Part VII—Sampling, Testing and Reporting Methods Part IX—Fees

Part IX—Fees

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[52 FR 43574, Nov. 13, 1987. Redesignated and amended at 70 FR 44480, Aug. 3, 2005]

APPENDIXES A-C TO PART 52 [RESERVED]

APPENDIX D TO PART 52—DETERMINA-TION OF SULFUR DIOXIDE EMISSIONS FROM STATIONARY SOURCES BY CON-TINUOUS MONITORS

1. Definitions.

1.1 Concentration Measurement System. The total equipment required for the continuous determination of SO_2 gas concentration in a given source effluent.

1.2 Span. The value of sulfur dioxide concentration at which the measurement system is set to produce the maximum data display output. For the purposes of this method, the span shall be set at the expected maximum sulfur dioxide concentration except as specified under section 5.2, Field Test for Accuracy.

1.3 Accuracy (Relative). The degree of correctness with which the measurement system yields the value of gas concentration of a sample relative to the value given by a defined reference method. This accuracy is expressed in terms of error which is the difference between the paired concentration measurements expressed as a percentage of the mean reference value.

1.4 Calibration Error. The difference between the pollutant concentration indicated by the measurement system and the known concentration of the test gas mixture.

1.5 Zero Drift. The change in measurement system output over a stated period of time of normal continuous operation when the pollutant concentration at the time for the measurement is zero.

1.6 Calibration Drift. The change in measurement system output over a stated period of time of normal continuous operation when the pollutant concentration at the time of the measurement is the same known upscale value.

1.7 Response Time. The time interval from a step change in pollutant concentration at the input to the measurement system to the time at which 95 percent of the corresponding final value is reached as dis1.8 Operational Period. A minimum period of time over which a measurement system is expected to operate within certain performance specifications without unscheduled maintenance, repair or adjustment.

1.9 Reference Method. The reference method for determination of SO_2 emissions shall be Method 8 as delineated in Part 60 of this chapter. The analytical and computational portions of Method 8 as they relate to determination of sulfuric acid mist and sulfur trioxide, as well as isokinetic sampling, may be omitted from the overall test procedure.

2. Principle and Applicability.

2.1 Principle. Gases are continuously sampled in the stack emissions and analyzed for sulfur dioxide by a continuously operating emission measurement system. Performance specifications for the continuous measurement systems are given. Test procedures are given to determine the capability of the measurement systems to conform to the performance specifications. Sampling may include either the extractive or nonextractive (in-situ) approach.

2.2 Applicability. The performance specifications are given for continuous sulfur dioxide measurement systems applied to nonferrous smelters.

3. Apparatus.

3.1 Calibration Gas Mixture. Mixture of a known concentrations of sulfur dioxide in oxygen-free nitrogen. Nominal volumetric concentrations of 50 percent and 90 percent of span are recommended. The mixture of 90 percent of span is to be used to set and to check the span and is referred to as the span gas. The gas mixtures shall be analyzed by the Reference Method at least two weeks prior to use or demonstrated to be accurate and stable by an alternate method subject to approval of the Administrator.

3.2 Zero Gas. A gas containing less than 1 ppm sulfur dioxide.

3.3 Equipment for measurement of sulfur dioxide concentration using the Reference Method.

3.4 *Chart Record.* Analog chart recorder, input voltage range compatible with analyzer system output.

3.5 Continuous measurement system for sulfur dioxide.

4. Measurement System Performance Specifications.

The following performance specifications shall be met in order that a measurement system shall be considered acceptable under this method.

TABLE I—PERFORMANCE SPECIFICATIONS

Parameter ^a	Specification
1. Accuracy ^a	≤20 percent of reference mean value.

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Parameter ^a	Specification
	≤4 percent of emission standard. ≤2 percent of emission standard. ≤5 percent of emission standard. ≤5 minutes maximum.

^a Expressed as sum of absolute mean value plus 95 percent confidence interval of a series of tests.

5. *Performance Specification Test Procedures*. The following test procedures shall be used to determine compliance with the requirements of paragraph 4:

5.1 Calibration test.

5.1.1 Analyze each calibration gas mixture (50 percent, 90 percent) for sulfur dioxide by the Reference method and record the results on the example sheet shown in Figure D-1. This step may be omitted for nonextractive monitors where dynamic calibration gas mixtures are not used (see section 5.1.2).

