited from underground injection at off-site injection facilities.

(d) Effective November 8, 1990, the wastes specified in paragraph (c) of this section are prohibited from underground injection at on-site injection facilities.

(e) The requirements of paragraphs (a) and (b) of this section do not apply:

(1) If the wastes meet or are treated to meet the applicable standards specified in subpart D of part 268; or

(2) If an exemption from a prohibition has been granted in response to a petition under subpart C of this part; or

(3) During the period of extension of the applicable effective date, if an extension has been granted under §148.4 of this part.

TABLE A

Acetone
n-Butyl alcohol
Carbon disulfide
Carbon tetrachloride
Chlorobenzene
Cresols and cresylic acid
Cyclohexanone
1,2-dichlorobenzene
Ethyl acetate
Ethyl benzene
Ethyl ether
Isobutanol
Methanol
Methylene chloride
Methylene chloride (from the pharmaceutical industry)
Methyl ethyl ketone
Methyl isobutyl ketone
Nitrobenzene
Pyridine
Tetrachloroethylene
Toulene
1,1,1-Trichloroethane
1,2,2-Trichloro-1,2,2-trifluoroethane
Trichloroethylene
Trichlorofluoromethane
Xylene

TABLE B

Benzene
2-Ethoxyethanol
2-Nitropropane
1,1,2-Trichloroethane

[53 FR 28154, July 26, 1988, as amended at 54 FR 25422, June 14, 1989; 56 FR 3876, Jan. 31, 1991; 57 FR 8088, Mar. 6, 1992]

§148.11 Waste specific prohibitions—dioxin-containing wastes.

(a) Effective August 8, 1988, the dioxin-containing wastes specified in §261.31 as EPA Hazardous Waste Nos. F020, F021, F022, F023, F026, F027, and F028, and prohibited from underground injection.

(b) The requirements of paragraph (a) of this section do not apply:

(1) If the wastes meet or are treated to meet the applicable standards specified in subpart D of part 268; or

(2) If an exemption from a prohibition has been granted in response to a petition under subpart C of this part; or

(3) During the period of extension of the applicable effective date, if an extension has been granted under §148.4 of this part.

[53 FR 28154, July 26, 1988, as amended at 54 FR 25422, June 14, 1989]

§148.12 Waste specific prohibitions—California list wastes.

(a) Effective August 8, 1988, the hazardous wastes listed in 40 CFR 268.32 containing polychlorinated biphenyls at concentrations greater than or equal to 50 ppm or halogenated organic compounds at concentrations greater than or equal to 10,000 mg/kg are prohibited from underground injection.

(b) Effective August 8, 1990, the following hazardous wastes are prohibited from underground injection:

(1) Liquid hazardous wastes, including free liquids associated with any solid or sludge, containing free cyanides at concentrations greater than or equal to 1,000 mg/l;

(2) Liquid hazardous wastes, including free liquids associated with any solid or sludge, containing the following metals (or elements) or compounds

of these metals (or elements) at concentrations greater than or equal to those specified below:

(i) Arsenic and/or compounds (as As) 500 mg/l;

(ii) Cadmium and/or compounds (as Cd) 100 mg/l;

(iii) Chromium (VI) and/or compounds (as Cr VI) 500 mg/l;

(iv) Lead and/or compounds (as Pb) 500 mg/l;

(v) Mercury and/or compounds (as Hg) 20 mg/l;

(vi) Nickel and/or compounds (as Ni) 134 mg/l;

(vii) Selenium and/or compounds (as Se) 100 mg/l; and

(viii) Thallium and/or compounds (as Tl) 130 mg/l;

(3) Liquid hazardous waste having a pH less than or equal to two (2.0); and

(4) Hazardous wastes containing halogenated organic compounds in total concentration less than 10,000 mg/kg but greater than or equal to 1,000 mg/kg.

(c) The requirements of paragraphs (a) and (b) of this section do not apply:

(1) If the wastes meet or are treated to meet the applicable standards specified in subpart D of part 268; or

(2) If an exemption from a prohibition has been granted in response to a petition under subpart C of this part; or

(3) During the period of extension of the applicable effective date, if an extension is granted under § 148.4 of this part.

[53 FR 30918, Aug. 16, 1988, as amended at 53 FR 41602, Oct. 24, 1988]

§ 148.14 Waste specific prohibitions—first third wastes.

(a) Effective June 7, 1989, the wastes specified in 40 CFR 261.31 as EPA Hazardous Waste numbers F006 (nonwastewaters) and the wastes specified in 40 CFR 261.32 as EPA Hazardous Waste numbers K001, K015 (wastewaters), K016 (at concentrations greater than or equal to 1%), K018, K019, K020, K021 (nonwastewaters generated by the process described in the waste listing description and disposed after August 17, 1988, and not generated in the course of treating wastewater forms of these wastes), K022 (nonwastewaters), K024, K030, K036 (nonwastewaters generated by the process described in the waste listing description and disposed after August 17, 1988, and not generated in the course of treating wastewater forms of these wastes), K037, K044, K045, nonexplosive K046 (nonwastewaters), K047, K048, K060 (nonwastewaters generated by the process described in the waste listing description and disposed after August 17, 1988, and not generated in the course of treating wastewater forms of these wastes), K061 (nonwastewaters), noncalcium sulfate K069 (nonwastewaters generated by the process described in the waste listing description and disposed after August 17, 1988, and not generated in the course of treating wastewater forms of these wastes), K086 solvent washes, K087, K099, K101 (all wastewaters and less than 1% total arsenic nonwastewaters), K102 (all wastewaters and less than 1% total arsenic nonwastewaters), and K103 are prohibited from underground injection.

