

Appendix A

National Ambient Air Quality Standards

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40 CFR Part 50 Appendix A-N

National Ambient Air Quality Standards

Revised as of March 28, 2008

Pollutant	Primary Standards		Secondary Standards	
	Level	Averaging Time	Level	Averaging Time
Carbon Monoxide	9 ppm (10 mg/m ³)	8-hour ⁽¹⁾	None	
	35 ppm (40 mg/m ³)	1-hour ⁽¹⁾		
Lead	1.5 µg/m ³	Quarterly Average	Same as Primary	
Nitrogen Dioxide	0.053 ppm (100 µg/m ³)	Annual (Arithmetic Mean)	Same as Primary	
Particulate Matter (PM ₁₀)	150 µg/m ³	24-hour ⁽²⁾	Same as Primary	
Particulate Matter (PM _{2.5})	15.0 µg/m ³	Annual ⁽³⁾ (Arithmetic Mean)	Same as Primary	
	35 µg/m ³	24-hour ⁽⁴⁾	Same as Primary	
Ozone	0.075 ppm (2008 std)	8-hour ⁽⁵⁾	Same as Primary	
	0.08 ppm (1997 std)	8-hour ⁽⁶⁾	Same as Primary	
	0.12 ppm	1-hour ⁽⁷⁾ (Applies only in limited areas)	Same as Primary	
Sulfur Dioxide	0.03 ppm	Annual (Arithmetic Mean)	0.5 ppm (1300 µg/m ³)	3-hour ⁽¹⁾
	0.14 ppm	24-hour ⁽¹⁾		

⁽¹⁾ Not to be exceeded more than once per year.

⁽²⁾ Not to be exceeded more than once per year on average over 3 years.

⁽³⁾ To attain this standard, the 3-year average of the weighted annual mean PM_{2.5} concentrations from single or multiple community-oriented monitors must not exceed 15.0 µg/m³.

⁽⁴⁾ To attain this standard, the 3-year average of the 98th percentile of 24-hour concentrations at each population-oriented monitor within an area must not exceed 35 µg/m³ (effective December 17, 2006).

⁽⁵⁾ To attain this standard, the 3-year average of the fourth-highest daily maximum 8-hour average ozone concentrations measured at each monitor within an area over each year must not exceed 0.075 ppm. (effective May 27, 2008)

⁽⁶⁾ (a) To attain this standard, the 3-year average of the fourth-highest daily maximum 8-hour average ozone concentrations measured at each monitor within an area over each year must not exceed 0.08 ppm.

(b) The 1997 standard—and the implementation rules for that standard—will remain in place for implementation purposes as EPA undertakes rulemaking to address the transition from the 1997 ozone standard to the 2008 ozone standard.

⁽⁷⁾ (a) The standard is attained when the expected number of days per calendar year with maximum hourly average concentrations above 0.12 ppm is ≤ 1.

(b) As of June 15, 2005 EPA revoked the [1-hour ozone standard](#) in all areas except the 8-hour ozone nonattainment [Early Action Compact \(EAC\) Areas](#).

Revised as of July 1, 2001

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APPENDIX N TO PART 50—INTERPRETATION OF THE NATIONAL AMBIENT AIR QUALITY STANDARDS FOR PARTICULATE MATTER

AUTHORITY: 42 U.S.C. 7401, *et seq.*

SOURCE: 36 FR 22384, Nov. 25, 1971, unless otherwise noted.

§ 50.1 Definitions.

(a) As used in this part, all terms not defined herein shall have the meaning given them by the Act.

(b) *Act* means the Clean Air Act, as amended (42 U.S.C. 1857–18571, as amended by Pub. L. 91–604).

(c) *Agency* means the Environmental Protection Agency.

(d) *Administrator* means the Administrator of the Environmental Protection Agency.

(e) *Ambient air* means that portion of the atmosphere, external to buildings, to which the general public has access.

(f) *Reference method* means a method of sampling and analyzing the ambient air for an air pollutant that is specified as a reference method in an appendix to this part, or a method that has been designated as a reference method in accordance with part 53 of this chapter; it does not include a method for which a reference method designation has been cancelled in accordance with § 53.11 or § 53.16 of this chapter.

(g) *Equivalent method* means a method of sampling and analyzing the ambient air for an air pollutant that has been designated as an equivalent method in accordance with part 53 of this chapter; it does not include a method for which an equivalent method designation has

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been cancelled in accordance with § 53.11 or § 53.16 of this chapter.

(h) *Traceable* means that a local standard has been compared and certified either directly or via not more than one intermediate standard, to a primary standard such as a National Bureau of Standards Standard Reference Material (NBS SRM), or a USEPA/NBS-approved Certified Reference Material (CRM).

(i) *Indian country* is as defined in 18 U.S.C. 1151.

[36 FR 22384, Nov. 25, 1971, as amended at 41 FR 11253, Mar. 17, 1976; 48 FR 2529, Jan. 20, 1983; 63 FR 7274, Feb. 12, 1998]

§ 50.2 Scope.

(a) National primary and secondary ambient air quality standards under section 109 of the Act are set forth in this part.

(b) National primary ambient air quality standards define levels of air quality which the Administrator judges are necessary, with an adequate margin of safety, to protect the public health. National secondary ambient air quality standards define levels of air quality which the Administrator judges necessary to protect the public welfare from any known or anticipated adverse effects of a pollutant. Such standards are subject to revision, and additional primary and secondary standards may be promulgated as the Administrator deems necessary to protect the public health and welfare.

(c) The promulgation of national primary and secondary ambient air quality standards shall not be considered in any manner to allow significant deterioration of existing air quality in any portion of any State or Indian country.

(d) The proposal, promulgation, or revision of national primary and secondary ambient air quality standards shall not prohibit any State or Indian country from establishing ambient air quality standards for that State or area under a tribal CAA program or any portion thereof which are more stringent than the national standards.

[36 FR 22384, Nov. 25, 1971, as amended at 63 FR 7274, Feb. 12, 1998]

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§ 50.3 Reference conditions.

All measurements of air quality that are expressed as mass per unit volume (e.g., micrograms per cubic meter) other than for the particulate matter (PM₁₀ and PM_{2.5}) standards contained in § 50.7 shall be corrected to a reference temperature of 25 °C and a reference pressure of 760 millimeters of mercury (1,013.2 millibars). Measurements of PM₁₀ and PM_{2.5} for purposes of comparison to the standards contained in § 50.7 shall be reported based on actual ambient air volume measured at the actual ambient temperature and pressure at the monitoring site during the measurement period.

[62 FR 38711, July 18, 1997]

§ 50.4 National primary ambient air quality standards for sulfur oxides (sulfur dioxide).

(a) The level of the annual standard is 0.030 parts per million (ppm), not to be exceeded in a calendar year. The annual arithmetic mean shall be rounded to three decimal places (fractional parts equal to or greater than 0.0005 ppm shall be rounded up).

(b) The level of the 24-hour standard is 0.14 parts per million (ppm), not to be exceeded more than once per calendar year. The 24-hour averages shall be determined from successive non-overlapping 24-hour blocks starting at midnight each calendar day and shall be rounded to two decimal places (fractional parts equal to or greater than 0.005 ppm shall be rounded up).

(c) Sulfur oxides shall be measured in the ambient air as sulfur dioxide by the reference method described in appendix A to this part or by an equivalent method designated in accordance with part 53 of this chapter.

(d) To demonstrate attainment, the annual arithmetic mean and the second-highest 24-hour averages must be based upon hourly data that are at least 75 percent complete in each calendar quarter. A 24-hour block average shall be considered valid if at least 75 percent of the hourly averages for the 24-hour period are available. In the event that only 18, 19, 20, 21, 22, or 23 hourly averages are available, the 24-hour block average shall be computed as the sum of the available hourly

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averages using 18, 19, etc. as the divisor. If fewer than 18 hourly averages are available, but the 24-hour average would exceed the level of the standard when zeros are substituted for the missing values, subject to the rounding rule of paragraph (b) of this section, then this shall be considered a valid 24-hour average. In this case, the 24-hour block average shall be computed as the sum of the available hourly averages divided by 24.

[61 FR 25579, May 22, 1996]

§ 50.5 National secondary ambient air quality standard for sulfur oxides (sulfur dioxide).

(a) The level of the 3-hour standard is 0.5 parts per million (ppm), not to be exceeded more than once per calendar year. The 3-hour averages shall be determined from successive nonoverlapping 3-hour blocks starting at midnight each calendar day and shall be rounded to 1 decimal place (fractional parts equal to or greater than 0.05 ppm shall be rounded up).

(b) Sulfur oxides shall be measured in the ambient air as sulfur dioxide by the reference method described in appendix A of this part or by an equivalent method designated in accordance with part 53 of this chapter.

(c) To demonstrate attainment, the second-highest 3-hour average must be based upon hourly data that are at least 75 percent complete in each calendar quarter. A 3-hour block average shall be considered valid only if all three hourly averages for the 3-hour period are available. If only one or two hourly averages are available, but the 3-hour average would exceed the level of the standard when zeros are substituted for the missing values, subject to the rounding rule of paragraph (a) of this section, then this shall be considered a valid 3-hour average. In all cases, the 3-hour block average shall be computed as the sum of the hourly averages divided by 3.

[61 FR 25580, May 22, 1996]

§ 50.6 National primary and secondary ambient air quality standards for PM₁₀.

(a) The level of the national primary and secondary 24-hour ambient air quality standards for particulate mat-

ter is 150 micrograms per cubic meter ($\mu\text{g}/\text{m}^3$), 24-hour average concentration. The standards are attained when the expected number of days per calendar year with a 24-hour average concentration above $150 \mu\text{g}/\text{m}^3$, as determined in accordance with appendix K to this part, is equal to or less than one.

(b) The level of the national primary and secondary annual standards for particulate matter is 50 micrograms per cubic meter ($\mu\text{g}/\text{m}^3$), annual arithmetic mean. The standards are attained when the expected annual arithmetic mean concentration, as determined in accordance with appendix K to this part, is less than or equal to $50 \mu\text{g}/\text{m}^3$.

(c) For the purpose of determining attainment of the primary and secondary standards, particulate matter shall be measured in the ambient air as PM₁₀ (particles with an aerodynamic diameter less than or equal to a nominal 10 micrometers) by:

(1) A reference method based on appendix J and designated in accordance with part 53 of this chapter, or

(2) An equivalent method designated in accordance with part 53 of this chapter.

[52 FR 24663, July 1, 1987, as amended at 62 FR 38711, July 18, 1997; 65 FR 80779, Dec. 22, 2000]

§ 50.7 National primary and secondary ambient air quality standards for particulate matter.

(a) The national primary and secondary ambient air quality standards for particulate matter are:

(1) 15.0 micrograms per cubic meter ($\mu\text{g}/\text{m}^3$) annual arithmetic mean concentration, and $65 \mu\text{g}/\text{m}^3$ 24-hour average concentration measured in the ambient air as PM_{2.5} (particles with an aerodynamic diameter less than or equal to a nominal 2.5 micrometers) by either:

(i) A reference method based on appendix L of this part and designated in accordance with part 53 of this chapter; or

(ii) An equivalent method designated in accordance with part 53 of this chapter.

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(2) 50 micrograms per cubic meter ($\mu\text{g}/\text{m}^3$) annual arithmetic mean concentration, and 150 $\mu\text{g}/\text{m}^3$ 24-hour average concentration measured in the ambient air as PM_{10} (particles with an aerodynamic diameter less than or equal to a nominal 10 micrometers) by either:

(i) A reference method based on appendix M of this part and designated in accordance with part 53 of this chapter; or

(ii) An equivalent method designated in accordance with part 53 of this chapter.

(b) The annual primary and secondary $\text{PM}_{2.5}$ standards are met when the annual arithmetic mean concentration, as determined in accordance with appendix N of this part, is less than or equal to 15.0 micrograms per cubic meter.

(c) The 24-hour primary and secondary $\text{PM}_{2.5}$ standards are met when the 98th percentile 24-hour concentration, as determined in accordance with appendix N of this part, is less than or equal to 65 micrograms per cubic meter.

(d) The annual primary and secondary PM_{10} standards are met when the annual arithmetic mean concentration, as determined in accordance with appendix N of this part, is less than or equal to 50 micrograms per cubic meter.

(e) The 24-hour primary and secondary PM_{10} standards are met when the 99th percentile 24-hour concentration, as determined in accordance with appendix N of this part, is less than or equal to 150 micrograms per cubic meter.

[62 FR 38711, July 18, 1997]

§ 50.8 National primary ambient air quality standards for carbon monoxide.

(a) The national primary ambient air quality standards for carbon monoxide are:

(1) 9 parts per million (10 milligrams per cubic meter) for an 8-hour average concentration not to be exceeded more than once per year and

(2) 35 parts per million (40 milligrams per cubic meter) for a 1-hour average

concentration not to be exceeded more than once per year.

(b) The levels of carbon monoxide in the ambient air shall be measured by:

(1) A reference method based on appendix C and designated in accordance with part 53 of this chapter, or

(2) An equivalent method designated in accordance with part 53 of this chapter.

(c) An 8-hour average shall be considered valid if at least 75 percent of the hourly average for the 8-hour period are available. In the event that only six (or seven) hourly averages are available, the 8-hour average shall be computed on the basis of the hours available using six (or seven) as the divisor.

(d) When summarizing data for comparison with the standards, averages shall be stated to one decimal place. Comparison of the data with the levels of the standards in parts per million shall be made in terms of integers with fractional parts of 0.5 or greater rounding up.

[50 FR 37501, Sept. 13, 1985]

§ 50.9 National 1-hour primary and secondary ambient air quality standards for ozone.

(a) The level of the national 1-hour primary and secondary ambient air quality standards for ozone measured by a reference method based on appendix D to this part and designated in accordance with part 53 of this chapter, is 0.12 parts per million (235 $\mu\text{g}/\text{m}^3$). The standard is attained when the expected number of days per calendar year with maximum hourly average concentrations above 0.12 parts per million (235 $\mu\text{g}/\text{m}^3$) is equal to or less than 1, as determined by appendix H to this part.

(b) The 1-hour standards set forth in this section will remain applicable to all areas notwithstanding the promulgation of 8-hour ozone standards under § 50.10. In addition, after the 8-hour standard has become fully enforceable under part D of title I of the CAA and subject to no further legal challenge, the 1-hour standards set forth in this section will no longer apply to an area once EPA determines that the area has

air quality meeting the 1-hour standard. Area designations and classifications with respect to the 1-hour standards are codified in 40 CFR part 81.

[62 FR 38894, July 18, 1997, as amended at 65 FR 45200, July 20, 2000]

§ 50.10 National 8-hour primary and secondary ambient air quality standards for ozone.

(a) The level of the national 8-hour primary and secondary ambient air quality standards for ozone, measured by a reference method based on appendix D to this part and designated in accordance with part 53 of this chapter, is 0.08 parts per million (ppm), daily maximum 8-hour average.

(b) The 8-hour primary and secondary ozone ambient air quality standards are met at an ambient air quality monitoring site when the average of the annual fourth-highest daily maximum 8-hour average ozone concentration is less than or equal to 0.08 ppm, as determined in accordance with appendix I to this part.

[62 FR 38894, July 18, 1997]

§ 50.11 National primary and secondary ambient air quality standards for nitrogen dioxide.

(a) The level of the national primary ambient air quality standard for nitrogen dioxide is 0.053 parts per million (100 micrograms per cubic meter), annual arithmetic mean concentration.

(b) The level of national secondary ambient air quality standard for nitrogen dioxide is 0.053 parts per million (100 micrograms per cubic meter), annual arithmetic mean concentration.

(c) The levels of the standards shall be measured by:

(1) A reference method based on appendix F and designated in accordance with part 53 of this chapter, or

(2) An equivalent method designated in accordance with part 53 of this chapter.

(d) The standards are attained when the annual arithmetic mean concentration in a calendar year is less than or equal to 0.053 ppm, rounded to three decimal places (fractional parts equal to or greater than 0.0005 ppm must be rounded up). To demonstrate attainment, an annual mean must be based upon hourly data that are at least 75

percent complete or upon data derived from manual methods that are at least 75 percent complete for the scheduled sampling days in each calendar quarter.

[50 FR 25544, June 19, 1985]

§ 50.12 National primary and secondary ambient air quality standards for lead.

National primary and secondary ambient air quality standards for lead and its compounds, measured as elemental lead by a reference method based on appendix G to this part, or by an equivalent method, are: 1.5 micrograms per cubic meter, maximum arithmetic mean averaged over a calendar quarter.

(Secs. 109, 301(a) Clean Air Act as amended (42 U.S.C. 7409, 7601(a)))

[43 FR 46258, Oct. 5, 1978]

APPENDIX A TO PART 50—REFERENCE METHOD FOR THE DETERMINATION OF SULFUR DIOXIDE IN THE ATMOSPHERE (PARAROSANILINE METHOD)

1.0 Applicability.

1.1 This method provides a measurement of the concentration of sulfur dioxide (SO₂) in ambient air for determining compliance with the primary and secondary national ambient air quality standards for sulfur oxides (sulfur dioxide) as specified in § 50.4 and § 50.5 of this chapter. The method is applicable to the measurement of ambient SO₂ concentrations using sampling periods ranging from 30 minutes to 24 hours. Additional quality assurance procedures and guidance are provided in part 58, appendixes A and B, of this chapter and in references 1 and 2.

2.0 Principle.

2.1 A measured volume of air is bubbled through a solution of 0.04 M potassium tetrachloromercurate (TCM). The SO₂ present in the air stream reacts with the TCM solution to form a stable monochloro-sulfonatomercurate(3) complex. Once formed, this complex resists air oxidation(4, 5) and is stable in the presence of strong oxidants such as ozone and oxides of nitrogen. During subsequent analysis, the complex is reacted with acid-bleached pararosaniline dye and formaldehyde to form an intensely colored pararosaniline methyl sulfonic acid.(6) The optical density of this species is determined spectrophotometrically at 548 nm and is directly related to the amount of SO₂ collected. The total volume of air sampled, corrected to EPA reference conditions (25 °C, 760 mm Hg [101 kPa]), is determined from the measured flow rate and the sampling time. The concentration of SO₂ in

the ambient air is computed and expressed in micrograms per standard cubic meter ($\mu\text{g}/\text{std m}^3$).

3.0 Range.

3.1 The lower limit of detection of SO_2 in 10 mL of TCM is $0.75 \mu\text{g}$ (based on collaborative test results).⁽⁷⁾ This represents a concentration of $25 \mu\text{g SO}_2/\text{m}^3$ (0.01 ppm) in an air sample of 30 standard liters (short-term sampling) and a concentration of $13 \mu\text{g SO}_2/\text{m}^3$ (0.005 ppm) in an air sample of 288 standard liters (long-term sampling). Concentrations less than $25 \mu\text{g SO}_2/\text{m}^3$ can be measured by sampling larger volumes of ambient air; however, the collection efficiency falls off rapidly at low concentrations.^(8, 9) Beer's law is adhered to up to $34 \mu\text{g}$ of SO_2 in 25 mL of final solution. This upper limit of the analysis range represents a concentration of $1,130 \mu\text{g SO}_2/\text{m}^3$ (0.43 ppm) in an air sample of 30 standard liters and a concentration of $590 \mu\text{g SO}_2/\text{m}^3$ (0.23 ppm) in an air sample of 288 standard liters. Higher concentrations can be measured by collecting a smaller volume of air, by increasing the volume of absorbing solution, or by diluting a suitable portion of the collected sample with absorbing solution prior to analysis.

4.0 Interferences.

4.1 The effects of the principal potential interferences have been minimized or eliminated in the following manner: Nitrogen oxides by the addition of sulfamic acid,^(10, 11) heavy metals by the addition of ethylenediamine tetracetic acid disodium salt (EDTA) and phosphoric acid,^(10, 12) and ozone by time delay.⁽¹⁰⁾ Up to $60 \mu\text{g Fe}$ (III), $22 \mu\text{g V}$ (V), $10 \mu\text{g Cu}$ (II), $10 \mu\text{g Mn}$ (II), and $10 \mu\text{g Cr}$ (III) in 10 mL absorbing reagent can be tolerated in the procedure.⁽¹⁰⁾ No significant interference has been encountered with $2.3 \mu\text{g NH}_3$.⁽¹³⁾

5.0 Precision and Accuracy.

5.1 The precision of the analysis is 4.6 percent (at the 95 percent confidence level) based on the analysis of standard sulfite samples.⁽¹⁰⁾

5.2 Collaborative test results⁽¹⁴⁾ based on the analysis of synthetic test atmospheres (SO_2 in scrubbed air) using the 24-hour sampling procedure and the sulfite-TCM calibration procedure show that:

- The replication error varies linearly with concentration from $\pm 2.5 \mu\text{g}/\text{m}^3$ at concentrations of $100 \mu\text{g}/\text{m}^3$ to $\pm 7 \mu\text{g}/\text{m}^3$ at concentrations of $400 \mu\text{g}/\text{m}^3$.
- The day-to-day variability within an individual laboratory (repeatability) varies linearly with concentration from $\pm 18.1 \mu\text{g}/\text{m}^3$ at levels of $100 \mu\text{g}/\text{m}^3$ to $\pm 50.9 \mu\text{g}/\text{m}^3$ at levels of $400 \mu\text{g}/\text{m}^3$.
- The day-to-day variability between two or more laboratories (reproducibility) varies linearly with concentration from $\pm 36.9 \mu\text{g}/\text{m}^3$ at levels of $100 \mu\text{g}/\text{m}^3$ to $\pm 103.5 \mu\text{g}/\text{m}^3$ at levels of $400 \mu\text{g}/\text{m}^3$.

- The method has a concentration-dependent bias, which becomes significant at the 95 percent confidence level at the high concentration level. Observed values tend to be lower than the expected SO_2 concentration level.

6.0 Stability.

6.1 By sampling in a controlled temperature environment of $15^\circ \pm 10^\circ \text{C}$, greater than 98.9 percent of the SO_2 -TCM complex is retained at the completion of sampling.⁽¹⁵⁾ If kept at 5°C following the completion of sampling, the collected sample has been found to be stable for up to 30 days.⁽¹⁰⁾ The presence of EDTA enhances the stability of SO_2 in the TCM solution and the rate of decay is independent of the concentration of SO_2 .⁽¹⁶⁾

7.0 Apparatus.

7.1 Sampling.

7.1.1 *Sample probe:* A sample probe meeting the requirements of section 7 of 40 CFR part 58, appendix E (Teflon[®] or glass with residence time less than 20 sec.) is used to transport ambient air to the sampling train location. The end of the probe should be designed or oriented to preclude the sampling of precipitation, large particles, etc. A suitable probe can be constructed from Teflon[®] tubing connected to an inverted funnel.

7.1.2 *Absorber—short-term sampling:* An all glass midget impinger having a solution capacity of 30 mL and a stem clearance of 4 ± 1 mm from the bottom of the vessel is used for sampling periods of 30 minutes and 1 hour (or any period considerably less than 24 hours). Such an impinger is shown in Figure 1. These impingers are commercially available from distributors such as Ace Glass, Incorporated.

7.1.3 *Absorber—24-hour sampling:* A polypropylene tube 32 mm in diameter and 164 mm long (available from Bel Art Products, Pequannock, NJ) is used as the absorber. The cap of the absorber must be a polypropylene cap with two ports (rubber stoppers are unacceptable because the absorbing reagent can react with the stopper to yield erroneously high SO_2 concentrations). A glass impinger stem, 6 mm in diameter and 158 mm long, is inserted into one port of the absorber cap. The tip of the stem is tapered to a small diameter orifice (0.4 ± 0.1 mm) such that a No. 79 jeweler's drill bit will pass through the opening but a No. 78 drill bit will not. Clearance from the bottom of the absorber to the tip of the stem must be 6 ± 2 mm. Glass stems can be fabricated by any reputable glass blower or can be obtained from a scientific supply firm. Upon receipt, the orifice test should be performed to verify the orifice size. The 50 mL volume level should be permanently marked on the absorber. The assembled absorber is shown in Figure 2.

7.1.4 *Moisture trap:* A moisture trap constructed of a glass trap as shown in Figure 1 or a polypropylene tube as shown in Figure 2 is placed between the absorber tube and

flow control device to prevent entrained liquid from reaching the flow control device. The tube is packed with indicating silica gel as shown in Figure 2. Glass wool may be substituted for silica gel when collecting short-term samples (1 hour or less) as shown in Figure 1, or for long term (24 hour) samples

if flow changes are not routinely encountered.

7.1.5 Cap seals: The absorber and moisture trap caps must seal securely to prevent leaks during use. Heat-shrink material as shown in Figure 2 can be used to retain the cap seals if there is any chance of the caps coming loose during sampling, shipment, or storage.

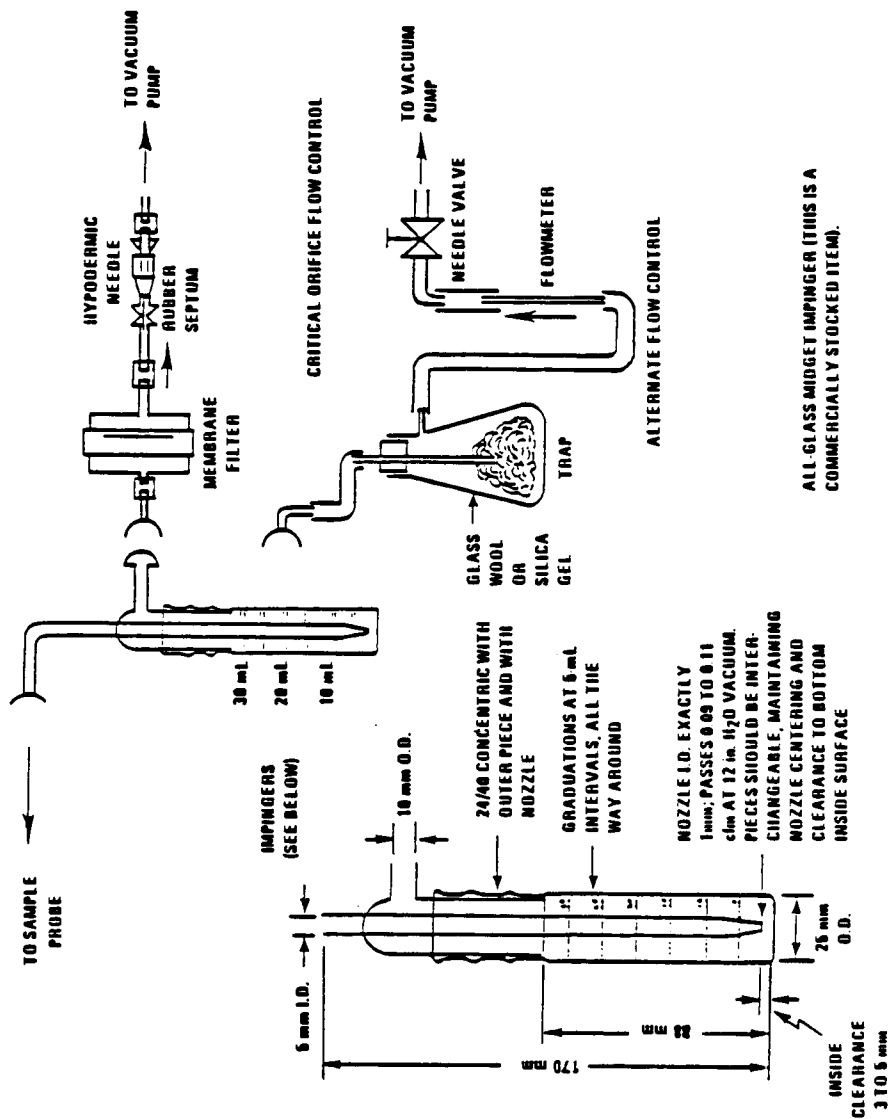


Figure 1. Short term sampling train.

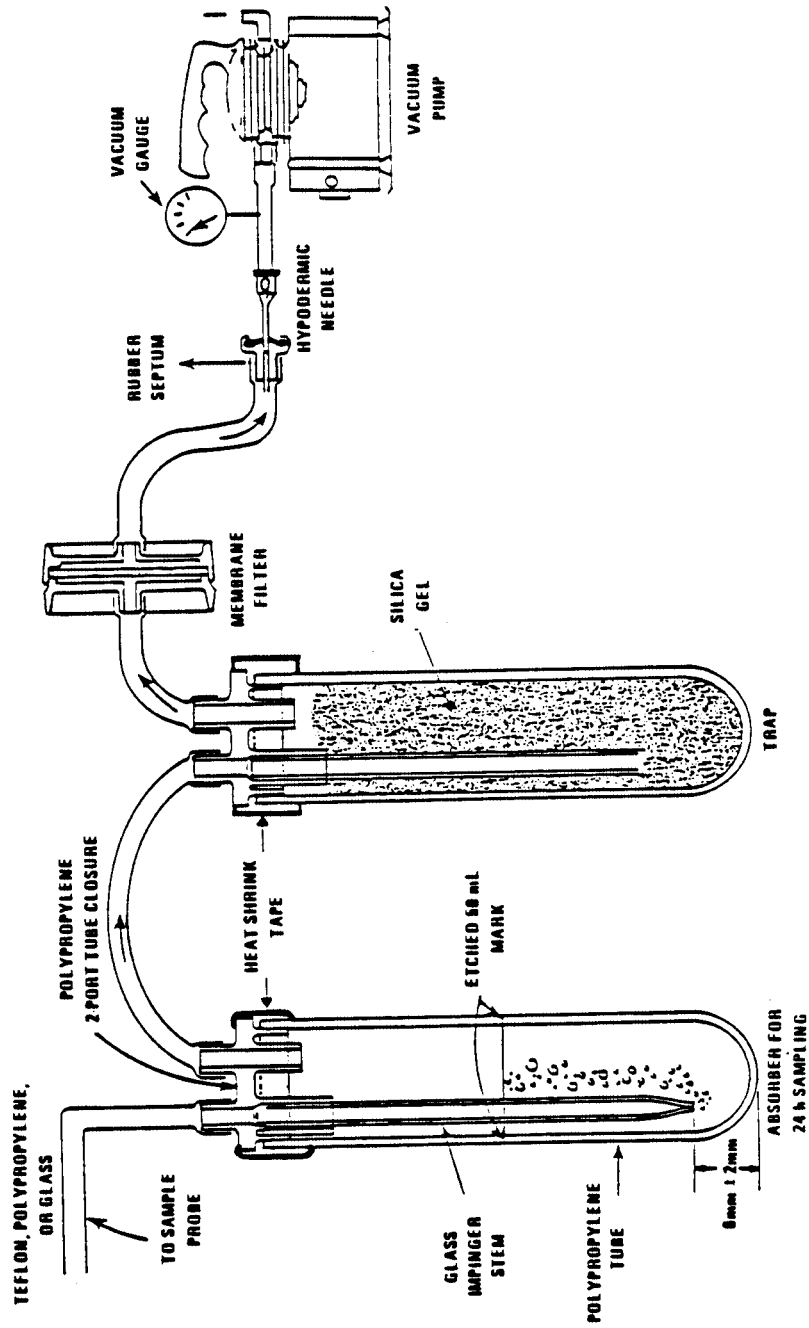


Figure 2. 24-Hour sampling system.

NOTE - A MIDGET IMPINGER IS USED FOR 1 HOUR SAMPLING.

7.1.6 *Flow control device*: A calibrated rotameter and needle valve combination capable of maintaining and measuring air flow to within ± 2 percent is suitable for short-term sampling but may not be used for long-term sampling. A critical orifice can be used for regulating flow rate for both long-term and short-term sampling. A 22-gauge hypodermic needle 25 mm long may be used as a critical orifice to yield a flow rate of approximately 1 L/min for a 30-minute sampling period. When sampling for 1 hour, a 23-gauge hypodermic needle 16 mm in length will provide a flow rate of approximately 0.5 L/min. Flow control for a 24-hour sample may be provided by a 27-gauge hypodermic needle critical orifice that is 9.5 mm in length. The flow rate should be in the range of 0.18 to 0.22 L/min.

7.1.7 *Flow measurement device*: Device calibrated as specified in 9.4.1 and used to measure sample flow rate at the monitoring site.

7.1.8 *Membrane particle filter*: A membrane filter of 0.8 to 2 μm porosity is used to protect the flow controller from particles during long-term sampling. This item is optional for short-term sampling.

7.1.9 *Vacuum pump*: A vacuum pump equipped with a vacuum gauge and capable of maintaining at least 70 kPa (0.7 atm) vacuum differential across the flow control device at the specified flow rate is required for sampling.

7.1.10 *Temperature control device*: The temperature of the absorbing solution during sampling must be maintained at $15^\circ \pm 10^\circ \text{C}$. As soon as possible following sampling and until analysis, the temperature of the collected sample must be maintained at $5^\circ \pm 5^\circ \text{C}$. Where an extended period of time may elapse before the collected sample can be moved to the lower storage temperature, a collection temperature near the lower limit of the $15 \pm 10^\circ \text{C}$ range should be used to minimize losses during this period. Thermoelectric coolers specifically designed for this temperature control are available commercially and normally operate in the range of 5° to 15°C . Small refrigerators can be modified to provide the required temperature control; however, inlet lines must be insulated from the lower temperatures to prevent condensation when sampling under humid conditions. A small heating pad may be necessary when sampling at low temperatures ($<7^\circ \text{C}$) to prevent the absorbing solution from freezing. (17)

7.1.11 *Sampling train container*: The absorbing solution must be shielded from light during and after sampling. Most commercially available sampler trains are enclosed in a light-proof box.

7.1.12 *Timer*: A timer is recommended to initiate and to stop sampling for the 24-hour period. The timer is not a required piece of equipment; however, without the timer a technician would be required to start and stop the sampling manually. An elapsed time

meter is also recommended to determine the duration of the sampling period.

7.2 Shipping.

7.2.1 *Shipping container*: A shipping container that can maintain a temperature of $5^\circ \pm 5^\circ \text{C}$ is used for transporting the sample from the collection site to the analytical laboratory. Ice coolers or refrigerated shipping containers have been found to be satisfactory. The use of eutectic cold packs instead of ice will give a more stable temperature control. Such equipment is available from Cole-Parmer Company, 7425 North Oak Park Avenue, Chicago, IL 60648.

7.3 Analysis.

7.3.1 *Spectrophotometer*: A spectrophotometer suitable for measurement of absorbances at 548 nm with an effective spectral bandwidth of less than 15 nm is required for analysis. If the spectrophotometer reads out in transmittance, convert to absorbance as follows:

$$A = \log_{10}(1/T) \quad (1)$$

where:

A = absorbance, and

T = transmittance ($0 < T < 1$).

A standard wavelength filter traceable to the National Bureau of Standards is used to verify the wavelength calibration according to the procedure enclosed with the filter. The wavelength calibration must be verified upon initial receipt of the instrument and after each 160 hours of normal use or every 6 months, whichever occurs first.

7.3.2 *Spectrophotometer cells*: A set of 1-cm path length cells suitable for use in the visible region is used during analysis. If the cells are unmatched, a matching correction factor must be determined according to Section 10.1.

7.3.3 *Temperature control device*: The color development step during analysis must be conducted in an environment that is in the range of 20° to 30°C and controlled to $\pm 1^\circ \text{C}$. Both calibration and sample analysis must be performed under identical conditions (within 1°C). Adequate temperature control may be obtained by means of constant temperature baths, water baths with manual temperature control, or temperature controlled rooms.

7.3.4 *Glassware*: Class A volumetric glassware of various capacities is required for preparing and standardizing reagents and standards and for dispensing solutions during analysis. These included pipets, volumetric flasks, and burets.

7.3.5 *TCM waste receptacle*: A glass waste receptacle is required for the storage of spent TCM solution. This vessel should be stoppered and stored in a hood at all times.