5.1.2 Set up and calibrate the complete measurement system according to the manufacturer's written instructions. This may be accomplished either in the laboratory or in the field. Make a series of five nonconsecutive readings with span gas mixtures alternately at each concentration (example, 50 percent, 90 percent, 50 percent, 90 percent, 50 percent). For nonextractive measurement systems, this test may be performed using procedures specified by the manufacturer and two or more calibration gases whose concentrations are certified by the manufacturer and differ by a factor of two or more. Convert the measurement system output readings to ppm and record the results on the example sheet shown in Figure D-2.

5.2 Field Test for Accuracy (Relative), Zero Drift and Calibration Drift. Install and operate the measurement system in accordance with the manufacturer's written instructions and drawings as follows:

5.2.1 Conditioning Period. Offset the zero setting at least 10 percent of span so that negative zero drift may be quantified. Operate the system for an initial 168-hour conditioning period. During this period the system should measure the SO_2 content of the effluent in a normal operational manner.

5.2.2 Operational Test Period. Operate the system for an additional 168-hour period. The system shall be monitoring the source effluent at all times when not being zeroed, calibrated or backpurged.

5.2.2.1 Field Test for Accuracy (Relative). The analyzer output for the following test shall be maintained between 20 percent and 90 percent of span. It is recommended that a calibrated gas mixture be used to verify the span setting utilized. During this 168-hour test period, make a minimum of nine (9) SO_2

concentration measurements using the Reference Method with a sampling period of one hour. If a measurement system operates across the stack or a portion of it, the Reference Method test shall make a four-point traverse over the measurement system operating path. Isokinetic sampling and analysis for SO₃ and H₂ SO₄ mist are not required. For measurement systems employing extractive sampling, place the measurement system and the Reference Method probe tips adjacent to each other in the duct. One test will consist of two simultaneous samples with not less than two analyses on each sample. Record the test data and measurement system concentrations on the example sheet shown in Figure D-3.

5.2.2.2 Field Test for Zero Drift and Calibration Drift. Determine the values given by zero and span gas SO_2 concentrations at 2-hour intervals until 15 sets of data are obtained. Alternatively, for nonextractive measurement systems, determine the values given by an electrically or mechanically produced zero condition, and by inserting a certified calibration gas concentration equivalent to not less than 20 percent of span, into the measurement system. Record these readings on the example sheet shown in Figure D-4. These 2-hour periods need not be consecutive but may not overlap. If the analyzer span is set at the expected maximum concentration for the tests performed under section 5.2.2, then the zero and span determinations to be made under this paragraph may be made concurrent with the tests under section 5.2.2.1. Zero and calibration corrections and adjustments are allowed only at 24-hour intervals (except as required under section 5.2.2) or at such shorter intervals as the manufacturer's written instructions specify. Automatic corrections made by the measurement system without operator intervention or initiation are allowable at any time. During the entire 168-hour test period, record the values given by zero and span gas SO₂ concentrations before and after adjustment at 24-hour intervals in the example sheet shown in Figure D-5.

5.3 Field Test for Response Time.

5.3.1 This test shall be accomplished using the entire measurement system as installed including sample transport lines if used.

Flow rates, line diameters, pumping rates, pressures (do not allow the pressurized calibration gas to change the normal operating pressure in the sample line), etc., shall be at the nominal values for normal operation as specified in the manufacturer's written instructions. In the case of cyclic analyzers, the response time test shall include one cycle.

5.3.2 Introduce a zero concentration of SO₂ into the measurement system sampling interface or as close to the sampling interface as possible. When the system output reading has stabilized, switch quickly to a known concentration of SO_2 at 70 to 90 percent of span. Record the time from concentration switching to final stable response. After the system response has stabilized at the upper level, switch quickly to a zero concentration of SO_2 . Record the time from concentration switching to final stable response. Alternatively, for nonextractive monitors, a calibration gas concentration equivalent to 20 percent of span or more may be switched into and out of the sample path and response times recorded. Perform this test sequence three (3) times. For each test record the results on the example sheet shown in Figure D-6.

6. Calculations, Data Analysis and Reporting. 6.1 Procedure for determination of mean values and confidence intervals.

6.1.1 The mean value of a data set is calculated according to equation D-1.

$$\overline{X} \frac{\sum_{i=1}^{n} x_{i}}{n}$$
 Equation D-1

Where:

 x_i =individual values. Σ =sum of the individual values. x=mean value.

n=number of data points.

 $6.1.2\,$ The 95 percent confidence interval (two-sided) is calculated according to equation D-2.

C.I.₉₅ =
$$\frac{t_{.975}}{n\sqrt{n-1}}\sqrt{n(\sum \chi_i^2) - (\sum \chi_i)^2}$$

Equation D-2

Where:

 Σx_i =sum of all data points.