(b) Effective June 8, 1989, the waste specified in 40 CFR 261.32 as EPA Hazardous Waste number K036 (wastewaters); and the wastes specified in 40 CFR 261.33 as P030, P039, P041, P063, P071, P089, P094, P097, U221, and U223 are prohibited from underground injection.

(c) Effective July 8, 1989, the wastes specified in 40 CFR 261.31 as EPA Hazardous Waste numbers F008 and F009 are prohibited from underground injection.

(d) Effective August 8, 1990, the wastes specified in 40 CFR 261.31 as EPA Hazardous Waste Number F006 (wastewaters) and F019; the wastes specified in 40 CFR 261.32 as EPA Hazardous Waste Numbers K004, K008, K015 (nonwastewaters), K017, K021 (wastewaters), K022 (wastewaters), K031, K035, K046 (reactive nonwastewaters and all wastewaters), K060 (wastewaters), K061 (wastewaters), K069 (calcium sulfate nonwastewaters and all wastewaters), K073, K083, K084, K085, K086 (all but solvent washes), K101 (high arsenic nonwastewaters), K102 (high arsenic nonwastewaters), and K106; and the wastes specified in 40 CFR part 261.33 as EPA Hazardous Waste Numbers P001, P004, P005, P010, P011, P012, P015, P016, P018, P020, P036, P037, P048, P050, P058, P059, P068, P069,

P070, P081, P082, P084, P087, P092, P102, P105, P108, P110, P115, P120, P122, P123, U007, U009, U010, U012, U016, U018, U019, U022, U029, U031, U036, U037, U041, U043, U044, U046, U050, U051, U053, U061, U063, U064, U066, U067, U074, U077, U078, U086, U089, U103, U105, U108, U115, U122, U124, U129, U130, U133, U134, U137, U151, U154, U155, U157, U158, U159, U171, U177, U180, U185, U188, U192, U200, U209, U210, U211, U219, U220, U226, U227, U228, U237, U238, U248, and U249 are prohibited from underground injection at off-site injection facilities.

(e) Effective August 8, 1990, the wastes specified in 40 CFR 261.32 as EPA Hazardous Waste numbers K049, K050, K051, K052, K062, K071, and K104 are prohibited from underground injection.

(f) Effective November 8, 1990, the wastes specified in paragraph (d) of this section are prohibited from underground injection at on-site injection facilities.

(g) Effective June 7, 1991, the wastes specified in 40 CFR 261.32 as EPA Hazardous Waste numbers K016 (at concentrations less than 1%) are prohibited from underground injection.

(h) Effective June 8, 1991, the waste specified in 40 CFR 261.31 as EPA Hazardous Waste number F007; and the wastes specified in 40 CFR 261.32 as K011 (nonwastewaters) and K013 (nonwastewaters) are prohibited from underground injection.

(i) Effective May 8, 1992, the wastes specified in 40 CFR 261.32 and 261.33 as EPA Hazardous Waste Numbers K011 (wastewaters), K013 (wastewaters), and K014 are prohibited from underground injection.

(j) The requirements of paragraphs (a) through (i) of this section do not apply:

(1) If the wastes meet or are treated to meet the applicable standards specified in subpart D of part 268; or

(2) If an exemption from a prohibition has been granted in response to a petition under subpart C of this part; or

(3) During the period of extension of the applicable effective date, if an extension has been granted under §148.4 of this part.

[54 FR 25423, June 14, 1989, as amended at 54 FR 26647, June 23, 1989; 54 FR 35328, Aug. 25, 1989; 55 FR 22683, June 1, 1990]

§148.15 Waste specific prohibitions—Second third wastes.

(a) Effective June 7, 1989, the wastes specified in 40 CFR 261.32 as EPA Hazardous Waste numbers K025 (nonwastewaters generated by the process described in the waste listing description and disposed after August 17, 1988, and not generated in the course of treating wastewater forms of these wastes) are prohibited from underground injection.

(b) Effective June 8, 1989, the wastes specified in 40 CFR 261.31 as EPA Hazardous Waste numbers F010, F024; the wastes specified in 40 CFR 261.32 as K009 (nonwastewaters), K010, K027, K028, K029 (nonwastewaters), K038, K039, K040, K043, K095 (nonwastewaters), K096 (nonwastewaters), K113, K114, K115, K116; and wastes specified in 40 CFR 261.33 as P029, P040, P043, P044, P062, P074, P085, P098, P104, P106, P111, U028, U058, U107, and U235 are prohibited from underground injection.