8.0 Reagents.

8.1 Sampling.

8.1.1 *Distilled water*: Purity of distilled water must be verified by the following procedure:⁽¹⁸⁾

- Place 0.20 mL of potassium permanganate solution (0.316 g/L), 500 mL of distilled water, and 1mL of concentrated sulfuric acid in a chemically resistant glass bottle, stopper the bottle, and allow to stand.
- If the permanganate color (pink) does not disappear completely after a period of 1 hour at room temperature, the water is suitable for use.
- If the permanganate color does disappear, the water can be purified by redistilling with one crystal each of barium hydroxide and potassium permanganate in an all glass still.

8.1.2 *Absorbing reagent* (0.04 M potassium tetrachloromercurate [TCM]): Dissolve 10.86 g mercuric chloride, 0.066 g EDTA, and 6.0 g potassium chloride in distilled water and dilute to volume with distilled water in a 1,000-mL volumetric flask. (*Caution*: Mercuric chloride is highly poisonous. If spilled on skin, flush with water immediately.) The pH of this reagent should be between 3.0 and 5.0 (10) Check the pH of the absorbing solution by using pH indicating paper or a pH meter. If the pH of the solution is not between 3.0 and 5.0, dispose of the solution according to one of the disposal techniques described in Section 13.0. The absorbing reagent is normally stable for 6 months. If a precipitate forms, dispose of the reagent according to one of the procedures described in Section 13.0.

8.2 *Analysis*.

8.2.1 *Sulfamic acid* (0.6%): Dissolve 0.6 g sulfamic acid in 100 mL distilled water. Prepare fresh daily.

8.2.2 *Formaldehyde* (0.2%): Dilute 5 mL formaldehyde solution (36 to 38 percent) to 1,000 mL with distilled water. Prepare fresh daily.

8.2.3 *Stock iodine solution* (0.1 N): Place 12.7 g resublimed iodine in a 250-mL beaker and add 40 g potassium iodide and 25 mL water. Stir until dissolved, transfer to a 1,000 mL volumetric flask and dilute to volume with distilled water.

8.2.4 *Iodine solution* (0.01 N): Prepare approximately 0.01 N iodine solution by diluting 50 mL of stock iodine solution (Section 8.2.3) to 500 mL with distilled water.

8.2.5 *Starch indicator solution*: Triturate 0.4 g soluble starch and 0.002 g mercuric iodide (preservative) with enough distilled water to form a paste. Add the paste slowly to 200 mL of boiling distilled water and continue boiling until clear. Cool and transfer the solution to a glass stoppered bottle.

8.2.6 *1 N hydrochloric acid*: Slowly and while stirring, add 86 mL of concentrated hydrochloric acid to 500 mL of distilled water. Allow to cool and dilute to 1,000 mL with distilled water.

8.2.7 *Potassium iodate solution*: Accurately weigh to the nearest 0.1 mg, 1.5 g (record weight) of primary standard grade potassium iodate that has been previously dried at 180 °C for at least 3 hours and cooled in a dessicator. Dissolve, then dilute to volume in a 500-mL volumetric flask with distilled water.

8.2.8 *Stock sodium thiosulfate solution* (0.1 N): Prepare a stock solution by dissolving 25 g sodium thiosulfate ($\text{Na}_2\text{S}_2\text{O}_3 \cdot 5\text{H}_2\text{O}$) in 1,000 mL freshly boiled, cooled, distilled water and adding 0.1 g sodium carbonate to the solution. Allow the solution to stand at least 1 day before standardizing. To standardize, accurately pipet 50 mL of potassium iodate solution (Section 8.2.7) into a 500-mL iodine flask and add 2.0 g of potassium iodide and 10 mL of 1 N HCl. Stopper the flask and allow to stand for 5 minutes. Titrate the solution with stock sodium thiosulfate solution (Section 8.2.8) to a pale yellow color. Add 5 mL of starch solution (Section 8.2.5) and titrate until the blue color just disappears. Calculate the normality (N_s) of the stock sodium thiosulfate solution as follows:

$$N_s = \frac{W}{M} \times 2.80 \quad (2)$$

where:

M = volume of thiosulfate required in mL, and

W = weight of potassium iodate in g (recorded weight in Section 8.2.7).

$$2.80 = \frac{10^3(\text{conversion of g to mg}) \times 0.1(\text{fraction iodate used})}{35.67(\text{equivalent weight of potassium iodate})}$$

8.2.9 *Working sodium thiosulfate titrant* (0.01 N): Accurately pipet 100 mL of stock sodium thiosulfate solution (Section 8.2.8) into a 1,000-mL volumetric flask and dilute to volume with freshly boiled, cooled, distilled water. Calculate the normality of the working sodium thiosulfate titrant (N_T) as follows:

$$N_T = N_s \times 0.100 \quad (3)$$

8.2.10 *Standardized sulfite solution for the preparation of working sulfite-TCM solution*: Dissolve 0.30 g sodium metabisulfite ($\text{Na}_2\text{S}_2\text{O}_5$) or 0.40 g sodium sulfite (Na_2SO_3) in 500 mL of recently boiled, cooled, distilled water. (Sulfite solution is unstable; it is therefore important to use water of the highest purity to minimize this instability.) This solution contains the equivalent of 320 to 400 $\mu\text{g SO}_2/\text{mL}$. The actual concentration of the solution is determined by adding excess iodine and back-titrating with standard sodium thiosulfate solution. To back-titrate, pipet 50 mL of the 0.01 N iodine solution (Section 8.2.4) into each of two 500-mL iodine flasks (A and B). To flask A (blank) add 25 mL distilled water, and to flask B (sample)

pipet 25 mL sulfite solution. Stopper the flasks and allow to stand for 5 minutes. Prepare the working sulfite-TCM solution (Section 8.2.11) immediately prior to adding the iodine solution to the flasks. Using a buret containing standardized 0.01 N thiosulfate titrant (Section 8.2.9), titrate the solution in each flask to a pale yellow color. Then add 5 mL starch solution (Section 8.2.5) and con-

tinue the titration until the blue color just disappears.

8.2.11 *Working sulfite-TCM solution:* Accurately pipet 5 mL of the standard sulfite solution (Section 8.2.10) into a 250-mL volumetric flask and dilute to volume with 0.04 M TCM. Calculate the concentration of sulfur dioxide in the working solution as follows:

$$C_{\text{TCM}/\text{SO}_2}(\mu\text{g SO}_2/\text{mL}) = \frac{(A - B)(N_T)(32,000)}{25} \times 0.02 \quad (4)$$

where:

A = volume of thiosulfate titrant required for the blank, mL;

B = volume of thiosulfate titrant required for the sample, mL;

N_T = normality of the thiosulfate titrant, from equation (3);

32,000 = milliequivalent weight of SO_2 , μg ;

25 = volume of standard sulfite solution, mL;

and

0.02 = dilution factor.

This solution is stable for 30 days if kept at 5 °C. (16) If not kept at 5 °C, prepare fresh daily.

8.2.12 *Purified pararosaniline (PRA) stock solution* (0.2% nominal):

8.2.12.1 *Dye specifications*—

- The dye must have a maximum absorbance at a wavelength of 540 nm when assayed in a buffered solution of 0.1 M sodium acetate-acetic acid;
- The absorbance of the reagent blank, which is temperature sensitive (0.015 absorbance unit/°C), must not exceed 0.170 at 22 °C with a 1-cm optical path length when the blank is prepared according to the specified procedure;
- The calibration curve (Section 10.0) must have a slope equal to 0.030 ± 0.002 absorbance unit/ $\mu\text{g SO}_2$ with a 1-cm optical path length when the dye is pure and the sulfite solution is properly standardized.

8.2.12.2 *Preparation of stock PRA solution*— A specially purified (99 to 100 percent pure) solution of pararosaniline, which meets the above specifications, is commercially available in the required 0.20 percent concentration (Harleco Co.). Alternatively, the dye may be purified, a stock solution prepared, and then assayed according to the procedure as described below.(10)

8.2.12.3 *Purification procedure for PRA*—

1. Place 100 mL each of 1-butanol and 1 N HCl in a large separatory funnel (250-mL) and allow to equilibrate. Note: Certain batches of 1-butanol contain oxidants that create an SO_2 demand. Before using, check by placing 20 mL of 1-butanol and 5 mL of 20

percent potassium iodide (KI) solution in a 50-mL separatory funnel and shake thoroughly. If a yellow color appears in the alcohol phase, redistill the 1-butanol from silver oxide and collect the middle fraction or purchase a new supply of 1-butanol.

2. Weigh 100 mg of pararosaniline hydrochloride dye (PRA) in a small beaker. Add 50 mL of the equilibrated acid (drain in acid from the bottom of the separatory funnel in 1.) to the beaker and let stand for several minutes. Discard the remaining acid phase in the separatory funnel.

3. To a 125-mL separatory funnel, add 50 mL of the equilibrated 1-butanol (draw the 1-butanol from the top of the separatory funnel in 1.). Transfer the acid solution (from 2.) containing the dye to the funnel and shake carefully to extract. The violet impurity will transfer to the organic phase.

4. Transfer the lower aqueous phase into another separatory funnel, add 20 mL of equilibrated 1-butanol, and extract again.

5. Repeat the extraction procedure with three more 10-mL portions of equilibrated 1-butanol.

6. After the final extraction, filter the acid phase through a cotton plug into a 50-mL volumetric flask and bring to volume with 1 N HCl. This stock reagent will be a yellowish red.

7. To check the purity of the PRA, perform the assay and adjustment of concentration (Section 8.2.12.4) and prepare a reagent blank (Section 11.2); the absorbance of this reagent blank at 540 nm should be less than 0.170 at 22 °C. If the absorbance is greater than 0.170 under these conditions, further extractions should be performed.

8.2.12.4 *PRA assay procedure*— The concentration of pararosaniline hydrochloride (PRA) need be assayed only once after purification. It is also recommended that commercial solutions of pararosaniline be assayed when first purchased. The assay procedure is as follows:(10)

1. Prepare 1 M acetate-acetic acid buffer stock solution with a pH of 4.79 by dissolving

13.61 g of sodium acetate trihydrate in distilled water in a 100-mL volumetric flask. Add 5.70 mL of glacial acetic acid and dilute to volume with distilled water.

2. Pipet 1 mL of the stock PRA solution obtained from the purification process or from a commercial source into a 100-mL volumetric flask and dilute to volume with distilled water.

3. Transfer a 5-mL aliquot of the diluted PRA solution from 2. into a 50-mL volumetric flask. Add 5 mL of 1 M acetate-acetic acid buffer solution from 1. and dilute the mixture to volume with distilled water. Let the mixture stand for 1 hour.

4. Measure the absorbance of the above solution at 540 nm with a spectrophotometer against a distilled water reference. Compute the percentage of nominal concentration of PRA by

$$\% \text{PRA} = \frac{A \times K}{W} \quad (5)$$

where:

A = measured absorbance of the final mixture (absorbance units);

W = weight in grams of the PRA dye used in the assay to prepare 50 mL of stock solution (for example, 0.100 g of dye was used to prepare 50 mL of solution in the purification procedure; when obtained from commercial sources, use the stated concentration to compute W; for 98% PRA, W=.098 g.); and

K = 21.3 for spectrophotometers having a spectral bandwidth of less than 15 nm and a path length of 1 cm.

8.2.13 *Pararosaniline reagent*: To a 250-mL volumetric flask, add 20 mL of stock PRA solution. Add an additional 0.2 mL of stock solution for each percentage that the stock assays below 100 percent. Then add 25 mL of 3 M phosphoric acid and dilute to volume with distilled water. The reagent is stable for at least 9 months. Store away from heat and light.

9.0 Sampling Procedure.

9.1 *General Considerations*. Procedures are described for short-term sampling (30-minute and 1-hour) and for long-term sampling (24-hour). Different combinations of absorbing reagent volume, sampling rate, and sampling time can be selected to meet special needs. For combinations other than those specifi-

cally described, the conditions must be adjusted so that linearity is maintained between absorbance and concentration over the dynamic range. Absorbing reagent volumes less than 10 mL are not recommended. The collection efficiency is above 98 percent for the conditions described; however, the efficiency may be substantially lower when sampling concentrations below $25 \mu\text{gSO}_2/\text{m}^3$.(8,9)

9.2 *30-Minute and 1-Hour Sampling*. Place 10 mL of TCM absorbing reagent in a midjet impinger and seal the impinger with a thin film of silicon stopcock grease (around the ground glass joint). Insert the sealed impinger into the sampling train as shown in Figure 1, making sure that all connections between the various components are leak tight. Greaseless ball joint fittings, heat shrinkable Teflon® tubing, or Teflon® tube fittings may be used to attain leakfree conditions for portions of the sampling train that come into contact with air containing SO_2 . Shield the absorbing reagent from direct sunlight by covering the impinger with aluminum foil or by enclosing the sampling train in a light-proof box. Determine the flow rate according to Section 9.4.2. Collect the sample at 1 ± 0.10 L/min for 30-minute sampling or 0.500 ± 0.05 L/min for 1-hour sampling. Record the exact sampling time in minutes, as the sample volume will later be determined using the sampling flow rate and the sampling time. Record the atmospheric pressure and temperature.

9.3 *24-Hour Sampling*. Place 50 mL of TCM absorbing solution in a large absorber, close the cap, and, if needed, apply the heat shrink material as shown in Figure 3. Verify that the reagent level is at the 50 mL mark on the absorber. Insert the sealed absorber into the sampling train as shown in Figure 2. At this time verify that the absorber temperature is controlled to 15 ± 10 °C. During sampling, the absorber temperature must be controlled to prevent decomposition of the collected complex. From the onset of sampling until analysis, the absorbing solution must be protected from direct sunlight. Determine the flow rate according to Section 9.4.2. Collect the sample for 24 hours from midnight to midnight at a flow rate of 0.200 ± 0.020 L/min. A start/stop timer is helpful for initiating and stopping sampling and an elapsed time meter will be useful for determining the sampling time.

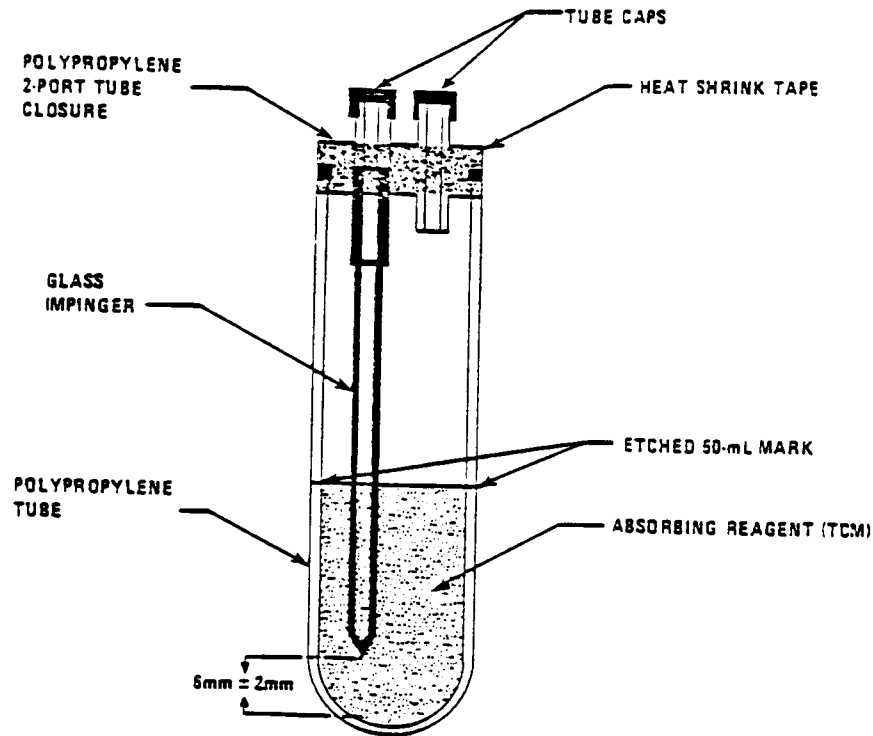


Figure 3. An absorber (24-hour sample) filled and assembled for shipment.

9.4 Flow Measurement.

9.4.1 *Calibration:* Flow measuring devices used for the on-site flow measurements required in 9.4.2 must be calibrated against a reliable flow or volume standard such as an NBS traceable bubble flowmeter or calibrated wet test meter. Rotameters or critical orifices used in the sampling train may be calibrated, if desired, as a quality control check, but such calibration shall not replace the on-site flow measurements required by 9.4.2. In-line rotameters, if they are to be calibrated, should be calibrated in situ, with the appropriate volume of solution in the absorber.

9.4.2 *Determination of flow rate at sampling site:* For short-term samples, the standard flow rate is determined at the sampling site at the initiation and completion of sample collection with a calibrated flow measuring device connected to the inlet of the absorber.

For 24-hour samples, the standard flow rate is determined at the time the absorber is placed in the sampling train and again when the absorber is removed from the train for shipment to the analytical laboratory with a calibrated flow measuring device connected to the inlet of the sampling train. The flow rate determination must be made with all components of the sampling system in operation (e.g., the absorber temperature controller and any sample box heaters must also be operating). Equation 6 may be used to determine the standard flow rate when a calibrated positive displacement meter is used as the flow measuring device. Other types of calibrated flow measuring devices may also be used to determine the flow rate at the sampling site provided that the user applies any appropriate corrections to devices for which output is dependent on temperature or pressure.

$$Q_{\text{std}} = Q_{\text{act}} \times \frac{P_b - (1 - \text{RH})P_{\text{H}_2\text{O}}}{P_{\text{std}}} \times \frac{298.16}{(T_{\text{meter}} + 273.16)} \quad (6)$$

where:

Q_{std} = flow rate at standard conditions, std L/min (25 °C and 760 mm Hg);

Q_{act} = flow rate at monitoring site conditions, L/min;

P_b = barometric pressure at monitoring site conditions, mm Hg or kPa;

RH = fractional relative humidity of the air being measured;

$P_{\text{H}_2\text{O}}$ = vapor pressure of water at the temperature of the air in the flow or volume standard, in the same units as P_b , (for wet

volume standards only, i.e., bubble flowmeter or wet test meter; for dry standards, i.e., dry test meter, $P_{\text{H}_2\text{O}}=0$);

P_{std} = standard barometric pressure, in the same units as P_b (760 mm Hg or 101 kPa); and

T_{meter} = temperature of the air in the flow or volume standard, °C (e.g., bubble flowmeter).

If a barometer is not available, the following equation may be used to determine the barometric pressure:

$$P_b = 760 - .076(H) \text{ mm Hg, or } P_b = 101 - .01(H) \text{ kPa} \quad (7)$$

where:

H = sampling site elevation above sea level in meters.

If the initial flow rate (Q_i) differs from the flow rate of the critical orifice or the flow rate indicated by the flowmeter in the sampling train (Q_c) by more than 5 percent as determined by equation (8), check for leaks and redetermine Q_i .

$$\% \text{ Diff} = \frac{Q_i - Q_c}{Q_c} \times 100 \quad (8)$$

Invalidate the sample if the difference between the initial (Q_i) and final (Q_f) flow rates is more than 5 percent as determined by equation (9):

$$\% \text{ Diff} = \frac{Q_i - Q_f}{Q_f} \times 100 \quad (9)$$

9.5 Sample Storage and Shipment. Remove the impinger or absorber from the sampling train and stopper immediately. Verify that the temperature of the absorber is not above 25 °C. Mark the level of the solution with a temporary (e.g., grease pencil) mark. If the sample will not be analyzed within 12 hours of sampling, it must be stored at 5° ±5 °C until analysis. Analysis must occur within 30 days. If the sample is transported or shipped for a period exceeding 12 hours, it is recommended that thermal coolers using eutectic ice packs, refrigerated shipping containers, etc., be used for periods up to 48 hours. (17) Measure the temperature of the absorber solution when the shipment is received. Invalidate the sample if the tempera-

ture is above 10 °C. Store the sample at 5° ±5 °C until it is analyzed.

10.0 Analytical Calibration.

10.1 Spectrophotometer Cell Matching. If unmatched spectrophotometer cells are used, an absorbance correction factor must be determined as follows:

1. Fill all cells with distilled water and designate the one that has the lowest absorbance at 548 nm as the reference. (This reference cell should be marked as such and continually used for this purpose throughout all future analyses.)

2. Zero the spectrophotometer with the reference cell.

3. Determine the absorbance of the remaining cells (A_c) in relation to the reference cell and record these values for future use. Mark all cells in a manner that adequately identifies the correction.

The corrected absorbance during future analyses using each cell is determining as follows:

$$A = A_{\text{obs}} - A_c \quad (10)$$

where:

A = corrected absorbance,

A_{obs} = uncorrected absorbance, and

A_c = cell correction.

10.2 Static Calibration Procedure (Option 1). Prepare a dilute working sulfite-TCM solution by diluting 10 mL of the working sulfite-TCM solution (Section 8.2.11) to 100 mL with TCM absorbing reagent. Following the table below, accurately pipet the indicated volumes of the sulfite-TCM solutions into a series of 25-mL volumetric flasks. Add TCM absorbing reagent as indicated to bring the volume in each flask to 10 mL.

Sulfite-TCM solution	Volume of sulfite-TCM solution	Volume of TCM, mL	Total $\mu\text{g SO}_2$ (approx.*
Working	4.0	6.0	28.8
Working	3.0	7.0	21.6
Working	2.0	8.0	14.4
Dilute working	10.0	0.0	7.2
Dilute working	5.0	5.0	3.6
	0.0	10.0	0.0

*Based on working sulfite-TCM solution concentration of 7.2 $\mu\text{g SO}_2/\text{mL}$; the actual total $\mu\text{g SO}_2$ must be calculated using equation 11 below.

To each volumetric flask, add 1 mL 0.6% sulfamic acid (Section 8.2.1), accurately pipet 2 mL 0.2% formaldehyde solution (Section 8.2.2), then add 5 mL pararosaniline solution (Section 8.2.13). Start a laboratory

timer that has been set for 30 minutes. Bring all flasks to volume with recently boiled and cooled distilled water and mix thoroughly. The color must be developed (during the 30-minute period) in a temperature environment in the range of 20° to 30 °C, which is controlled to ± 1 °C. For increased precision, a constant temperature bath is recommended during the color development step. After 30 minutes, determine the corrected absorbance of each standard at 548 nm against a distilled water reference (Section 10.1). Denote this absorbance as (A). Distilled water is used in the reference cell rather than the reagent blank because of the temperature sensitivity of the reagent blank. Calculate the total micrograms SO_2 in each solution:

$$\mu\text{g SO}_2 = V_{\text{TCM}/\text{SO}_2} \times C_{\text{TCM}/\text{SO}_2} \times D \quad (11)$$

where:

$V_{\text{TCM}/\text{SO}_2}$ = volume of sulfite-TCM solution used, mL;

$C_{\text{TCM}/\text{SO}_2}$ = concentration of sulfur dioxide in the working sulfite-TCM, $\mu\text{g SO}_2/\text{mL}$ (from equation 4); and

D = dilution factor (D = 1 for the working sulfite-TCM solution; D = 0.1 for the diluted working sulfite-TCM solution).

A calibration equation is determined using the method of linear least squares (Section 12.1). The total micrograms SO_2 contained in each solution is the x variable, and the corrected absorbance (eq. 10) associated with each solution is the y variable. For the calibration to be valid, the slope must be in the range of 0.030 ± 0.002 absorbance unit/ $\mu\text{g SO}_2$, the intercept as determined by the least squares method must be equal to or less than 0.170 absorbance unit when the color is developed at 22 °C (add 0.015 to this 0.170 specification for each °C above 22 °C) and the correlation coefficient must be greater than 0.998. If these criteria are not met, it may be the result of an impure dye and/or an improperly standardized sulfite-TCM solution. A calibration factor (B_s) is determined by calculating the reciprocal of the slope and is subsequently used for calculating the sample concentration (Section 12.3).

10.3 *Dynamic Calibration Procedures* (Option 2). Atmospheres containing accurately known concentrations of sulfur dioxide are prepared using permeation devices. In the systems for generating these atmospheres, the permeation device emits gaseous SO_2 at a known, low, constant rate, provided the temperature of the device is held constant (± 0.1 °C) and the device has been accurately calibrated at the temperature of use. The SO_2 permeating from the device is carried by

a low flow of dry carrier gas to a mixing chamber where it is diluted with SO_2 -free air to the desired concentration and supplied to a vented manifold. A typical system is shown schematically in Figure 4 and this system and other similar systems have been described in detail by O'Keefe and Ortman; (19) Scaringelli, Frey, and Saltzman, (20) and Scaringelli, O'Keefe, Rosenberg, and Bell. (21) Permeation devices may be prepared or purchased and in both cases must be traceable either to a National Bureau of Standards (NBS) Standard Reference Material (SRM 1625, SRM 1626, SRM 1627) or to an NBS/EPA-approved commercially available Certified Reference Material (CRM). CRM's are described in Reference 22, and a list of CRM sources is available from the address shown for Reference 22. A recommended protocol for certifying a permeation device to an NBS SRM or CRM is given in Section 2.0.7 of Reference 2. Device permeation rates of 0.2 to 0.4 $\mu\text{g}/\text{min}$, inert gas flows of about 50 mL/min, and dilution air flow rates from 1.1 to 15 L/min conveniently yield standard atmospheres in the range of 25 to 600 $\mu\text{g SO}_2/\text{m}^3$ (0.010 to 0.230 ppm).

10.3.1 *Calibration Option 2A* (30-minute and 1-hour samples): Generate a series of six standard atmospheres of SO_2 (e.g., 0, 50, 100, 200, 350, 500, 750 $\mu\text{g}/\text{m}^3$) by adjusting the dilution flow rates appropriately. The concentration of SO_2 in each atmosphere is calculated as follows:

$$C_a = \frac{P_r \times 10^3}{Q_d + Q_p} \quad (12)$$

where:

C_a = concentration of SO_2 at standard conditions, $\mu g/m^3$;
 P_r = permeation rate, $\mu g/min$;

Q_d = flow rate of dilution air, std L/min; and
 Q_p = flow rate of carrier gas across permeation device, std L/min.

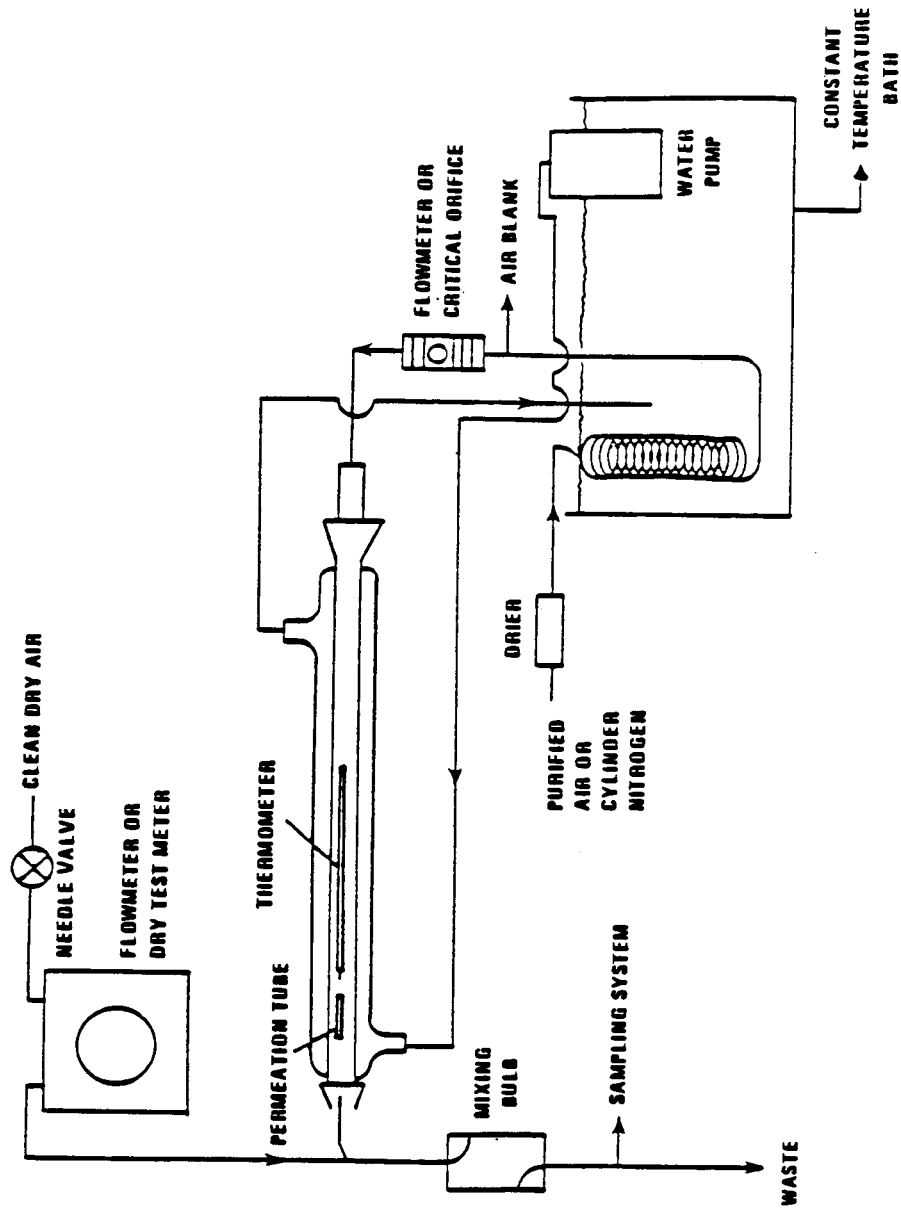


Figure 4. Permeation tube schematic for laboratory use.

Be sure that the total flow rate of the standard exceeds the flow demand of the sample train, with the excess flow vented at atmospheric pressure. Sample each atmosphere using similar apparatus as shown in Figure 1 and under the same conditions as field sampling (i.e., use same absorbing reagent volume and sample same volume of air at an equivalent flow rate). Due to the length of the sampling periods required, this method is not recommended for 24-hour sampling. At the completion of sampling, quantitatively transfer the contents of each impinger to one of a series of 25-mL volumetric flasks (if 10 mL of absorbing solution was used) using small amounts of distilled water for rinse (<5mL). If >10 mL of absorbing solution was used, bring the absorber solution in each impinger to original volume with distilled H₂O and pipet 10-mL portions from each impinger into a series of 25-mL volumetric flasks. If the color development steps are not to be started within 12 hours of sampling, store the solutions at 5° ± 5 °C. Calculate the total micrograms SO₂ in each solution as follows:

$$\mu\text{gSO}_2 = \frac{C_a \times Q_s \times t \times V_a \times 10^{-3}}{V_b} \quad (13)$$

where:

C_a = concentration of SO₂ in the standard atmosphere, µg/m³;

Q_s = sampling flow rate, std L/min;

t = sampling time, min;

V_a = volume of absorbing solution used for color development (10 mL); and

V_b = volume of absorbing solution used for sampling, mL.

Add the remaining reagents for color development in the same manner as in Section 10.2 for static solutions. Calculate a calibration equation and a calibration factor (B₂) according to Section 10.2, adhering to all the specified criteria.

10.3.2 Calibration Option 2B (24-hour samples): Generate a standard atmosphere containing approximately 1,050 µg SO₂/m³ and calculate the exact concentration according to equation 12. Set up a series of six absorbers according to Figure 2 and connect to a common manifold for sampling the standard atmosphere. Be sure that the total flow rate of the standard exceeds the flow demand at the sample manifold, with the excess flow vented at atmospheric pressure. The absorbers are then allowed to sample the atmosphere for varying time periods to yield solutions containing 0, 0.2, 0.6, 1.0, 1.4, 1.8, and 2.2 µg SO₂/mL solution. The sampling times required to attain these solution concentrations are calculated as follows:

$$t = \frac{V_b \times C_s}{C_a \times Q_s \times 10^{-3}} \quad (14)$$

where:

t = sampling time, min;

V_b = volume of absorbing solution used for sampling (50 mL);

C_s = desired concentration of SO₂ in the absorbing solution, µg/mL;

C_a = concentration of the standard atmosphere calculated according to equation 12, µg/m³; and

Q_s = sampling flow rate, std L/min.

At the completion of sampling, bring the absorber solutions to original volume with distilled water. Pipet a 10-mL portion from each absorber into one of a series of 25-mL volumetric flasks. If the color development steps are not to be started within 12 hours of sampling, store the solutions at 5° ± 5 °C. Add the remaining reagents for color development in the same manner as in Section 10.2 for static solutions. Calculate the total µg SO₂ in each standard as follows:

$$\mu\text{gSO}_2 = \frac{C_a \times Q_s \times t \times V_a \times 10^{-3}}{V_b} \quad (15)$$

where:

V_a = volume of absorbing solution used for color development (10 mL).

All other parameters are defined in equation 14.

Calculate a calibration equation and a calibration factor (B₂) according to Section 10.2 adhering to all the specified criteria.

11.0 Sample Preparation and Analysis.

11.1 *Sample Preparation.* Remove the samples from the shipping container. If the shipment period exceeded 12 hours from the completion of sampling, verify that the temperature is below 10 °C. Also, compare the solution level to the temporary level mark on the absorber. If either the temperature is above 10 °C or there was significant loss (more than 10 mL) of the sample during shipping, make an appropriate notation in the record and invalidate the sample. Prepare the samples for analysis as follows:

1. For 30-minute or 1-hour samples: Quantitatively transfer the entire 10 mL amount of absorbing solution to a 25-mL volumetric flask and rinse with a small amount (<5 mL) of distilled water.

2. For 24-hour samples: If the volume of the sample is less than the original 50-mL volume (permanent mark on the absorber), adjust the volume back to the original volume with distilled water to compensate for water lost to evaporation during sampling. If the final volume is greater than the original volume, the volume must be measured using a graduated cylinder. To analyze, pipet 10 mL

of the solution into a 25-mL volumetric flask.

11.2 *Sample Analysis.* For each set of determinations, prepare a reagent blank by adding 10 mL TCM absorbing solution to a 25-mL volumetric flask, and two control standards containing approximately 5 and 15 $\mu\text{g SO}_2$, respectively. The control standards are prepared according to Section 10.2 or 10.3. The analysis is carried out as follows:

1. Allow the sample to stand 20 minutes after the completion of sampling to allow any ozone to decompose (if applicable).

2. To each 25-mL volumetric flask containing reagent blank, sample, or control standard, add 1 mL of 0.6% sulfamic acid (Section 8.2.1) and allow to react for 10 min.

3. Accurately pipet 2 mL of 0.2% formaldehyde solution (Section 8.2.2) and then 5 mL of pararosaniline solution (Section 8.2.13) into each flask. Start a laboratory timer set at 30 minutes.

4. Bring each flask to volume with recently boiled and cooled distilled water and mix thoroughly.

5. During the 30 minutes, the solutions must be in a temperature controlled environment in the range of 20° to 30 °C maintained to ± 1 °C. This temperature must also be within 1 °C of that used during calibration.

6. After 30 minutes and before 60 minutes, determine the corrected absorbances (equation 10) of each solution at 548 nm using 1-cm optical path length cells against a distilled water reference (Section 10.1). (*Distilled water is used as a reference instead of the reagent blank because of the sensitivity of the reagent blank to temperature.*)

7. Do not allow the colored solution to stand in the cells because a film may be deposited. Clean the cells with isopropyl alcohol after use.

8. The reagent blank must be within 0.03 absorbance units of the intercept of the calibration equation determined in Section 10.

11.3 *Absorbance range.* If the absorbance of the sample solution ranges between 1.0 and 2.0, the sample can be diluted 1:1 with a portion of the reagent blank and the absorbance redetermined within 5 minutes. Solutions with higher absorbances can be diluted up to sixfold with the reagent blank in order to obtain scale readings of less than 1.0 absorbance unit. However, it is recommended that a smaller portion (<10 mL) of the original sample be reanalyzed (if possible) if the sample requires a dilution greater than 1:1.

