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(e) Tests for all performance parameters shall be completed on the same test analyzer, except that use of multiple test analyzers to accelerate testing will be permitted when alternate ranges of a multi-range candidate method are being tested.

[62 FR 38784, July 18, 1997, as amended at 75 FR 35599, June 22, 2010]

§ 53.22 Generation of test atmospheres.

(a) Table B–2 specifies preferred methods for generating test atmospheres and suggested methods of verifying the concentrations. Only one means of establishing the concentration of a test atmosphere is normally required. If the method of generation can produce reproducible concentrations, verification is optional. If the method of generation is not reproducible, then establishment of the concentration by some verification meth-

od is required. However, when a method of generation other than that given in table B–2 is used, the test concentration shall be verified.

(b) The test atmosphere delivery system shall be designed and constructed so as not to significantly alter the test atmosphere composition or concentration during the period of the test. The delivery system shall be fabricated from borosilicate glass or FEP Teflon.

(c) The output of the test atmosphere generation system shall be sufficiently stable to obtain stable response during the required tests. If a permeation device is used for generation of a test atmosphere, the device, as well as the air passing over it, shall be controlled to ± 0.1 °C.

(d) All diluent air shall be zero air free of contaminants likely to cause a detectable response on the test analyzer.

TABLE B–2—TEST ATMOSPHERES

Test gas	Generation	Verification
Ammonia	Permeation device. Similar to system described in references 1 and 2.	Indophenol method, reference 3.
Carbon dioxide	Cylinder of zero air or nitrogen containing CO ₂ as required to obtain the concentration specified in Table B–3.	Use NIST-certified standards whenever possible. If NIST standards are not available, obtain 2 standards from independent sources which agree within 2 percent, or obtain one standard and submit it to an independent laboratory for analysis, which must agree within 2 percent of the supplier's nominal analysis.
Carbon monoxide	Cylinder of zero air or nitrogen containing CO as required to obtain the concentration specified in Table B–3.	Use a FRM CO analyzer as described in reference 8.
Ethane	Cylinder of zero air or nitrogen containing ethane as required to obtain the concentration specified in Table B–3.	Gas chromatography, ASTM D2820, reference 10. Use NIST-traceable gaseous methane or propane standards for calibration.
Ethylene	Cylinder of pre-purified nitrogen containing ethylene as required to obtain the concentration specified in Table B–3.	Do.
Hydrogen chloride	Cylinder ¹ of pre-purified nitrogen containing approximately 100 ppm of gaseous HCL. Dilute with zero air to concentration specified in Table B–3.	Collect samples in bubbler containing distilled water and analyze by the mercuric thiocyanate method, ASTM (D612), p. 29, reference 4.
Hydrogen sulfide	Permeation device system described in references 1 and 2.	Tentative method of analysis for H ₂ S content of the atmosphere, p. 426, reference 5.
Methane	Cylinder of zero air containing methane as required to obtain the concentration specified in Table B–3.	Gas chromatography ASTM D2820, reference 10. Use NIST-traceable methane standards for calibration.
Naphthalene	1. Permeation device as described in references 1 and 2. 2. Cylinder of pre-purified nitrogen containing 100 ppm naphthalene. Dilute with zero air to concentration specified in Table B–3.	Use NIST-certified standards whenever possible. If NIST standards are not available, obtain 2 standards from independent sources which agree within 2 percent, or obtain one standard and submit it to an independent laboratory for analysis, which must agree within 2 percent of the supplier's nominal analysis.