(c) Effective July 8, 1989, and continuing until December 8, 1989, the wastes specified in 40 CFR 261.31 as EPA Hazardous Waste numbers F011 and F012 are prohibited from underground injection pursuant to the treatment standards specified in §§268.41 and 268.43 applicable to F007, F008, and F009 wastewaters and nonwastewaters. Effective December 8, 1989, F011 (nonwastewaters) and F012 (nonwastewaters) are prohibited pursuant to the treatment standards specified in §§268.41 and 268.43 applicable to F011 and F012 wastewaters and nonwastewaters.

(d) Effective August 8, 1990, the wastes specified in 40 CFR 261.32 as EPA Hazardous Waste Number K025 (wastewaters), K029 (wastewaters), K041, K042, K095 (wastewaters), K096 (wastewaters), K097, K098, and K105; and the wastes specified in 40 CFR part 261.33 as P002, P003, P007, P008, P014, P026, P027, P049, P054, P057, P060, P066, P067, P072, P107, P112, P113, P114, U002, U003, U005, U008, U011, U014, U015, U020,

U021, U023, U025, U026, U032, U035, U047, U049, U057, U059, U060, U062, U070, U073, U080, U083, U092, U093, U094, U095, U097, U098, U099, U101, U106, U109, U110, U111, U114, U116, U119, U127, U128, U131, U135, U138, U140, U142, U143, U144, U146, U147, U149, U150, U161, U162, U163, U164, U165, U168, U169, U170, U172, U173, U174, U176, U178, U179, U189, U193, U196, U203, U205, U206, U208, U213, U214, U215, U216, U217, U218, U239, and U244 are prohibited from underground injection at off-site injection facilities.

(e) Effective June 8, 1991, the waste specified in 40 CFR 261.32 as EPA Hazardous Waste number K009 (wastewaters) is prohibited from underground injection.

(f) Effective November 8, 1990, the wastes specified in paragraph (d) of this section are prohibited from underground injection at on-site injection facilities.

(g) The requirements of paragraphs (a) through (f) of this section do not apply:

(1) If the wastes meet or are treated to meet the applicable standards specified in subpart D of part 268; or

(2) If an exemption from a prohibition has been granted in response to a petition under subpart C of this part; or

(3) During the period of extension of the applicable effective date, if an extension has been granted under § 148.4 of this part.

[54 FR 25423, June 14, 1989, as amended at 54 FR 26647, June 23, 1989; 55 FR 22683, June 1, 1990]

§ 148.16 Waste specific prohibitions—third third wastes.

(a) Effective June 7, 1989, the wastes specified in 40 CFR 261.32 as EPA Hazardous Waste numbers K100 (nonwastewaters generated by the process described in the waste listing description and disposed after August 17, 1988, and not generated in the course of treating wastewater forms of these wastes) are prohibited from underground injection.

(b) Effective June 8, 1989, the wastes specified in 40 CFR 261.32 as EPA Hazardous Waste numbers K005 (nonwastewaters), K007 (nonwastewaters), K023, K093, K094; and the wastes specified in 40 CFR 261.33 as P013, P021, P099, P109, P121, U069, U087, U088, U102, and U190 are prohibited from underground injection.

(c) Effective August 8, 1990, the wastes identified in 40 CFR 261.31 as EPA Hazardous Waste Number F039 (nonwastewaters); the wastes specified in 40 CFR 261.32 as EPA Hazardous Waste Numbers K002, K003, K005 (wastewaters), K006, K007 (wastewaters), K026, K032, K033, K034, and K100 (wastewaters); the wastes specified in 40 CFR 261.33 as P006, P009, P017, P022, P023, P024, P028, P031, P033, P034, P038, P042, P045, P046, P047, P051, P056, P064, P065, P073, P075, P076, P077, P078, P088, P093, P095, P096, P101, P103, P116, P118, P119, U001, U004, U006, U017, U024, U027, U030, U033, U034, U038, U039, U042, U045, U048, U052, U055, U056, U068, U071, U072, U075, U076, U079, U081, U082, U084, U085, U090, U091, U096, U112, U113, U117, U118, U120, U121, U123, U125, U126, U132, U136, U141, U145, U148, U152, U153, U156, U160, U166, U167, U181, U182, U183, U184, U186, U187, U191, U194, U197, U201, U202, U204, U207, U222, U225, U234, U236, U240, U243, U246, and U247; and the wastes identified in 40 CFR 261.21, 261.23 or 261.24 as hazardous based on a characteristic alone, designated as D001, D004, D005, D006, D008, D009 (wastewaters), D010, D011, D012, D013, D014, D015, D016, D017, and newly listed waste F025 are prohibited from underground injection at off-site injection facilities.

(d) Effective August 8, 1990, mixed radioactive/hazardous waste in 40 CFR 268.10, 268.11, and 268.12, that are mixed radioactive and hazardous wastes, are prohibited from underground injection.

(e) Effective November 8, 1990, the wastes specified in paragraph (c) of this section are prohibited from underground injection at on-site injection facilities. These effective dates do not apply to the wastes listed in 40 CFR 148.12(b) which are prohibited from underground injection on August 8, 1990.

(f) Effective May 8, 1992, the waste identified in 40 CFR 261.31 as EPA Hazardous Waste Number F039 (wastewaters); the wastes identified in 40 CFR 261.22, 261.23 or 261.24 as hazardous based on a characteristic alone, designated as D002 (wastewaters and nonwastewaters), D003 (wastewaters and nonwastewaters), D007 (wastewaters

and nonwastewaters), and D009 (nonwastewaters) are prohibited from underground injection. These effective dates do not apply to the wastes listed in 40 CFR 148.12(b) which are prohibited from underground injection on August 8, 1990.