11.4 *Reagent disposal.* All reagents containing mercury compounds must be stored and disposed of using one of the procedures contained in Section 13. Until disposal, the discarded solutions can be stored in closed glass containers and should be left in a fume hood.

12.0 Calculations.

12.1 *Calibration Slope, Intercept, and Correlation Coefficient.* The method of least squares

is used to calculate a calibration equation in the form of:

$$y = mx + b \quad (16)$$

where:

y = corrected absorbance,

m = slope, absorbance unit/ $\mu\text{g SO}_2$,

x = micrograms of SO_2 ,

b = y intercept (absorbance units).

The slope (m), intercept (b), and correlation coefficient (r) are calculated as follows:

$$m = \frac{n \sum xy - (\sum x)(\sum y)}{n \sum x^2 - (\sum x)^2} \quad (17)$$

$$b = \frac{\sum y - m \sum x}{n} \quad (18)$$

$$r = \frac{m(\sum xy - \sum x \sum y / n)}{\sqrt{\sum y^2 - (\sum y)^2 / n}} \quad (19)$$

where n is the number of calibration points.

A data form (Figure 5) is supplied for easily organizing calibration data when the slope, intercept, and correlation coefficient are calculated by hand.

12.2 *Total Sample Volume.* Determine the sampling volume at standard conditions as follows:

$$V_{\text{std}} = \frac{Q_i + Q_f}{2} \times t \quad (20)$$

where:

V_{std} = sampling volume in std L,

Q_i = standard flow rate determined at the initiation of sampling in std L/min,

Q_f = standard flow rate determined at the completion of sampling in std L/min, and

t = total sampling time, min.

12.3 *Sulfur Dioxide Concentration.* Calculate and report the concentration of each sample as follows:

$$\mu\text{g SO}_2/\text{m}^3 = \frac{(A - A_o)(B_x)(10^3)}{V_{\text{std}}} \times \frac{V_b}{V_a} \quad (21)$$

where:

A = corrected absorbance of the sample solution, from equation (10);

A_o = corrected absorbance of the reagent blank, using equation (10);

B_x = calibration factor equal to B_s , B_g , or B_i depending on the calibration procedure used, the reciprocal of the slope of the calibration equation;

V_a = volume of absorber solution analyzed, mL;

V_b = total volume of solution in absorber (see 11.1-2), mL; and

V_{std} = standard air volume sampled, std L (from Section 12.2).

DATA FORM
[For hand calculations]

Calibration point no.	Micrograms SO ₂	Absorbance units			
	(x)	(y)	x ²	xy	y ²
1
2
3
4
5
6

Σ x= _____ Σ y= _____ Σ x²= _____ Σ xy _____
 Σ y² _____
 n= _____ (number of pairs of coordinates.)

FIGURE 5. Data form for hand calculations.

12.4 Control Standards. Calculate the analyzed micrograms of SO₂ in each control standard as follows:

$$C_q = (A - A_o) \times B_x \quad (22)$$

where:

C_q = analyzed μg SO₂ in each control standard,

A = corrected absorbance of the control standard, and

A_o = corrected absorbance of the reagent blank.

The difference between the true and analyzed values of the control standards must not be greater than 1 μg. If the difference is greater than 1 μg, the source of the discrepancy must be identified and corrected.

12.5 Conversion of μg/m³ to ppm (v/v). If desired, the concentration of sulfur dioxide at reference conditions can be converted to ppm SO₂ (v/v) as follows:

$$\text{ppm SO}_2 = \frac{\mu\text{g SO}_2}{\text{m}^3} \times 3.82 \times 10^{-4} \quad (23)$$

13.0 The TCM absorbing solution and any reagents containing mercury compounds must be treated and disposed of by one of the methods discussed below. Both methods remove greater than 99.99 percent of the mercury.

13.1 *Disposal of Mercury-Containing Solutions.*

13.2 *Method for Forming an Amalgam.*

1. Place the waste solution in an uncapped vessel in a hood.

2. For each liter of waste solution, add approximately 10 g of sodium carbonate until neutralization has occurred (NaOH may have to be used).

3. Following neutralization, add 10 g of granular zinc or magnesium.

4. Stir the solution in a hood for 24 hours. Caution must be exercised as hydrogen gas is evolved by this treatment process.

5. After 24 hours, allow the solution to stand without stirring to allow the mercury amalgam (solid black material) to settle to the bottom of the waste receptacle.

6. Upon settling, decant and discard the supernatant liquid.

7. Quantitatively transfer the solid material to a container and allow to dry.

8. The solid material can be sent to a mercury reclaiming plant. It must not be discarded.

13.3 *Method Using Aluminum Foil Strips.*

1. Place the waste solution in an uncapped vessel in a hood.

2. For each liter of waste solution, add approximately 10 g of aluminum foil strips. If all the aluminum is consumed and no gas is evolved, add an additional 10 g of foil. Repeat until the foil is no longer consumed and allow the gas to evolve for 24 hours.

3. Decant the supernatant liquid and discard.

4. Transfer the elemental mercury that has settled to the bottom of the vessel to a storage container.

5. The mercury can be sent to a mercury reclaiming plant. It must not be discarded.

14.0 *References for SO₂ Method.*

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[47 FR 54899, Dec. 6, 1982; 48 FR 17355, Apr. 22, 1983]

APPENDIX B TO PART 50—REFERENCE METHOD FOR THE DETERMINATION OF SUSPENDED PARTICULATE MATTER IN THE ATMOSPHERE (HIGH-VOLUME METHOD)

1.0 *Applicability.*

1.1 This method provides a measurement of the mass concentration of total suspended particulate matter (TSP) in ambient air for determining compliance with the primary and secondary national ambient air quality standards for particulate matter as specified in §50.6 and §50.7 of this chapter. The measurement process is nondestructive, and the size of the sample collected is usually adequate for subsequent chemical analysis. Quality assurance procedures and guidance are provided in part 58, appendixes A and B, of this chapter and in References 1 and 2.

2.0 *Principle.*

2.1 An air sampler, properly located at the measurement site, draws a measured quantity of ambient air into a covered housing and through a filter during a 24-hr (nominal) sampling period. The sampler flow rate and the geometry of the shelter favor the collection of particles up to 25–50 μm (aerodynamic diameter), depending on wind speed and direction.⁽³⁾ The filters used are specified to have a minimum collection efficiency of 99 percent for 0.3 μm (DOP) particles (see Section 7.1.4).

2.2 The filter is weighed (after moisture equilibration) before and after use to determine the net weight (mass) gain. The total volume of air sampled, corrected to EPA standard conditions (25 °C, 760 mm Hg [101 kPa]), is determined from the measured flow rate and the sampling time. The concentration of total suspended particulate matter in the ambient air is computed as the mass of collected particles divided by the volume of air sampled, corrected to standard conditions, and is expressed in micrograms per standard cubic meter ($\mu\text{g}/\text{std m}^3$). For samples collected at temperatures and pressures significantly different than standard conditions, these corrected concentrations may differ substantially from actual concentrations (micrograms per actual cubic meter), particularly at high elevations. The actual particulate matter concentration can be calculated from the corrected concentration using the actual temperature and pressure during the sampling period.

3.0 *Range.*

3.1 The approximate concentration range of the method is 2 to 750 $\mu\text{g}/\text{std m}^3$. The upper limit is determined by the point at which the sampler can no longer maintain the specified

flow rate due to the increased pressure drop of the loaded filter. This point is affected by particle size distribution, moisture content of the collected particles, and variability from filter to filter, among other things. The lower limit is determined by the sensitivity of the balance (see Section 7.10) and by inherent sources of error (see Section 6).

3.2 At wind speeds between 1.3 and 4.5 m/sec (3 and 10 mph), the high-volume air sampler has been found to collect particles up to 25 to 50 μm , depending on wind speed and direction.(3) For the filter specified in Section 7.1, there is effectively no lower limit on the particle size collected.

4.0 Precision.

4.1 Based upon collaborative testing, the relative standard deviation (coefficient of variation) for single analyst precision (repeatability) of the method is 3.0 percent. The corresponding value for interlaboratory precision (reproducibility) is 3.7 percent.(4)

5.0 Accuracy.

5.1 The absolute accuracy of the method is undefined because of the complex nature of atmospheric particulate matter and the difficulty in determining the "true" particulate matter concentration. This method provides a measure of particulate matter concentration suitable for the purpose specified under Section 1.0, Applicability.

6.0 Inherent Sources of Error.

6.1 *Airflow variation.* The weight of material collected on the filter represents the (integrated) sum of the product of the instantaneous flow rate times the instantaneous particle concentration. Therefore, dividing this weight by the average flow rate over the sampling period yields the true particulate matter concentration only when the flow rate is constant over the period. The error resulting from a nonconstant flow rate depends on the magnitude of the instantaneous changes in the flow rate and in the particulate matter concentration. Normally, such errors are not large, but they can be greatly reduced by equipping the sampler with an automatic flow controlling mechanism that maintains constant flow during the sampling period. Use of a constant flow controller is recommended.*

6.2 *Air volume measurement.* If the flow rate changes substantially or nonuniformly during the sampling period, appreciable error in the estimated air volume may result from using the average of the presampling and postsampling flow rates. Greater air volume measurement accuracy may be achieved by (1) equipping the sampler with a flow controlling mechanism that maintains constant air flow during the sampling period,* (2)

using a calibrated, continuous flow rate recording device to record the actual flow rate during the sampling period and integrating the flow rate over the period, or (3) any other means that will accurately measure the total air volume sampled during the sampling period. Use of a continuous flow recorder is recommended, particularly if the sampler is not equipped with a constant flow controller.

6.3 *Loss of volatiles.* Volatile particles collected on the filter may be lost during subsequent sampling or during shipment and/or storage of the filter prior to the postsampling weighing.(5) Although such losses are largely unavoidable, the filter should be reweighed as soon after sampling as practical.

6.4 *Artifact particulate matter.* Artifact particulate matter can be formed on the surface of alkaline glass fiber filters by oxidation of acid gases in the sample air, resulting in a higher than true TSP determination.(6 7) This effect usually occurs early in the sample period and is a function of the filter pH and the presence of acid gases. It is generally believed to account for only a small percentage of the filter weight gain, but the effect may become more significant where relatively small particulate weights are collected.

6.5 *Humidity.* Glass fiber filters are comparatively insensitive to changes in relative humidity, but collected particulate matter can be hygroscopic.(8) The moisture conditioning procedure minimizes but may not completely eliminate error due to moisture.

6.6 *Filter handling.* Careful handling of the filter between the presampling and postsampling weighings is necessary to avoid errors due to loss of fibers or particles from the filter. A filter paper cartridge or cassette used to protect the filter can minimize handling errors. (See Reference 2, Section 2).

6.7 *Nonsampled particulate matter.* Particulate matter may be deposited on the filter by wind during periods when the sampler is inoperative. (9) It is recommended that errors from this source be minimized by an automatic mechanical device that keeps the filter covered during nonsampling periods, or by timely installation and retrieval of filters to minimize the nonsampling periods prior to and following operation.

6.8 *Timing errors.* Samplers are normally controlled by clock timers set to start and stop the sampler at midnight. Errors in the nominal 1,440-min sampling period may result from a power interruption during the sampling period or from a discrepancy between the start or stop time recorded on the filter information record and the actual start or stop time of the sampler. Such discrepancies may be caused by (1) poor resolution of the timer set-points, (2) timer error due to power interruption, (3) missetting of

*At elevated altitudes, the effectiveness of automatic flow controllers may be reduced because of a reduction in the maximum sampler flow.

the timer, or (4) timer malfunction. In general, digital electronic timers have much better set-point resolution than mechanical timers, but require a battery backup system to maintain continuity of operation after a power interruption. A continuous flow recorder or elapsed time meter provides an indication of the sampler run-time, as well as indication of any power interruption during the sampling period and is therefore recommended.

6.9 *Recirculation of sampler exhaust.* Under stagnant wind conditions, sampler exhaust air can be resampled. This effect does not appear to affect the TSP measurement substantially, but may result in increased carbon and copper in the collected sample. (10) This problem can be reduced by ducting the exhaust air well away, preferably downwind, from the sampler.

7.0 Apparatus.

(See References 1 and 2 for quality assurance information.)

NOTE: Samplers purchased prior to the effective date of this amendment are not subject to specifications preceded by (†).

7.1 *Filter.* (Filters supplied by the Environmental Protection Agency can be assumed to meet the following criteria. Additional specifications are required if the sample is to be analyzed chemically.)

7.1.1 *Size:* 20.3±0.2×25.4±0.2 cm (nominal 8×10 in).

7.1.2 *Nominal exposed area:* 406.5 cm² (63 in²).

7.1.3 *Material:* Glass fiber or other relatively inert, nonhygroscopic material. (8)

7.1.4 *Collection efficiency:* 99 percent minimum as measured by the DOP test (ASTM-2986) for particles of 0.3 μm diameter.

7.1.5 *Recommended pressure drop range:* 42–54 mm Hg (5.6–7.2 kPa) at a flow rate of 1.5 std m³/min through the nominal exposed area.

7.1.6 *pH:* 6 to 10. (11)

7.1.7 *Integrity:* 2.4 mg maximum weight loss. (11)

7.1.8 *Pinholes:* None.

7.1.9 *Tear strength:* 500 g minimum for 20 mm wide strip cut from filter in weakest dimension. (See ASTM Test D828–60).

7.1.10 *Brittleness:* No cracks or material separations after single lengthwise crease.

7.2 *Sampler.* The air sampler shall provide means for drawing the air sample, via reduced pressure, through the filter at a uniform face velocity.

7.2.1 The sampler shall have suitable means to:

a. Hold and seal the filter to the sampler housing.

b. Allow the filter to be changed conveniently.

c. Preclude leaks that would cause error in the measurement of the air volume passing through the filter.

d. (†) Manually adjust the flow rate to accommodate variations in filter pressure drop and site line voltage and altitude. The adjustment may be accomplished by an automatic flow controller or by a manual flow adjustment device. Any manual adjustment device must be designed with positive detents or other means to avoid unintentional changes in the setting.

7.2.2 *Minimum sample flow rate, heavily loaded filter:* 1.1 m³/min (39 ft³/min).‡

7.2.3 *Maximum sample flow rate, clean filter:* 1.7 m³/min (60 ft³/min).‡

7.2.4 *Blower Motor:* The motor must be capable of continuous operation for 24-hr periods.

7.3 Sampler shelter.

7.3.1 The sampler shelter shall:

a. Maintain the filter in a horizontal position at least 1 m above the sampler supporting surface so that sample air is drawn downward through the filter.

b. Be rectangular in shape with a gabled roof, similar to the design shown in Figure 1.

c. Cover and protect the filter and sampler from precipitation and other weather.

d. Discharge exhaust air at least 40 cm from the sample air inlet.

e. Be designed to minimize the collection of dust from the supporting surface by incorporating a baffle between the exhaust outlet and the supporting surface.

7.3.2 The sampler cover or roof shall overhang the sampler housing somewhat, as shown in Figure 1, and shall be mounted so as to form an air inlet gap between the cover and the sampler housing walls. † This sample air inlet should be approximately uniform on all sides of the sampler. † The area of the sample air inlet must be sized to provide an effective particle capture air velocity of between 20 and 35 cm/sec at the recommended operational flow rate. The capture velocity is the sample air flow rate divided by the inlet area measured in a horizontal plane at the lower edge of the cover. † Ideally, the inlet area and operational flow rate should be selected to obtain a capture air velocity of 25 ±2 cm/sec.

7.4 Flow rate measurement devices.

7.4.1 The sampler shall incorporate a flow rate measurement device capable of indicating the total sampler flow rate. Two common types of flow indicators covered in the calibration procedure are (1) an electronic mass flowmeter and (2) an orifice or orifices

(†) See note at beginning of Section 7 of this appendix.

‡ These specifications are in actual air volume units; to convert to EPA standard air volume units, multiply the specifications by $(P_b/P_{std})(298/T)$ where P_b and T are the barometric pressure in mm Hg (or kPa) and the temperature in K at the sampler, and P_{std} is 760 mm Hg (or 101 kPa).

located in the sample air stream together with a suitable pressure indicator such as a manometer, or aneroid pressure gauge. A pressure recorder may be used with an orifice to provide a continuous record of the flow. Other types of flow indicators (including rotameters) having comparable precision and accuracy are also acceptable.

7.4.2 † The flow rate measurement device must be capable of being calibrated and read in units corresponding to a flow rate which is readable to the nearest 0.02 std m³/min over the range 1.0 to 1.8 std m³/min.

7.5 *Thermometer*, to indicate the approximate air temperature at the flow rate measurement orifice, when temperature corrections are used.

7.5.1 *Range*: -40° to +50 °C (223-323 K).

7.5.2 *Resolution*: 2 °C (2 K).

7.6 *Barometer*, to indicate barometric pressure at the flow rate measurement orifice, when pressure corrections are used.

7.6.1 *Range*: 500 to 800 mm Hg (66-106 kPa).

7.6.2 *Resolution*: ±5 mm Hg (0.67 kPa).

7.7 *Timing/control device*.

7.7.1 The timing device must be capable of starting and stopping the sampler to obtain an elapsed run-time of 24 hr ±1 hr (1,440 ±60 min).

7.7.2 *Accuracy of time setting*: ±30 min, or better. (See Section 6.8).

7.8 *Flow rate transfer standard*, traceable to a primary standard. (See Section 9.2.)

7.8.1 *Approximate range*: 1.0 to 1.8 m³/min.

7.8.2 *Resolution*: 0.02 m³/min.

7.8.3 *Reproducibility*: ±2 percent (2 times coefficient of variation) over normal ranges of ambient temperature and pressure for the stated flow rate range. (See Reference 2, Section 2.)

7.8.4 *Maximum pressure drop at 1.7 std m³/min*; 50 cm H₂O (5 kPa).

7.8.5 The flow rate transfer standard must connect without leaks to the inlet of the sampler and measure the flow rate of the total air sample.

7.8.6 The flow rate transfer standard must include a means to vary the sampler flow rate over the range of 1.0 to 1.8 m³/min (35-64 ft³/min) by introducing various levels of flow resistance between the sampler and the transfer standard inlet.

7.8.7 The conventional type of flow transfer standard consists of: An orifice unit with adapter that connects to the inlet of the sampler, a manometer or other device to measure orifice pressure drop, a means to vary the flow through the sampler unit, a thermometer to measure the ambient temperature, and a barometer to measure ambient pressure. Two such devices are shown in Figures 2a and 2b. Figure 2a shows multiple fixed resistance plates, which necessitate disassembly of the unit each time the flow resistance is changed. A preferable design, illustrated in Figure 2b, has a variable flow restriction that can be adjusted externally

without disassembly of the unit. Use of a conventional, orifice-type transfer standard is assumed in the calibration procedure (Section 9). However, the use of other types of transfer standards meeting the above specifications, such as the one shown in Figure 2c, may be approved; see the note following Section 9.1.

7.9 *Filter conditioning environment*

7.9.1 *Controlled temperature*: between 15° and 30 °C with less than ±3 °C variation during equilibration period.

7.9.2 *Controlled humidity*: Less than 50 percent relative humidity, constant within ±5 percent.

7.10 *Analytical balance*.

7.10.1 *Sensitivity*: 0.1 mg.

7.10.2 Weighing chamber designed to accept an unfolded 20.3 x 25.4 cm (8 x 10 in) filter.

7.11 *Area light source*, similar to X-ray film viewer, to backlight filters for visual inspection.

7.12 *Numbering device*, capable of printing identification numbers on the filters before they are placed in the filter conditioning environment, if not numbered by the supplier.

8.0 *Procedure*.

(See References 1 and 2 for quality assurance information.)

8.1 Number each filter, if not already numbered, near its edge with a unique identification number.

8.2 Backlight each filter and inspect for pinholes, particles, and other imperfections; filters with visible imperfections must not be used.

8.3 Equilibrate each filter in the conditioning environment for at least 24-hr.

8.4 Following equilibration, weigh each filter to the nearest milligram and record this tare weight (W_i) with the filter identification number.

8.5 Do not bend or fold the filter before collection of the sample.

8.6 Open the shelter and install a numbered, preweighed filter in the sampler, following the sampler manufacturer's instructions. During inclement weather, precautions must be taken while changing filters to prevent damage to the clean filter and loss of sample from or damage to the exposed filter. Filter cassettes that can be loaded and unloaded in the laboratory may be used to minimize this problem (See Section 6.6).

8.7 Close the shelter and run the sampler for at least 5 min to establish run-temperature conditions.

8.8 Record the flow indicator reading and, if needed, the barometric pressure (P₃) and the ambient temperature (T₃) see NOTE following step 8.12). Stop the sampler. Determine the sampler flow rate (see Section 10.1); if it is outside the acceptable range (1.1 to 1.7 m³/min [39-60 ft³/min]), use a different filter, or adjust the sampler flow rate. Warning: Substantial flow adjustments may affect the

calibration of the orifice-type flow indicators and may necessitate recalibration.

8.9 Record the sampler identification information (filter number, site location or identification number, sample date, and starting time).

8.10 Set the timer to start and stop the sampler such that the sampler runs 24-hrs, from midnight to midnight (local time).

8.11 As soon as practical following the sampling period, run the sampler for at least 5 min to again establish run-temperature conditions.

8.12 Record the flow indicator reading and, if needed, the barometric pressure (P_3) and the ambient temperature (T_3).

NOTE: No onsite pressure or temperature measurements are necessary if the sampler flow indicator does not require pressure or temperature corrections (e.g., a mass flowmeter) or if average barometric pressure and seasonal average temperature for the site are incorporated into the sampler calibration (see step 9.3.9). For individual pressure and temperature corrections, the ambient pressure and temperature can be obtained by onsite measurements or from a nearby weather station. Barometric pressure readings obtained from airports must be station pressure, not corrected to sea level, and may need to be corrected for differences in elevation between the sampler site and the airport. For samplers having flow recorders but not constant flow controllers, the average temperature and pressure at the site *during the sampling period* should be estimated from weather bureau or other available data.

8.13 Stop the sampler and carefully remove the filter, following the sampler manufacturer's instructions. Touch only the outer edges of the filter. See the precautions in step 8.6.

8.14 Fold the filter in half lengthwise so that only surfaces with collected particulate matter are in contact and place it in the filter holder (glassine envelope or manila folder).

8.15 Record the ending time or elapsed time on the filter information record, either from the stop set-point time, from an elapsed time indicator, or from a continuous flow record. The sample period must be $1,440 \pm 60$ min. for a valid sample.

8.16 Record on the filter information record any other factors, such as meteorological conditions, construction activity, fires or dust storms, etc., that might be pertinent to the measurement. If the sample is known to be defective, void it at this time.

8.17 Equilibrate the exposed filter in the conditioning environment for at least 24-hrs.

8.18 Immediately after equilibration, reweigh the filter to the nearest milligram and record the gross weight with the filter identification number. See Section 10 for TSP concentration calculations.

9.0 Calibration.

9.1 Calibration of the high volume sampler's flow indicating or control device is necessary to establish traceability of the field measurement to a primary standard via a flow rate transfer standard. Figure 3a illustrates the certification of the flow rate transfer standard and Figure 3b illustrates its use in calibrating a sampler flow indicator. Determination of the corrected flow rate from the sampler flow indicator, illustrated in Figure 3c, is addressed in Section 10.1

NOTE: The following calibration procedure applies to a conventional orifice-type flow transfer standard and an orifice-type flow indicator in the sampler (the most common types). For samplers using a pressure recorder having a square-root scale, 3 other acceptable calibration procedures are provided in Reference 12. Other types of transfer standards may be used if the manufacturer or user provides an appropriately modified calibration procedure that has been approved by EPA under Section 2.8 of appendix C to part 58 of this chapter.

9.2 Certification of the flow rate transfer standard.

9.2.1 *Equipment required:* Positive displacement standard volume meter traceable to the National Bureau of Standards (such as a Roots meter or equivalent), stop-watch, manometer, thermometer, and barometer.

9.2.2 Connect the flow rate transfer standard to the inlet of the standard volume meter. Connect the manometer to measure the pressure at the inlet of the standard volume meter. Connect the orifice manometer to the pressure tap on the transfer standard. Connect a high-volume air pump (such as a high-volume sampler blower) to the outlet side of the standard volume meter. See Figure 3a.

9.2.3 Check for leaks by temporarily clamping both manometer lines (to avoid fluid loss) and blocking the orifice with a large-diameter rubber stopper, wide cellophane tape, or other suitable means. Start the high-volume air pump and note any change in the standard volume meter reading. The reading should remain constant. If the reading changes, locate any leaks by listening for a whistling sound and/or retightening all connections, making sure that all gaskets are properly installed.

9.2.4 After satisfactorily completing the leak check as described above, unclamp both manometer lines and zero both manometers.

9.2.5 Achieve the appropriate flow rate through the system, either by means of the variable flow resistance in the transfer standard or by varying the voltage to the air pump. (Use of resistance plates as shown in Figure 1a is discouraged because the above leak check must be repeated each time a new resistance plate is installed.) At least five different but constant flow rates, evenly distributed, with at least three in the specified

flow rate interval (1.1 to 1.7 m³/min [39–60 ft³/min]), are required.

9.2.6 Measure and record the certification data on a form similar to the one illustrated in Figure 4 according to the following steps.

9.2.7 Observe the barometric pressure and record as P₁ (item 8 in Figure 4).

9.2.8 Read the ambient temperature in the vicinity of the standard volume meter and record it as T₁ (item 9 in Figure 4).

9.2.9 Start the blower motor, adjust the flow, and allow the system to run for at least 1 min for a constant motor speed to be attained.

9.2.10 Observe the standard volume meter reading and simultaneously start a stopwatch. Record the initial meter reading (V_i) in column 1 of Figure 4.

9.2.11 Maintain this constant flow rate until at least 3 m³ of air have passed through the standard volume meter. Record the standard volume meter inlet pressure manometer reading as ΔP (column 5 in Figure 4), and the orifice manometer reading as ΔH (column 7 in Figure 4). Be sure to indicate the correct units of measurement.

9.2.12 After at least 3 m³ of air have passed through the system, observe the standard volume meter reading while simultaneously stopping the stopwatch. Record the final meter reading (V_f) in column 2 and the elapsed time (t) in column 3 of Figure 4.

9.2.13 Calculate the volume measured by the standard volume meter at meter conditions of temperature and pressures as V_m = V_f - V_i. Record in column 4 of Figure 4.

9.2.14 Correct this volume to standard volume (std m³) as follows:

$$V_{std} = V_m \frac{P_1 - \Delta P}{P_{std}} \frac{T_{std}}{T_1}$$

where:

V_{std} = standard volume, std m³;

V_m = actual volume measured by the standard volume meter;

P₁ = barometric pressure during calibration, mm Hg or kPa;

ΔP = differential pressure at inlet to volume meter, mm Hg or kPa;

P_{std} = 760 mm Hg or 101 kPa;

T_{std} = 298 K;

T₁ = ambient temperature during calibration, K.

Calculate the standard flow rate (std m³/min) as follows:

$$Q_{std} = \frac{V_{std}}{t}$$

where:

Q_{std} = standard volumetric flow rate, std m³/min

t = elapsed time, minutes.

Record Q_{std} to the nearest 0.01 std m³/min in column 6 of Figure 4.

9.2.15 Repeat steps 9.2.9 through 9.2.14 for at least four additional constant flow rates, evenly spaced over the approximate range of 1.0 to 1.8 std m³/min (35–64 ft³/min).

9.2.16 For each flow, compute

$$\sqrt{\Delta H} (P_1/P_{std})(298/T_1)$$

(column 7a of Figure 4) and plot these value against Q_{std} as shown in Figure 3a. Be sure to use consistent units (mm Hg or kPa) for barometric pressure. Draw the orifice transfer standard certification curve or calculate the linear least squares slope (m) and intercept (b) of the certification curve:

$$\sqrt{\Delta H} (P_1/P_{std})(298/T_1)$$

= mQ_{std}+b. See Figures 3 and 4. A certification graph should be readable to 0.02 std m³/min.

9.2.17 Recalibrate the transfer standard annually or as required by applicable quality control procedures. (See Reference 2.)

9.3 Calibration of sampler flow indicator.

NOTE: For samplers equipped with a flow controlling device, the flow controller must be disabled to allow flow changes during calibration of the sampler's flow indicator, or the alternate calibration of the flow controller given in 9.4 may be used. For samplers using an orifice-type flow indicator downstream of the motor, do not vary the flow rate by adjusting the voltage or power supplied to the sampler.

9.3.1 A form similar to the one illustrated in Figure 5 should be used to record the calibration data.

9.3.2 Connect the transfer standard to the inlet of the sampler. Connect the orifice manometer to the orifice pressure tap, as illustrated in Figure 3b. Make sure there are no leaks between the orifice unit and the sampler.

9.3.3 Operate the sampler for at least 5 minutes to establish thermal equilibrium prior to the calibration.

9.3.4 Measure and record the ambient temperature, T₂, and the barometric pressure, P₂, during calibration.

9.3.5 Adjust the variable resistance or, if applicable, insert the appropriate resistance plate (or no plate) to achieve the desired flow rate.

9.3.6 Let the sampler run for at least 2 min to re-establish the run-temperature conditions. Read and record the pressure drop across the orifice (ΔH) and the sampler flow rate indication (I) in the appropriate columns of Figure 5.

9.3.7 Calculate $\sqrt{\Delta H}(P_2/P_{std})(298/T_2)$ and determine the flow rate at standard conditions (Q_{std}) either graphically from the certification curve or by calculating Q_{std} from the least square slope and intercept of the transfer standard's transposed certification curve: Q_{std}=1/m $\sqrt{\Delta H}(P_2/P_{std})(298/T_2)$ - b. Record the value of Q_{std} on Figure 5.

9.3.8 Repeat steps 9.3.5, 9.3.6, and 9.3.7 for several additional flow rates distributed over a range that includes 1.1 to 1.7 std m³/min.

9.3.9 Determine the calibration curve by plotting values of the appropriate expression involving I, selected from table 1, against Q_{std}. The choice of expression from table 1 depends on the flow rate measurement device used (see Section 7.4.1) and also on whether the calibration curve is to incorporate geographic average barometric pressure (P_a) and seasonal average temperature (T_a) for the site to approximate actual pressure and temperature. Where P_a and T_a can be determined for a site for a seasonal period such that the actual barometric pressure and temperature at the site do not vary by more than ±60 mm Hg (8 kPa) from P_a or ±15 °C from T_a, respectively, then using P_a and T_a avoids the need for subsequent pressure and temperature calculation when the sampler is used. The geographic average barometric pressure (P_a) may be estimated from an altitude-pressure table or by making an (approximate) elevation correction of -26 mm Hg (-3.46 kPa) for each 305 m (1,000 ft) above sea level (760 mm Hg or 101 kPa). The seasonal average temperature (T_a) may be estimated from weather station or other records. Be sure to use consistent units (mm Hg or kPa) for barometric pressure.

9.3.10 Draw the sampler calibration curve or calculate the linear least squares slope (m), intercept (b), and correlation coefficient of the calibration curve: [Expression from table 1]= mQ_{std}+b. See Figures 3 and 5. Calibration curves should be readable to 0.02 std m³/min.

9.3.11 For a sampler equipped with a flow controller, the flow controlling mechanism should be re-enabled and set to a flow near the lower flow limit to allow maximum control range. The sample flow rate should be verified at this time with a clean filter installed. Then add two or more filters to the sampler to see if the flow controller maintains a constant flow; this is particularly important at high altitudes where the range of the flow controller may be reduced.

9.4 Alternate calibration of flow-controlled samplers. A flow-controlled sampler may be calibrated solely at its controlled flow rate, provided that previous operating history of the sampler demonstrates that the flow rate is stable and reliable. In this case, the flow indicator may remain uncalibrated but should be used to indicate any relative change between initial and final flows, and the sampler should be recalibrated more often to minimize potential loss of samples because of controller malfunction.

9.4.1 Set the flow controller for a flow near the lower limit of the flow range to allow maximum control range.

9.4.2 Install a clean filter in the sampler and carry out steps 9.3.2, 9.3.3, 9.3.4, 9.3.6, and 9.3.7.

9.4.3 Following calibration, add one or two additional clean filters to the sampler, reconnect the transfer standard, and operate the sampler to verify that the controller maintains the same calibrated flow rate; this is particularly important at high altitudes where the flow control range may be reduced.

TABLE 1. EXPRESSIONS FOR PLOTTING SAMPLER CALIBRATION CURVES

Type of sampler flow rate measuring device	Expression	
	For actual pressure and temperature corrections	For incorporation of geographic average pressure and seasonal average temperature
Mass flowmeter	I	I
Orifice and pressure indicator	$I \sqrt{\left(\frac{P_2}{P_{std}}\right) \left(\frac{298}{T_2}\right)}$	$I \sqrt{\left(\frac{P_2}{P_a}\right) \left(\frac{T_a}{T_2}\right)}$
Rotameter, or orifice and pressure recorder having square root scale*	$I \sqrt{\left(\frac{P_2}{P_{std}}\right) \left(\frac{298}{T_2}\right)}$	$I \sqrt{\left(\frac{P_2}{P_a}\right) \left(\frac{T_a}{T_2}\right)}$

*This scale is recognizable by its nonuniform divisions and is the most commonly available for high-volume samplers.

TABLE 2. EXPRESSIONS FOR DETERMINING FLOW RATE DURING SAMPLER OPERATION

Type of sampler flow rate measuring device	Expression	
	For actual pressure and temperature corrections	For use when geographic average pressure and seasonal average temperature have been incorporated into the sampler calibration
Mass flowmeter	I	I
Orifice and pressure indicator	$I \sqrt{\left(\frac{P_3}{P_{std}}\right) \left(\frac{298}{T_3}\right)}$	\sqrt{I}
Rotameter, or orifice and pressure recorder having square root scale*	$I \sqrt{\left(\frac{P_3}{P_{std}}\right) \left(\frac{298}{T_3}\right)}$	I

*This scale is recognizable by its nonuniform divisions and is the most commonly available for high-volume samplers.

10.0 Calculations of TSP Concentration.

10.1 Determine the average sampler flow rate during the sampling period according to either 10.1.1 or 10.1.2 below.

10.1.1 For a sampler without a continuous flow recorder, determine the appropriate expression to be used from table 2 corresponding to the one from table 1 used in step 9.3.9. Using this appropriate expression, determine Q_{std} for the initial flow rate from the sampler calibration curve, either graphically or from the transposed regression equation:

$$Q_{std} = 1/m \text{ ([Appropriate expression from table 2] - b)}$$

Similarly, determine Q_{std} from the final flow reading, and calculate the average flow Q_{std} as one-half the sum of the initial and final flow rates.

10.1.2 For a sampler with a continuous flow recorder, determine the average flow rate device reading, I, for the period. Determine the appropriate expression from table 2 corresponding to the one from table 1 used in step 9.3.9. Then using this expression and the average flow rate reading, determine Q_{std} from the sampler calibration curve, either graphically or from the transposed regression equation:

$$Q_{std} = 1/m \text{ ([Appropriate expression from table 2] - b)}$$

If the trace shows substantial flow change during the sampling period, greater accuracy

may be achieved by dividing the sampling period into intervals and calculating an average reading before determining Q_{std} .

10.2 Calculate the total air volume sampled as:

$$V = Q_{std} \times t$$

where:

V = total air volume sampled, in standard volume units, std m³;

Q_{std} = average standard flow rate, std m³/min;

t = sampling time, min.