TABLE B-2—TEST ATMOSPHERES—Continued

Test gas	Generation	Verification
Nitric oxide	Cylinder ¹ of pre-purified nitrogen containing approximately 100 ppm NO. Dilute with zero air to required concentration.	Use NIST-certified standards whenever possible. If NIST standards are not available, obtain 2 standards from independent sources which agree within 2 percent, or obtain one standard and submit it to an independent laboratory for analysis, which must agree within 2 percent of the supplier's nominal analysis.
Nitrogen dioxide	1. Gas phase titration as described in reference 6. 2. Permeation device, similar to system described in reference 6.	1. Use an FRM NO ₂ analyzer calibrated with a gravimetrically calibrated permeation device. 2. Use an FRM NO ₂ analyzer calibrated by gas-phase titration as described in reference 6.
Ozone	Calibrated ozone generator as described in reference 9.	Use an FEM ozone analyzer calibrated as described in reference 9.
Sulfur dioxide	1. Permeation device as described in references 1 and 2. 2. Dynamic dilution of a cylinder containing approximately 100 ppm SO ₂ as described in Reference 7.	Use an SO ₂ FRM or FEM analyzer as described in reference 7.
Water	Pass zero air through distilled water at a fixed known temperature between 20° and 30 °C such that the air stream becomes saturated. Dilute with zero air to concentration specified in Table B-3.	Measure relative humidity by means of a dew-point indicator, calibrated electrolytic or piezo electric hygrometer, or wet/dry bulb thermometer.
Xylene	Cylinder of pre-purified nitrogen containing 100 ppm xylene. Dilute with zero air to concentration specified in Table B-3.	Use NIST-certified standards whenever possible. If NIST standards are not available, obtain 2 standards from independent sources which agree within 2 percent, or obtain one standard and submit it to an independent laboratory for analysis, which must agree within 2 percent of the supplier's nominal analysis.
Zero air	1. Ambient air purified by appropriate scrubbers or other devices such that it is free of contaminants likely to cause a detectable response on the analyzer. 2. Cylinder of compressed zero air certified by the supplier or an independent laboratory to be free of contaminants likely to cause a detectable response on the analyzer.	

¹ Use stainless steel pressure regulator dedicated to the pollutant measured.
 Reference 1. O'Keefe, A. E., and Ortaman, G. C. "Primary Standards for Trace Gas Analysis," *Anal. Chem.* 38, 760 (1966).
 Reference 2. Scaringelli, F. P., A. E. Rosenberg, E., and Bell, J. P., "Primary Standards for Trace Gas Analysis." *Anal. Chem.* 42, 871 (1970).
 Reference 3. "Tentative Method of Analysis for Ammonia in the Atmosphere (Indophenol Method)", *Health Lab Sciences*, vol. 10, No. 2, 115-118, April 1973.
 Reference 4. 1973 Annual Book of ASTM Standards, American Society for Testing and Materials, 1916 Race St., Philadelphia, PA.
 Reference 5. *Methods for Air Sampling and Analysis*, Intersociety Committee, 1972, American Public Health Association, 1015.
 Reference 6. 40 CFR 50 Appendix F, "Measurement Principle and Calibration Principle for the Measurement of Nitrogen Dioxide in the Atmosphere (Gas Phase Chemiluminescence)."
 Reference 7. 40 CFR 50 Appendix A-1, "Measurement Principle and Calibration Procedure for the Measurement of Sulfur Dioxide in the Atmosphere (Ultraviolet Fluorescence)."
 Reference 8. 40 CFR 50 Appendix C, "Measurement Principle and Calibration Procedure for the Measurement of Carbon Monoxide in the Atmosphere" (Non-Dispersive Infrared Photometry)."
 Reference 9. 40 CFR 50 Appendix D, "Measurement Principle and Calibration Procedure for the Measurement of Ozone in the Atmosphere".
 Reference 10. "Standard Test Method for C, through C5 Hydrocarbons in the Atmosphere by Gas Chromatography", D 2820, 1987 Annual Book of ASTM Standards, vol 11.03, American Society for Testing and Materials, 1916 Race St., Philadelphia, PA 19103.

(e) The concentration of each test atmosphere shall be established and/or verified before or during each series of tests. Samples for verifying test concentrations shall be collected from the test atmosphere delivery system as close as possible to the sample intake port of the test analyzer.