(g) The requirements of paragraphs (a) through (f) of this section do not apply:

(1) If the wastes meet or are treated to meet the applicable standards specified in subpart D of part 268; or

(2) If an exemption from a prohibition has been granted in response to a petition under subpart C of this part; or

(3) During the period of extension of the applicable effective date, if an extension has been granted under §148.4 of this part.

[54 FR 25423, June 14, 1989, as amended at 54 FR 26647, June 23, 1989; 55 FR 22683, June 1, 1990; 55 FR 33694, Aug. 17, 1990; 56 FR 3876, Jan. 31, 1991]

§148.17 Waste specific prohibitions; newly listed wastes.

(a) Effective November 9, 1992, the wastes specified in 40 CFR part 261 as EPA hazardous waste numbers F037, F038, K107, K108, K109, K110, K111, K112, K117, K118, K123, K124, K125, K126, K131, K136, U328, U353, and U359 are prohibited from underground injection.

(b) Effective December 19, 1994 the wastes specified in 40 CFR 261.32 as EPA Hazardous waste numbers K141, K142, K143, K144, K145, K147, K148, K149, K150, and K151, are prohibited from underground injection.

(c) [Reserved]

(d) Effective June 30, 1995, the wastes specified in 40 CFR part 261 as EPA Hazardous waste numbers K117, K118, K131, and K132 are prohibited from underground injection.

(e) The requirements of paragraphs (a) and (b) of this section do not apply:

(1) If the wastes meet or are treated to meet the applicable standards specified in subpart D of part 268; or

(2) If an exemption from a prohibition has been granted in response to a petition under subpart C of this part; or

(3) During the period of extension of the applicable effective date, if an extension has been granted under §148.4 of this part.

[57 FR 37263, Aug. 18, 1992, as amended at 59 FR 48041, Sept. 19, 1994; 61 FR 15662, Apr. 8, 1996]

§148.18 Waste specific prohibitions—newly listed and identified wastes.

(a) Effective August 11, 1997, the wastes specified in 40 CFR part 261 as EPA Hazardous waste numbers F032, F034, F035 are prohibited from underground injection.

(b) Effective May 12, 1999, the wastes specified in 40 CFR part 261 as EPA Hazardous waste numbers F032, F034, F035 that are mixed with radioactive wastes are prohibited from underground injection.

(c) On July 8, 1996, the wastes specified in 40 CFR 261.32 as EPA Hazardous waste numbers K156–K161, P127, P128, P185, P188–P192, P194, P196–P199, P201–P205, U271, U277–U280, U364–U367, U372, U373, U375–U379, U381–387, U389–U396, U400–U404, U407, and U409–U411 are prohibited from underground injection.

(d) On January 8, 1997, the wastes specified in 40 CFR 261.32 as EPA Hazardous waste number K088 is prohibited from underground injection.

(e) On April 8, 1998, the wastes specified in 40 CFR part 261 as EPA Hazardous waste numbers D018–043, and Mixed TC/Radioactive wastes, are prohibited from underground injection.

[61 FR 15662, Apr. 8, 1996, as amended at 62 FR 26018, May 12, 1997]

EFFECTIVE DATE NOTE: At 62 FR 26018, May 12, 1997, §148.18 was amended by revising the heading and redesignating paragraphs (a) through (c) as (c) through (e) respectively, and adding a new paragraph (a) effective Aug. 11, 1997. A new paragraph (b) was added, effective May 12, 1999.

Subpart C—Petition Standards and Procedures

§148.20 Petitions to allow injection of a waste prohibited under subpart B.

(a) Any person seeking an exemption from a prohibition under subpart B of this part for the injection of a restricted hazardous waste into an injection well or wells shall submit a petition to the Director demonstrating

that, to a reasonable degree of certainty, there will be no migration of hazardous constituents from the injection zone for as long as the waste remains hazardous. This demonstration requires a showing that:

(1) The hydrogeological and geochemical conditions at the sites and the physiochemical nature of the waste stream(s) are such that reliable predictions can be made that:

(i) Fluid movement conditions are such that the injected fluids will not migrate within 10,000 years:

(A) Vertically upward out of the injection zone; or

(B) Laterally within the injection zone to a point of discharge or interface with an Underground Source of Drinking Water (USDW) as defined in 40 CFR part 146; or

(ii) Before the injected fluids migrate out of the injection zone or to a point of discharge or interface with USDW, the fluid will no longer be hazardous because of attenuation, transformation, or immobilization of hazardous constituents within the injection zone by hydrolysis, chemical interactions or other means; and

(2) For each well the petition has:

(i) Demonstrated that the injection well's area of review complies with the substantive requirements of § 146.63;

(ii) Located, identified, and ascertained the condition of all wells within the injection well's area of review (as specified in § 146.63) that penetrate the injection zone or the confining zone by use of a protocol acceptable to the Director that meets the substantive requirements of § 146.64;

(iii) Submitted a corrective action plan that meets the substantive requirements of § 146.64, the implementation of which shall become a condition of petition approval; and

(iv) Submitted the results of pressure and radioactive tracer tests performed within one year prior to submission of the petition demonstrating the mechanical integrity of the well's long string casing, injection tube, annular seal, and bottom hole cement. In cases where the petition has not been approved or denied within one year after the initial demonstration of mechanical integrity, the Director may require the owner or operator to perform the tests again and submit the results of the new tests.