10.3 Calculate and report the particulate matter concentration as:

$$TSP = \frac{(W_f - W_i) \times 10^6}{V}$$

where:

TSP = mass concentration of total suspended particulate matter, µg/std m³;

W_i = initial weight of clean filter, g;

W_f = final weight of exposed filter, g;

V = air volume sampled, converted to standard conditions, std m³;

10⁶ = conversion of g to µg.

10.4 If desired, the actual particulate matter concentration (see Section 2.2) can be calculated as follows:

$$(TSP)_a = TSP (P_3/P_{std})(298/T_3)$$

where:

$(TSP)_a$ = actual concentration at field conditions, µg/m³;

TSP = concentration at standard conditions, $\mu\text{g}/\text{std m}^3$;

P_3 = average barometric pressure during sampling period, mm Hg;

P_{std} = 760 mm Hg (or 101 kPa);

T_3 = average ambient temperature during sampling period, K.

11.0 References.

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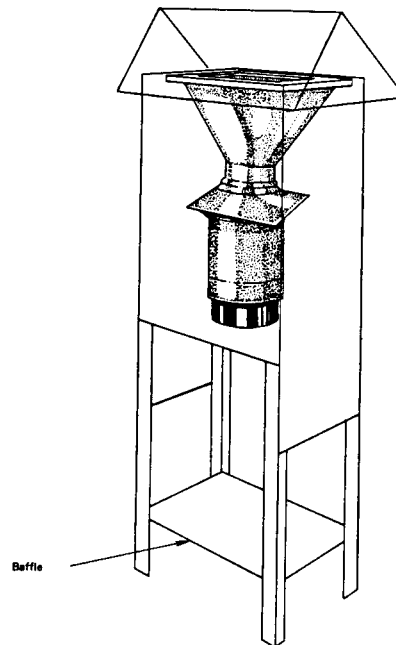
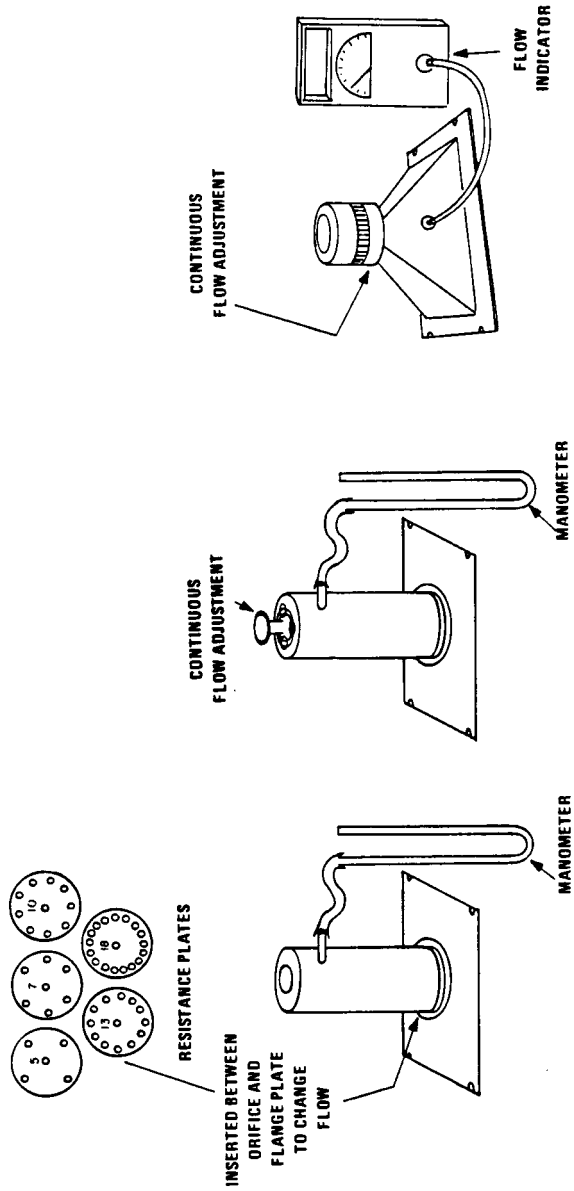


Figure 1. High-volume sampler in shelter.

**NONORIFICE TYPE FLOW
TRANSFER STANDARD**

**ORIFICE TYPE FLOW
TRANSFER STANDARDS**



2c. ELECTRONIC FLOWMETER WITH EXTERNALLY ADJUSTABLE RESISTANCE.

2b. PREFERABLE ORIFICE UNIT WITH EXTERNALLY ADJUSTABLE RESISTANCE.

2a. ORIFICE UNIT USING FIXED RESISTANCE PLATES.

Figure 2. Various types of flow transfer standards. Note that all devices are designed to mount to the filter inlet area of the sampler.

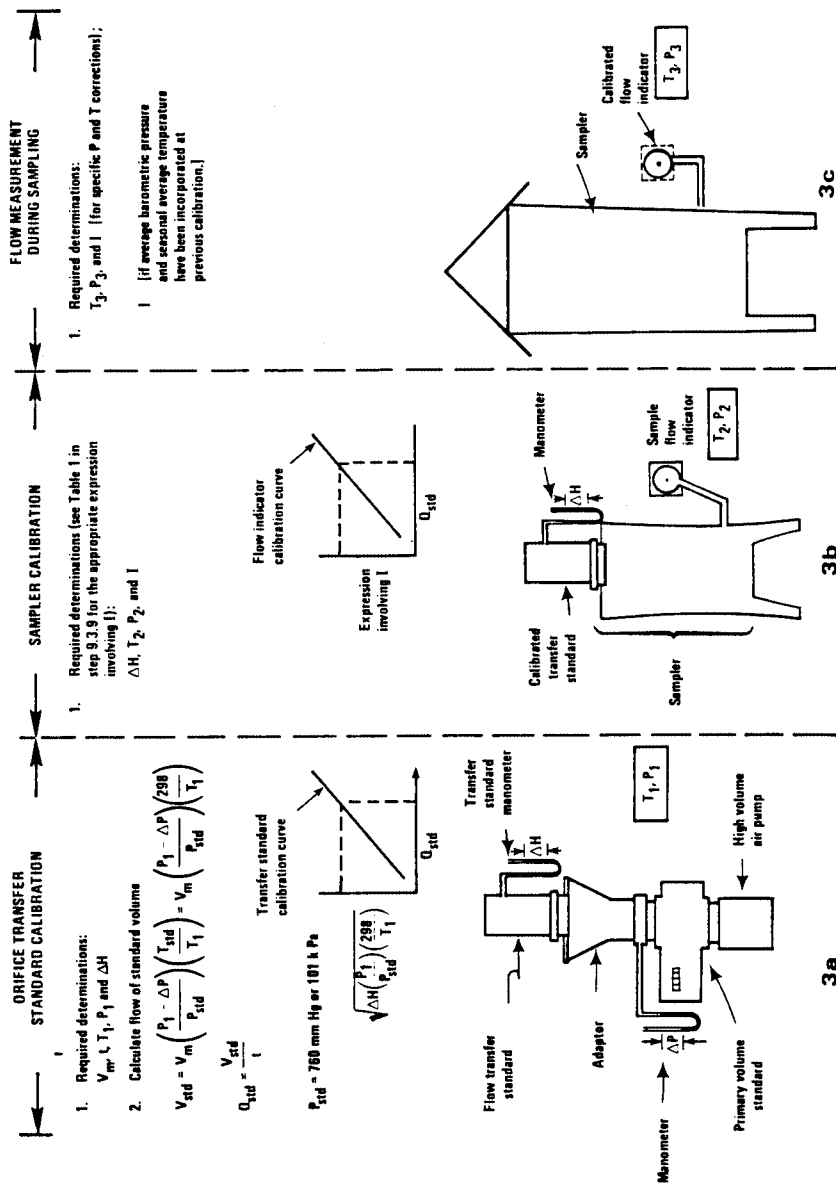


Figure 3. Illustration of the 3 steps in the flow measurement process.

ORIFICE TRANSFER STANDARD CERTIFICATION WORKSHEET

Run No.	(1) Meter reading start V_i (m^3)	(2) Meter reading stop V_f (m^3)	(3) Sampling time t (min)	(4) Volume measured V_m (m^3)	(5) Differential pressure (at inlet to volume meter) ΔP (mm Hg or kPa)	(6) Flow rate Q_{std} ($std\ m^3/min$)	(7) Pressure drop across orifice ΔH (in) or ΔP (cm) of water	(7a) $\sqrt{\Delta H \left(\frac{P_1}{P_{std}} \right) \left(\frac{298}{T_1} \right)}$ (\bar{y})
1								
2								
3								
4								
5								
6								

RECORDED CALIBRATION DATA

Standard volume meter no. _____
 Transfer standard type: orifice other
 Model No. _____ Serial No. _____
 (8) P_1 : _____ mm Hg (or kPa) (10) P_{std} : 760 mm Hg (or 101.3 kPa)
 (9) T_1 : _____ K (11) T_{std} : 298 K
 Calibration performed by: _____
 Date: _____

CALCULATION EQUATIONS

(1) $V_m = V_f - V_i$
 (2) $V_{std} = V_m \left(\frac{P_1 - \Delta P}{P_{std}} \right) \left(\frac{T_{std}}{T_1} \right)$
 (3) $Q_{std} = \frac{V_{std}}{t}$

LEAST SQUARES CALCULATIONS

Linear ($Y = mx + b$) regression equation of $Y = \sqrt{\Delta H(P_1/P_{std})(298/T_1)}$ on $X = Q_{std}$ for Orifice Calibration Unit (i.e., $\sqrt{\Delta H(P_1/P_{std})(298/T_1)} = mQ_{std} + b$)
 Slope (m) = _____ Intercept (b) = _____ Correlation coefficient (r) = _____

To use for subsequent calibration: $X = \frac{1}{m}(Y-b)$; $Q_{std} = \frac{1}{m} \left(\sqrt{\Delta H \left(\frac{P_1}{P_{std}} \right) \left(\frac{298}{T_1} \right)} - b \right)$

Figure 4. Example of orifice transfer standard certification worksheet.

HIGH-VOLUME AIR SAMPLER CALIBRATION WORKSHEET

Site Location: _____
 Date: _____ Barometric Pressure, P_a mm Hg (or kPa) _____
 Calibrated By: _____ Temperature, T_a (K) _____
 Sampler No. _____ Serial No. _____
 Transfer std. type: _____ Serial No. _____

(Y)

$P_{std} = 760$ mm Hg (or 101 kPa)

Optional: Average barometric pressure: $P_a =$ _____
 Seasonal average temperature: $T_a =$ _____

No.	Pressure drop Δh (cross orifice or water)	$\sqrt{\Delta h \left(\frac{P_a}{P_{std}} \right) \left(\frac{298}{T_a} \right)}$	Q_{std} (from orifice certification std m ³ /min)	Sampler flow rate indication (arbitrary)	For specific pressure and temperature cor- rections (see Table 1)		For incorporation of average pressure and seasonal average tem- perature (see Table 1)	
					<input type="checkbox"/> 1	<input type="checkbox"/> 2	<input type="checkbox"/> 1	<input type="checkbox"/> 2
1					<input type="checkbox"/> $\sqrt{\left(\frac{P_a}{P_{std}} \right) \left(\frac{298}{T_a} \right)}$	<input type="checkbox"/> $\sqrt{\left(\frac{P_a}{P_{std}} \right) \left(\frac{T_a}{298} \right)}$	<input type="checkbox"/> $\sqrt{\left(\frac{P_a}{P_{std}} \right) \left(\frac{T_a}{298} \right)}$	<input type="checkbox"/> $\sqrt{\left(\frac{P_a}{P_{std}} \right) \left(\frac{T_a}{298} \right)}$
2					<input type="checkbox"/> $\sqrt{\left(\frac{P_a}{P_{std}} \right) \left(\frac{298}{T_a} \right)}$	<input type="checkbox"/> $\sqrt{\left(\frac{P_a}{P_{std}} \right) \left(\frac{T_a}{298} \right)}$	<input type="checkbox"/> $\sqrt{\left(\frac{P_a}{P_{std}} \right) \left(\frac{T_a}{298} \right)}$	<input type="checkbox"/> $\sqrt{\left(\frac{P_a}{P_{std}} \right) \left(\frac{T_a}{298} \right)}$
3					<input type="checkbox"/> $\sqrt{\left(\frac{P_a}{P_{std}} \right) \left(\frac{298}{T_a} \right)}$	<input type="checkbox"/> $\sqrt{\left(\frac{P_a}{P_{std}} \right) \left(\frac{T_a}{298} \right)}$	<input type="checkbox"/> $\sqrt{\left(\frac{P_a}{P_{std}} \right) \left(\frac{T_a}{298} \right)}$	<input type="checkbox"/> $\sqrt{\left(\frac{P_a}{P_{std}} \right) \left(\frac{T_a}{298} \right)}$
4					<input type="checkbox"/> $\sqrt{\left(\frac{P_a}{P_{std}} \right) \left(\frac{298}{T_a} \right)}$	<input type="checkbox"/> $\sqrt{\left(\frac{P_a}{P_{std}} \right) \left(\frac{T_a}{298} \right)}$	<input type="checkbox"/> $\sqrt{\left(\frac{P_a}{P_{std}} \right) \left(\frac{T_a}{298} \right)}$	<input type="checkbox"/> $\sqrt{\left(\frac{P_a}{P_{std}} \right) \left(\frac{T_a}{298} \right)}$
5					<input type="checkbox"/> $\sqrt{\left(\frac{P_a}{P_{std}} \right) \left(\frac{298}{T_a} \right)}$	<input type="checkbox"/> $\sqrt{\left(\frac{P_a}{P_{std}} \right) \left(\frac{T_a}{298} \right)}$	<input type="checkbox"/> $\sqrt{\left(\frac{P_a}{P_{std}} \right) \left(\frac{T_a}{298} \right)}$	<input type="checkbox"/> $\sqrt{\left(\frac{P_a}{P_{std}} \right) \left(\frac{T_a}{298} \right)}$
6					<input type="checkbox"/> $\sqrt{\left(\frac{P_a}{P_{std}} \right) \left(\frac{298}{T_a} \right)}$	<input type="checkbox"/> $\sqrt{\left(\frac{P_a}{P_{std}} \right) \left(\frac{T_a}{298} \right)}$	<input type="checkbox"/> $\sqrt{\left(\frac{P_a}{P_{std}} \right) \left(\frac{T_a}{298} \right)}$	<input type="checkbox"/> $\sqrt{\left(\frac{P_a}{P_{std}} \right) \left(\frac{T_a}{298} \right)}$

LEAST SQUARES CALCULATIONS

Linear regression of Y on X: $Y = mx + b$; Y = appropriate expression from Table 1; $X = Q_{std}$
 Slope (m) = _____ Intercept (b) = _____ Correlation Coeff. (r) = _____

To determine subsequent flow rate during use: $X = \frac{1}{m}(Y-b)$; $Q_{std} = \frac{1}{m}([appropriate expression from Table 2] - b)$

Figure 5. Example of high-volume air sampler calibration worksheet.

[47 FR 54912, Dec. 6, 1982; 48 FR 17355, Apr. 22, 1983]

APPENDIX C TO PART 50—MEASUREMENT PRINCIPLE AND CALIBRATION PROCEDURE FOR THE MEASUREMENT OF CARBON MONOXIDE IN THE ATMOSPHERE (NON-DISPERSIVE INFRARED PHOTOMETRY)

MEASUREMENT PRINCIPLE

1. Measurements are based on the absorption of infrared radiation by carbon monoxide (CO) in a non-dispersive photometer. Infrared energy from a source is passed

through a cell containing the gas sample to be analyzed, and the quantitative absorption of energy by CO in the sample cell is measured by a suitable detector. The photometer is sensitized to CO by employing CO gas in either the detector or in a filter cell in the optical path, thereby limiting the measured absorption to one or more of the characteristic wavelengths at which CO strongly absorbs. Optical filters or other means may

also be used to limit sensitivity of the photometer to a narrow band of interest. Various schemes may be used to provide a suitable zero reference for the photometer. The measured absorption is converted to an electrical output signal, which is related to the concentration of CO in the measurement cell.

2. An analyzer based on this principle will be considered a reference method only if it has been designated as a reference method in accordance with part 53 of this chapter.

3. Sampling considerations.

The use of a particle filter on the sample inlet line of an NDIR CO analyzer is optional and left to the discretion of the user or the manufacturer. Use of filter should depend on the analyzer's susceptibility to interference, malfunction, or damage due to particles.

CALIBRATION PROCEDURE

1. *Principle.* Either of two methods may be used for dynamic multipoint calibration of CO analyzers:

(1) One method uses a single certified standard cylinder of CO, diluted as necessary with zero air, to obtain the various calibration concentrations needed.

(2) The other method uses individual certified standard cylinders of CO for each concentration needed. Additional information on calibration may be found in Section 2.0.9 of Reference 1.

2. *Apparatus.* The major components and typical configurations of the calibration systems for the two calibration methods are shown in Figures 1 and 2.

2.1 Flow controller(s). Device capable of adjusting and regulating flow rates. Flow rates for the dilution method (Figure 1) must be regulated to $\pm 1\%$.

2.2 Flow meter(s). Calibrated flow meter capable of measuring and monitoring flow rates. Flow rates for the dilution method (Figure 1) must be measured with an accuracy of $\pm 2\%$ of the measured value.

2.3 Pressure regulator(s) for standard CO cylinder(s). Regulator must have nonreactive diaphragm and internal parts and a suitable delivery pressure.

2.4 Mixing chamber. A chamber designed to provide thorough mixing of CO and diluent air for the dilution method.

2.5 Output manifold. The output manifold should be of sufficient diameter to insure an insignificant pressure drop at the analyzer connection. The system must have a vent designed to insure atmospheric pressure at the manifold and to prevent ambient air from entering the manifold.

3. *Reagents.*

3.1 CO concentration standard(s). Cylinder(s) of CO in air containing appropriate concentrations(s) of CO suitable for the selected operating range of the analyzer under calibration; CO standards for the dilution method may be contained in a nitrogen ma-

trix if the zero air dilution ratio is not less than 100:1. The assay of the cylinder(s) must be traceable either to a National Bureau of Standards (NBS) CO in air Standard Reference Material (SRM) or to an NBS/EPA-approved commercially available Certified Reference Material (CRM). CRM's are described in Reference 2, and a list of CRM sources is available from the address shown for Reference 2. A recommended protocol for certifying CO gas cylinders against either a CO SRM or a CRM is given in Reference 1. CO gas cylinders should be recertified on a regular basis as determined by the local quality control program.

3.2 Dilution gas (zero air). Air, free of contaminants which will cause a detectable response on the CO analyzer. The zero air should contain <0.1 ppm CO. A procedure for generating zero air is given in Reference 1.

4. *Procedure Using Dynamic Dilution Method.*

4.1 Assemble a dynamic calibration system such as the one shown in Figure 1. All calibration gases including zero air must be introduced into the sample inlet of the analyzer system. For specific operating instructions refer to the manufacturer's manual.

4.2 Insure that all flowmeters are properly calibrated, under the conditions of use, if appropriate, against an authoritative standard such as a soap-bubble meter or wet-test meter. All volumetric flowrates should be corrected to 25 °C and 760 mm Hg (101 kPa). A discussion on calibration of flowmeters is given in Reference 1.

4.3 Select the operating range of the CO analyzer to be calibrated.

4.4 Connect the signal output of the CO analyzer to the input of the strip chart recorder or other data collection device. All adjustments to the analyzer should be based on the appropriate strip chart or data device readings. References to analyzer responses in the procedure given below refer to recorder or data device responses.

4.5 Adjust the calibration system to deliver zero air to the output manifold. The total air flow must exceed the total demand of the analyzer(s) connected to the output manifold to insure that no ambient air is pulled into the manifold vent. Allow the analyzer to sample zero air until a stable response is obtained. After the response has stabilized, adjust the analyzer zero control. Offsetting the analyzer zero adjustments to +5 percent of scale is recommended to facilitate observing negative zero drift. Record the stable zero air response as Z_{CO} .

4.6 Adjust the zero air flow and the CO flow from the standard CO cylinder to provide a diluted CO concentration of approximately 80 percent of the upper range limit (URL) of the operating range of the analyzer. The total air flow must exceed the total demand of the analyzer(s) connected to the output manifold to insure that no ambient air is

pulled into the manifold vent. The exact CO concentration is calculated from:

$$[\text{CO}]_{\text{OUT}} = \frac{[\text{CO}]_{\text{STD}} \times F_{\text{CO}}}{F_{\text{D}} + F_{\text{CO}}} \quad (1)$$

where:

$[\text{CO}]_{\text{OUT}}$ = diluted CO concentration at the output manifold, ppm;

$[\text{CO}]_{\text{STD}}$ = concentration of the undiluted CO standard, ppm;

F_{CO} = flow rate of the CO standard corrected to 25 °C and 760 mm Hg, (101 kPa), L/min; and

F_{D} = flow rate of the dilution air corrected to 25 °C and 760 mm Hg, (101 kPa), L/min.

Sample this CO concentration until a stable response is obtained. Adjust the analyzer span control to obtain a recorder response as indicated below:

Recorder response (percent scale) =

$$\frac{[\text{CO}]_{\text{OUT}}}{\text{URL}} \times 100 + Z_{\text{CO}} \quad (2)$$

where:

URL = nominal upper range limit of the analyzer's operating range, and

Z_{CO} = analyzer response to zero air, % scale.

If substantial adjustment of the analyzer span control is required, it may be necessary to recheck the zero and span adjustments by repeating Steps 4.5 and 4.6. Record the CO concentration and the analyzer's response. 4.7 Generate several additional concentrations (at least three evenly spaced points across the remaining scale are suggested to

verify linearity) by decreasing F_{CO} or increasing F_{D} . Be sure the total flow exceeds the analyzer's total flow demand. For each concentration generated, calculate the exact CO concentration using Equation (1). Record the concentration and the analyzer's response for each concentration. Plot the analyzer responses versus the corresponding CO concentrations and draw or calculate the calibration curve.

5. *Procedure Using Multiple Cylinder Method.* Use the procedure for the dynamic dilution method with the following changes:

5.1 Use a multi-cylinder system such as the typical one shown in Figure 2.

5.2 The flowmeter need not be accurately calibrated, provided the flow in the output manifold exceeds the analyzer's flow demand.

5.3 The various CO calibration concentrations required in Steps 4.6 and 4.7 are obtained without dilution by selecting the appropriate certified standard cylinder.

REFERENCES

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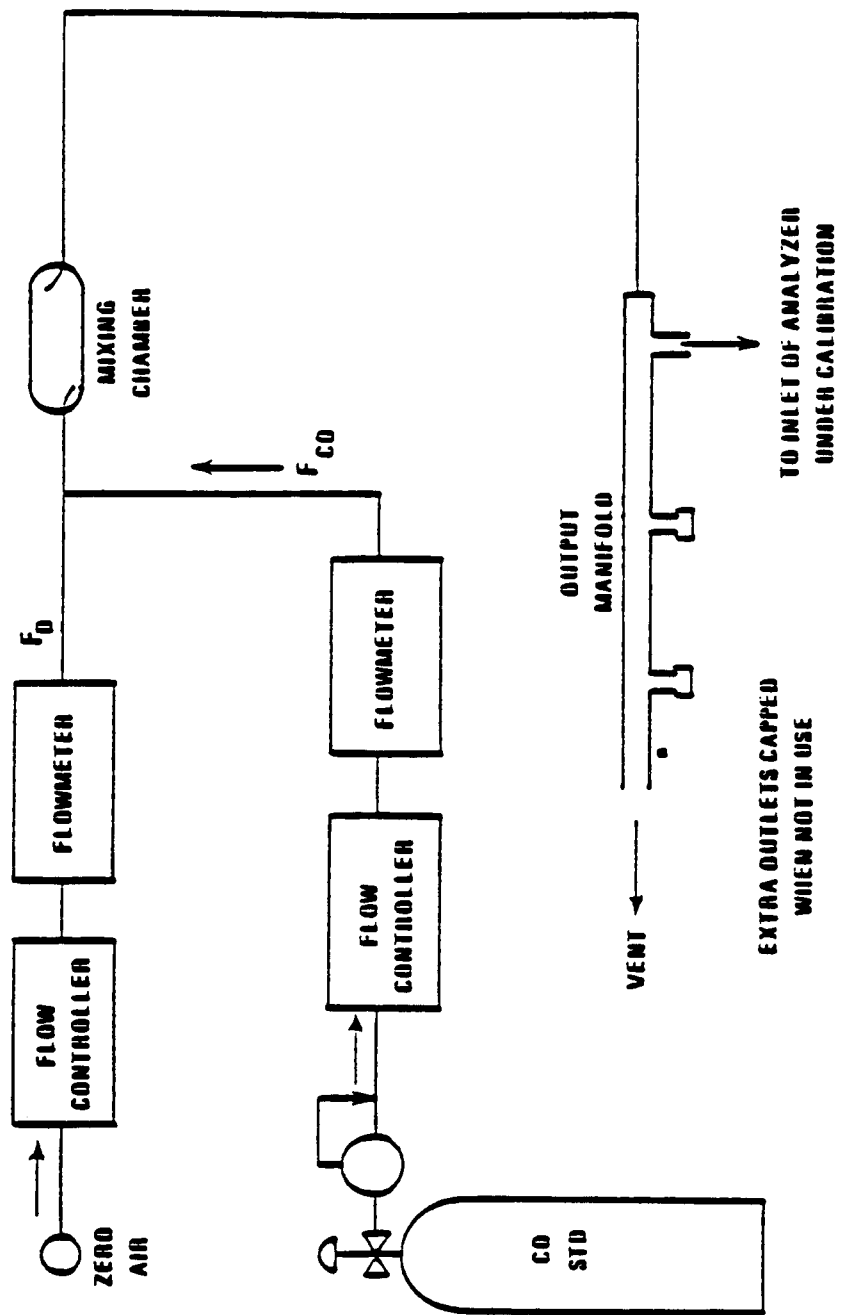


Figure 1. Dilution method for calibration of CO analyzers.

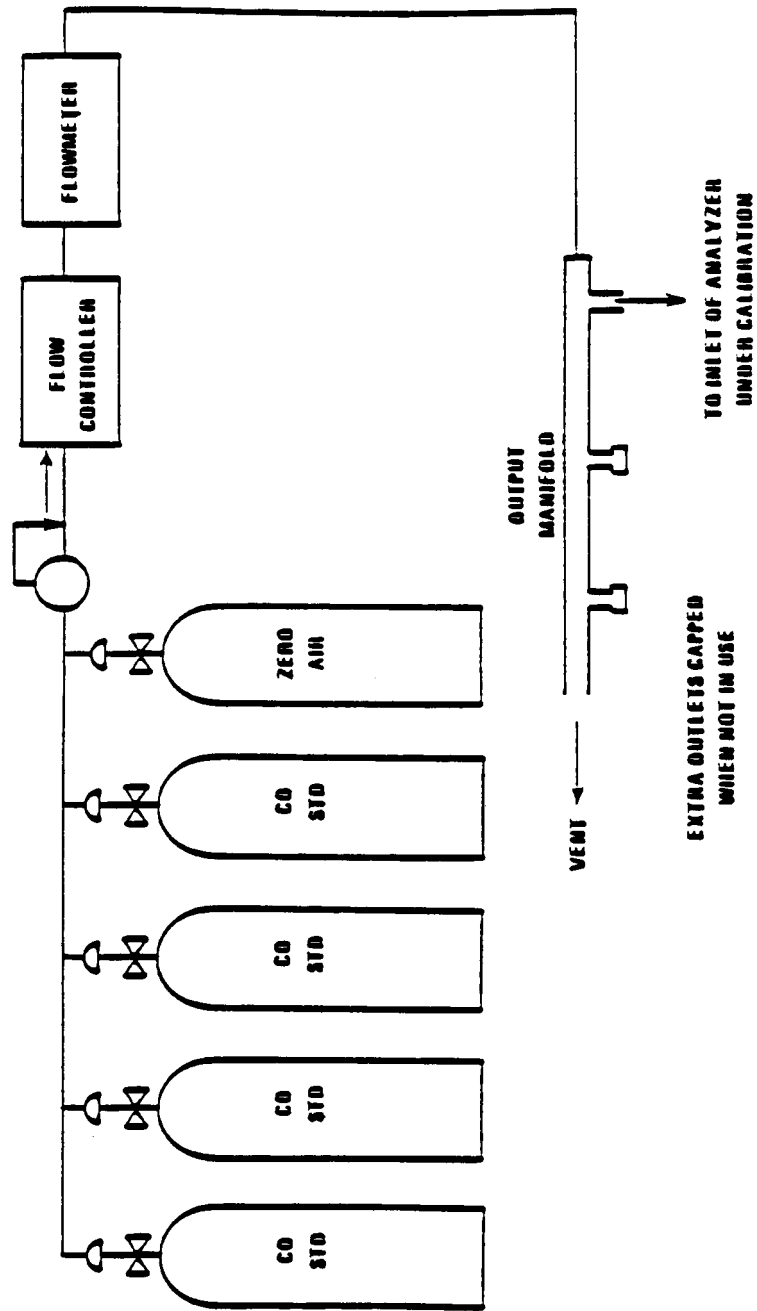


Figure 2. Multiple cylinder method for calibration of CO analyzers.

[47 FR 54922, Dec. 6, 1982; 48 FR 17355, Apr. 22, 1983]

APPENDIX D TO PART 50—MEASUREMENT
PRINCIPLE AND CALIBRATION PROCEDURE
FOR THE MEASUREMENT OF
OZONE IN THE ATMOSPHERE

MEASUREMENT PRINCIPLE

1. Ambient air and ethylene are delivered simultaneously to a mixing zone where the ozone in the air reacts with the ethylene to emit light, which is detected by a photomultiplier tube. The resulting photocurrent is amplified and is either read directly or displayed on a recorder.

2. An analyzer based on this principle will be considered a reference method only if it has been designated as a reference method in accordance with part 53 of this chapter and calibrated as follows:

CALIBRATION PROCEDURE

1. *Principle.* The calibration procedure is based on the photometric assay of ozone (O₃) concentrations in a dynamic flow system. The concentration of O₃ in an absorption cell is determined from a measurement of the amount of 254 nm light absorbed by the sample. This determination requires knowledge of (1) the absorption coefficient (α) of O₃ at 254 nm, (2) the optical path length (l) through the sample, (3) the transmittance of the sample at a wavelength of 254 nm, and (4) the temperature (T) and pressure (P) of the sample. The transmittance is defined as the ratio I/I_0 , where I is the intensity of light which passes through the cell and is sensed by the detector when the cell contains an O₃ sample, and I_0 is the intensity of light which passes through the cell and is sensed by the detector when the cell contains zero air. It is assumed that all conditions of the system, except for the contents of the absorption cell, are identical during measurement of I and I_0 . The quantities defined above are related by the Beer-Lambert absorption law,

$$\text{Transmittance} = \frac{I}{I_0} = e^{-\alpha cl} \quad (1)$$

where:

α = absorption coefficient of O₃ at 254 nm = 3084 atm⁻¹ cm⁻¹ at 0 °C and 760 torr. (1, 2, 3, 4, 5, 6, 7)

c = O₃ concentration in atmospheres

l = optical path length in cm

In practice, a stable O₃ generator is used to produce O₃ concentrations over the required range. Each O₃ concentration is determined from the measurement of the transmittance (I/I_0) of the sample at 254 nm with a photometer of path length l and calculated from the equation,

$$c(\text{atm}) = -\frac{1}{\alpha l} (\ln I/I_0) \quad (2a)$$

or

$$c(\text{ppm}) = -\frac{10^6}{\alpha l} (\ln I/I_0) \quad (2b)$$

The calculated O₃ concentrations must be corrected for O₃ losses which may occur in the photometer and for the temperature and pressure of the sample.

2. *Applicability.* This procedure is applicable to the calibration of ambient air O₃ analyzers, either directly or by means of a transfer standard certified by this procedure. Transfer standards must meet the requirements and specifications set forth in Reference 8.

3. *Apparatus.* A complete UV calibration system consists of an ozone generator, an output port or manifold, a photometer, an appropriate source of zero air, and other components as necessary. The configuration must provide a stable ozone concentration at the system output and allow the photometer to accurately assay the output concentration to the precision specified for the photometer (3.1). Figure 1 shows a commonly used configuration and serves to illustrate the calibration procedure which follows. Other configurations may require appropriate variations in the procedural steps. All connections between components in the calibration system downstream of the O₃ generator should be of glass, Teflon, or other relatively inert materials. Additional information regarding the assembly of a UV photometric calibration apparatus is given in Reference 9. For certification of transfer standards which provide their own source of O₃, the transfer standard may replace the O₃ generator and possibly other components shown in Figure 1; see Reference 8 for guidance.

3.1 *UV photometer.* The photometer consists of a low-pressure mercury discharge lamp, (optional) collimation optics, an absorption cell, a detector, and signal-processing electronics, as illustrated in Figure 1. It must be capable of measuring the transmittance, I/I_0 , at a wavelength of 254 nm with sufficient precision such that the standard deviation of the concentration measurements does not exceed the greater of 0.005 ppm or 3% of the concentration. Because the low-pressure mercury lamp radiates at several wavelengths, the photometer must incorporate suitable means to assure that no O₃ is generated in the cell by the lamp, and that at least 99.5% of the radiation sensed by the detector is 254 nm radiation. (This can be readily achieved by prudent selection of optical filter and detector response characteristics.) The length of the light path through the absorption cell must be known with an accuracy of at least 99.5%. In addition, the cell and associated plumbing must be designed to

minimize loss of O₃ from contact with cell walls and gas handling components. See Reference 9 for additional information.

3.2 *Air flow controllers.* Devices capable of regulating air flows as necessary to meet the output stability and photometer precision requirements.

3.3 *Ozone generator.* Device capable of generating stable levels of O₃ over the required concentration range.

3.4 *Output manifold.* The output manifold should be constructed of glass, Teflon, or other relatively inert material, and should be of sufficient diameter to insure a negligible pressure drop at the photometer connection and other output ports. The system must have a vent designed to insure atmospheric pressure in the manifold and to prevent ambient air from entering the manifold.

3.5 *Two-way valve.* Manual or automatic valve, or other means to switch the photometer flow between zero air and the O₃ concentration.

3.6 *Temperature indicator.* Accurate to ±1 °C.

3.7 *Barometer or pressure indicator.* Accurate to ±2 torr.

4. Reagents.

4.1 *Zero air.* The zero air must be free of contaminants which would cause a detectable response from the O₃ analyzer, and it should be free of NO, C₂H₄, and other species which react with O₃. A procedure for generating suitable zero air is given in Reference 9. As shown in Figure 1, the zero air supplied to the photometer cell for the I₀ reference measurement must be derived from the same source as the zero air used for generation of the ozone concentration to be assayed (I measurement). When using the photometer to certify a transfer standard having its own source of ozone, see Reference 8 for guidance on meeting this requirement.

5. Procedure.

5.1 *General operation.* The calibration photometer must be dedicated exclusively to use as a calibration standard. It should always be used with clean, filtered calibration gases, and never used for ambient air sampling. Consideration should be given to locating the calibration photometer in a clean laboratory where it can be stationary, protected from physical shock, operated by a responsible analyst, and used as a common standard for all field calibrations via transfer standards.

5.2 *Preparation.* Proper operation of the photometer is of critical importance to the accuracy of this procedure. The following steps will help to verify proper operation. The steps are not necessarily required prior to each use of the photometer. Upon initial operation of the photometer, these steps should be carried out frequently, with all quantitative results or indications recorded in a chronological record either in tabular form or plotted on a graphical chart. As the performance and stability record of the pho-

tometer is established, the frequency of these steps may be reduced consistent with the documented stability of the photometer.

5.2.1 *Instruction manual:* Carry out all set up and adjustment procedures or checks as described in the operation or instruction manual associated with the photometer.

