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If the candidate method does not include an integral strip chart recorder, connect the output signal of the test analyzer to a suitable strip chart recorder of the servo, null-balance type. This recorder shall have a chart width of at least 25 centimeters, chart speeds up to 10 cm per hour, a response time of 1 second or less, a deadband of not more than 0.25 percent of full scale, and capability either of reading measurements at least 5 percent below zero or of offsetting the zero by at least 5 percent.

NOTE: Other data acquisition components may be used along with the chart recorder during conduct of these tests. Use of the chart recorder is intended only to facilitate evaluation of data submitted.

(b) *Calibration* of the test analyzer shall be as indicated in the manual referred to in § 53.4(b)(3) and as follows: If the chart recorder does not have below zero capability, adjust either the controls of the test analyzer or the chart recorder to obtain a +5% offset zero reading on the recorder chart to facilitate observing negative response or drift. If the candidate method is not capable of negative response, the test analyzer (not recorder) shall be operated with an offset zero. Construct and submit a calibration curve showing a plot of recorder scale readings (ordinate) against pollutant concentrations (abscissa). A plot of output units (volts, millivolts, milliamps, etc.) against pollutant concentrations shall also be shown for methods not including an integral chart recorder. All such plots shall consist of at least seven (7) approximately equally spaced, identifiable points, including 0 and 90±5 percent of full scale.

(c) Once the test analyzer has been set up and calibrated and the tests started, manual adjustment or normal periodic maintenance is permitted only every 3 days. Automatic adjustments which the test analyzer performs by itself are permitted at any time. The submitted records shall show clearly when any manual adjustment or periodic maintenance was made and describe the operations performed.

(d) If the test analyzer should malfunction during any of the performance tests, the tests for that parameter shall be repeated. A detailed explanation of the malfunction, remedial action taken, and whether recalibration was necessary (along with all pertinent records and charts) shall be submitted. If more than one malfunction occurs, all performance test procedures for all parameters shall be repeated.

(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.

§ 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 method 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 NBS-certified standards whenever possible. If NBS 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.	Do.
Ethane	Cylinder of zero air or nitrogen containing ethane as required to obtain the concentration specified in table B–3.	Do.
Ethylene	Cylinder of prepurified nitrogen containing ethylene as required to obtain the concentration specified in table B–3.	Do.
Hydrogen chloride.	Cylinder[1] of prepurified nitrogen containing approximately 100 p/m 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 (D512), 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.	Use NBS-certified standards whenever possible. If NBS 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 an analysis which must agree within 2 percent of the supplier's nominal analysis.
Nitric oxide	Cylinder[1] of prepurified nitrogen containing approximately 100 p/m NO. Dilute with zero air to required concentration.	Gas-phase titration as described in reference 6, section 7.1.
Nitrogen dioxide.	1. Gas phase titration as described in reference 6 2. Permeation device, similar to system described in references 1 and 2.	1. Use an NO_2 analyzer calibrated with a gravimetrically calibrated permeation device. 2. Use an NO_2 analyzer calibrated by gas-phase titration as described in reference 6.
Ozone	Calibrated ozone generator as described in reference 7, appendix D.	Use an ozone analyzer calibrated by gas-phase titration as described in reference 6.
Sulfur dioxide ..	Permeation device Similar to system described in reference method for SO_2, reference 7, appendix A.	P-rosaniline method. Reference 7, appendix A.
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 prepurified nitrogen containing 100 p/m xylene. Dilute with zero air to concentration specified in table B–3.	Use NBS-certified standards whenever possible. If NBS 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 an 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'Keeffe, 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. *Federal Register,* vol. 38, No. 110, Tentative Method for the Continuous Measurement of Nitrogen Dioxide (Chemiluminescent) addenda C. (June 8, 1973).
Reference 7. *Federal Register,* vol. 36, No. 228, National Primary and Secondary Ambient Air Quality Standards, Nov. 25, 1971.

(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 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]

§ 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 the test analyzer's calibration curve as determined in § 53.21(b). Label the converted DM readings r_1, r_2, r_3 . . . r_i . . . 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_o; compare S_o 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