NOTE: The requirements of § 148.20(a)(2) need not be incorporated in a permit at the time of petition approval.

(b) A demonstration under § 148.20(a)(1)(i) shall identify the strata within the injection zone which will confine fluid movement above the injection interval and include a showing that this strata is free of known transmissive faults of fractures and that there is a confining zone above the injection zone.

(c) A demonstration under § 148.20(a)(1)(ii) shall identify the strata within the injection zone where waste transformation will be accomplished and include a showing that this strata is free of known transmissive faults or fractures and that there is a confining zone above the injection zone.

(d) A demonstration may include a showing that:

(1) Treatment methods, the implementation of which shall become a condition of petition approval, will be utilized that reduce the toxicity or mobility of the wastes; or

(2) A monitoring plan, the implementation of which shall become a condition of petition approval, will be utilized to enhance confidence in one or more aspects of the demonstration.

(e) Any person who has been granted an exemption pursuant to this section may submit a petition for reissuance of the exemption to include an additional restricted waste or wastes or to modify any conditions placed on the exemption by the Director. The Director shall reissue the petition if the petitioner complies with the requirements of paragraphs (a), (b) and (c) of this section.

(f) Any person who has been granted an exemption pursuant to this section may submit a petition to modify an exemption to include an additional (hazardous) waste or wastes. The Director may grant the modification if he determines, to a reasonable degree of certainty, that the additional waste or wastes will behave hydraulically and chemically in a manner similar to previously included wastes and that it will not interfere with the containment capability of the injection zone.

§148.21 Information to be submitted in support of petitions.

(a) Information submitted in support of §148.20 must meet the following criteria:

(1) All waste analysis and any new testing performed by the petitioner shall be accurate and reproducible and performed in accordance with quality assurance standards;

(2) Estimation techniques shall be appropriate, and EPA-certified test protocols shall be used where available and appropriate;

(3) Predictive models shall have been verified and validated, shall be appropriate for the specific site, waste streams, and injection conditions of the operation, and shall be calibrated for existing sites where sufficient data are available;

(4) An approved quality assurance and quality control plan shall address all aspects of the demonstration;

(5) Reasonably conservative values shall be used whenever values taken from the literature or estimated on the basis of known information are used instead of site-specific measurements; and

(6) An analysis shall be performed to identify and assess aspects of the demonstration that contribute significantly to uncertainty. The petitioner shall conduct a sensitivity analysis to determine the effect that significant uncertainty may contribute to the demonstration. The demonstration shall then be based on conservative assumptions identified in the analysis.

(b) Any petitioner under §148.20(a)(1)(i) shall provide sufficient site-specific information to support the demonstration, such as:

(1) Thickness, porosity, permeability and extent of the various strata in the injection zone;

(2) Thickness, porosity, permeability, extent, and continuity of the confining zone;

(3) Hydraulic gradient in the injection zone;

(4) Hydrostatic pressure in the injection zone; and

(5) Geochemical conditions of the site.

(c) In addition to the information in §148.21(b), any petitioner under §148.20(a)(1)(ii) shall provide sufficient waste-specific information to ensure reasonably reliant predictions about the waste transformation. The petitioner shall provide the information necessary to support the demonstration, such as:

(1) Description of the chemical processes or other means that will lead to waste transformation; and

(2) Results of laboratory experiments verifying the waste transformation.

§148.22 Requirements for petition submission, review and approval or denial.

(a) Any petition submitted to the Director pursuant to §148.20(a) shall include the following components:

(1) An identification of the specific waste or wastes and the specific injection well or wells for which the demonstration will be made;

(2) A waste analysis to describe fully the chemical and physical characteristics of the subject wastes;

(3) Such additional information as is required by the Director to support the petition under §§148.20 and 148.21; and

(4) This statement signed by the petitioner or an authorized representative:

> I certify under penalty of law that I have personally examined and am familiar with the information submitted in this petition and all attached documents, and that, based on my inquiry of those individuals immediately responsible for obtaining the information, I believe that submitted information is true, accurate, and complete. I am aware that there are significant penalties for submitting false information, including the possibility of fine and imprisonment.

(b) The Director shall provide public notice and an opportunity for public comment in accordance with the procedures in §124.10 of the intent to approve or deny a petition. The final decision on a petition will be published in the FEDERAL REGISTER.

(c) If an exemption is granted it will apply only to the underground injection of the specific restricted waste or wastes identified in the petition into a Class I hazardous waste injection well or wells specifically identified in the petition (unless the exemption is modified or reissued pursuant to §148.20(e) or (f).

(d) Upon request by any petitioner who obtains an exemption for a well

under this subpart, the Director shall initiate and reasonably expedite the necessary procedures to issue or reissue a permit or permits for the hazardous waste well or wells covered by the exemption for a term not to exceed ten years.