5.2.2 *System check:* Check the photometer system for integrity, leaks, cleanliness, proper flowrates, etc. Service or replace filters and zero air scrubbers or other consumable materials, as necessary.

5.2.3 *Linearity:* Verify that the photometer manufacturer has adequately established that the linearity error of the photometer is less than 3%, or test the linearity by dilution as follows: Generate and assay an O₃ concentration near the upper range limit of the system (0.5 or 1.0 ppm), then accurately dilute that concentration with zero air and reassay it. Repeat at several different dilution ratios. Compare the assay of the original concentration with the assay of the diluted concentration divided by the dilution ratio, as follows

$$E = \frac{A_1 - A_2 / R}{A_1} \times 100\% \quad (3)$$

where:

E = linearity error, percent

A₁ = assay of the original concentration

A₂ = assay of the diluted concentration

R = dilution ratio = flow of original concentration divided by the total flow

The linearity error must be less than 5%. Since the accuracy of the measured flowrates will affect the linearity error as measured this way, the test is not necessarily conclusive. Additional information on verifying linearity is contained in Reference 9.

5.2.4 *Intercomparison:* When possible, the photometer should be occasionally intercompared, either directly or via transfer standards, with calibration photometers used by other agencies or laboratories.

5.2.5 *Ozone losses:* Some portion of the O₃ may be lost upon contact with the photometer cell walls and gas handling components. The magnitude of this loss must be determined and used to correct the calculated O₃ concentration. This loss must not exceed 5%. Some guidelines for quantitatively determining this loss are discussed in Reference 9.

5.3 Assay of O₃ concentrations.

5.3.1 Allow the photometer system to warm up and stabilize.

5.3.2 Verify that the flowrate through the photometer absorption cell, F allows the cell to be flushed in a reasonably short period of time (2 liter/min is a typical flow). The precision of the measurements is inversely related to the time required for flushing, since the photometer drift error increases with time.

5.3.3 Insure that the flowrate into the output manifold is at least 1 liter/min greater than the total flowrate required by the photometer and any other flow demand connected to the manifold.

5.3.4 Insure that the flowrate of zero air, F_z , is at least 1 liter/min greater than the flowrate required by the photometer.

5.3.5 With zero air flowing in the output manifold, actuate the two-way valve to allow the photometer to sample first the manifold zero air, then F_z . The two photometer readings must be equal ($I=I_0$).

NOTE: In some commercially available photometers, the operation of the two-way valve and various other operations in section 5.3 may be carried out automatically by the photometer.

5.3.6 Adjust the O_3 generator to produce an O_3 concentration as needed.

5.3.7 Actuate the two-way valve to allow the photometer to sample zero air until the absorption cell is thoroughly flushed and record the stable measured value of I_0 .

5.3.8 Actuate the two-way valve to allow the photometer to sample the ozone concentration until the absorption cell is thoroughly flushed and record the stable measured value of I .

5.3.9 Record the temperature and pressure of the sample in the photometer absorption cell. (See Reference 9 for guidance.)

5.3.10 Calculate the O_3 concentration from equation 4. An average of several determinations will provide better precision.

$$[O_3]_{OUT} = \left(\frac{-1}{\alpha l} \ln \frac{I}{I_0} \right) \left(\frac{T}{273} \right) \left(\frac{760}{P} \right) \times \frac{10^6}{L} \quad (4)$$

where:

$[O_3]_{OUT}$ = O_3 concentration, ppm

α = absorption coefficient of O_3 at 254 nm = 308 atm⁻¹ cm⁻¹ at 0 °C and 760 torr

l = optical path length, cm

T = sample temperature, K

P = sample pressure, torr

L = correction factor for O_3 losses from 5.2.5=(1-fraction O_3 lost).

NOTE: Some commercial photometers may automatically evaluate all or part of equation 4. It is the operator's responsibility to verify that all of the information required for equation 4 is obtained, either automatically by the photometer or manually. For "automatic" photometers which evaluate the first term of equation 4 based on a linear approximation, a manual correction may be required, particularly at higher O_3 levels. See the photometer instruction manual and Reference 9 for guidance.

5.3.11 Obtain additional O_3 concentration standards as necessary by repeating steps 5.3.6 to 5.3.10 or by Option 1.

5.4 *Certification of transfer standards.* A transfer standard is certified by relating the output of the transfer standard to one or

more ozone standards as determined according to section 5.3. The exact procedure varies depending on the nature and design of the transfer standard. Consult Reference 8 for guidance.

5.5 *Calibration of ozone analyzers.* Ozone analyzers are calibrated as follows, using ozone standards obtained directly according to section 5.3 or by means of a certified transfer standard.

5.5.1 Allow sufficient time for the O_3 analyzer and the photometer or transfer standard to warmup and stabilize.

5.5.2 Allow the O_3 analyzer to sample zero air until a stable response is obtained and adjust the O_3 analyzer's zero control. Offsetting the analyzer's zero adjustment to +5% of scale is recommended to facilitate observing negative zero drift. Record the stable zero air response as "Z".

5.5.3 Generate an O_3 concentration standard of approximately 80% of the desired upper range limit (URL) of the O_3 analyzer. Allow the O_3 analyzer to sample this O_3 concentration standard until a stable response is obtained.

5.5.4 Adjust the O_3 analyzer's span control to obtain a convenient recorder response as indicated below:

recorder response (%scale) =

$$\left(\frac{[O_3]_{OUT}}{URL} \times 100 \right) + Z \quad (5)$$

where:

URL = upper range limit of the O_3 analyzer, ppm

Z = recorder response with zero air, % scale

Record the O_3 concentration and the corresponding analyzer response. If substantial adjustment of the span control is necessary, recheck the zero and span adjustments by repeating steps 5.5.2 to 5.5.4.

5.5.5 Generate several other O_3 concentration standards (at least 5 others are recommended) over the scale range of the O_3 analyzer by adjusting the O_3 source or by Option 1. For each O_3 concentration standard, record the O_3 and the corresponding analyzer response.

5.5.6 Plot the O_3 analyzer responses versus the corresponding O_3 concentrations and draw the O_3 analyzer's calibration curve or calculate the appropriate response factor.

5.5.7 *Option 1:* The various O_3 concentrations required in steps 5.3.11 and 5.5.5 may be obtained by dilution of the O_3 concentration generated in steps 5.3.6 and 5.5.3. With this option, accurate flow measurements are required. The dynamic calibration system may be modified as shown in Figure 2 to allow for dilution air to be metered in downstream of the O_3 generator. A mixing chamber between the O_3 generator and the output manifold is also required. The flowrate through the O_3 generator (F_o) and the dilution air flowrate

(F_D) are measured with a reliable flow or volume standard traceable to NBS. Each O_3 concentration generated by dilution is calculated from:

$$[O_3]_{OUT}' = [O_3]_{OUT} \left(\frac{F_O}{F_O + F_D} \right) \quad (6)$$

where:

$[O_3]_{OUT}'$ = diluted O_3 concentration, ppm

F_O = flowrate through the O_3 generator, liter/min

F_D = diluent air flowrate, liter/min

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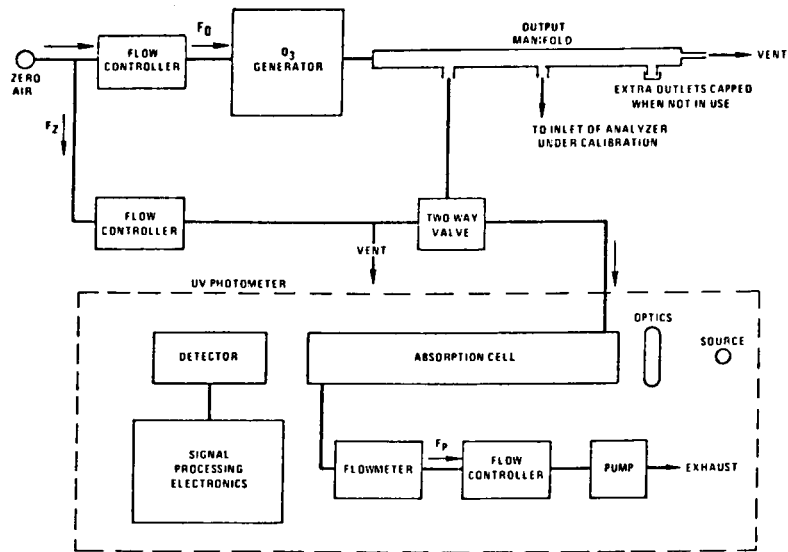


Figure 1. Schematic diagram of a typical UV photometric calibration system.

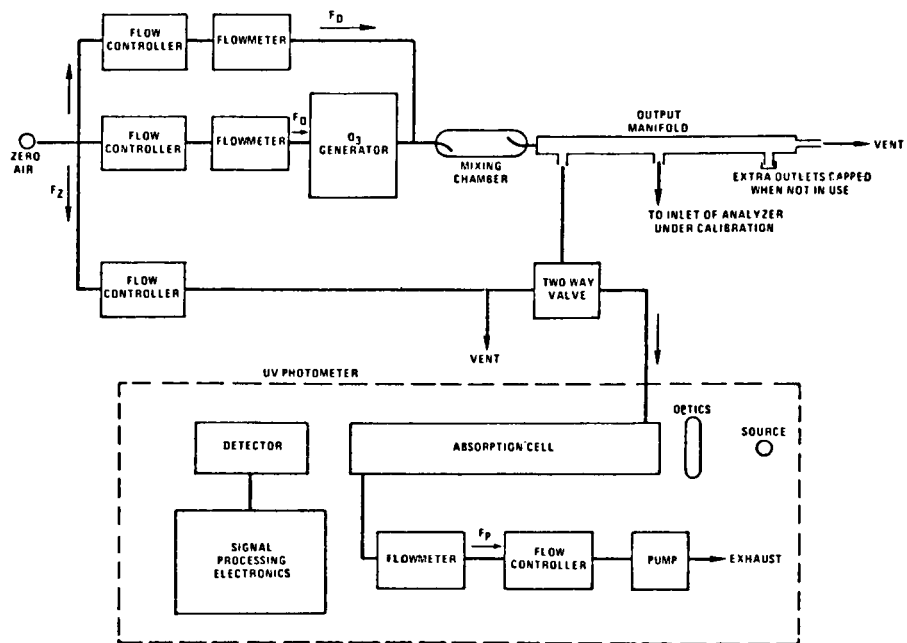


Figure 2. Schematic diagram of a typical UV photometric calibration system (OPTION 1)

[44 FR 8224, Feb. 8, 1979, as amended at 62 FR 38895, July 18, 1997]

APPENDIX E TO PART 50 [RESERVED]

APPENDIX F TO PART 50—MEASUREMENT PRINCIPLE AND CALIBRATION PROCEDURE FOR THE MEASUREMENT OF NITROGEN DIOXIDE IN THE ATMOSPHERE (GAS PHASE CHEMILUMINESCENCE)

PRINCIPLE AND APPLICABILITY

1. Atmospheric concentrations of nitrogen dioxide (NO₂) are measured indirectly by photometrically measuring the light intensity, at wavelengths greater than 600 nanometers, resulting from the chemiluminescent reaction of nitric oxide (NO) with ozone (O₃). (1,2,3) NO₂ is first quantitatively reduced to NO(4,5,6) by means of a converter. NO, which commonly exists in ambient air together with NO₂, passes through the converter unchanged causing a resultant total NO_x concentration equal to NO+NO₂. A sample of the input air is also measured without having passed through the converter. This latter NO measurement is subtracted from the former measurement (NO+NO₂) to yield the final NO₂ measurement. The NO and NO+NO₂ measurements may be made concurrently with dual systems, or cyclically with the same system provided the cycle time does not exceed 1 minute.

2. *Sampling considerations.*

2.1 Chemiluminescence NO/NO_x/NO₂ analyzers will respond to other nitrogen containing compounds, such as peroxyacetyl nitrate (PAN), which might be reduced to NO in the thermal converter. (7) Atmospheric concentrations of these potential interferences are generally low relative to NO₂ and valid NO₂ measurements may be obtained. In certain geographical areas, where the concentration of these potential interferences is known or suspected to be high relative to NO₂, the use of an equivalent method for the measurement of NO₂ is recommended.

2.2 The use of integrating flasks on the sample inlet line of chemiluminescence NO/NO_x/NO₂ analyzers is optional and left to the discretion of the user. The sample residence time between the sampling point and the analyzer should be kept to a minimum to avoid erroneous NO₂ measurements resulting from the reaction of ambient levels of NO and O₃ in the sampling system.

2.3 The use of particulate filters on the sample inlet line of chemiluminescence NO/NO_x/NO₂ analyzers is optional and left to the discretion of the user or the manufacturer. Use of the filter should depend on the analyzer's susceptibility to interference, malfunction, or damage due to particulates. Users are cautioned that particulate matter concentrated on a filter may cause erroneous

NO₂ measurements and therefore filters should be changed frequently.

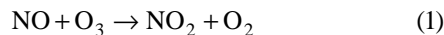
3. An analyzer based on this principle will be considered a reference method only if it has been designated as a reference method in accordance with part 53 of this chapter.

CALIBRATION

1. *Alternative A*—Gas phase titration (GPT) of an NO standard with O₃.

Major equipment required: Stable O₃ generator. Chemiluminescence NO/NO_x/NO₂ analyzer with strip chart recorder(s). NO concentration standard.

1.1 *Principle.* This calibration technique is based upon the rapid gas phase reaction between NO and O₃ to produce stoichiometric quantities of NO₂ in accordance with the following equation: (8)



The quantitative nature of this reaction is such that when the NO concentration is known, the concentration of NO₂ can be determined. Ozone is added to excess NO in a dynamic calibration system, and the NO channel of the chemiluminescence NO/NO_x/NO₂ analyzer is used as an indicator of changes in NO concentration. Upon the addition of O₃, the decrease in NO concentration observed on the calibrated NO channel is equivalent to the concentration of NO₂ produced. The amount of NO₂ generated may be varied by adding variable amounts of O₃ from a stable uncalibrated O₃ generator. (9)

1.2 *Apparatus.* Figure 1, a schematic of a typical GPT apparatus, shows the suggested configuration of the components listed below. All connections between components in the calibration system downstream from the O₃ generator should be of glass, Teflon®, or other non-reactive material.

1.2.1 *Air flow controllers.* Devices capable of maintaining constant air flows within ±2% of the required flowrate.

1.2.2 *NO flow controller.* A device capable of maintaining constant NO flows within ±2% of the required flowrate. Component parts in contact with the NO should be of a non-reactive material.

1.2.3 *Air flowmeters.* Calibrated flowmeters capable of measuring and monitoring air flowrates with an accuracy of ±2% of the measured flowrate.

1.2.4 *NO flowmeter.* A calibrated flowmeter capable of measuring and monitoring NO flowrates with an accuracy of ±2% of the measured flowrate. (Rotameters have been reported to operate unreliably when measuring low NO flows and are not recommended.)

1.2.5 *Pressure regulator for standard NO cylinder.* This regulator must have a nonreactive diaphragm and internal parts and a suitable delivery pressure.

1.2.6 *Ozone generator.* The generator must be capable of generating sufficient and stable levels of O₃ for reaction with NO to generate NO₂ concentrations in the range required. Ozone generators of the electric discharge type may produce NO and NO₂ and are not recommended.

1.2.7 *Valve.* A valve may be used as shown in Figure 1 to divert the NO flow when zero air is required at the manifold. The valve should be constructed of glass, Teflon[®], or other nonreactive material.

1.2.8 *Reaction chamber.* A chamber, constructed of glass, Teflon[®], or other nonreactive material, for the quantitative reaction of O₃ with excess NO. The chamber should be of sufficient volume (V_{RC}) such that the residence time (t_R) meets the requirements specified in 1.4. For practical reasons, t_R should be less than 2 minutes.

1.2.9 *Mixing chamber.* A chamber constructed of glass, Teflon[®], or other nonreactive material and designed to provide thorough mixing of reaction products and diluent air. The residence time is not critical when the dynamic parameter specification given in 1.4 is met.

1.2.10 *Output manifold.* The output manifold should be constructed of glass, Teflon[®], or other non-reactive material and should be of sufficient diameter to insure an insignificant pressure drop at the analyzer connection. The system must have a vent designed to insure atmospheric pressure at the manifold and to prevent ambient air from entering the manifold.

1.3 Reagents.

1.3.1 *NO concentration standard.* Gas cylinder standard containing 50 to 100 ppm NO in N₂ with less than 1 ppm NO₂. This standard must be traceable to a National Bureau of Standards (NBS) NO in N₂ Standard Reference Material (SRM 1683 or SRM 1684), an NBS NO₂ Standard Reference Material (SRM 1629), or an NBS/EPA-approved commercially available Certified Reference Material (CRM). CRM's are described in Reference 14, and a list of CRM sources is available from the address shown for Reference 14. A recommended protocol for certifying NO gas cylinders against either an NO SRM or CRM is given in section 2.0.7 of Reference 15. Reference 13 gives procedures for certifying an NO gas cylinder against an NBS NO₂ SRM and for determining the amount of NO₂ impurity in an NO cylinder.

1.3.2 *Zero air.* Air, free of contaminants which will cause a detectable response on the NO/NO_x/NO₂ analyzer or which might react with either NO, O₃, or NO₂ in the gas phase titration. A procedure for generating zero air is given in reference 13.

1.4 Dynamic parameter specification.

1.4.1 The O₃ generator air flowrate (F_O) and NO flowrate (F_{NO}) (see Figure 1) must be adjusted such that the following relationship holds:

$$P_R = [\text{NO}]_{RC} \times t_R \quad 2.75 \text{ ppm-minutes} \quad (2)$$

$$[\text{NO}]_{RC} = [\text{NO}]_{\text{STD}} \left(\frac{F_{NO}}{F_O + F_{NO}} \right) \quad (3)$$

$$t_R = \frac{V_{RC}}{F_O + F_{NO}} < 2 \text{ minutes} \quad (4)$$

where:

P_R = dynamic parameter specification, determined empirically, to insure complete reaction of the available O₃, ppm-minute

$[\text{NO}]_{RC}$ = NO concentration in the reaction chamber, ppm

t_R = residence time of the reactant gases in the reaction chamber, minute

$[\text{NO}]_{\text{STD}}$ = concentration of the undiluted NO standard, ppm

F_{NO} = NO flowrate, scm³/min

F_O = O₃ generator air flowrate, scm³/min

V_{RC} = volume of the reaction chamber, scm³

1.4.2 The flow conditions to be used in the GPT system are determined by the following procedure:

(a) Determine F_T , the total flow required at the output manifold (F_T =analyzer demand plus 10 to 50% excess).

(b) Establish $[\text{NO}]_{\text{OUT}}$ as the highest NO concentration (ppm) which will be required at the output manifold. $[\text{NO}]_{\text{OUT}}$ should be approximately equivalent to 90% of the upper range limit (URL) of the NO₂ concentration range to be covered.

(c) Determine F_{NO} as

$$F_{NO} = \frac{[\text{NO}]_{\text{OUT}} \times F_T}{[\text{NO}]_{\text{STD}}} \quad (5)$$

(d) Select a convenient or available reaction chamber volume. Initially, a trial V_{RC} may be selected to be in the range of approximately 200 to 500 scm³.

(e) Compute F_O as

$$F_O = \sqrt{\frac{[\text{NO}]_{\text{STD}} \times F_{NO} \times V_{RC}}{2.75}} - F_{NO} \quad (6)$$

(f) Compute t_R as

$$t_R = \frac{V_{RC}}{F_O + F_{NO}} \quad (7)$$

Verify that $t_R < 2$ minutes. If not, select a reaction chamber with a smaller V_{RC} .

(g) Compute the diluent air flowrate as

$$F_D = F_T - F_O - F_{NO} \quad (8)$$

where:

F_D = diluent air flowrate, scm³/min

(h) If F_O turns out to be impractical for the desired system, select a reaction chamber having a different V_{RC} and recompute F_O and F_D .

NOTE: A dynamic parameter lower than 2.75 ppm-minutes may be used if it can be determined empirically that quantitative reaction of O_3 with NO occurs. A procedure for making this determination as well as a more detailed discussion of the above requirements and other related considerations is given in reference 13.

1.5 Procedure.

1.5.1 Assemble a dynamic calibration system such as the one shown in Figure 1.

1.5.2 Insure that all flowmeters are calibrated under the conditions of use against a reliable standard such as a soap-bubble meter or wet-test meter. All volumetric flowrates should be corrected to 25 °C and 760 mm Hg. A discussion on the calibration of flowmeters is given in reference 13.

1.5.3 Precautions must be taken to remove O_2 and other contaminants from the NO pressure regulator and delivery system prior to the start of calibration to avoid any conversion of the standard NO to NO_2 . Failure to do so can cause significant errors in calibration. This problem may be minimized by (1) carefully evacuating the regulator, when possible, after the regulator has been connected to the cylinder and before opening the cylinder valve; (2) thoroughly flushing the regulator and delivery system with NO after opening the cylinder valve; (3) not removing the regulator from the cylinder between calibrations unless absolutely necessary. Further discussion of these procedures is given in reference 13.

1.5.4 Select the operating range of the NO/ NO_x / NO_2 analyzer to be calibrated. In order to obtain maximum precision and accuracy for NO_2 calibration, all three channels of the analyzer should be set to the same range. If operation of the NO and NO_x channels on higher ranges is desired, subsequent recalibration of the NO and NO_x channels on the higher ranges is recommended.

NOTE: Some analyzer designs may require identical ranges for NO, NO_x , and NO_2 during operation of the analyzer.

1.5.5 Connect the recorder output cable(s) of the NO/ NO_x / NO_2 analyzer to the input terminals of the strip chart recorder(s). All adjustments to the analyzer should be performed based on the appropriate strip chart readings. References to analyzer responses in the procedures given below refer to recorder responses.

1.5.6 Determine the GPT flow conditions required to meet the dynamic parameter specification as indicated in 1.4.

1.5.7 Adjust the diluent air and O_3 generator air flows to obtain the flows determined in section 1.4.2. The total air flow must exceed the total demand of the analyzer(s) connected to the output manifold to insure that

no ambient air is pulled into the manifold vent. Allow the analyzer to sample zero air until stable NO, NO_x , and NO_2 responses are obtained. After the responses have stabilized, adjust the analyzer zero control(s).

NOTE: Some analyzers may have separate zero controls for NO, NO_x , and NO_2 . Other analyzers may have separate zero controls only for NO and NO_x , while still others may have only one zero control common to all three channels.

Offsetting the analyzer zero adjustments to +5 percent of scale is recommended to facilitate observing negative zero drift. Record the stable zero air responses as Z_{NO} , Z_{NO_x} , and Z_{NO_2} .

1.5.8 Preparation of NO and NO_x calibration curves.

1.5.8.1 Adjustment of NO span control. Adjust the NO flow from the standard NO cylinder to generate an NO concentration of approximately 80 percent of the upper range limit (URL) of the NO range. This exact NO concentration is calculated from:

$$[NO]_{OUT} = \frac{F_{NO} \times [NO]_{STD}}{F_{NO} + F_O + F_D} \quad (9)$$

where:

$[NO]_{OUT}$ = diluted NO concentration at the output manifold, ppm

Sample this NO concentration until the NO and NO_x responses have stabilized. Adjust the NO span control to obtain a recorder response as indicated below:

recorder response (percent scale) =

$$\left(\frac{[NO]_{OUT} \times 100}{URL} \right) + Z_{NO} \quad (10)$$

where:

URL = nominal upper range limit of the NO channel, ppm

NOTE: Some analyzers may have separate span controls for NO, NO_x , and NO_2 . Other analyzers may have separate span controls only for NO and NO_x , while still others may have only one span control common to all three channels. When only one span control is available, the span adjustment is made on the NO channel of the analyzer.

If substantial adjustment of the NO span control is necessary, it may be necessary to recheck the zero and span adjustments by repeating steps 1.5.7 and 1.5.8.1. Record the NO concentration and the analyzer's NO response.

1.5.8.2 Adjustment of NO_x span control. When adjusting the analyzer's NO_x span control, the presence of any NO_2 impurity in the standard NO cylinder must be taken into account. Procedures for determining the amount of NO_2 impurity in the standard NO

cylinder are given in reference 13. The exact NO_x concentration is calculated from:

$$[\text{NO}_x]_{\text{OUT}} = \frac{F_{\text{NO}} \times ([\text{NO}]_{\text{STD}} + [\text{NO}_2]_{\text{IMP}})}{F_{\text{NO}} + F_{\text{O}} + F_{\text{D}}} \quad (11)$$

where:

[NO_x]_{OUT} = diluted NO_x concentration at the output manifold, ppm

[NO₂]_{IMP} = concentration of NO₂ impurity in the standard NO cylinder, ppm

Adjust the NO_x span control to obtain a recorder response as indicated below:

recorder response (% scale) =

$$\left(\frac{[\text{NO}_x]_{\text{OUT}} \times 100}{\text{URL}} \right) + Z_{\text{NO}_x} \quad (12)$$

NOTE: If the analyzer has only one span control, the span adjustment is made on the NO channel and no further adjustment is made here for NO_x.

If substantial adjustment of the NO_x span control is necessary, it may be necessary to recheck the zero and span adjustments by repeating steps 1.5.7 and 1.5.8.2. Record the NO_x concentration and the analyzer's NO_x response.

1.5.8.3 Generate several additional concentrations (at least five evenly spaced points across the remaining scale are suggested to verify linearity) by decreasing F_{NO} or increasing F_D. For each concentration generated, calculate the exact NO and NO_x

concentrations using equations (9) and (11) respectively. Record the analyzer's NO and NO_x responses for each concentration. Plot the analyzer responses versus the respective calculated NO and NO_x concentrations and draw or calculate the NO and NO_x calibration curves. For subsequent calibrations where linearity can be assumed, these curves may be checked with a two-point calibration consisting of a zero air point and NO and NO_x concentrations of approximately 80% of the URL.

1.5.9 Preparation of NO₂ calibration curve.

1.5.9.1 Assuming the NO₂ zero has been properly adjusted while sampling zero air in step 1.5.7, adjust F_O and F_D as determined in section 1.4.2. Adjust F_{NO} to generate an NO concentration near 90% of the URL of the NO range. Sample this NO concentration until the NO and NO_x responses have stabilized. Using the NO calibration curve obtained in section 1.5.8, measure and record the NO concentration as [NO]_{orig}. Using the NO_x calibration curve obtained in section 1.5.8, measure and record the NO_x concentration as [NO_x]_{orig}.

1.5.9.2 Adjust the O₃ generator to generate sufficient O₃ to produce a decrease in the NO concentration equivalent to approximately 80% of the URL of the NO₂ range. The decrease must not exceed 90% of the NO concentration determined in step 1.5.9.1. After the analyzer responses have stabilized, record the resultant NO and NO_x concentrations as [NO]_{rem} and [NO_x]_{rem}.

1.5.9.3 Calculate the resulting NO₂ concentration from:

$$[\text{NO}_2]_{\text{OUT}} = [\text{NO}]_{\text{orig}} - [\text{NO}]_{\text{rem}} + \frac{F_{\text{NO}} \times [\text{NO}_2]_{\text{IMP}}}{F_{\text{NO}} + F_{\text{O}} + F_{\text{D}}} \quad (13)$$

where:

[NO₂]_{OUT} = diluted NO₂ concentration at the output manifold, ppm

[NO]_{orig} = original NO concentration, prior to addition of O₃, ppm

[NO]_{rem} = NO concentration remaining after addition of O₃, ppm

Adjust the NO₂ span control to obtain a recorder response as indicated below:

recorder response (% scale) =

$$\left(\frac{[\text{NO}_2]_{\text{OUT}} \times 100}{\text{URL}} \right) + Z_{\text{NO}_2} \quad (14)$$

NOTE: If the analyzer has only one or two span controls, the span adjustments are made on the NO channel or NO and NO_x channels and no further adjustment is made here for NO₂.

If substantial adjustment of the NO₂ span control is necessary, it may be necessary to recheck the zero and span adjustments by repeating steps 1.5.7 and 1.5.9.3. Record the NO₂ concentration and the corresponding analyzer NO₂ and NO_x responses.

1.5.9.4 Maintaining the same F_{NO}, F_O, and F_D as in section 1.5.9.1, adjust the ozone generator to obtain several other concentrations of NO₂ over the NO₂ range (at least five evenly spaced points across the remaining scale are suggested). Calculate each NO₂ concentration using equation (13) and record the corresponding analyzer NO₂ and NO_x responses. Plot the analyzer's NO₂ responses versus the corresponding calculated NO₂ concentrations and draw or calculate the NO₂ calibration curve.

1.5.10 Determination of converter efficiency.

1.5.10.1 For each NO₂ concentration generated during the preparation of the NO₂

calibration curve (see section 1.5.9) calculate the concentration of NO₂ converted from:

$$[\text{NO}_2]_{\text{CONV}} = [\text{NO}_2]_{\text{OUT}} \left(\frac{[\text{NO}_X]_{\text{orig}}}{[\text{NO}_X]_{\text{rem}}} \right) \quad (15)$$

where:

[NO₂]_{CONV} = concentration of NO₂ converted, ppm

[NO_X]_{orig} = original NO_X concentration prior to addition of O₃, ppm

[NO_X]_{rem} = NO_X concentration remaining after addition of O₃, ppm

NOTE: Supplemental information on calibration and other procedures in this method are given in reference 13.

Plot [NO₂]_{CONV} (y-axis) versus [NO₂]_{OUT} (x-axis) and draw or calculate the converter efficiency curve. The slope of the curve times 100 is the average converter efficiency, Ec. The average converter efficiency must be greater than 96%; if it is less than 96%, replace or service the converter.

2. Alternative B—NO₂ permeation device.

Major equipment required:

Stable O₃ generator.

Chemiluminescence NO/NO_X/NO₂ analyzer with strip chart recorder(s).

NO concentration standard.

NO₂ concentration standard.

2.1 *Principle.* Atmospheres containing accurately known concentrations of nitrogen dioxide are generated by means of a permeation device. (10) The permeation device emits NO₂ at a known constant rate provided the temperature of the device is held constant (±0.1 °C) and the device has been accurately calibrated at the temperature of use. The NO₂ emitted from the device is diluted with zero air to produce NO₂ concentrations suitable for calibration of the NO₂ channel of the NO/NO_X/NO₂ analyzer. An NO concentration standard is used for calibration of the NO and NO_X channels of the analyzer.

2.2 *Apparatus.* A typical system suitable for generating the required NO and NO₂ concentrations is shown in Figure 2. All connections between components downstream from the permeation device should be of glass, Teflon®, or other non-reactive material.

2.2.1 *Air flow controllers.* Devices capable of maintaining constant air flows within ±2% of the required flowrate.

2.2.2 *NO flow controller.* A device capable of maintaining constant NO flows within ±2% of the required flowrate. Component parts in contact with the NO must be of a non-reactive material.

2.2.3 *Air flowmeters.* Calibrated flowmeters capable of measuring and monitoring air flowrates with an accuracy of ±2% of the measured flowrate.

2.2.4 *NO flowmeter.* A calibrated flowmeter capable of measuring and monitoring NO flowrates with an accuracy of ±2% of the measured flowrate. (Rotameters have been reported to operate unreliably when measuring low NO flows and are not recommended.)

2.2.5 *Pressure regulator for standard NO cylinder.* This regulator must have a non-reactive diaphragm and internal parts and a suitable delivery pressure.

2.2.6 *Drier.* Scrubber to remove moisture from the permeation device air system. The use of the drier is optional with NO₂ permeation devices not sensitive to moisture. (Refer to the supplier's instructions for use of the permeation device.)

2.2.7 *Constant temperature chamber.* Chamber capable of housing the NO₂ permeation device and maintaining its temperature to within ±0.1 °C.

2.2.8 *Temperature measuring device.* Device capable of measuring and monitoring the temperature of the NO₂ permeation device with an accuracy of ±0.05 °C.

2.2.9 *Valves.* A valve may be used as shown in Figure 2 to divert the NO₂ from the permeation device when zero air or NO is required at the manifold. A second valve may be used to divert the NO flow when zero air or NO₂ is required at the manifold.

The valves should be constructed of glass, Teflon®, or other nonreactive material.

2.2.10 *Mixing chamber.* A chamber constructed of glass, Teflon®, or other nonreactive material and designed to provide thorough mixing of pollutant gas streams and diluent air.

2.2.11 *Output manifold.* The output manifold should be constructed of glass, Teflon®, or other non-reactive material and should be of sufficient diameter to insure an insignificant pressure drop at the analyzer connection. The system must have a vent designed to insure atmospheric pressure at the manifold and to prevent ambient air from entering the manifold.

2.3 Reagents.

2.3.1 *Calibration standards.* Calibration standards are required for both NO and NO₂. The reference standard for the calibration may be either an NO or NO₂ standard, and must be traceable to a National Bureau of Standards (NBS) NO in N₂ Standard Reference Material (SRM 1683 or SRM 1684), and NBS NO₂ Standard Reference Material (SRM 1629), or an NBS/EPA-approved commercially

available Certified Reference Material (CRM). CRM's are described in Reference 14, and a list of CRM sources is available from the address shown for Reference 14. Reference 15 gives recommended procedures for certifying an NO gas cylinder against an NO SRM or CRM and for certifying an NO₂ permeation device against an NO₂ SRM. Reference 13 contains procedures for certifying an NO gas cylinder against an NO₂ SRM and for certifying an NO₂ permeation device against an NO SRM or CRM. A procedure for determining the amount of NO₂ impurity in an NO cylinder is also contained in Reference 13. The NO or NO₂ standard selected as the reference standard must be used to certify the other standard to ensure consistency between the two standards.

2.3.1.1 *NO₂ Concentration standard.* A permeation device suitable for generating NO₂ concentrations at the required flow-rates over the required concentration range. If the permeation device is used as the reference standard, it must be traceable to an SRM or CRM as specified in 2.3.1. If an NO cylinder is used as the reference standard, the NO₂ permeation device must be certified against the NO standard according to the procedure given in Reference 13. The use of the permeation device should be in strict accordance with the instructions supplied with the device. Additional information regarding the use of permeation devices is given by Scaringelli et al. (11) and Rook et al. (12).

2.3.1.2 *NO Concentration standard.* Gas cylinder containing 50 to 100 ppm NO in N₂ with less than 1 ppm NO₂. If this cylinder is used as the reference standard, the cylinder must be traceable to an SRM or CRM as specified in 2.3.1. If an NO₂ permeation device is used as the reference standard, the NO cylinder must be certified against the NO₂ standard according to the procedure given in Reference 13. The cylinder should be recertified on a regular basis as determined by the local quality control program.

2.3.3 *Zero air.* Air, free of contaminants which might react with NO or NO₂ or cause a detectable response on the NO/NO_x/NO₂ analyzer. When using permeation devices that are sensitive to moisture, the zero air passing across the permeation device must be dry to avoid surface reactions on the device. (Refer to the supplier's instructions for use of the permeation device.) A procedure for generating zero air is given in reference 13.

2.4 Procedure.

2.4.1 Assemble the calibration apparatus such as the typical one shown in Figure 2.

2.4.2 Insure that all flowmeters are calibrated under the conditions of use against a reliable standard such as a soap bubble meter or wet-test meter. All volumetric flowrates should be corrected to 25 °C and 760 mm Hg. A discussion on the calibration of flowmeters is given in reference 13.

2.4.3 Install the permeation device in the constant temperature chamber. Provide a small fixed air flow (200-400 scm³/min) across the device. The permeation device should always have a continuous air flow across it to prevent large buildup of NO₂ in the system and a consequent restabilization period. Record the flowrate as FP. Allow the device to stabilize at the calibration temperature for at least 24 hours. The temperature must be adjusted and controlled to within ±0.1 °C or less of the calibration temperature as monitored with the temperature measuring device.