1.975 = 1 - a/2, and

 $C.I._{95}=95$ percent confidence interval estimated of the average mean value.

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IYPICAL	VALUES	FOB 11	A/2

n	t.975	n	t.975	n	t.975
2	12.706	7	2.447	12	2.201

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TYPICAL VALUES FOR $T_1 - A/2$	2-Continued
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n	t.975	n	t.975	n	t.975
3	4.303	8	2.365	13	2.179
4	3.182	9	2.306	14	2.160
5	2.776	10	2.262	15	2.145
6	2.571	11	2.228	16	2.131

The values in this table are already corrected for n-1 degrees of freedom. Use *n* equal to the number of samples as data points.

6.2 Data Analysis and Reporting.

6.2.1 Accuracy (Relative). For each of the nine reference method testing periods, determine the average sulfur dioxide concentration reported by the continuous measurement system. These average concentrations shall be determined from the measurement system data recorded under section 5.2.2.1 by integrating the pollutant concentrations over each of the time intervals concurrent with each reference method test, then dividing by the cumulative time of each applicable reference method testing period. Before proceeding to the next step, determine the basis (wet or dry) of the measurement system data and reference method test data concentrations.

If the bases are not consistent, apply a moisture correction to either the referenced method concentrations or the measurement system concentrations, as appropriate. Determine the correction factor by moisture tests concurrent with the reference method testing periods. Report the moisture test method and the correction procedure employed. For each of the nine test runs, subtract the Reference Method test concentrations from the continuous monitoring system average concentrations. Using these data, compute the mean difference and the 95 percent confidence interval using equations D-1 and D-2. Accuracy is reported as the sum of the absolute value of the mean difference and the 95 percent confidence interval expressed as a percentage of the mean reference method value. Use the example sheet shown in Figure D-3.

6.2.2 Calibration Error. Using the data from section 5.1 of this appendix, subtract the measured SO₂ value determined under section 5.1.1 (Figure D-1) from the value shown by the measurement system for each of the 5 readings at each concentration measured under section 5.1.2 (Figure D-2). Calculate the mean of these difference values and the 95 percent confidence intervals according to equations D-1 and D-2. The calibration error is reported as the sum of absolute value of the mean difference and the 95 percent confidence interval as a percentage of each respective calibration gas concentration. Use example sheet shown in Figure D-2.

6.2.3 Zero Drift (2-hour). Using the zero concentration values measured each two

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hours during the field test, calculate the differences between *consecutive* two-hour readings expressed in ppm. Calculate the mean difference and the confidence interval using Equations D-1 and D-2. Report the zero drift as the sum of the absolute mean value and the confidence interval as a percentage of the emission standard. Use example sheet shown in Figure D-4.

6.2.4 Zero Drift (24-hour). Using the zero concentration values measured every 24 hours during the field test, calculate the differences between the zero point after zero adjustment and the zero value 24 hours later just prior to zero adjustment. Calculate the mean value of these points and the confidence interval using Equations D-1 and D-2. Report the zero drift as the sum of the absolute mean and confidence interval as a percentage of the emission standard. Use example sheet shown in Figure D-5.

6.2.5 Calibration Drift (2-hour). Using the calibration values obtained at two-hour intervals during the field test, calculate the differences between consecutive two-hour readings expressed as ppm. These values should be corrected for the corresponding zero drift during that two-hour period. Calculate the mean and confidence interval of these corrected difference values using Equations D-1 and D-2. Do not use the differences between non-consecutive readings. Report the calibration drift as the sum of the absolute mean and confidence interval as a percentage of the emission standard. Use the example sheet shown in Figure D-4.

6.2.6 Calibration Drift (24-hour). Using the calibration values measured every 24 hours during the field test, calculate the differences between the calibration concentration reading after zero and calibration adjustment and the calibration concentration reading 24 hours later after zero adjustment but before calibration adjustment. Calculate the mean value of these differences and the confidence interval using equations D-1 and D-2. Report the sum of the absolute mean and confidence interval as a percentage of the emission standard. Use the example sheet shown in Figure D-5.

6.2.7 Response Time. Using the charts from section 5.3 of this Appendix, calculate the time interval from concentration switching to 95 percent to the final stable value for all upscale and downscale tests. Report the mean of the three upscale test times and the mean of the three downscale test times. For nonextractive instruments using a calibration gas cell to determine response time, the observed times shall be extrapolated to 90 percent of full scale response time. For example, if the observed time for a 20 percent of span gas cell is one minute, this would be equivalent to a $4\frac{1}{2}$ -minute response time when extrapolated to 90 percent of span. The two average times should not differ by more than 15 percent of the slower time. Report

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the slower time as the system response time. Use the example sheet shown in Figure D–6.