§ 148.23 Review of exemptions granted pursuant to a petition.

(a) When considering whether to reissue a permit for the operation of a Class I hazardous waste injection well, the Director shall review any petition filed pursuant to § 148.20 and require a new demonstration if information shows that the basis for granting the exemption may no longer be valid.

(b) Whenever the Director determines that the basis for approval of a petition may no longer be valid, the Director shall require a new demonstration in accordance with § 148.20.

§ 148.24 Termination of approved petition.

(a) The Director may terminate an exemption granted under § 148.20 for the following causes:

(1) Noncompliance by the petitioner with any condition of the exemption;

(2) The petitioner's failure in the petition or during the review and approval to disclose fully all relevant facts, or the petitioner's misrepresentation of any relevant facts at any time; or

(3) A determination that new information shows that the basis for approval of the petition is no longer valid.

(b) The Director shall terminate an exemption granted under § 148.20 for the following causes:

(1) The petitioner's willful withholding during the review and approval of the petition of facts directly and materially relevant to the Director's decision on the petition;

(2) A determination that there has been migration from the injection zone or the well that is not in accordance with the terms of the exemption, except that the Director may at his discretion decide not to terminate where:

(i) The migration resulted from a mechanical failure of the well that can be corrected promptly through a repair to the injection well itself or from an undetected well or conduit that can be plugged promptly; and

(ii) The requirements of § 146.67(i) are satisfied.

(c) The Director shall follow the procedures in § 124.5 in terminating any exemption under this section.

PART 149—SOLE SOURCE AQUIFERS

Subpart A—Criteria for Identifying Critical Aquifer Protection Areas

Sec.
149.1 Purpose.
149.2 Definitions.
149.3 Critical Aquifer Protection Areas.

Subpart B—Review of Projects Affecting the Edwards Underground Reservoir, A Designated Sole Source Aquifer in the San Antonio, Texas Area

149.100 Applicability.
149.101 Definitions.
149.102 Project review authority.
149.103 Public information.
149.104 Submission of petitions.
149.105 Decision to review.
149.106 Notice of review.
149.107 Request for information.
149.108 Public hearing.
149.109 Decision under section 1424(e).
149.110 Resubmittal of redesigned projects.
149.111 Funding to redesigned projects.

AUTHORITY: Sec. 1424(e), Safe Drinking Water Act (42 U.S.C. 300h–3(e); sec. 1427 of the Safe Drinking Water Act, (42 U.S.C. 300h–6).

Subpart A—Criteria for Identifying Critical Aquifer Protection Areas

SOURCE: 52 FR 23986, June 26, 1987, unless otherwise noted.

§ 149.1 Purpose.

The purpose of this subpart is to provide criteria for identifying critical aquifer protection areas, pursuant to section 1427 of the Safe Drinking Water Act (SDWA).

§ 149.2 Definitions.

(a) *Aquifer* means a geological formation, group of formations, or part of a formation that is capable of yielding a significant amount of water to a well or spring.

(b) *Recharge* means a process, natural or artificial, by which water is added to the saturated zone of an aquifer.

(c) *Recharge Area* means an area in which water reaches the zone of saturation (ground water) by surface infiltration; in addition, a *major recharge area* is an area where a major part of the recharge to an aquifer occurs.

(d) *Sole or Principal Source Aquifer* (SSA) means an aquifer which is designated as an SSA under section 1424(e) of the SDWA.

[54 FR 6843, Feb. 14, 1989]

§ 149.3 Critical Aquifer Protection Areas.

A Critical Aquifer Protection Area is either:

(a) All or part of an area which was designated as a sole or principal source aquifer prior to June 19, 1986, and for which an areawide ground-water quality protection plan was approved, under section 208 of the Clean Water Act, prior to that date; or

(b) All or part of a major recharge area of a sole or principal source aquifer, designated before June 19, 1988, for which:

(1) The sole or principal source aquifer is particularly vulnerable to contamination due to the hydrogeologic characteristics of the unsaturated or saturated zone within the suggested critical aquifer protection area; and

(2) Contamination of the sole or principal source aquifer is reasonably likely to occur, unless a program to reduce or prevent such contamination is implemented; and

(3) In the absence of any program to reduce or prevent contamination, reasonably foreseeable contamination would result in significant cost, taking into account:

(i) The cost of replacing the drinking water supply from the sole or principal source aquifer, and

(ii) Other economic costs and environmental and social costs resulting from such contamination.

[54 FR 6843, Feb. 14, 1989]

Subpart B—Review of Projects Affecting the Edwards Underground Reservoir, A Designated Sole Source Aquifer in the San Antonio, Texas Area

SOURCE: 42 FR 51574, Sept. 29, 1977, unless otherwise noted. Redesignated at 52 FR 23986, June 26, 1987.

§ 149.100 Applicability.

This subpart sets forth, pursuant to sections 1424(e) and 1450 of the Public Health Service Act, as amended by the Safe Drinking Water Act, Pub. L. 93–523, regulations relating the Edwards Underground Reservoir which is the sole or principal drinking water source for the San Antonio area and which, if contaminated, would create a significant hazard to public health.

[42 FR 51574, Sept. 29, 1977. Redesignated and amended at 52 FR 23986, June 26, 1987]

§ 149.101 Definitions.