2.4.4 Precautions must be taken to remove O₂ and other contaminants from the NO pressure regulator and delivery system prior to the start of calibration to avoid any conversion of the standard NO to NO₂. Failure to do so can cause significant errors in calibration. This problem may be minimized by

- (1) Carefully evacuating the regulator, when possible, after the regulator has been connected to the cylinder and before opening the cylinder valve;

- (2) Thoroughly flushing the regulator and delivery system with NO after opening the cylinder valve;

- (3) Not removing the regulator from the cylinder between calibrations unless absolutely necessary. Further discussion of these procedures is given in reference 13.

2.4.5 Select the operating range of the NO/NO_x NO₂ analyzer to be calibrated. In order to obtain maximum precision and accuracy for NO₂ calibration, all three channels of the analyzer should be set to the same range. If operation of the NO and NO_x channels on higher ranges is desired, subsequent recalibration of the NO and NO_x channels on the higher ranges is recommended.

NOTE: Some analyzer designs may require identical ranges for NO, NO_x, and NO₂ during operation of the analyzer.

2.4.6 Connect the recorder output cable(s) of the NO/NO_x/NO₂ analyzer to the input terminals of the strip chart recorder(s). All adjustments to the analyzer should be performed based on the appropriate strip chart readings. References to analyzer responses in the procedures given below refer to recorder responses.

2.4.7 Switch the valve to vent the flow from the permeation device and adjust the diluent air flowrate, F_D, to provide zero air at the output manifold. The total air flow must exceed the total demand of the analyzer(s) connected to the output manifold to insure that no ambient air is pulled into the manifold vent. Allow the analyzer to sample zero air until stable NO, NO_x, and NO₂ responses are obtained. After the responses have stabilized, adjust the analyzer zero control(s).

NOTE: Some analyzers may have separate zero controls for NO, NO_x, and NO₂. Other analyzers may have separate zero controls only for NO and NO_x, while still others may

have only one zero common control to all three channels.

Offsetting the analyzer zero adjustments to +5% of scale is recommended to facilitate observing negative zero drift. Record the stable zero air responses as Z_{NO} , Z_{NO_X} , and Z_{NO_2} .

2.4.8 Preparation of NO and NO_X calibration curves.

2.4.8.1 Adjustment of NO span control. Adjust the NO flow from the standard NO cylinder to generate an NO concentration of approximately 80% of the upper range limit (URL) of the NO range. The exact NO concentration is calculated from:

$$[NO]_{OUT} = \frac{F_{NO} \times [NO]_{STD}}{F_{NO} + F_D} \quad (16)$$

where:

$[NO]_{OUT}$ = diluted NO concentration at the output manifold, ppm

F_{NO} = NO flowrate, scm³/min

$[NO]_{STD}$ = concentration of the undiluted NO standard, ppm

F_D = diluent air flowrate, scm³/min

Sample this NO concentration until the NO and NO_X responses have stabilized. Adjust the NO span control to obtain a recorder response as indicated below:

recorder response (% scale) =

$$= \left(\frac{[NO]_{OUT}}{URL} \times 100 \right) + Z_{NO} \quad (17)$$

$$= \left(\frac{[NO_X]_{OUT}}{URL} \times 100 \right) + Z_{NO_X} \quad (19)$$

where:

URL = nominal upper range limit of the NO channel, ppm

NOTE: Some analyzers may have separate span controls for NO, NO_X, and NO₂. Other analyzers may have separate span controls only for NO and NO_X, while still others may have only one span control common to all three channels. When only one span control is available, the span adjustment is made on the NO channel of the analyzer.

If substantial adjustment of the NO span control is necessary, it may be necessary to recheck the zero and span adjustments by repeating steps 2.4.7 and 2.4.8.1. Record the NO concentration and the analyzer's NO response.

2.4.8.2 Adjustment of NO_X span control. When adjusting the analyzer's NO_X span control, the presence of any NO₂ impurity in the standard NO cylinder must be taken into account. Procedures for determining the amount of NO₂ impurity in the standard NO

cylinder are given in reference 13. The exact NO_X concentration is calculated from:

$$[NO_X]_{OUT} = \frac{F_{NO} \times ([NO]_{STD} + [NO_2]_{IMP})}{F_{NO} + F_D} \quad (18)$$

where:

$[NO_X]_{OUT}$ = diluted NO_X concentration at the output manifold, ppm

$[NO_2]_{IMP}$ = concentration of NO₂ impurity in the standard NO cylinder, ppm

Adjust the NO_X span control to obtain a convenient recorder response as indicated below:

recorder response (% scale)

$$= \left(\frac{[NO_X]_{OUT}}{URL} \times 100 \right) + Z_{NO_X} \quad (19)$$

NOTE: If the analyzer has only one span control, the span adjustment is made on the NO channel and no further adjustment is made here for NO_X.

If substantial adjustment of the NO_X span control is necessary, it may be necessary to recheck the zero and span adjustments by repeating steps 2.4.7 and 2.4.8.2. Record the NO_X concentration and the analyzer's NO_X response.

2.4.8.3 Generate several additional concentrations (at least five evenly spaced points across the remaining scale are suggested to verify linearity) by decreasing F_{NO} or increasing F_D . For each concentration generated, calculate the exact NO and NO_X concentrations using equations (16) and (18) respectively. Record the analyzer's NO and NO_X responses for each concentration. Plot the analyzer responses versus the respective calculated NO and NO_X concentrations and draw or calculate the NO and NO_X calibration curves. For subsequent calibrations where linearity can be assumed, these curves may be checked with a two-point calibration consisting of a zero point and NO and NO_X concentrations of approximately 80 percent of the URL.

2.4.9 Preparation of NO₂ calibration curve.

2.4.9.1 Remove the NO flow. Assuming the NO₂ zero has been properly adjusted while sampling zero air in step 2.4.7, switch the valve to provide NO₂ at the output manifold.

2.4.9.2 Adjust F_D to generate an NO₂ concentration of approximately 80 percent of the URL of the NO₂ range. The total air flow must exceed the demand of the analyzer(s) under calibration. The actual concentration of NO₂ is calculated from:

$$[NO_2]_{OUT} = \frac{R \times K}{F_P + F_D} \quad (20)$$

where:

$[\text{NO}_2]_{\text{OUT}}$ = diluted NO_2 concentration at the output manifold, ppm

R = permeation rate, $\mu\text{g}/\text{min}$

$K = 0.532 \mu\text{l NO}_2/\mu\text{g NO}_2$ (at 25 °C and 760 mm Hg)

F_p = air flowrate across permeation device, scm^3/min

F_D = diluent air flowrate, scm^3/min

Sample this NO_2 concentration until the NO_x and NO_2 responses have stabilized. Adjust the NO_2 span control to obtain a recorder response as indicated below:

recorder response (% scale)

$$= \left(\frac{[\text{NO}_2]_{\text{OUT}}}{URL} \times 100 \right) + Z_{\text{NO}_2} \quad (21)$$

NOTE: If the analyzer has only one or two span controls, the span adjustments are made on the NO channel or NO and NO_x channels and no further adjustment is made here for NO_2 .

If substantial adjustment of the NO_2 span control is necessary it may be necessary to recheck the zero and span adjustments by repeating steps 2.4.7 and 2.4.9.2. Record the NO_2 concentration and the analyzer's NO_2 response. Using the NO_x calibration curve obtained in step 2.4.8, measure and record the NO_x concentration as $[\text{NO}_x]_M$.

2.4.9.3 Adjust F_D to obtain several other concentrations of NO_2 over the NO_2 range (at least five evenly spaced points across the remaining scale are suggested). Calculate each NO_2 concentration using equation (20) and record the corresponding analyzer NO_2 and NO_x responses. Plot the analyzer's NO_2 responses versus the corresponding calculated NO_2 concentrations and draw or calculate the NO_2 calibration curve.

2.4.10 *Determination of converter efficiency.*

2.4.10.1 Plot $[\text{NO}_x]_M$ (y-axis) versus $[\text{NO}_2]_{\text{OUT}}$ (x-axis) and draw or calculate the converter efficiency curve. The slope of the curve times 100 is the average converter efficiency, E_C . The average converter efficiency must be greater than 96 percent; if it is less than 96 percent, replace or service the converter.

NOTE: Supplemental information on calibration and other procedures in this method are given in reference 13.

3. *Frequency of calibration.* The frequency of calibration, as well as the number of points necessary to establish the calibration curve and the frequency of other performance checks, will vary from one analyzer to another. The user's quality control program should provide guidelines for initial establishment of these variables and for subsequent alteration as operational experience is accumulated. Manufacturers of analyzers should include in their instruction/operation manuals information and guidance as to

these variables and on other matters of operation, calibration, and quality control.

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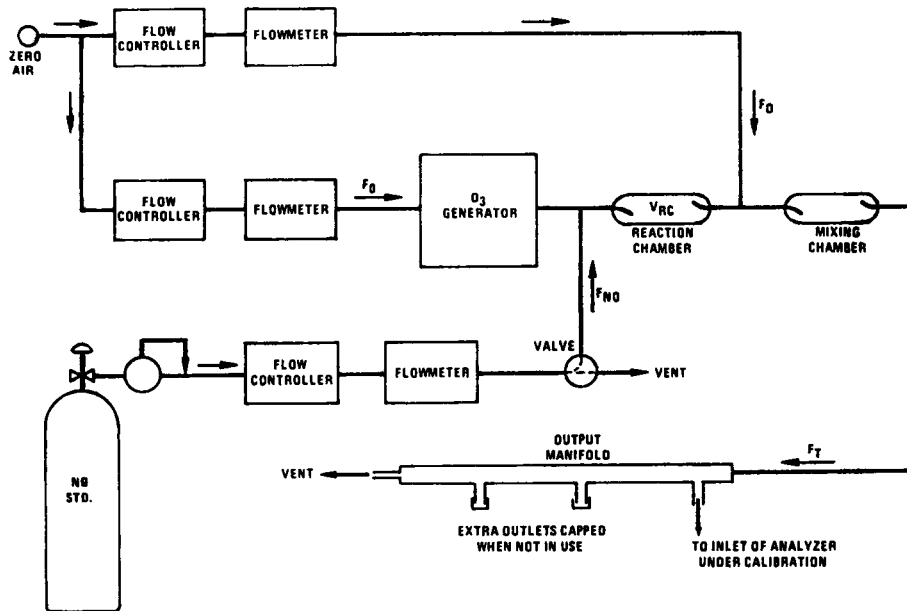


Figure 1. Schematic diagram of a typical GPT calibration system.

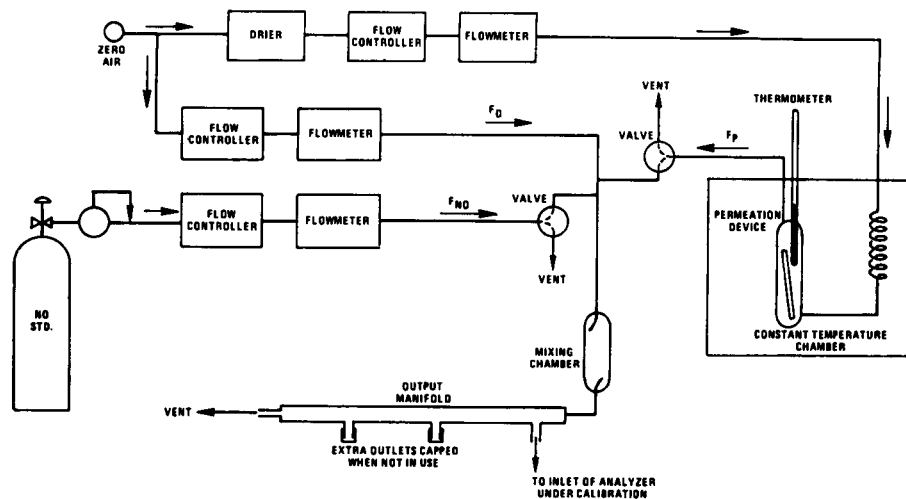


Figure 2. Schematic diagram of a typical calibration apparatus using an NO₂ permeation device.

[41 FR 52688, Dec. 1, 1976, as amended at 48 FR 2529, Jan 20, 1983]

APPENDIX G TO PART 50—REFERENCE METHOD FOR THE DETERMINATION OF LEAD IN SUSPENDED PARTICULATE MATTER COLLECTED FROM AMBIENT AIR

1. Principle and applicability.

1.1 Ambient air suspended particulate matter is collected on a glass-fiber filter for 24 hours using a high volume air sampler. The analysis of the 24-hour samples may be performed for either individual samples or composites of the samples collected over a calendar month or quarter, provided that the compositing procedure has been approved in accordance with section 2.8 of appendix C to part 58 of this chapter—*Modifications of methods by users*. (Guidance or assistance in requesting approval under Section 2.8 can be obtained from the address given in section 2.7 of appendix C to part 58 of this chapter.)

1.2 Lead in the particulate matter is solubilized by extraction with nitric acid (HNO₃), facilitated by heat or by a mixture of HNO₃ and hydrochloric acid (HCl) facilitated by ultrasonication.

1.3 The lead content of the sample is analyzed by atomic absorption spectrometry using an air-acetylene flame, the 283.3 or 217.0 nm lead absorption line, and the optimum instrumental conditions recommended by the manufacturer.

1.4 The ultrasonication extraction with HNO₃/HCl will extract metals other than lead from ambient particulate matter.

2. Range, sensitivity, and lower detectable limit. The values given below are typical of the methods capabilities. Absolute values will vary for individual situations depending on the type of instrument used, the lead line, and operating conditions.

2.1 Range. The typical range of the method is 0.07 to 7.5 µg Pb/m³ assuming an upper linear range of analysis of 15 µg/ml and an air volume of 2,400 m³.

2.2 Sensitivity. Typical sensitivities for a 1 percent change in absorption (0.0044 absorbance units) are 0.2 and 0.5 µg Pb/ml for the 217.0 and 283.3 nm lines, respectively.

2.3 Lower detectable limit (LDL). A typical LDL is 0.07 µg Pb/m³. The above value was calculated by doubling the between-laboratory standard deviation obtained for the lowest measurable lead concentration in a collaborative test of the method.⁽¹⁵⁾ An air volume of 2,400 m³ was assumed.

3. Interferences. Two types of interferences are possible: chemical and light scattering.

3.1 Chemical. Reports on the absence (1, 2, 3, 4, 5) of chemical interferences far outweigh those reporting their presence, (6) therefore, no correction for chemical interferences is given here. If the analyst suspects that the sample matrix is causing a chemical interference, the interference can be verified and corrected for by carrying out the analysis with and without the method of standard additions.⁽⁷⁾

3.2 *Light scattering.* Nonatomic absorption or light scattering, produced by high concentrations of dissolved solids in the sample, can produce a significant interference, especially at low lead concentrations. (2) The interference is greater at the 217.0 nm line than at the 283.3 nm line. No interference was observed using the 283.3 nm line with a similar method.(1)

Light scattering interferences can, however, be corrected for instrumentally. Since the dissolved solids can vary depending on the origin of the sample, the correction may be necessary, especially when using the 217.0 nm line. Dual beam instruments with a continuum source give the most accurate correction. A less accurate correction can be obtained by using a nonabsorbing lead line that is near the lead analytical line. Information on use of these correction techniques can be obtained from instrument manufacturers' manuals.

If instrumental correction is not feasible, the interference can be eliminated by use of the ammonium pyrrolidinecarbodithioate-methylisobutyl ketone, chelation-solvent extraction technique of sample preparation.(8)

4. Precision and bias.

4.1 The high-volume sampling procedure used to collect ambient air particulate matter has a between-laboratory relative standard deviation of 3.7 percent over the range 80 to 125 $\mu\text{g}/\text{m}^3$.(9) The combined extraction-analysis procedure has an average within-laboratory relative standard deviation of 5 to 6 percent over the range 1.5 to 15 $\mu\text{g Pb}/\text{ml}$, and an average between laboratory relative standard deviation of 7 to 9 percent over the same range. These values include use of either extraction procedure.

4.2 Single laboratory experiments and collaborative testing indicate that there is no significant difference in lead recovery between the hot and ultrasonic extraction procedures.(15)

5. Apparatus.

5.1 Sampling.

5.1.1 *High-Volume Sampler.* Use and calibrate the sampler as described in appendix B to this part.

5.2 Analysis.

5.2.1 *Atomic absorption spectrophotometer.* Equipped with lead hollow cathode or electrodeless discharge lamp.

5.2.1.1 *Acetylene.* The grade recommended by the instrument manufacturer should be used. Change cylinder when pressure drops below 50-100 psig.

5.2.1.2 *Air.* Filtered to remove particulate, oil, and water.

5.2.2 *Glassware.* Class A borosilicate glassware should be used throughout the analysis.

5.2.2.1 *Beakers.* 30 and 150 ml. graduated, Pyrex.

5.2.2.2 *Volumetric flasks.* 100-ml.

5.2.2.3 *Pipettes.* To deliver 50, 30, 15, 8, 4, 2, 1 ml.

5.2.2.4 *Cleaning.* All glassware should be scrupulously cleaned. The following procedure is suggested. Wash with laboratory detergent, rinse, soak for 4 hours in 20 percent (w/w) HNO_3 , rinse 3 times with distilled-deionized water, and dry in a dust free manner.

5.2.3 Hot plate.

5.2.4 *Ultrasonication water bath, unheated.* Commercially available laboratory ultrasonic cleaning baths of 450 watts or higher "cleaning power," i.e., actual ultrasonic power output to the bath have been found satisfactory.

5.2.5 *Template.* To aid in sectioning the glass-fiber filter. See figure 1 for dimensions.

5.2.6 *Pizza cutter.* Thin wheel. Thickness 1mm.

5.2.7 Watch glass.

5.2.8 *Polyethylene bottles.* For storage of samples. Linear polyethylene gives better storage stability than other polyethylenes and is preferred.

5.2.9 Parafilm "M".¹ American Can Co., Marathon Products, Neenah, Wis., or equivalent.

6. Reagents.

6.1 Sampling.

6.1.1 *Glass fiber filters.* The specifications given below are intended to aid the user in obtaining high quality filters with reproducible properties. These specifications have been met by EPA contractors.

6.1.1.1 *Lead content.* The absolute lead content of filters is not critical, but low values are, of course, desirable. EPA typically obtains filters with a lead content of 75 $\mu\text{g}/\text{filter}$.

It is important that the variation in lead content from filter to filter, within a given batch, be small.

6.1.1.2 Testing.

6.1.1.2.1 For large batches of filters (>500 filters) select at random 20 to 30 filters from a given batch. For small batches (>500 filters) a lesser number of filters may be taken. Cut one $\frac{3}{4}\times 8$ " strip from each filter anywhere in the filter. Analyze all strips, separately, according to the directions in sections 7 and 8.

6.1.1.2.2 Calculate the total lead in each filter as

$$F_b = \mu\text{g Pb}/\text{ml} \times \frac{100 \text{ ml}}{\text{strip}} \times \frac{12 \text{ strips}}{\text{filter}}$$

where:

F_b = Amount of lead per 72 square inches of filter, μg .

6.1.1.2.3 Calculate the mean, F_b , of the values and the relative standard deviation (standard deviation/mean $\times 100$). If the relative standard deviation is high enough so

¹Mention of commercial products does not imply endorsement by the U.S. Environmental Protection Agency.

that, in the analysts opinion, subtraction of F_b , (section 10.3) may result in a significant error in the $\mu\text{g Pb/m}^3$. the batch should be rejected.

6.1.1.2.4 For acceptable batches, use the value of F_b to correct all lead analyses (section 10.3) of particulate matter collected using that batch of filters. If the analyses are below the LDL (section 2.3) no correction is necessary.

6.2 Analysis.

6.2.1 Concentrated (15.6 M) HNO_3 . ACS reagent grade HNO_3 and commercially available redistilled HNO_3 has found to have sufficiently low lead concentrations.

6.2.2 Concentrated (11.7 M) HCl . ACS reagent grade.

6.2.3 Distilled-deionized water. (D.I. water).

6.2.4 3 M HNO_3 . This solution is used in the hot extraction procedure. To prepare, add 192 ml of concentrated HNO_3 to D.I. water in a 1 l volumetric flask. Shake well, cool, and dilute to volume with D.I. water. *Caution:* Nitric acid fumes are toxic. Prepare in a well ventilated fume hood.

6.2.5 0.45 M HNO_3 . This solution is used as the matrix for calibration standards when using the hot extraction procedure. To prepare, add 29 ml of concentrated HNO_3 to D.I. water in a 1 l volumetric flask. Shake well, cool, and dilute to volume with D.I. water.

6.2.6 2.6 M HNO_3+0 to 0.9 M HCl . This solution is used in the ultrasonic extraction procedure. The concentration of HCl can be varied from 0 to 0.9 M. Directions are given for preparation of a 2.6 M $\text{HNO}_3+0.9$ M HCl solution. Place 167 ml of concentrated HNO_3 into a 1 l volumetric flask and add 77 ml of concentrated HCl . Stir 4 to 6 hours, dilute to nearly 1 l with D.I. water, cool to room temperature, and dilute to 1 l.

6.2.7 0.40 M $\text{HNO}_3 + X$ M HCl . This solution is used as the matrix for calibration standards when using the ultrasonic extraction procedure. To prepare, add 26 ml of concentrated HNO_3 , plus the ml of HCl required, to a 1 l volumetric flask. Dilute to nearly 1 l with D.I. water, cool to room temperature, and dilute to 1 l. The amount of HCl required can be determined from the following equation:

$$y = \frac{77 \text{ ml} \times 0.15 \times}{0.9 \text{ M}}$$

where:

y = ml of concentrated HCl required.

x = molarity of HCl in 6.2.6.

0.15 = dilution factor in 7.2.2.

6.2.8 Lead nitrate, $\text{Pb}(\text{NO}_3)_2$. ACS reagent grade, purity 99.0 percent. Heat for 4 hours at 120 °C and cool in a desiccator.

6.3 Calibration standards.

6.3.1 Master standard, 1000 $\mu\text{g Pb/ml}$ in HNO_3 . Dissolve 1.598 g of $\text{Pb}(\text{NO}_3)_2$ in 0.45 M

HNO_3 contained in a 1 l volumetric flask and dilute to volume with 0.45 M HNO_3 .

6.3.2 Master standard, 1000 $\mu\text{g Pb/ml}$ in HNO_3/HCl . Prepare as in section 6.3.1 except use the HNO_3/HCl solution in section 6.2.7.

Store standards in a polyethylene bottle. Commercially available certified lead standard solutions may also be used.

7. Procedure.

7.1 Sampling. Collect samples for 24 hours using the procedure described in reference 10 with glass-fiber filters meeting the specifications in section 6.1.1. Transport collected samples to the laboratory taking care to minimize contamination and loss of sample. (16).

7.2 Sample preparation.

7.2.1 Hot extraction procedure.

7.2.1.1 Cut a $\frac{3}{4} \times 8$ " strip from the exposed filter using a template and a pizza cutter as described in Figures 1 and 2. Other cutting procedures may be used.

Lead in ambient particulate matter collected on glass fiber filters has been shown to be uniformly distributed across the filter.^{1,3,11} Another study¹² has shown that when sampling near a roadway, strip position contributes significantly to the overall variability associated with lead analyses. Therefore, when sampling near a roadway, additional strips should be analyzed to minimize this variability.

7.2.1.2 Fold the strip in half twice and place in a 150-ml beaker. Add 15 ml of 3 M HNO_3 to cover the sample. The acid should completely cover the sample. Cover the beaker with a watch glass.

7.2.1.3 Place beaker on the hot-plate, contained in a fume hood, and boil gently for 30 min. Do not let the sample evaporate to dryness. *Caution:* Nitric acid fumes are toxic.

7.2.1.4 Remove beaker from hot plate and cool to near room temperature.

7.2.1.5 Quantitatively transfer the sample as follows:

7.2.1.5.1 Rinse watch glass and sides of beaker with D.I. water.

7.2.1.5.2 Decant extract and rinsings into a 100-ml volumetric flask.

7.2.1.5.3 Add D.I. water to 40 ml mark on beaker, cover with watch glass, and set aside for a minimum of 30 minutes. This is a critical step and cannot be omitted since it allows the HNO_3 trapped in the filter to diffuse into the rinse water.

7.2.1.5.4 Decant the water from the filter into the volumetric flask.

7.2.1.5.5 Rinse filter and beaker twice with D.I. water and add rinsings to volumetric flask until total volume is 80 to 85 ml.

7.2.1.5.6 Stopper flask and shake vigorously. Set aside for approximately 5 minutes or until foam has dissipated.

7.2.1.5.7 Bring solution to volume with D.I. water. Mix thoroughly.

7.2.1.5.8 Allow solution to settle for one hour before proceeding with analysis.

7.2.1.5.9 If sample is to be stored for subsequent analysis, transfer to a linear polyethylene bottle.

7.2.2 *Ultrasonic extraction procedure.*

7.2.2.1 Cut a 3/4"x8" strip from the exposed filter as described in section 7.2.1.1.

7.2.2.2 Fold the strip in half twice and place in a 30 ml beaker. Add 15 ml of the HNO₃/HCl solution in section 6.2.6. The acid should completely cover the sample. Cover the beaker with parafilm.

The parafilm should be placed over the beaker such that none of the parafilm is in contact with water in the ultrasonic bath. Otherwise, rinsing of the parafilm (section 7.2.2.4.1) may contaminate the sample.

7.2.2.3 Place the beaker in the ultrasonication bath and operate for 30 minutes.

7.2.2.4 Quantitatively transfer the sample as follows:

7.2.2.4.1 Rinse parafilm and sides of beaker with D.I. water.

7.2.2.4.2 Decant extract and rinsings into a 100 ml volumetric flask.

7.2.2.4.3 Add 20 ml D.I. water to cover the filter strip, cover with parafilm, and set aside for a minimum of 30 minutes. This is a critical step and cannot be omitted. The sample is then processed as in sections 7.2.1.5.4 through 7.2.1.5.9.

NOTE: Samples prepared by the hot extraction procedure are now in 0.45 M HNO₃. Samples prepared by the ultrasonication procedure are in 0.40 M HNO₃ + X M HCl.

8. *Analysis.*

8.1 Set the wavelength of the monochromator at 283.3 or 217.0 nm. Set or align other instrumental operating conditions as recommended by the manufacturer.

8.2 The sample can be analyzed directly from the volumetric flask, or an appropriate amount of sample decanted into a sample analysis tube. In either case, care should be taken not to disturb the settled solids.

8.3 Aspirate samples, calibration standards and blanks (section 9.2) into the flame and record the equilibrium absorbance.

8.4 Determine the lead concentration in µg Pb/ml, from the calibration curve, section 9.3.

8.5 Samples that exceed the linear calibration range should be diluted with acid of the same concentration as the calibration standards and reanalyzed.

9. *Calibration.*

9.1 *Working standard.* 20 µg Pb/ml. Prepared by diluting 2.0 ml of the master standard

(section 6.3.1 if the hot acid extraction was used or section 6.3.2 if the ultrasonic extraction procedure was used) to 100 ml with acid of the same concentration as used in preparing the master standard.

9.2 *Calibration standards.* Prepare daily by diluting the working standard, with the same acid matrix, as indicated below. Other lead concentrations may be used.

Volume of 20 µg/ml working standard, ml	Final volume, ml	Concentration µg Pb/ml
0	100	0
1.0	200	0.1
2.0	200	0.2
2.0	100	0.4
4.0	100	0.8
8.0	100	1.6
15.0	100	3.0
30.0	100	6.0
50.0	100	10.0
100.0	100	20.0

9.3 *Preparation of calibration curve.* Since the working range of analysis will vary depending on which lead line is used and the type of instrument, no one set of instructions for preparation of a calibration curve can be given. Select standards (plus the reagent blank), in the same acid concentration as the samples, to cover the linear absorption range indicated by the instrument manufacturer. Measure the absorbance of the blank and standards as in section 8.0. Repeat until good agreement is obtained between replicates. Plot absorbance (y-axis) versus concentration in µg Pb/ml (x-axis). Draw (or compute) a straight line through the linear portion of the curve. Do not force the calibration curve through zero. Other calibration procedures may be used.

To determine stability of the calibration curve, remeasure—alternately—one of the following calibration standards for every 10th sample analyzed: Concentration ≤ 1µg Pb/ml; concentration ≤ 10 µg Pb/ml. If either standard deviates by more than 5 percent from the value predicted by the calibration curve, recalibrate and repeat the previous 10 analyses.

10. *Calculation.*

10.1 *Measured air volume.* Calculate the measured air volume at Standard Temperature and Pressure as described in Reference 10.

10.2 *Lead concentration.* Calculate lead concentration in the air sample.

$$C = \frac{(\mu\text{g Pb/ml} \times 100 \text{ ml/strip} \times 12 \text{ strips/filter}) - \bar{F}_b}{V_{\text{STP}}}$$

where:

C = Concentration, $\mu\text{g Pb}/\text{sm}^3$.
 $\mu\text{g Pb}/\text{ml}$ = Lead concentration determined from section 8.
 100 ml/strip = Total sample volume.
 12 strips = Total useable filter area, 8"×9". Exposed area of one strip, 3/4"×7".
 Filter = Total area of one strip, 3/4"×8".
 F_b = Lead concentration of blank filter, μg , from section 6.1.1.2.3.
 V_{STP} = Air volume from section 10.2.

11. *Quality control.*

3/4"×8" glass fiber filter strips containing 80 to 2000 $\mu\text{g Pb}/\text{strip}$ (as lead salts) and blank strips with zero Pb content should be used to determine if the method—as being used—has any bias. Quality control charts should be established to monitor differences between measured and true values. The frequency of such checks will depend on the local quality control program.

To minimize the possibility of generating unreliable data, the user should follow practices established for assuring the quality of air pollution data, (13) and take part in EPA's semiannual audit program for lead analyses.

12. *Trouble shooting.*

1. During extraction of lead by the hot extraction procedure, it is important to keep the sample covered so that corrosion products—formed on fume hood surfaces which may contain lead—are not deposited in the extract.

2. The sample acid concentration should minimize corrosion of the nebulizer. However, different nebulizers may require lower acid concentrations. Lower concentrations can be used provided samples and standards have the same acid concentration.

3. Ashing of particulate samples has been found, by EPA and contractor laboratories, to be unnecessary in lead analyses by atomic absorption. Therefore, this step was omitted from the method.

4. Filtration of extracted samples, to remove particulate matter, was specifically excluded from sample preparation, because some analysts have observed losses of lead due to filtration.

5. If suspended solids should clog the nebulizer during analysis of samples, centrifuge the sample to remove the solids.

13. *Authority.*

(Secs. 109 and 301(a), Clean Air Act, as amended (42 U.S.C. 7409, 7601(a)))

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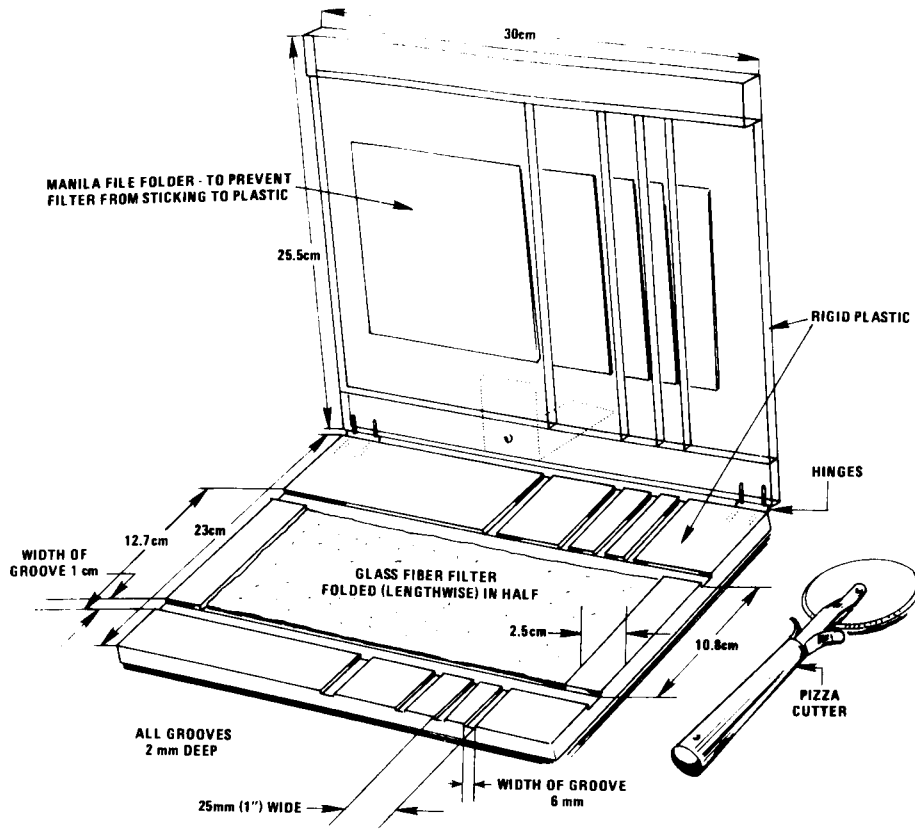


Figure 1

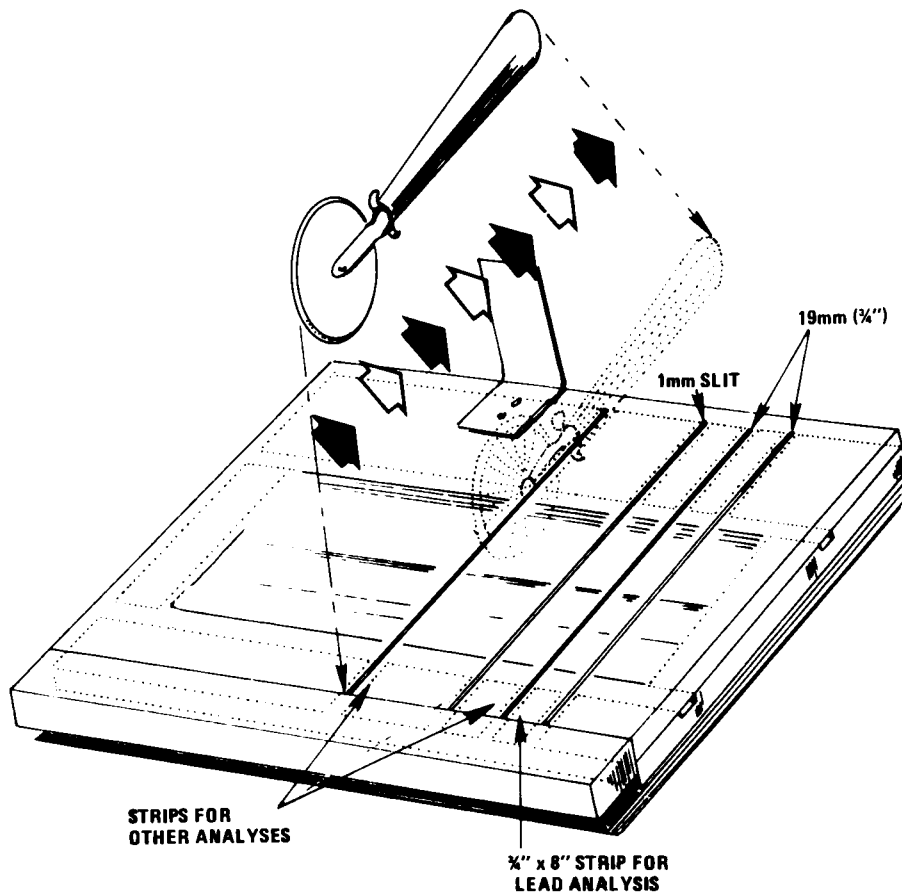


Figure 2

(Secs. 109, 301(a) of the Clean Air Act, as amended (42 U.S.C. 7409, 7601(a)); secs. 110, 301(a) and 319 of the Clean Air Act (42 U.S.C. 7410, 7601(a), 7619))

[43 FR 46258, Oct. 5, 1978; 44 FR 37915, June 29, 1979, as amended at 46 FR 44163, Sept. 3, 1981; 52 FR 24664, July 1, 1987]

APPENDIX H TO PART 50—INTERPRETATION OF THE 1-HOUR PRIMARY AND SECONDARY NATIONAL AMBIENT AIR QUALITY STANDARDS FOR OZONE

1. GENERAL

This appendix explains how to determine when the expected number of days per calendar year with maximum hourly average concentrations above 0.12 ppm (235 $\mu\text{g}/\text{m}^3$) is equal to or less than 1. An expanded discussion of these procedures and associated ex-

amples are contained in the "Guideline for Interpretation of Ozone Air Quality Standards." For purposes of clarity in the following discussion, it is convenient to use the term "exceedance" to describe a daily maximum hourly average ozone measurement that is greater than the level of the standard. Therefore, the phrase "expected number of days with maximum hourly average ozone concentrations above the level of the standard" may be simply stated as the "expected number of exceedances."