(f) The accuracy of all flow measurements used to calculate test atmosphere concentrations shall be documented and referenced to a primary standard (such as a spirometer, bubble meter, etc.). Any corrections shall be clearly shown. All flow measurements

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given in volume units shall be standardized to 25 °C. and 760 mm Hg.

(g) Schematic drawings and other information showing complete procedural details of the test atmosphere generation, verification, and delivery system shall be provided. All pertinent calculations shall be clearly indicated.

[40 FR 7049, Feb. 18, 1975, as amended at 40 FR 18168, Apr. 25, 1975; 75 FR 35599, June 22, 2010]

§ 53.23 Test procedures.

(a) *Range*—(1) *Technical definition.* Nominal minimum and maximum concentrations which a method is capable of measuring.

NOTE: The nominal range is specified at the lower and upper range limits in concentration units, for example, 0–0.5 p/m.

(2) *Test procedure.* Submit a suitable calibration curve, as specified in § 53.21(b), showing the test analyzer's response over at least 95 percent of the required range.

NOTE: A single calibration curve will normally suffice.

(b) *Noise*—(1) *Technical definition.* Spontaneous, short duration deviations in output, about the mean output, which are not caused by input concentration changes. Noise is determined as the standard deviation about the mean and is expressed in concentration units.

(2) *Test procedure.* (i) Allow sufficient time for the test analyzer to warm up and stabilize. Determine at two concentrations, first using zero air and then a pollutant test gas concentration as indicated below. The noise specification in table B-1 shall apply to both of these tests.

(ii) Connect an integrating-type digital meter (DM) suitable for the test analyzer's output and accurate to three significant digits, to measure the analyzer's output signal.

NOTE: Use of a chart recorder in addition to the DM is optional.

(iii) Measure zero air for 60 minutes. During this 60-minute interval, record twenty-five (25) readings at 2-minute intervals. (See Figure B-2 in appendix A.)

(iv) Convert each DM reading to concentration units (p/m) by reference to

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the test analyzer's calibration curve as determined in § 53.21(b). Label the converted DM readings $r_1, r_2, r_3 \dots r_i \dots r_{25}$.

(v) Calculate the standard deviation, S , as follows:

$$S = \sqrt{\frac{\sum_{i=1}^{25} (r_i)^2 - \frac{1}{25} \left(\sum_{i=1}^{25} r_i \right)^2}{24}} (p/m)$$

where i indicates the i -th DM reading in ppm.

(vi) Let S at 0 ppm be identified as S_0 ; compare S_0 to the noise specification given in table B-1.

(vii) Repeat steps (iii) through (vi) of this section using a pollutant test atmosphere concentration of 80±5 percent of the upper range limit (URL) instead of zero gas, and let S at 80 percent of the URL be identified as S_{80} . Compare S_{80} to the noise specification given in table B-1.

(viii) Both S_0 and S_{80} must be less than or equal to the specification for noise to pass the test for the noise parameter.

(c) *Lower detectable limit*—(1) *Technical definition.* The minimum pollutant concentration which produces a signal of twice the noise level.

(2) *Test procedure.* (i) Allow sufficient time for the test analyzer to warm up and stabilize. Measure zero air and record the stable reading in ppm as B_Z . (See Figure B-3 in appendix A.)

(ii) Generate and measure a pollutant test atmosphere concentration equal to the value for the lower detectable limit specified in table B-1.

NOTE: If necessary, the test atmosphere concentration may be generated or verified at a higher concentration, then accurately diluted with zero air to the final required concentration.

(iii) Record the test analyzer's stable indicated reading, in ppm, as B_L .

(iv) Determine the Lower Detectable Limit (LDL) as $LDL = B_L - B_Z$. Compare this LDL value with the noise level, S_0 , determined in § 53.23(b), for 0 concentration test atmosphere. LDL must be equal to or higher than $2S_0$ to pass this test.