As used in this subpart and except as otherwise specifically provided, the term(s):

(a) *Act* means the Public Health Service Act, as amended by the Safe Drinking Water Act, Public Law 93–523.

(b) *Contaminant* means any physical, chemical, biological, or radiological substance or matter in water.

(c) *Recharge zone* means the area through which water enters the Edwards Underground Reservoir as defined in the December 16, 1975, Notice of Determination.

(d) *Administrator* (Regional Administrator) means the Administrator (Regional Administrator) of the United States Environmental Protection Agency.

(e) *Person* means an individual, corporation, company, association, partnership, State, or municipality.

(f) *Project* means a program or action for which an application for Federal financial assistance has been made.

(g) *Federal financial assistance* means any financial benefits provided directly as aid to a project by a department, agency, or instrumentality of the Federal government in any form including contracts, grants, and loan guarantees.

Actions or programs carried out by the Federal government itself such as dredging performed by the Army Corps of Engineers do not involve Federal financial assistance. Actions performed for the Federal government by contractors, such as construction of roads on Federal lands by a contractor under the supervision of the Bureau of Land Management, should be distinguished from contracts entered into specifically for the purpose of providing financial assistance, and will not be considered programs or actions receiving Federal financial assistance. Federal financial assistance is limited to benefits earmarked for a specific program or action and directly awarded to the program or action. Indirect assistance, e.g., in the form of a loan to a developer by a lending institution which in turn receives Federal assistance not specifically related to the project in question is not Federal financial assistance under section 1424(e).

(h) *Commitment of Federal financial assistance* means a written agreement entered into by a department, agency, or instrumentality of the Federal Government to provide financial assistance as defined in paragraph (g) of this section. Renewal of a commitment which the issuing agency determines has lapsed shall not constitute a new commitment unless the Regional Administrator determines that the project's impact on the aquifer has not been previously reviewed under section 1424(e). The determination of a Federal agency that a certain written agreement constitutes a commitment shall be conclusive with respect to the existence of such a commitment.

(i) *Streamflow source zone* means the upstream headwaters area which drains into the recharge zone as defined in the December 16, 1975, Notice of Determination.

(j) *Significant hazard to public health* means any level of contaminant which causes or may cause the aquifer to exceed any maximum contaminant level set forth in any promulgated National Primary Drinking Water Standard at any point where the water may be used for drinking purposes or which may otherwise adversely affect the health of persons, or which may require a public water system to install additional treatment to prevent such adverse effect.

(k) *Aquifer* means the Edwards Underground Reservoir.

[42 FR 51574, Sept. 29, 1977. Redesignated and amended at 52 FR 23986, June 26, 1987]

§ 149.102 Project review authority.

(a) Once an area is designated, no subsequent commitments of Federal financial assistance may be made to projects which the Administrator determines may contaminate the aquifer so as to create a significant hazard to public health.

(b) The Regional Administrator is hereby delegated the authority and assigned responsibility for carrying out the project review process assigned to the Administrator under section 1424(e) of the Act, except the final determination that a project may contaminate the aquifer through its recharge zone so as to create a significant hazard to public health.

(c) The Regional Administrator may review any project which he considers may potentially contaminate the aquifer through its recharge zone so as to create a significant hazard to public health.

§ 149.103 Public information.

After the area is designated under section 1424(e), Federal agencies, for projects, located in the recharge zone and streamflow source zones, are required to:

(a) Maintain a list of projects for which environmental impact statements will be prepared in accordance with the National Environmental Policy Act (NEPA);

(b) Revise the list at regular intervals and submit to EPA; and

(c) Make the list available to the public upon request.

§ 149.104 Submission of petitions.

Any person may submit a petition requesting the Regional Administrator to review a project to determine if such project may contaminate the aquifer through its recharge zone so as to create a significant hazard to public health. Any such petition shall identify:

(a) The name, address, and telephone number of the individual, organization,

or other entity submitting the petition;

(b) A brief statement of the requesting person's interest in the Regional Administrator's determination;

(c) The name of the project and Federal agency involved;

In addition, the petitioner is requested to submit to EPA available information on:

(d) Applicable action already taken by State and local agencies including establishment of regulations to prevent contamination of the aquifer and why, in the petitioner's judgment, the action was inadequate.

(e) Any actions taken under the National Environmental Policy Act and why, in the petitioner's judgment, that action was inadequate in regard to evaluation of potential effect on the aquifer.

(f) The potential contaminants involved;

(g) The means by which the contaminant might enter the aquifer; and

(h) The potential impact of the proposed project.

§ 149.105 Decision to review.

(a) The Regional Administrator shall review under section 1424(e) all projects located in the recharge or streamflow source zone of the aquifer for which a draft or final EIS is submitted which may have an impact on ground water quality and which involve Federal financial assistance as defined in these regulations.

(b) Upon receipt of a public petition, the Regional Administrator shall decide whether the project which is the subject of the petition should be reviewed under section 1424(e).

(c) The Regional Administrator may decide to review a project upon his own motion.