The basic principle in making this determination is relatively straightforward. Most of the complications that arise in determining the expected number of annual exceedances relate to accounting for incomplete sampling. In general, the average number of exceedances per calendar year must be less than or equal to 1. In its simplest form, the number of exceedances at a monitoring site would be recorded for each calendar year and then averaged over the past 3 calendar years to determine if this average is less than or equal to 1.

2. INTERPRETATION OF EXPECTED EXCEEDANCES

The ozone standard states that the expected number of exceedances per year must be less than or equal to 1. The statistical term "expected number" is basically an arithmetic average. The following example explains what it would mean for an area to be in compliance with this type of standard. Suppose a monitoring station records a valid daily maximum hourly average ozone value for every day of the year during the past 3 years. At the end of each year, the number of days with maximum hourly concentrations above 0.12 ppm is determined and this number is averaged with the results of previous years. As long as this average remains "less than or equal to 1," the area is in compliance.

3. ESTIMATING THE NUMBER OF EXCEEDANCES FOR A YEAR

In general, a valid daily maximum hourly average value may not be available for each day of the year, and it will be necessary to account for these missing values when estimating the number of exceedances for a particular calendar year. The purpose of these computations is to determine if the expected number of exceedances per year is less than or equal to 1. Thus, if a site has two or more observed exceedances each year, the standard is not met and it is not necessary to use the procedures of this section to account for incomplete sampling.

The term "missing value" is used here in the general sense to describe all days that do not have an associated ozone measurement. In some cases, a measurement might actually have been missed but in other cases no measurement may have been scheduled for that day. A daily maximum ozone value is defined to be the highest hourly ozone value recorded for the day. This daily maximum value is considered to be valid if 75 percent of the hours from 9:01 a.m. to 9:00 p.m. (LST) were measured or if the highest hour is greater than the level of the standard.

In some areas, the seasonal pattern of ozone is so pronounced that entire months need not be sampled because it is extremely unlikely that the standard would be exceed-

ed. Any such waiver of the ozone monitoring requirement would be handled under provisions of 40 CFR, part 58. Some allowance should also be made for days for which valid daily maximum hourly values were not obtained but which would quite likely have been below the standard. Such an allowance introduces a complication in that it becomes necessary to define under what conditions a missing value may be assumed to have been less than the level of the standard. The following criterion may be used for ozone:

A missing daily maximum ozone value may be assumed to be less than the level of the standard if the valid daily maxima on both the preceding day and the following day do not exceed 75 percent of the level of the standard.

Let z denote the number of missing daily maximum values that may be assumed to be less than the standard. Then the following formula shall be used to estimate the expected number of exceedances for the year:

$$e = v + [(v/n)8(N-n-z)] \quad (1)$$

(*Indicates multiplication.)

where:

e = the estimated number of exceedances for the year,

N = the number of required monitoring days in the year,

n = the number of valid daily maxima,

v = the number of daily values above the level of the standard, and

z = the number of days assumed to be less than the standard level.

This estimated number of exceedances shall be rounded to one decimal place (fractional parts equal to 0.05 round up).

It should be noted that N will be the total number of days in the year unless the appropriate Regional Administrator has granted a waiver under the provisions of 40 CFR part 58.

The above equation may be interpreted intuitively in the following manner. The estimated number of exceedances is equal to the observed number of exceedances (v) plus an increment that accounts for incomplete sampling. There were $(N-n)$ missing values for the year but a certain number of these, namely z , were assumed to be less than the standard. Therefore, $(N-n-z)$ missing values are considered to include possible exceedances. The fraction of measured values that are above the level of the standard is v/n . It is assumed that this same fraction applies to the $(N-n-z)$ missing values and that $(v/n)*(N-n-z)$ of these values would also have exceeded the level of the standard.

[44 FR 8220, Feb. 8, 1979, as amended at 62 FR 38895, July 18, 1997]

APPENDIX I TO PART 50—INTERPRETATION OF THE 8-HOUR PRIMARY AND SECONDARY NATIONAL AMBIENT AIR QUALITY STANDARDS FOR OZONE

1. *General.*

This appendix explains the data handling conventions and computations necessary for determining whether the national 8-hour primary and secondary ambient air quality standards for ozone specified in §50.10 are met at an ambient ozone air quality monitoring site. Ozone is measured in the ambient air by a reference method based on appendix D of this part. Data reporting, data handling, and computation procedures to be used in making comparisons between reported ozone concentrations and the level of the ozone standard are specified in the following sections. Whether to exclude, retain, or make adjustments to the data affected by stratospheric ozone intrusion or other natural events is subject to the approval of the appropriate Regional Administrator.

2. *Primary and Secondary Ambient Air Quality Standards for Ozone.*2.1 *Data Reporting and Handling Conventions.*

2.1.1 *Computing 8-hour averages.* Hourly average concentrations shall be reported in parts per million (ppm) to the third decimal place, with additional digits to the right being truncated. Running 8-hour averages shall be computed from the hourly ozone concentration data for each hour of the year and the result shall be stored in the first, or start, hour of the 8-hour period. An 8-hour average shall be considered valid if at least 75% of the hourly averages for the 8-hour period are available. In the event that only 6 (or 7) hourly averages are available, the 8-hour average shall be computed on the basis of the hours available using 6 (or 7) as the divisor. (8-hour periods with three or more missing hours shall not be ignored if, after substituting one-half the minimum detectable limit for the missing hourly concentrations, the 8-hour average concentration is greater than the level of the standard.) The computed 8-hour average ozone concentrations shall be reported to three decimal places (the insignificant digits to the right of the third decimal place are truncated, consistent with the data handling procedures for the reported data.)

2.1.2 *Daily maximum 8-hour average concentrations.* (a) There are 24 possible running 8-hour average ozone concentrations for each calendar day during the ozone monitoring season. (Ozone monitoring seasons vary by geographic location as designated in part 58, appendix D to this chapter.) The daily maximum 8-hour concentration for a given calendar day is the highest of the 24 possible 8-hour average concentrations computed for that day. This process is repeated, yielding a daily maximum 8-hour average ozone con-

centration for each calendar day with ambient ozone monitoring data. Because the 8-hour averages are recorded in the start hour, the daily maximum 8-hour concentrations from two consecutive days may have some hourly concentrations in common. Generally, overlapping daily maximum 8-hour averages are not likely, except in those non-urban monitoring locations with less pronounced diurnal variation in hourly concentrations.

(b) An ozone monitoring day shall be counted as a valid day if valid 8-hour averages are available for at least 75% of possible hours in the day (i.e., at least 18 of the 24 averages). In the event that less than 75% of the 8-hour averages are available, a day shall also be counted as a valid day if the daily maximum 8-hour average concentration for that day is greater than the level of the ambient standard.

2.2 *Primary and Secondary Standard-related Summary Statistic.* The standard-related summary statistic is the annual fourth-highest daily maximum 8-hour ozone concentration, expressed in parts per million, averaged over three years. The 3-year average shall be computed using the three most recent, consecutive calendar years of monitoring data meeting the data completeness requirements described in this appendix. The computed 3-year average of the annual fourth-highest daily maximum 8-hour average ozone concentrations shall be expressed to three decimal places (the remaining digits to the right are truncated.)

2.3 *Comparisons with the Primary and Secondary Ozone Standards.* (a) The primary and secondary ozone ambient air quality standards are met at an ambient air quality monitoring site when the 3-year average of the annual fourth-highest daily maximum 8-hour average ozone concentration is less than or equal to 0.08 ppm. The number of significant figures in the level of the standard dictates the rounding convention for comparing the computed 3-year average annual fourth-highest daily maximum 8-hour average ozone concentration with the level of the standard. The third decimal place of the computed value is rounded, with values equal to or greater than 5 rounding up. Thus, a computed 3-year average ozone concentration of 0.085 ppm is the smallest value that is greater than 0.08 ppm.

(b) This comparison shall be based on three consecutive, complete calendar years of air quality monitoring data. This requirement is met for the three year period at a monitoring site if daily maximum 8-hour average concentrations are available for at least 90%, on average, of the days during the designated ozone monitoring season, with a minimum data completeness in any one year of at least 75% of the designated sampling days. When

computing whether the minimum data completeness requirements have been met, meteorological or ambient data may be sufficient to demonstrate that meteorological conditions on missing days were not conducive to concentrations above the level of the standard. Missing days assumed less than the level of the standard are counted for the purpose of meeting the data completeness requirement, subject to the approval of the appropriate Regional Administrator.

(c) Years with concentrations greater than the level of the standard shall not be ignored on the ground that they have less than complete data. Thus, in computing the 3-year average fourth maximum concentration, calendar years with less than 75% data completeness shall be included in the computa-

tion if the average annual fourth maximum 8-hour concentration is greater than the level of the standard.

(d) Comparisons with the primary and secondary ozone standards are demonstrated by examples 1 and 2 in paragraphs (d)(1) and (d)(2) respectively as follows:

(1) As shown in example 1, the primary and secondary standards are met at this monitoring site because the 3-year average of the annual fourth-highest daily maximum 8-hour average ozone concentrations (i.e., 0.084 ppm) is less than or equal to 0.08 ppm. The data completeness requirement is also met because the average percent of days with valid ambient monitoring data is greater than 90%, and no single year has less than 75% data completeness.

EXAMPLE 1. AMBIENT MONITORING SITE ATTAINING THE PRIMARY AND SECONDARY OZONE STANDARDS

Year	Percent Valid Days	1st Highest Daily Max 8-hour Conc. (ppm)	2nd Highest Daily Max 8-hour Conc. (ppm)	3rd Highest Daily Max 8-hour Conc. (ppm)	4th Highest Daily Max 8-hour Conc. (ppm)	5th Highest Daily Max 8-hour Conc. (ppm)
1993	100%	0.092	0.091	0.090	0.088	0.085
1994	96%	0.090	0.089	0.086	0.084	0.080
1995	98%	0.087	0.085	0.083	0.080	0.075
Average	98%					

(2) As shown in example 2, the primary and secondary standards are not met at this monitoring site because the 3-year average of the fourth-highest daily maximum 8-hour average ozone concentrations (i.e., 0.093 ppm) is greater than 0.08 ppm. Note that the ozone

concentration data for 1994 is used in these computations, even though the data capture is less than 75%, because the average fourth-highest daily maximum 8-hour average concentration is greater than 0.08 ppm.

EXAMPLE 2. AMBIENT MONITORING SITE FAILING TO MEET THE PRIMARY AND SECONDARY OZONE STANDARDS

Year	Percent Valid Days	1st Highest Daily Max 8-hour Conc. (ppm)	2nd Highest Daily Max 8-hour Conc. (ppm)	3rd Highest Daily Max 8-hour Conc. (ppm)	4th Highest Daily Max 8-hour Conc. (ppm)	5th Highest Daily Max 8-hour Conc. (ppm)
1993	96%	0.105	0.103	0.103	0.102	0.102
1994	74%	0.090	0.085	0.082	0.080	0.078
1995	98%	0.103	0.101	0.101	0.097	0.095
Average	89%					

3. *Design Values for Primary and Secondary Ambient Air Quality Standards for Ozone.* The air quality design value at a monitoring site is defined as that concentration that when reduced to the level of the standard ensures that the site meets the standard. For a concentration-based standard, the air quality design value is simply the standard-related test statistic. Thus, for the primary and secondary ozone standards, the 3-year average annual fourth-highest daily maximum 8-hour

average ozone concentration is also the air quality design value for the site.

[62 FR 38895, July 18, 1997]

APPENDIX J TO PART 50—REFERENCE METHOD FOR THE DETERMINATION OF PARTICULATE MATTER AS PM₁₀ IN THE ATMOSPHERE

1.0 *Applicability.*

1.1 This method provides for the measurement of the mass concentration of particulate matter with an aerodynamic diameter less than or equal to a nominal 10 micrometers (PM₁₀) in ambient air over a 24-hour period for purposes of determining attainment and maintenance of the primary and secondary national ambient air quality standards for particulate matter specified in §50.6 of this chapter. The measurement process is nondestructive, and the PM₁₀ sample can be subjected to subsequent physical or chemical analyses. Quality assurance procedures and guidance are provided in part 58, appendices A and B, of this chapter and in References 1 and 2.

2.0 Principle.

2.1 An air sampler draws ambient air at a constant flow rate into a specially shaped inlet where the suspended particulate matter is inertially separated into one or more size fractions within the PM₁₀ size range. Each size fraction in the PM₁₀ size range is then collected on a separate filter over the specified sampling period. The particle size discrimination characteristics (sampling effectiveness and 50 percent cutpoint) of the sampler inlet are prescribed as performance specifications in part 53 of this chapter.

2.2 Each filter is weighed (after moisture equilibration) before and after use to determine the net weight (mass) gain due to collected PM₁₀. The total volume of air sampled, corrected to EPA reference conditions (25 C, 101.3 kPa), is determined from the measured flow rate and the sampling time. The mass concentration of PM₁₀ in the ambient air is computed as the total mass of collected particles in the PM₁₀ size range divided by the volume of air sampled, and is expressed in micrograms per standard cubic meter (µg/std m³). For PM₁₀ samples collected at temperatures and pressures significantly different from EPA reference conditions, these corrected concentrations sometimes differ substantially from actual concentrations (in micrograms per actual cubic meter), particularly at high elevations. Although not required, the actual PM₁₀ concentration can be calculated from the corrected concentration, using the average ambient temperature and barometric pressure during the sampling period.

2.3 A method based on this principle will be considered a reference method only if (a) the associated sampler meets the requirements specified in this appendix and the requirements in part 53 of this chapter, and (b) the method has been designated as a reference method in accordance with part 53 of this chapter.

3.0 Range.

3.1 The lower limit of the mass concentration range is determined by the repeatability of filter tare weights, assuming the nominal air sample volume for the sampler. For samplers having an automatic filter-changing

mechanism, there may be no upper limit. For samplers that do not have an automatic filter-changing mechanism, the upper limit is determined by the filter mass loading beyond which the sampler no longer maintains the operating flow rate within specified limits due to increased pressure drop across the loaded filter. This upper limit cannot be specified precisely because it is a complex function of the ambient particle size distribution and type, humidity, filter type, and perhaps other factors. Nevertheless, all samplers should be capable of measuring 24-hour PM₁₀ mass concentrations of at least 300 µg/std m³ while maintaining the operating flow rate within the specified limits.

4.0 Precision.

4.1 The precision of PM₁₀ samplers must be 5 µg/m³ for PM₁₀ concentrations below 80 µg/m³ and 7 percent for PM₁₀ concentrations above 80 µg/m³, as required by part 53 of this chapter, which prescribes a test procedure that determines the variation in the PM₁₀ concentration measurements of identical samplers under typical sampling conditions. Continual assessment of precision via collocated samplers is required by part 58 of this chapter for PM₁₀ samplers used in certain monitoring networks.

5.0 Accuracy.

5.1 Because the size of the particles making up ambient particulate matter varies over a wide range and the concentration of particles varies with particle size, it is difficult to define the absolute accuracy of PM₁₀ samplers. Part 53 of this chapter provides a specification for the sampling effectiveness of PM₁₀ samplers. This specification requires that the expected mass concentration calculated for a candidate PM₁₀ sampler, when sampling a specified particle size distribution, be within ±10 percent of that calculated for an ideal sampler whose sampling effectiveness is explicitly specified. Also, the particle size for 50 percent sampling effectiveness is required to be 10±0.5 micrometers. Other specifications related to accuracy apply to flow measurement and calibration, filter media, analytical (weighing) procedures, and artifact. The flow rate accuracy of PM₁₀ samplers used in certain monitoring networks is required by part 58 of this chapter to be assessed periodically via flow rate audits.

6.0 Potential Sources of Error.

6.1 *Volatile Particles.* Volatile particles collected on filters are often lost during shipment and/or storage of the filters prior to the post-sampling weighing³. Although shipment or storage of loaded filters is sometimes unavoidable, filters should be reweighed as soon as practical to minimize these losses.

6.2 *Artifacts.* Positive errors in PM₁₀ concentration measurements may result from retention of gaseous species on filters^{4,5}. Such errors include the retention of sulfur

dioxide and nitric acid. Retention of sulfur dioxide on filters, followed by oxidation to sulfate, is referred to as artifact sulfate formation, a phenomenon which increases with increasing filter alkalinity⁶. Little or no artifact sulfate formation should occur using filters that meet the alkalinity specification in section 7.2.4. Artifact nitrate formation, resulting primarily from retention of nitric acid, occurs to varying degrees on many filter types, including glass fiber, cellulose ester, and many quartz fiber filters^{5, 7, 8, 9, 10}. Loss of true atmospheric particulate nitrate during or following sampling may also occur due to dissociation or chemical reaction. This phenomenon has been observed on Teflon[®] filters⁸ and inferred for quartz fiber filters^{11, 12}. The magnitude of nitrate artifact errors in PM₁₀ mass concentration measurements will vary with location and ambient temperature; however, for most sampling locations, these errors are expected to be small.

6.3 *Humidity*. The effects of ambient humidity on the sample are unavoidable. The filter equilibration procedure in section 9.0 is designed to minimize the effects of moisture on the filter medium.

6.4 *Filter Handling*. Careful handling of filters between presampling and postsampling weighings is necessary to avoid errors due to damaged filters or loss of collected particles from the filters. Use of a filter cartridge or cassette may reduce the magnitude of these errors. Filters must also meet the integrity specification in section 7.2.3.

6.5 *Flow Rate Variation*. Variations in the sampler's operating flow rate may alter the particle size discrimination characteristics of the sampler inlet. The magnitude of this error will depend on the sensitivity of the inlet to variations in flow rate and on the particle distribution in the atmosphere during the sampling period. The use of a flow control device (section 7.1.3) is required to minimize this error.

6.6 *Air Volume Determination*. Errors in the air volume determination may result from errors in the flow rate and/or sampling time measurements. The flow control device serves to minimize errors in the flow rate determination, and an elapsed time meter (section 7.1.5) is required to minimize the error in the sampling time measurement.

7.0 Apparatus.

7.1 PM₁₀ Sampler.

7.1.1 The sampler shall be designed to:

- a. Draw the air sample into the sampler inlet and through the particle collection filter at a uniform face velocity.
- b. Hold and seal the filter in a horizontal position so that sample air is drawn downward through the filter.
- c. Allow the filter to be installed and removed conveniently.

d. Protect the filter and sampler from precipitation and prevent insects and other debris from being sampled.

e. Minimize air leaks that would cause error in the measurement of the air volume passing through the filter.

f. Discharge exhaust air at a sufficient distance from the sampler inlet to minimize the sampling of exhaust air.

g. Minimize the collection of dust from the supporting surface.

7.1.2 The sampler shall have a sample air inlet system that, when operated within a specified flow rate range, provides particle size discrimination characteristics meeting all of the applicable performance specifications prescribed in part 53 of this chapter. The sampler inlet shall show no significant wind direction dependence. The latter requirement can generally be satisfied by an inlet shape that is circularly symmetrical about a vertical axis.

7.1.3 The sampler shall have a flow control device capable of maintaining the sampler's operating flow rate within the flow rate limits specified for the sampler inlet over normal variations in line voltage and filter pressure drop.

7.1.4 The sampler shall provide a means to measure the total flow rate during the sampling period. A continuous flow recorder is recommended but not required. The flow measurement device shall be accurate to ± 2 percent.

7.1.5 A timing/control device capable of starting and stopping the sampler shall be used to obtain a sample collection period of 24 ± 1 hr ($1,440 \pm 60$ min). An elapsed time meter, accurate to within ± 15 minutes, shall be used to measure sampling time. This meter is optional for samplers with continuous flow recorders if the sampling time measurement obtained by means of the recorder meets the ± 15 minute accuracy specification.

7.1.6 The sampler shall have an associated operation or instruction manual as required by part 53 of this chapter which includes detailed instructions on the calibration, operation, and maintenance of the sampler.

7.2 Filters.

7.2.1 *Filter Medium*. No commercially available filter medium is ideal in all respects for all samplers. The user's goals in sampling determine the relative importance of various filter characteristics (e.g., cost, ease of handling, physical and chemical characteristics, etc.) and, consequently, determine the choice among acceptable filters. Furthermore, certain types of filters may not be suitable for use with some samplers, particularly under heavy loading conditions (high mass concentrations), because of high or rapid increase in the filter flow resistance that would exceed the capability of the sampler's flow control device. However, samplers equipped with automatic filter-changing

mechanisms may allow use of these types of filters. The specifications given below are minimum requirements to ensure acceptability of the filter medium for measurement of PM₁₀ mass concentrations. Other filter evaluation criteria should be considered to meet individual sampling and analysis objectives.

7.2.2 *Collection Efficiency.* ≥ 99 percent, as measured by the DOP test (ASTM-2986) with 0.3 μm particles at the sampler's operating face velocity.

7.2.3 *Integrity.* $\pm 5 \mu\text{g}/\text{m}^3$ (assuming sampler's nominal 24-hour air sample volume). Integrity is measured as the PM₁₀ concentration equivalent corresponding to the average difference between the initial and the final weights of a random sample of test filters that are weighed and handled under actual or simulated sampling conditions, but have no air sample passed through them (i.e., filter blanks). As a minimum, the test procedure must include initial equilibration and weighing, installation on an inoperative sampler, removal from the sampler, and final equilibration and weighing.

7.2.4 *Alkalinity.* < 25 microequivalents/gram of filter, as measured by the procedure given in Reference 13 following at least two months storage in a clean environment (free from contamination by acidic gases) at room temperature and humidity.

7.3 *Flow Rate Transfer Standard.* The flow rate transfer standard must be suitable for the sampler's operating flow rate and must be calibrated against a primary flow or volume standard that is traceable to the National Bureau of Standards (NBS). The flow rate transfer standard must be capable of measuring the sampler's operating flow rate with an accuracy of ± 2 percent.

7.4 *Filter Conditioning Environment.*

7.4.1 Temperature range: 15 to 30 C.

7.4.2 Temperature control: ± 3 C.

7.4.3 Humidity range: 20% to 45% RH.

7.4.4 Humidity control: $\pm 5\%$ RH.

7.5 *Analytical Balance.* The analytical balance must be suitable for weighing the type and size of filters required by the sampler. The range and sensitivity required will depend on the filter tare weights and mass loadings. Typically, an analytical balance with a sensitivity of 0.1 mg is required for high volume samplers (flow rates $> 0.5 \text{ m}^3/\text{min}$). Lower volume samplers (flow rates $< 0.5 \text{ m}^3/\text{min}$) will require a more sensitive balance.

8.0 *Calibration.*

8.1 *General Requirements.*

8.1.1 Calibration of the sampler's flow measurement device is required to establish traceability of subsequent flow measurements to a primary standard. A flow rate transfer standard calibrated against a primary flow or volume standard shall be used to calibrate or verify the accuracy of the sampler's flow measurement device.

8.1.2 Particle size discrimination by inertial separation requires that specific air velocities be maintained in the sampler's air inlet system. Therefore, the flow rate through the sampler's inlet must be maintained throughout the sampling period within the design flow rate range specified by the manufacturer. Design flow rates are specified as actual volumetric flow rates, measured at existing conditions of temperature and pressure (Q_a). In contrast, mass concentrations of PM₁₀ are computed using flow rates corrected to EPA reference conditions of temperature and pressure (Q_{std}).

8.2 *Flow Rate Calibration Procedure.*

8.2.1 PM₁₀ samplers employ various types of flow control and flow measurement devices. The specific procedure used for flow rate calibration or verification will vary depending on the type of flow controller and flow indicator employed. Calibration in terms of actual volumetric flow rates (Q_a) is generally recommended, but other measures of flow rate (e.g., Q_{std}) may be used provided the requirements of section 8.1 are met. The general procedure given here is based on actual volumetric flow units (Q_a) and serves to illustrate the steps involved in the calibration of a PM₁₀ sampler. Consult the sampler manufacturer's instruction manual and Reference 2 for specific guidance on calibration. Reference 14 provides additional information on the use of the commonly used measures of flow rate and their interrelationships.

8.2.2 Calibrate the flow rate transfer standard against a primary flow or volume standard traceable to NBS. Establish a calibration relationship (e.g., an equation or family of curves) such that traceability to the primary standard is accurate to within 2 percent over the expected range of ambient conditions (i.e., temperatures and pressures) under which the transfer standard will be used. Recalibrate the transfer standard periodically.

8.2.3 Following the sampler manufacturer's instruction manual, remove the sampler inlet and connect the flow rate transfer standard to the sampler such that the transfer standard accurately measures the sampler's flow rate. Make sure there are no leaks between the transfer standard and the sampler.

8.2.4 Choose a minimum of three flow rates (actual m^3/min), spaced over the acceptable flow rate range specified for the inlet (see 7.1.2) that can be obtained by suitable adjustment of the sampler flow rate. In accordance with the sampler manufacturer's instruction manual, obtain or verify the calibration relationship between the flow rate (actual m^3/min) as indicated by the transfer standard and the sampler's flow indicator response. Record the ambient temperature and barometric pressure. Temperature and pressure corrections to subsequent flow indicator readings may be required for certain types of

flow measurement devices. When such corrections are necessary, correction on an individual or daily basis is preferable. However, seasonal average temperature and average barometric pressure for the sampling site may be incorporated into the sampler calibration to avoid daily corrections. Consult the sampler manufacturer's instruction manual and Reference 2 for additional guidance.

8.2.5 Following calibration, verify that the sampler is operating at its design flow rate (actual m³/min) with a clean filter in place.

8.2.6 Replace the sampler inlet.

9.0 Procedure.

9.1 The sampler shall be operated in accordance with the specific guidance provided in the sampler manufacturer's instruction manual and in Reference 2. The general procedure given here assumes that the sampler's flow rate calibration is based on flow rates at ambient conditions (\bar{Q}_a) and serves to illustrate the steps involved in the operation of a PM₁₀ sampler.

9.2 Inspect each filter for pinholes, particles, and other imperfections. Establish a filter information record and assign an identification number to each filter.

9.3 Equilibrate each filter in the conditioning environment (see 7.4) for at least 24 hours.

9.4 Following equilibration, weigh each filter and record the presampling weight with the filter identification number.

9.5 Install a preweighed filter in the sampler following the instructions provided in the sampler manufacturer's instruction manual.

9.6 Turn on the sampler and allow it to establish run-temperature conditions. Record the flow indicator reading and, if needed, the ambient temperature and barometric pressure. Determine the sampler flow rate (actual m³/min) in accordance with the instructions provided in the sampler manufacturer's instruction manual. NOTE.—No onsite temperature or pressure measurements are necessary if the sampler's flow indicator does not require temperature or pressure corrections or if seasonal average temperature and average barometric pressure for the sampling site are incorporated into the sampler calibration (see step 8.2.4). If individual or daily temperature and pressure corrections are required, ambient temperature and barometric pressure can be obtained by on-site measurements or from a nearby weather station. Barometric pressure readings obtained from airports must be station pressure, not corrected to sea level, and may need to be corrected for differences in elevation between the sampling site and the airport.

9.7 If the flow rate is outside the acceptable range specified by the manufacturer, check for leaks, and if necessary, adjust the flow rate to the specified setpoint. Stop the sampler.

9.8 Set the timer to start and stop the sampler at appropriate times. Set the elapsed time meter to zero or record the initial meter reading.

9.9 Record the sample information (site location or identification number, sample date, filter identification number, and sampler model and serial number).

9.10 Sample for 24±1 hours.

9.11 Determine and record the average flow rate (\bar{Q}_a) in actual m³/min for the sampling period in accordance with the instructions provided in the sampler manufacturer's instruction manual. Record the elapsed time meter final reading and, if needed, the average ambient temperature and barometric pressure for the sampling period (see note following step 9.6).

9.12 Carefully remove the filter from the sampler, following the sampler manufacturer's instruction manual. Touch only the outer edges of the filter.

9.13 Place the filter in a protective holder or container (e.g., petri dish, glassine envelope, or manila folder).

9.14 Record any factors such as meteorological conditions, construction activity, fires or dust storms, etc., that might be pertinent to the measurement on the filter information record.

9.15 Transport the exposed sample filter to the filter conditioning environment as soon as possible for equilibration and subsequent weighing.

9.16 Equilibrate the exposed filter in the conditioning environment for at least 24 hours under the same temperature and humidity conditions used for presampling filter equilibration (see 9.3).

9.17 Immediately after equilibration, reweigh the filter and record the postsampling weight with the filter identification number.

10.0 Sampler Maintenance.

10.1 The PM₁₀ sampler shall be maintained in strict accordance with the maintenance procedures specified in the sampler manufacturer's instruction manual.

11.0 Calculations.

11.1 Calculate the average flow rate over the sampling period corrected to EPA reference conditions as \bar{Q}_{std} . When the sampler's flow indicator is calibrated in actual volumetric units (\bar{Q}_a), \bar{Q}_{std} is calculated as:

$$\bar{Q}_{std} = \bar{Q}_a \times (P_{av}/T_{av})(T_{std}/P_{std})$$

where

\bar{Q}_{std} = average flow rate at EPA reference conditions, std m³/min;

\bar{Q}_a = average flow rate at ambient conditions, m³/min;

P_{av} = average barometric pressure during the sampling period or average barometric pressure for the sampling site, kPa (or mm Hg);

T_{av} = average ambient temperature during the sampling period or seasonal average

ambient temperature for the sampling site, K;

T_{std} = standard temperature, defined as 298 K;
 P_{std} = standard pressure, defined as 101.3 kPa (or 760 mm Hg).

11.2 Calculate the total volume of air sampled as:

$$V_{std} = \bar{Q}_{std} \times t$$

where

V_{std} = total air sampled in standard volume units, std m³;
 t = sampling time, min.

11.3 Calculate the PM₁₀ concentration as:

$$PM_{10} = (W_f - W_i) \times 10^6 / V_{std}$$

where

PM_{10} = mass concentration of PM₁₀, µg/std m³;

W_f, W_i = final and initial weights of filter collecting PM₁₀ particles, g;

10^6 = conversion of g to µg.

NOTE: If more than one size fraction in the PM₁₀ size range is collected by the sampler, the sum of the net weight gain by each collection filter [$\Sigma(W_f - W_i)$] is used to calculate the PM₁₀ mass concentration.

12.0 References.

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APPENDIX K TO PART 50—INTERPRETATION OF THE NATIONAL AMBIENT AIR QUALITY STANDARDS FOR PARTICULATE MATTER

1.0 General.

- (a) This appendix explains the computations necessary for analyzing particulate matter data to determine attainment of the 24-hour and annual standards specified in 40 CFR 50.6. For the primary and secondary standards, particulate matter is measured in the ambient air as PM₁₀ (particles with an aerodynamic diameter less than or equal to a nominal 10 micrometers) by a reference method based on appendix J of this part and designated in accordance with part 53 of this chapter, or by an equivalent method designated in accordance with part 53 of this chapter. The required frequency of measurements is specified in part 58 of this chapter.
- (b) The terms used in this appendix are defined as follows:

Average refers to an arithmetic mean. All particulate matter standards are expressed in terms of expected annual values: Expected number of exceedances per year for the 24-hour standards and expected annual arithmetic mean for the annual standards.

Daily value for PM₁₀ refers to the 24-hour average concentration of PM₁₀ calculated or measured from midnight to midnight (local time).

Exceedance means a daily value that is above the level of the 24-hour standard after

rounding to the nearest 10 $\mu\text{g}/\text{m}^3$ (i.e., values ending in 5 or greater are to be rounded up).

Expected annual value is the number approached when the annual values from an increasing number of years are averaged, in the absence of long-term trends in emissions or meteorological conditions.

Year refers to a calendar year.

(c) Although the discussion in this appendix focuses on monitored data, the same principles apply to modeling data, subject to EPA modeling guidelines.

2.0 Attainment Determinations.

2.1 24-Hour Primary and Secondary Standards.

(a) Under 40 CFR 50.6(a) the 24-hour primary and secondary standards are attained when the expected number of exceedances per year at each monitoring site is less than or equal to one. In the simplest case, the number of expected exceedances at a site is determined by recording the number of exceedances in each calendar year and then averaging them over the past 3 calendar years. Situations in which 3 years of data are not available and possible adjustments for unusual events or trends are discussed in sections 2.3 and 2.4 of this appendix. Further, when data for a year are incomplete, it is necessary to compute an estimated number of exceedances for that year by adjusting the observed number of exceedances. This procedure, performed by calendar quarter, is described in section 3.0 of this appendix. The expected number of exceedances is then estimated by averaging the individual annual estimates for the past 3 years.

(b) The comparison with the allowable expected exceedance rate of one per year is made in terms of a number rounded to the nearest tenth (fractional values equal to or greater than 0.05 are to be rounded up; e.g., an exceedance rate of 1.05 would be rounded to 1.1, which is the lowest rate for nonattainment).

2.2 *Annual Primary and Secondary Standards.* Under 40 CFR 50.6(b), the annual primary and secondary standards are attained when the expected annual arithmetic mean PM_{10} concentration is less than or equal to the level of the standard. In the simplest case, the expected annual arithmetic mean is determined by averaging the annual arithmetic mean PM_{10} concentrations for the past 3 calendar years. Because of the potential for incomplete data and the possible seasonality in PM_{10} concentrations, the annual mean shall be calculated by averaging the four quarterly means of PM_{10} concentrations within the calendar year. The equations for calculating the annual arithmetic mean are given in section 4.0 of this appendix. Situations in which 3 years of data are not available and possible adjustments for unusual events or trends are discussed in sections 2.3 and 2.4 of this appendix. The expected annual arithmetic mean is rounded to the nearest 1

$\mu\text{g}/\text{m}^3$ before comparison with the annual standards (fractional values equal to or greater than 0.5 are to be rounded up).

2.3 Data Requirements.

(a) 40 CFR 58.13 specifies the required minimum frequency of sampling for PM_{10} . For the purposes of making comparisons with the particulate matter standards, all data produced by National Air Monitoring Stations (NAMS), State and Local Air Monitoring Stations (SLAMS) and other sites submitted to EPA in accordance with the part 58 requirements must be used, and a minimum of 75 percent of the scheduled PM_{10} samples per quarter are required.

(b) To demonstrate attainment of either the annual or 24-hour standards at a monitoring site, the monitor must provide sufficient data to perform the required calculations of sections 3.0 and 4.0 of this appendix. The amount of data required varies with the sampling frequency, data capture rate and the number of years of record. In all cases, 3 years of representative monitoring data that meet the 75 percent criterion of the previous paragraph should be utilized, if available, and would suffice. More than 3 years may be considered, if all additional representative years of data meeting the 75 percent criterion are utilized. Data not meeting these criteria may also suffice to show attainment; however, such exceptions will have to be approved by the appropriate Regional Administrator in accordance with EPA guidance.