6.2.8 Operational Period. During the 168hour performance and operational test period, the measurement system shall not require any corrective maintenance, repair, replacement, or adjustment other than that clearly specified as required in the operation and maintenance manuals as routine and expected during a one-week period. If the measurement system operates within the specified performance parameters and does not require corrective maintenance, repair. replacement or adjustment other than specified above, during the 168-hour test period. the operational period will be successfully concluded. Failure of the measurement to meet this requirement shall call for a repetition of the 168-hour test period. Portions of the test which were satisfactorily completed need not be repeated. Failure to meet any performance specifications shall call for a repetition of the one-week performance test period and that portion of the testing which is related to the failed specification. All maintenance and adjustments required shall be recorded. Output readings shall be recorded before and after all adjustments.

6.2.9 Performance Specifications Testing Frequency. In the event that significant repair work is performed in the system, the company shall demonstrate to the Administrator that the system still meets the performance specifications listed in Table I of this appendix. The Administrator may require a performance test at any time he determines that such test is necessary to verify the performance of the measurement system.

7. References.

7.1 Monitoring Instrumentation for the Measurement of Sulfur Dioxide in Stationary Source Emissions, Environmental Protection Agency, Research Triangle Park, N.C., February 1973.

7.2 Instrumentation for the Determination of Nitrogen Oxides Content of Stationary Source Emissions, Environmental Protection Agency, Research Triangle Park, N.C., APTD 0847, Vol. I, October 1971; APTD 0942, Vol. II, January 1972.

7.3 *Experimental Statistics*, Department of Commerce, Handbook 91, 1963, p. 3-31, paragraphs 3-3.1.4.

7.4 Performance Specifications for Stationary-Source Monitoring Systems for Gases, and Visible Emissions, Environmental Protection Agency, Research Triangle Park, N.C., EPA-650/2-74-013, January 1974.

FIGURE D-1—ANALYSIS OF CALIBRATION GAS MIXTURES

Date Reference Method Used

Mid Range Calibration Gas Mixture	
Sample 1	ppm
Sample 2	ppm
Sample 3	ppm
Average	ppm

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FIGURE I	D-1—ANALYSIS OF CALIBRATION C MIXTURES—Continued	Gas	FIGURE D-	-1—ANALYSIS OF CALIBRATION	Gas
Date	Reference Method Used		Date	Reference Method Used	
Hig	h Range (span) Calibration Gas Mixture				ppm ppm
Sample 1		ppm			ppm

FIGURE D-2-CALIBRATION ERROR DETERMINATION

Calibration gas mixture data (from fig. D-1): Mid (50 percent) average ___ p/m, high (90 percent) average ___ p/m [See footnotes at end of table]

Run No.	Calibration gas concentration ¹	Measurement system reading, p/m	Differences, p/m ²
1			
2			
3			
4			
5			
6			
7			
8			
9			
1			
11			
12			
13			
14			
15			

	read	
	50% mid	90% high
Mean difference		
Confidence interval	±	±
Calibration error=Mean difference ³ + C.I. / Average calibration gas concentration × 100	%	%

¹ Mid or high. ² Calibration gas concentration—measurement system reading. ³ Absolute value.

FIGURE D-3-ACCURACY

Date and time	Test No.	Reference method samples	Analyzer 1-hour average ¹ (p/m)	Difference ² (p/m)
1.				
2.				
3.				
4.				
5.				
6.				
7.				

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FIGURE D	-3-ACCURACY	-Continued

Date and time	Test No.	Reference method samples	Analyzer 1-hour average ¹ (p/m)	Difference ² (p/m)
8.				
9.				

Mean difference= ____p/m. 95 percent confidence interval=+ _____p/m. Mean Reference method value= _____p/m. Accuracy=Mean difference (absolute value)+95 percent confidence interval/Mean reference method value×100__ percent

¹ Explain method used to determine average. ² Difference=the 1-h average minus the reference method average.

Date	Tir	Time		Zero	Zero	Span		Calibra- tion drift
	Begin	End	Date set No.	reading	drift (∆ zero)	Span reading	(∆ span)	$(\Delta \text{ span-} \Delta \text{ zero})$
			1					
			23					
			45					
			6					
			7					
			8					
			9					
			10					
			11					
			12					
			13 14					
			14					
Zero drift=[mean zero drift ¹ +	CI (zero)	+emissio	n standard]:	<100=		1	1	

Calibration drift=[mean span drift 1 _____ CI + CI (span) _____ +emission standard]×100=

¹ Absolute value.