(d) In determining whether to review a project upon receipt of a public petition or upon his own motion, the Regional Administrator shall consider whether the project is likely to directly or indirectly cause contamination of the aquifer through its recharge zone, taking into account any factors he deems relevant, including:

(1) The location of the project, and

(2) The nature of the project.

(e) In determining whether to review a project upon receipt of a public petition or upon his own motion, the Regional Administrator may consult with, or request information from, the Federal agency to which the project application has been made, the applicant seeking Federal assistance, appropriate State and local agencies, and other appropriate persons or entities.

(f) In determining whether to review a project which is the subject of a public petition, the Regional Administrator may request such additional information from the petitioner as he deems necessary.

§ 149.106 Notice of review.

(a) *Notice to Federal agency.* If the Regional Administrator decides upon receipt of a public petition or upon his own motion to review a project under section 1424(e), he shall give written notification of the decision to the Federal agency from which financial assistance is sought. The notification shall include a description and identification of the project.

(b) *Notice to public.* When the Regional Administrator undertakes to review a project pursuant to § 149.13 above, he shall provide public notice of project review by such means as he deems appropriate. The notice shall set forth the availability for public review of all data and information available, and shall solicit comments, data and information with respect to the determination of impact under section 1424(e). The period for public comment shall be 30 days after public notice unless the Regional Administrator extends the period at his discretion or a public hearing is held under § 149.16.

§ 149.107 Request for information.

In reviewing a project under section 1424(e), the Regional Administrator may request any additional information from the funding Federal agency which is pertinent to reaching a decision. If full evaluation of the groundwater impact of a project has not been submitted in accordance with the agency's NEPA procedures, the Regional Administrator may specifically request that the Federal agency submit a groundwater impact evaluation of

whether the proposed project may contaminate the aquifer through its recharge zone so as to create a significant hazard to public health.

§ 149.108 Public hearing.

If there is significant public interest, the Regional Administrator may hold a public hearing with respect to any project or projects to be reviewed if he finds that such a hearing is necessary and would be helpful in clarifying the issues. Public hearings held under this section should be coordinated, if possible, with other Federal public hearings held pursuant to applicable laws and regulations. Any such hearing shall be conducted by the Regional Administrator or designee in an informal, orderly and expeditious manner. Where appropriate, limits may be placed upon the time allowed for oral statements, and statements may be required to be submitted in writing. The record will be held open for further public comment for seven (7) days following the close of the public hearing.

§ 149.109 Decision under section 1424(e).

(a) As soon as practicable after the submission of public comments under section 1424(e) and information requested by the Environmental Protection Agency from the originating Federal agency, on the basis of such information as is available to him, the Regional Administrator shall review the project taking all relevant factors into account including:

(1) The extent of possible public health hazard presented by the project;

(2) Planning, design, construction, operation, maintenance and monitoring measures included in the project which would prevent or mitigate the possible health hazard;

(3) The extent and effectiveness of State or local control over possible contaminant releases to the aquifer;

(4) The cumulative and secondary impacts of the proposed project; and

(5) The expected environmental benefits of the proposed project.

(b) After reviewing the available information, the Regional Administrator shall:

(1) Determine that the risk of contamination of the aquifer through the recharge zone so as to create a significant hazard to public health is not sufficiently great so as to prevent commitment of Federal funding to the project; or

(2) Forward the information to the Administrator with his recommendation that the project may contaminate the aquifer through the recharge zone so as to create a significant hazard to public health.

(c) After receiving the available information forwarded by the Regional Administrator, the Administrator shall:

(1) Determine that the risk of contamination of the aquifer through the recharge zone so as to create a significant hazard to public health is not sufficiently great so as to prevent commitment of Federal funding to the project; or

(2) Determine that the project may contaminate the aquifer through the recharge zone so as to create a significant hazard to public health.

(d) Notice of any decisions by the Regional Administrator under paragraph (b)(1) of this section or by the Administrator under paragraphs (c)(1) and (2) of this section to prevent a commitment of Federal funding shall be published in the FEDERAL REGISTER. Such notices shall include a description of the propsed project, and a statement of decision with an accompanying statement of facts and reasons.

§ 149.110 Resubmittal of redesigned projects.

If a project is redesigned in response to EPA's objections, the applicant for Federal financial assistance or the grantor agency may file a petition with the Regional Administrator for withdrawal of the determination that the project may contaminate the aquifer through the recharge zone so as to create a significant hazard to public health. Any such petition shall demonstrate how the project has been redesigned so as to justify the withdrawal of EPA's objections. If appropriate, the Regional Administrator may request public comments or hold an informal public hearing to consider the petition. After review of pertinent information,

the Regional Administrator shall either deny the petition or recommend to the Administrator that the initial determination that a project may contaminate the aquifer be vacated. Upon receipt of a recommendation from the Regional Administrator that a determination be vacated, the Administrator shall either deny the petition or order that the initial determination be vacated. The final decision regarding a petition shall be published in the FEDERAL REGISTER with an accompanying statement of reasons.

§ 149.111 Funding to redesigned projects.

After publication of a decision that a proposed project may contaminate a sole or principal source aquifer in a designated area through its recharge zone so as to create a significant hazard to public health, a commitment for Federal financial assistance may be entered into, if authorized under another provision of law, to plan or redesign such project to assure that it will not so contaminate the aquifer.