FIGURE D-5-ZERO AND CALIBRATION DRIFT (24 HR)

Date and time	Zero reading	Zero drift (∆ zero)	Span reading (after zero adjustment)	Calibration drift (∆ span)
Zero drift=[mean zero dri Calibration drift=[mean s		+emission standard]×10 +emission standard		
¹ Absolute value.				

FIGURE D-6—RESPON Date of Test	Average upsca Downscale:		
Span Gas Concentration Analyzer Span Setting Upscale:	ppm. ppm.	$\begin{array}{c}1\\2\\3\end{array}$	se se se
1		Average System onds.	

ale response ____ seconds.

econds.

econds.

econds.

 Average downscale response ______ seconds.

 System response time=slower time _____ sec
 onds.

Percent deviation from slowest time = average upscale - average downscale \times 100% / slower time

[40 FR 5517, Feb. 6, 1975]

APPENDIX E TO PART 52—PERFORMANCE SPECIFICATIONS AND, SPECIFICATION TEST PROCEDURES FOR MONITORING SYSTEMS FOR EFFLUENT STREAM GAS VOLUMETRIC FLOW RATE

1. Principle and applicability.

1.1 Principle. Effluent stream gas volumetric flow rates are sampled and analyzed by a continuous measurement system. To verify the measurement system performance, values obtained from the measurement system are compared against simultaneous values obtained using the reference method. These comparison tests will be performed to determine the relative accuracy, and drift of the measurement system over the range of operating conditions expected to occur during normal operation of the source. If the measurement system is such that the specified tests in section 5.1 for drift do not apply, those test procedures shall be disregarded.

1.2 Applicability. This method is applicable to subparts which require continuous gas volumetric flow rate measurement. Specifications are given in terms of performance. Test procedures are given for determining compliance with performance specifications. 2. Apparatus.

2.1 Continuous measurement system for determining stack gas volumetric flow rate.

2.2 Equipment for measurement of stack gas volumetric flow rate as specified in the reference method.

3. Definitions.

3.1 Measurement system. The total equipment required for the determination of the gas volumetric flow rate in a duct or stack. The system consists of three major subsystems:

3.1.1 Sampling interface. That portion of the measurement system that performs one or more of the following operations: Delineation, acquisition, transportation, and conditioning of a signal from the stack gas and protection of the analyzer from any hostile aspects of the source environment.

3.1.2 Analyzer. That portion of the measurement system which senses the stack gas flow rate or velocity pressure and generates a signal output that is a function of the flow rate or velocity of the gases.

3.1.3 Data presentation. That portion of the measurement system that provides a display of the output signal in terms of volumetric flow rate units, or other units which are convertible to volumetric flow rate units.

3.2 Span. The value of gas volumetric flow rate at which the measurement system is set to produce the maximum data display output. For the purposes of this method, the span shall be set at 1.5 times the maximum volumetric flow rate expected under varying operating conditions of the source.

3.3 Zero drift. The change in measurement system output over a stated period of time of normal continuous operation when gas volumetric flow rate at the time of the measurements is zero.

3.4 Calibration drift. The change in measurement system output over a stated time period of normal continuous operation when the gas volumetric flow rate at the time of the measurement is 67 percent of the span value.

3.5 Operation period. A minimum period of time over which a measurement system is expected to operate within certain performance specifications without unscheduled maintenance, repair, or adjustment.

3.6 Orientation sensitivity. The angular tolerance to which the sensor can be misaligned from its correct orientation to measure the flow rate vector before a specified error occurs in the indicated flow rate compared to the reference flow rate.

3.7 *Reference method*. Method 2 as delineated in 40 CFR Part 60.

4. Measurement system performance specifications. A measurement system must meet the performance specifications in Table E-1 to be considered acceptable under this method.

TABLE E-1

Parameter	Specifications
Accuracy (relative) Zero drift (24 hours) Calibration drift (24 hours) Operational period	<3 percent of span (paragraph 6.3.2).

5. Test procedures.

5.1 Field test for accuracy, zero drift, calibration drift, and operation period.

5.1.1 System conditioning. Set up and operate the measurement system in accordance with the manufacturer's written instructions and drawings. Offset the zero point of the chart recorder so that negative values up to 5 percent of the span value may be registered. Operate the system for an initial 168hour conditioning period. During this initial period, the system should measure the gas