(c) There are less stringent data requirements for showing that a monitor has failed an attainment test and thus has recorded a violation of the particulate matter standards. Although it is generally necessary to meet the minimum 75 percent data capture requirement per quarter to use the computational equations described in sections 3.0 and 4.0 of this appendix, this criterion does not apply when less data is sufficient to unambiguously establish nonattainment. The following examples illustrate how nonattainment can be demonstrated when a site fails to meet the completeness criteria. Nonattainment of the 24-hour primary standards can be established by the observed annual number of exceedances (e.g., four observed exceedances in a single year), or by the estimated number of exceedances derived from the observed number of exceedances and the required number of scheduled samples (e.g., two observed exceedances with every other day sampling). Nonattainment of the annual standards can be demonstrated on the basis of quarterly mean concentrations developed from observed data combined with one-half the minimum detectable concentration substituted for missing values. In both cases, expected annual values must exceed the levels allowed by the standards.

2.4 Adjustment for Exceptional Events and Trends.

(a) An exceptional event is an uncontrollable event caused by natural sources of particulate matter or an event that is not expected to recur at a given location. Inclusion of such a value in the computation of exceedances or averages could result in inappropriate estimates of their respective expected annual values. To reduce the effect of unusual events, more than 3 years of representative data may be used. Alternatively, other techniques, such as the use of statistical models or the use of historical data could be considered so that the event may be discounted or weighted according to the likelihood that it will recur. The use of such techniques is subject to the approval of the appropriate Regional Administrator in accordance with EPA guidance.

(b) In cases where long-term trends in emissions and air quality are evident, mathematical techniques should be applied to account for the trends to ensure that the expected annual values are not inappropriately biased by unrepresentative data. In the simplest case, if 3 years of data are available under stable emission conditions, this data should be used. In the event of a trend or shift in emission patterns, either the most recent representative year(s) could be used or statistical techniques or models could be used in conjunction with previous years of data to adjust for trends. The use of less than 3 years of data, and any adjustments are subject to the approval of the appropriate Regional Administrator in accordance with EPA guidance.

3.0 Computational Equations for the 24-hour Standards.

3.1 Estimating Exceedances for a Year.

(a) If PM₁₀ sampling is scheduled less frequently than every day, or if some scheduled samples are missed, a PM₁₀ value will not be available for each day of the year. To account for the possible effect of incomplete data, an adjustment must be made to the data collected at each monitoring location to estimate the number of exceedances in a calendar year. In this adjustment, the assumption is made that the fraction of missing values that would have exceeded the standard level is identical to the fraction of measured values above this level. This computation is to be made for all sites that are scheduled to monitor throughout the entire year and meet the minimum data requirements of section 2.3 of this appendix. Because of possible seasonal imbalance, this adjustment shall be applied on a quarterly basis. The estimate of the expected number of exceedances for the quarter is equal to the observed number of exceedances plus an increment associated with the missing data. The following equation must be used for these computations:

Equation 1

$$e_q = v_q + \left[\left(v_q / n_q \right) \times \left(N_q - n_q \right) \right] = v_q \times N_q / n_q$$

where:

e_q = the estimated number of exceedances for calendar quarter q ;

v_q = the observed number of exceedances for calendar quarter q ;

N_q = the number of days in calendar quarter q ;

n_q = the number of days in calendar quarter q with PM₁₀ data; and

q = the index for calendar quarter, $q=1, 2, 3$ or 4.

(b) The estimated number of exceedances for a calendar quarter must be rounded to the nearest hundredth (fractional values equal to or greater than 0.005 must be rounded up).

(c) The estimated number of exceedances for the year, e , is the sum of the estimates for each calendar quarter.

Equation 2

$$e = \sum_{q=1}^4 e_q$$

(d) The estimated number of exceedances for a single year must be rounded to one decimal place (fractional values equal to or greater than 0.05 are to be rounded up). The expected number of exceedances is then estimated by averaging the individual annual estimates for the most recent 3 or more representative years of data. The expected number of exceedances must be rounded to one decimal place (fractional values equal to or greater than 0.05 are to be rounded up).

(e) The adjustment for incomplete data will not be necessary for monitoring or modeling data which constitutes a complete record, i.e., 365 days per year.

(f) To reduce the potential for overestimating the number of expected exceedances, the correction for missing data will not be required for a calendar quarter in which the first observed exceedance has occurred if:

(1) There was only one exceedance in the calendar quarter;

(2) Everyday sampling is subsequently initiated and maintained for 4 calendar quarters in accordance with 40 CFR 58.13; and

(3) Data capture of 75 percent is achieved during the required period of everyday sampling. In addition, if the first exceedance is observed in a calendar quarter in which the monitor is already sampling every day, no adjustment for missing data will be made to the first exceedance if a 75 percent data capture rate was achieved in the quarter in which it was observed.

Example 1

a. During a particular calendar quarter, 39 out of a possible 92 samples were recorded, with one observed exceedance of the 24-hour standard. Using Equation 1, the estimated number of exceedances for the quarter is:

$$e_q = 1 \times 92 / 39 = 2.359 \text{ or } 2.36.$$

b. If the estimated exceedances for the other 3 calendar quarters in the year were 2.30, 0.0 and 0.0, then, using Equation 2, the estimated number of exceedances for the year is $2.36 = 2.30 + 0.0 = 2.30$ which equals 4.66 or 4.7. If no exceedances were observed for the 2 previous years, then the expected number of exceedances is estimated by: $(1/3) \times (4.7 + 0 + 0) = 1.57$ or 1.6. Since 1.6 exceeds the allowable number of expected exceedances, this monitoring site would fail the attainment test.

Example 2

In this example, everyday sampling was initiated following the first observed exceedance as required by 40 CFR 58.13. Accordingly, the first observed exceedance would not be adjusted for incomplete sampling. During the next three quarters, 1.2 exceedances were estimated. In this case, the estimated exceedances for the year would be $1.0 = 1.2 + 0.0 = 1.2$ which equals 2.2. If, as before, no exceedances were observed for the two previous years, then the estimated exceedances for the 3-year period would then be $(1/3) \times (2.2 + 0.0 + 0.0) = 0.7$, and the monitoring site would *not* fail the attainment test.

3.2 Adjustments for Non-Scheduled Sampling Days.

(a) If a systematic sampling schedule is used and sampling is performed on days in addition to the days specified by the systematic sampling schedule, e.g., during episodes of high pollution, then an adjustment must be made in the equation for the estimation of exceedances. Such an adjustment is needed to eliminate the bias in the estimate of the quarterly and annual number of exceedances that would occur if the chance of an exceedance is different for scheduled than for non-scheduled days, as would be the case with episode sampling.

(b) The required adjustment treats the systematic sampling schedule as a stratified sampling plan. If the period from one scheduled sample until the day preceding the next scheduled sample is defined as a sampling stratum, then there is one stratum for each scheduled sampling day. An average number of observed exceedances is computed for each of these sampling strata. With nonscheduled sampling days, the estimated number of exceedances is defined as:

Equation 3

$$e_q = (N_q / m_q) \times \sum_{j=1}^{m_q} (v_j / k_j)$$

where:

e_q = the estimated number of exceedances for the quarter;

N_q = the number of days in the quarter;

m_q = the number of strata with samples during the quarter;

v_j = the number of observed exceedances in stratum j ; and

k_j = the number of actual samples in stratum j .

(c) Note that if only one sample value is recorded in each stratum, then Equation 3 reduces to Equation 1.

Example 3

A monitoring site samples according to a systematic sampling schedule of one sample every 6 days, for a total of 15 scheduled samples in a quarter out of a total of 92 possible samples. During one 6-day period, potential episode levels of PM_{10} were suspected, so 5 additional samples were taken. One of the regular scheduled samples was missed, so a total of 19 samples in 14 sampling strata were measured. The one 6-day sampling stratum with 6 samples recorded 2 exceedances. The remainder of the quarter with one sample per stratum recorded zero exceedances. Using Equation 3, the estimated number of exceedances for the quarter is:

$$e_q = (92/14) \times (2/6 + 0 + \dots + 0) = 2.19.$$

4.0 Computational Equations for Annual Standards.

4.1 Calculation of the Annual Arithmetic Mean. (a) An annual arithmetic mean value for PM_{10} is determined by averaging the quarterly means for the 4 calendar quarters of the year. The following equation is to be used for calculation of the mean for a calendar quarter:

Equation 4

$$\bar{x}_q = (1/n_q) \times \sum_{i=1}^{n_q} x_i$$

where:

\bar{x}_q = the quarterly mean concentration for quarter q , $q=1, 2, 3, \text{ or } 4$,

n_q = the number of samples in the quarter, and

x_i = the i th concentration value recorded in the quarter.

(b) The quarterly mean, expressed in $\mu\text{g}/\text{m}^3$, must be rounded to the nearest tenth (fractional values of 0.05 should be rounded up).

(c) The annual mean is calculated by using the following equation:

Equation 5

$$\bar{x} = \left(\frac{1}{4}\right) \times \sum_{q=1}^4 \bar{x}_q$$

where:

\bar{x} = the annual mean; and
 \bar{x}_q = the mean for calendar quarter q.

(d) The average of quarterly means must be rounded to the nearest tenth (fractional values of 0.05 should be rounded up).

(e) The use of quarterly averages to compute the annual average will not be necessary for monitoring or modeling data which results in a complete record, i.e., 365 days per year.

(f) The expected annual mean is estimated as the average of three or more annual means. This multi-year estimate, expressed in $\mu\text{g}/\text{m}^3$, shall be rounded to the nearest integer for comparison with the annual standard (fractional values of 0.5 should be rounded up).

Example 4

Using Equation 4, the quarterly means are calculated for each calendar quarter. If the quarterly means are 52.4, 75.3, 82.1, and 63.2 $\mu\text{g}/\text{m}^3$, then the annual mean is:

$$\bar{x} = (1/4) \times (52.4 + 75.3 + 82.1 + 63.2) = 68.25 \text{ or } 68.3.$$

4.2 *Adjustments for Non-scheduled Sampling Days.* (a) An adjustment in the calculation of the annual mean is needed if sampling is performed on days in addition to the days specified by the systematic sampling schedule. For the same reasons given in the discussion of estimated exceedances, under section 3.2 of this appendix, the quarterly averages would be calculated by using the following equation:

Equation 6

$$\bar{x}_q = \left(\frac{1}{m_q}\right) \times \sum_{j=1}^{m_q} \sum_{i=1}^{k_j} (x_{ij}/k_j)$$

where:

\bar{x}_q = the quarterly mean concentration for quarter q, q=1, 2, 3, or 4;
 x_{ij} = the ith concentration value recorded in stratum j;
 k_j = the number of actual samples in stratum j; and
 m_q = the number of strata with data in the quarter.

(b) If one sample value is recorded in each stratum, Equation 6 reduces to a simple arithmetic average of the observed values as described by Equation 4.

Example 5

a. During one calendar quarter, 9 observations were recorded. These samples were distributed among 7 sampling strata, with 3 observations in one stratum. The concentrations of the 3 observations in the single stratum were 202, 242, and 180 $\mu\text{g}/\text{m}^3$. The remaining 6 observed concentrations were 55, 68, 73, 92, 120, and 155 $\mu\text{g}/\text{m}^3$. Applying the weighting factors specified in Equation 6, the quarterly mean is:

$$\bar{x}_q = (1/7) \times [(1/3) \times (202 + 242 + 180) + 155 + 68 + 73 + 92 + 120 + 155] = 110.1$$

b. Although 24-hour measurements are rounded to the nearest 10 $\mu\text{g}/\text{m}^3$ for determinations of exceedances of the 24-hour standard, note that these values are rounded to the nearest 1 $\mu\text{g}/\text{m}^3$ for the calculation of means.

[62 FR 38712, July 18, 1997]

APPENDIX L TO PART 50—REFERENCE METHOD FOR THE DETERMINATION OF FINE PARTICULATE MATTER AS $\text{PM}_{2.5}$ IN THE ATMOSPHERE

1.0 *Applicability.*

1.1 This method provides for the measurement of the mass concentration of fine particulate matter having an aerodynamic diameter less than or equal to a nominal 2.5 micrometers ($\text{PM}_{2.5}$) in ambient air over a 24-hour period for purposes of determining whether the primary and secondary national ambient air quality standards for fine particulate matter specified in §50.7 of this part are met. The measurement process is considered to be nondestructive, and the $\text{PM}_{2.5}$ sample obtained can be subjected to subsequent physical or chemical analyses. Quality assessment procedures are provided in part 58, appendix A of this chapter, and quality assurance guidance are provided in references 1, 2, and 3 in section 13.0 of this appendix.

1.2 This method will be considered a reference method for purposes of part 58 of this chapter only if:

(a) The associated sampler meets the requirements specified in this appendix and the applicable requirements in part 53 of this chapter, and

(b) The method and associated sampler have been designated as a reference method in accordance with part 53 of this chapter.

1.3 $\text{PM}_{2.5}$ samplers that meet nearly all specifications set forth in this method but have minor deviations and/or modifications of the reference method sampler will be designated as "Class I" equivalent methods for $\text{PM}_{2.5}$ in accordance with part 53 of this chapter.

2.0 *Principle.*

2.1 An electrically powered air sampler draws ambient air at a constant volumetric flow rate into a specially shaped inlet and

through an inertial particle size separator (impactor) where the suspended particulate matter in the PM_{2.5} size range is separated for collection on a polytetrafluoroethylene (PTFE) filter over the specified sampling period. The air sampler and other aspects of this reference method are specified either explicitly in this appendix or generally with reference to other applicable regulations or quality assurance guidance.

2.2 Each filter is weighed (after moisture and temperature conditioning) before and after sample collection to determine the net gain due to collected PM_{2.5}. The total volume of air sampled is determined by the sampler from the measured flow rate at actual ambient temperature and pressure and the sampling time. The mass concentration of PM_{2.5} in the ambient air is computed as the total mass of collected particles in the PM_{2.5} size range divided by the actual volume of air sampled, and is expressed in micrograms per cubic meter of air ($\mu\text{g}/\text{m}^3$).

3.0 *PM_{2.5} Measurement Range.*

3.1 *Lower concentration limit.* The lower detection limit of the mass concentration measurement range is estimated to be approximately 2 $\mu\text{g}/\text{m}^3$, based on noted mass changes in field blanks in conjunction with the 24 m³ nominal total air sample volume specified for the 24-hour sample.

3.2 *Upper concentration limit.* The upper limit of the mass concentration range is determined by the filter mass loading beyond which the sampler can no longer maintain the operating flow rate within specified limits due to increased pressure drop across the loaded filter. This upper limit cannot be specified precisely because it is a complex function of the ambient particle size distribution and type, humidity, the individual filter used, the capacity of the sampler flow rate control system, and perhaps other factors. Nevertheless, all samplers are estimated to be capable of measuring 24-hour PM_{2.5} mass concentrations of at least 200 $\mu\text{g}/\text{m}^3$ while maintaining the operating flow rate within the specified limits.

3.3 *Sample period.* The required sample period for PM_{2.5} concentration measurements by this method shall be 1,380 to 1500 minutes (23 to 25 hours). However, when a sample period is less than 1,380 minutes, the measured concentration (as determined by the collected PM_{2.5} mass divided by the actual sampled air volume), multiplied by the actual number of minutes in the sample period and divided by 1,440, may be used as if it were a valid concentration measurement for the specific purpose of determining a violation of the NAAQS. This value assumes that the PM_{2.5} concentration is zero for the remaining portion of the sample period and therefore represents the minimum concentration that could have been measured for the full 24-hour sample period. Accordingly, if the value thus calculated is high enough to be an exceed-

ance, such an exceedance would be a valid exceedance for the sample period. When reported to AIRS, this data value should receive a special code to identify it as not to be commingled with normal concentration measurements or used for other purposes.

4.0 *Accuracy.*

4.1 Because the size and volatility of the particles making up ambient particulate matter vary over a wide range and the mass concentration of particles varies with particle size, it is difficult to define the accuracy of PM_{2.5} measurements in an absolute sense. The accuracy of PM_{2.5} measurements is therefore defined in a relative sense, referenced to measurements provided by this reference method. Accordingly, accuracy shall be defined as the degree of agreement between a subject field PM_{2.5} sampler and a collocated PM_{2.5} reference method audit sampler operating simultaneously at the monitoring site location of the subject sampler and includes both random (precision) and systematic (bias) errors. The requirements for this field sampler audit procedure are set forth in part 58, appendix A of this chapter.

4.2 *Measurement system bias.* Results of collocated measurements where the duplicate sampler is a reference method sampler are used to assess a portion of the measurement system bias according to the schedule and procedure specified in part 58, appendix A of this chapter.

4.3 *Audits with reference method samplers to determine system accuracy and bias.* According to the schedule and procedure specified in part 58, appendix A of this chapter, a reference method sampler is required to be located at each of selected PM_{2.5} SLAMS sites as a duplicate sampler. The results from the primary sampler and the duplicate reference method sampler are used to calculate accuracy of the primary sampler on a quarterly basis, bias of the primary sampler on an annual basis, and bias of a single reporting organization on an annual basis. Reference 2 in section 13.0 of this appendix provides additional information and guidance on these reference method audits.

4.4 *Flow rate accuracy and bias.* Part 58, appendix A of this chapter requires that the flow rate accuracy and bias of individual PM_{2.5} samplers used in SLAMS monitoring networks be assessed periodically via audits of each sampler's operational flow rate. In addition, part 58, appendix A of this chapter requires that flow rate bias for each reference and equivalent method operated by each reporting organization be assessed quarterly and annually. Reference 2 in section 13.0 of this appendix provides additional information and guidance on flow rate accuracy audits and calculations for accuracy and bias.

5.0 *Precision.* A data quality objective of 10 percent coefficient of variation or better has

been established for the operational precision of PM_{2.5} monitoring data.

5.1 Tests to establish initial operational precision for each reference method sampler are specified as a part of the requirements for designation as a reference method under §53.58 of this chapter.

5.2 *Measurement System Precision.* Collocated sampler results, where the duplicate sampler is not a reference method sampler but is a sampler of the same designated method as the primary sampler, are used to assess measurement system precision according to the schedule and procedure specified in part 58, appendix A of this chapter. Part 58, appendix A of this chapter requires that these collocated sampler measurements be used to calculate quarterly and annual precision estimates for each primary sampler and for each designated method employed by each reporting organization. Reference 2 in section 13.0 of this appendix provides additional information and guidance on this requirement.

6.0 *Filter for PM_{2.5} Sample Collection.* Any filter manufacturer or vendor who sells or offers to sell filters specifically identified for use with this PM_{2.5} reference method shall certify that the required number of filters from each lot of filters offered for sale as such have been tested as specified in this section 6.0 and meet all of the following design and performance specifications.

6.1 *Size.* Circular, 46.2 mm diameter ± 0.25 mm.

6.2 *Medium.* Polytetrafluoroethylene (PTFE Teflon), with integral support ring.

6.3 *Support ring.* Polymethylpentene (PMP) or equivalent inert material, 0.38 ± 0.04 mm thick, outer diameter 46.2 mm ± 0.25 mm, and width of 3.68 mm (± 0.00 , -0.51 mm).

6.4 *Pore size.* 2 μ m as measured by ASTM F 316-94.

6.5 *Filter thickness.* 30 to 50 μ m.

6.6 *Maximum pressure drop (clean filter).* 30 cm H₂O column @ 16.67 L/min clean air flow.

6.7 *Maximum moisture pickup.* Not more than 10 μ g weight increase after 24-hour exposure to air of 40 percent relative humidity, relative to weight after 24-hour exposure to air of 35 percent relative humidity.

6.8 *Collection efficiency.* Greater than 99.7 percent, as measured by the DOP test (ASTM D 2986-91) with 0.3 μ m particles at the sampler's operating face velocity.

6.9 *Filter weight stability.* Filter weight loss shall be less than 20 μ g, as measured in each of the following two tests specified in sections 6.9.1 and 6.9.2 of this appendix. The following conditions apply to both of these tests: Filter weight loss shall be the average difference between the initial and the final filter weights of a random sample of test filters selected from each lot prior to sale. The number of filters tested shall be not less than 0.1 percent of the filters of each manufacturing lot, or 10 filters, whichever is

greater. The filters shall be weighed under laboratory conditions and shall have had no air sample passed through them, i.e., filter blanks. Each test procedure must include initial conditioning and weighing, the test, and final conditioning and weighing. Conditioning and weighing shall be in accordance with sections 8.0 through 8.2 of this appendix and general guidance provided in reference 2 of section 13.0 of this appendix.

6.9.1 *Test for loose, surface particle contamination.* After the initial weighing, install each test filter, in turn, in a filter cassette (Figures L-27, L-28, and L-29 of this appendix) and drop the cassette from a height of 25 cm to a flat hard surface, such as a particle-free wood bench. Repeat two times, for a total of three drop tests for each test filter. Remove the test filter from the cassette and weigh the filter. The average change in weight must be less than 20 μ g.

6.9.2 *Test for temperature stability.* After weighing each filter, place the test filters in a drying oven set at 40 °C ± 2 °C for not less than 48 hours. Remove, condition, and reweigh each test filter. The average change in weight must be less than 20 μ g.

6.10 *Alkalinity.* Less than 25 microequivalents/gram of filter, as measured by the guidance given in reference 2 in section 13.0 of this appendix.

6.11 *Supplemental requirements.* Although not required for determination of PM_{2.5} mass concentration under this reference method, additional specifications for the filter must be developed by users who intend to subject PM_{2.5} filter samples to subsequent chemical analysis. These supplemental specifications include background chemical contamination of the filter and any other filter parameters that may be required by the method of chemical analysis. All such supplemental filter specifications must be compatible with and secondary to the primary filter specifications given in this section 6.0 of this appendix.

7.0 *PM_{2.5} Sampler.*

7.1 *Configuration.* The sampler shall consist of a sample air inlet, downtube, particle size separator (impactor), filter holder assembly, air pump and flow rate control system, flow rate measurement device, ambient and filter temperature monitoring system, barometric pressure measurement system, timer, outdoor environmental enclosure, and suitable mechanical, electrical, or electronic control capability to meet or exceed the design and functional performance as specified in this section 7.0 of this appendix. The performance specifications require that the sampler:

- (a) Provide automatic control of sample volumetric flow rate and other operational parameters.
- (b) Monitor these operational parameters as well as ambient temperature and pressure.
- (c) Provide this information to the sampler operator at the end of each sample period in

digital form, as specified in table L-1 of section 7.4.19 of this appendix.

7.2 *Nature of specifications.* The PM_{2.5} sampler is specified by a combination of design and performance requirements. The sample inlet, downtube, particle size discriminator, filter cassette, and the internal configuration of the filter holder assembly are specified explicitly by design figures and associated mechanical dimensions, tolerances, materials, surface finishes, assembly instructions, and other necessary specifications. All other aspects of the sampler are specified by required operational function and performance, and the design of these other aspects (including the design of the lower portion of the filter holder assembly) is optional, subject to acceptable operational performance. Test procedures to demonstrate compliance with both the design and performance requirements are set forth in subpart E of part 53 of this chapter.

7.3 *Design specifications.* Except as indicated in this section 7.3 of this appendix, these components must be manufactured or reproduced exactly as specified, in an ISO 9001-registered facility, with registration initially approved and subsequently maintained during the period of manufacture. See §53.1(t) of this chapter for the definition of an ISO-registered facility. Minor modifications or variances to one or more components that clearly would not affect the aerodynamic performance of the inlet, downtube, impactor, or filter cassette will be considered for specific approval. Any such proposed modifications shall be described and submitted to the EPA for specific individual acceptability either as part of a reference or equivalent method application under part 53 of this chapter or in writing in advance of such an intended application under part 53 of this chapter.

7.3.1 *Sample inlet assembly.* The sample inlet assembly, consisting of the inlet, downtube, and impactor shall be configured and assembled as indicated in Figure L-1 of this appendix and shall meet all associated requirements. A portion of this assembly shall also be subject to the maximum overall sampler leak rate specification under section 7.4.6 of this appendix.

7.3.2 *Inlet.* The sample inlet shall be fabricated as indicated in Figures L-2 through L-18 of this appendix and shall meet all associated requirements.

7.3.3 *Downtube.* The downtube shall be fabricated as indicated in Figure L-19 of this appendix and shall meet all associated requirements.

7.3.4 *Impactor.*

7.3.4.1 The impactor (particle size separator) shall be fabricated as indicated in Figures L-20 through L-24 of this appendix and shall meet all associated requirements. Following the manufacture and finishing of each upper impactor housing (Figure L-21 of this

appendix), the dimension of the impaction jet must be verified by the manufacturer using Class ZZ go/no-go plug gauges that are traceable to NIST.

7.3.4.2 *Impactor filter specifications:*

- (a) Size. Circular, 35 to 37 mm diameter.
- (b) Medium. Borosilicate glass fiber, without binder.
- (c) Pore size. 1 to 1.5 micrometer, as measured by ASTM F 316-80.
- (d) Thickness. 300 to 500 micrometers.

7.3.4.3 *Impactor oil specifications:*

- (a) Composition. Tetramethyltetraphenyltrisiloxane, single-compound diffusion oil.
- (b) Vapor pressure. Maximum 2×10^{-8} mm Hg at 25 °C.
- (c) Viscosity. 36 to 40 centistokes at 25 °C.
- (d) Density. 1.06 to 1.07 g/cm³ at 25 °C.
- (e) Quantity. 1 mL \pm 0.1 mL.

7.3.5 *Filter holder assembly.* The sampler shall have a sample filter holder assembly to adapt and seal to the down tube and to hold and seal the specified filter, under section 6.0 of this appendix, in the sample air stream in a horizontal position below the downtube such that the sample air passes downward through the filter at a uniform face velocity. The upper portion of this assembly shall be fabricated as indicated in Figures L-25 and L-26 of this appendix and shall accept and seal with the filter cassette, which shall be fabricated as indicated in Figures L-27 through L-29 of this appendix.

(a) The lower portion of the filter holder assembly shall be of a design and construction that:

- (1) Mates with the upper portion of the assembly to complete the filter holder assembly.
- (2) Completes both the external air seal and the internal filter cassette seal such that all seals are reliable over repeated filter changings, and
- (3) Facilitates repeated changing of the filter cassette by the sampler operator.

(b) Leak-test performance requirements for the filter holder assembly are included in section 7.4.6 of this appendix.

(c) If additional or multiple filters are stored in the sampler as part of an automatic sequential sample capability, all such filters, unless they are currently and directly installed in a sampling channel or sampling configuration (either active or inactive), shall be covered or (preferably) sealed in such a way as to:

- (1) Preclude significant exposure of the filter to possible contamination or accumulation of dust, insects, or other material that may be present in the ambient air, sampler, or sampler ventilation air during storage periods either before or after sampling; and
- (2) To minimize loss of volatile or semi-volatile PM sample components during storage of the filter following the sample period.

7.3.6 *Flow rate measurement adapter.* A flow rate measurement adapter as specified in

Figure L-30 of this appendix shall be furnished with each sampler.

7.3.7 Surface finish. All internal surfaces exposed to sample air prior to the filter shall be treated electrolytically in a sulfuric acid bath to produce a clear, uniform anodized surface finish of not less than 1000 mg/ft² (1.08 mg/cm²) in accordance with military standard specification (mil. spec.) 8625F, Type II, Class 1 in reference 4 of section 13.0 of this appendix. This anodic surface coating shall not be dyed or pigmented. Following anodization, the surfaces shall be sealed by immersion in boiling deionized water for not less than 15 minutes. Section 53.51(d)(2) of this chapter should also be consulted.

7.3.8 Sampling height. The sampler shall be equipped with legs, a stand, or other means to maintain the sampler in a stable, upright position and such that the center of the sample air entrance to the inlet, during sample collection, is maintained in a horizontal plane and is 2.0 ±0.2 meters above the floor or other horizontal supporting surface. Suitable bolt holes, brackets, tie-downs, or other means should be provided to facilitate mechanically securing the sample to the supporting surface to prevent toppling of the sampler due to wind.

7.4 Performance specifications.

7.4.1 Sample flow rate. Proper operation of the impactor requires that specific air velocities be maintained through the device. Therefore, the design sample air flow rate through the inlet shall be 16.67 L/min (1,000 m³/hour) measured as actual volumetric flow rate at the temperature and pressure of the sample air entering the inlet.

7.4.2 Sample air flow rate control system. The sampler shall have a sample air flow rate control system which shall be capable of providing a sample air volumetric flow rate within the specified range, under section 7.4.1 of this appendix, for the specified filter, under section 6.0 of this appendix, at any atmospheric conditions specified, under section 7.4.7 of this appendix, at a filter pressure drop equal to that of a clean filter plus up to 75 cm water column (55 mm Hg), and over the specified range of supply line voltage, under section 7.4.15.1 of this appendix. This flow control system shall allow for operator adjustment of the operational flow rate of the sampler over a range of at least ±15 percent of the flow rate specified in section 7.4.1 of this appendix.

7.4.3 Sample flow rate regulation. The sample flow rate shall be regulated such that for the specified filter, under section 6.0 of this appendix, at any atmospheric conditions specified, under section 7.4.7 of this appendix, at a filter pressure drop equal to that of a clean filter plus up to 75 cm water column (55 mm Hg), and over the specified range of supply line voltage, under section 7.4.15.1 of this appendix, the flow rate is regulated as follows:

7.4.3.1 The volumetric flow rate, measured or averaged over intervals of not more than 5 minutes over a 24-hour period, shall not vary more than ±5 percent from the specified 16.67 L/min flow rate over the entire sample period.

7.4.3.2 The coefficient of variation (sample standard deviation divided by the mean) of the flow rate, measured over a 24-hour period, shall not be greater than 2 percent.

7.4.3.3 The amplitude of short-term flow rate pulsations, such as may originate from some types of vacuum pumps, shall be attenuated such that they do not cause significant flow measurement error or affect the collection of particles on the particle collection filter.

7.4.4 Flow rate cut off. The sampler's sample air flow rate control system shall terminate sample collection and stop all sample flow for the remainder of the sample period in the event that the sample flow rate deviates by more than 10 percent from the sampler design flow rate specified in section 7.4.1 of this appendix for more than 60 seconds. However, this sampler cut-off provision shall not apply during periods when the sampler is inoperative due to a temporary power interruption, and the elapsed time of the inoperative period shall not be included in the total sample time measured and reported by the sampler, under section 7.4.13 of this appendix.

7.4.5 Flow rate measurement.

7.4.5.1 The sampler shall provide a means to measure and indicate the instantaneous sample air flow rate, which shall be measured as volumetric flow rate at the temperature and pressure of the sample air entering the inlet, with an accuracy of ±2 percent. The measured flow rate shall be available for display to the sampler operator at any time in either sampling or standby modes, and the measurement shall be updated at least every 30 seconds. The sampler shall also provide a simple means by which the sampler operator can manually start the sample flow temporarily during non-sampling modes of operation, for the purpose of checking the sample flow rate or the flow rate measurement system.

7.4.5.2 During each sample period, the sampler's flow rate measurement system shall automatically monitor the sample volumetric flow rate, obtaining flow rate measurements at intervals of not greater than 30 seconds.

(a) Using these interval flow rate measurements, the sampler shall determine or calculate the following flow-related parameters, scaled in the specified engineering units:

(1) The instantaneous or interval-average flow rate, in L/min.

(2) The value of the average sample flow rate for the sample period, in L/min.

(3) The value of the coefficient of variation (sample standard deviation divided by the

average) of the sample flow rate for the sample period, in percent.

(4) The occurrence of any time interval during the sample period in which the measured sample flow rate exceeds a range of ± 5 percent of the average flow rate for the sample period for more than 5 minutes, in which case a warning flag indicator shall be set.

(5) The value of the integrated total sample volume for the sample period, in m^3 .

(b) Determination or calculation of these values shall properly exclude periods when the sampler is inoperative due to temporary interruption of electrical power, under section 7.4.13 of this appendix, or flow rate cut off, under section 7.4.4 of this appendix.

(c) These parameters shall be accessible to the sampler operator as specified in table L-1 of section 7.4.19 of this appendix. In addition, it is strongly encouraged that the flow rate for each 5-minute interval during the sample period be available to the operator following the end of the sample period.

7.4.6 Leak test capability.

7.4.6.1 *External leakage.* The sampler shall include an external air leak-test capability consisting of components, accessory hardware, operator interface controls, a written procedure in the associated Operation/Instruction Manual, under section 7.4.18 of this appendix, and all other necessary functional capability to permit and facilitate the sampler operator to conveniently carry out a leak test of the sampler at a field monitoring site without additional equipment. The sampler components to be subjected to this leak test include all components and their interconnections in which external air leakage would or could cause an error in the sampler's measurement of the total volume of sample air that passes through the sample filter.

(a) The suggested technique for the operator to use for this leak test is as follows:

(1) Remove the sampler inlet and install the flow rate measurement adapter supplied with the sampler, under section 7.3.6 of this appendix.

(2) Close the valve on the flow rate measurement adapter and use the sampler air pump to draw a partial vacuum in the sampler, including (at least) the impactor, filter holder assembly (filter in place), flow measurement device, and interconnections between these devices, of at least 55 mm Hg (75 cm water column), measured at a location downstream of the filter holder assembly.

(3) Plug the flow system downstream of these components to isolate the components under vacuum from the pump, such as with a built-in valve.

(4) Stop the pump.

(5) Measure the trapped vacuum in the sampler with a built-in pressure measuring device.

(6) (i) Measure the vacuum in the sampler with the built-in pressure measuring device

again at a later time at least 10 minutes after the first pressure measurement.

(ii) CAUTION: Following completion of the test, the adaptor valve should be opened slowly to limit the flow rate of air into the sampler. Excessive air flow rate may blow oil out of the impactor.

(7) Upon completion of the test, open the adaptor valve, remove the adaptor and plugs, and restore the sampler to the normal operating configuration.

(b) The associated leak test procedure shall require that for successful passage of this test, the difference between the two pressure measurements shall not be greater than the number of mm of Hg specified for the sampler by the manufacturer, based on the actual internal volume of the sampler, that indicates a leak of less than 80 mL/min.

(c) Variations of the suggested technique or an alternative external leak test technique may be required for samplers whose design or configuration would make the suggested technique impossible or impractical. The specific proposed external leak test procedure, or particularly an alternative leak test technique, proposed for a particular candidate sampler may be described and submitted to the EPA for specific individual acceptability either as part of a reference or equivalent method application under part 53 of this chapter or in writing in advance of such an intended application under part 53 of this chapter.

7.4.6.2 *Internal, filter bypass leakage.* The sampler shall include an internal, filter bypass leak-check capability consisting of components, accessory hardware, operator interface controls, a written procedure in the Operation/Instruction Manual, and all other necessary functional capability to permit and facilitate the sampler operator to conveniently carry out a test for internal filter bypass leakage in the sampler at a field monitoring site without additional equipment. The purpose of the test is to determine that any portion of the sample flow rate that leaks past the sample filter without passing through the filter is insignificant relative to the design flow rate for the sampler.

(a) The suggested technique for the operator to use for this leak test is as follows:

(1) Carry out an external leak test as provided under section 7.4.6.1 of this appendix which indicates successful passage of the prescribed external leak test.

(2) Install a flow-impervious membrane material in the filter cassette, either with or without a filter, as appropriate, which effectively prevents air flow through the filter.

(3) Use the sampler air pump to draw a partial vacuum in the sampler, downstream of the filter holder assembly, of at least 55 mm Hg (75 cm water column).

(4) Plug the flow system downstream of the filter holder to isolate the components under

vacuum from the pump, such as with a built-in valve.

(5) Stop the pump.

(6) Measure the trapped vacuum in the sampler with a built-in pressure measuring device.

(7) Measure the vacuum in the sampler with the built-in pressure measuring device again at a later time at least 10 minutes after the first pressure measurement.

(8) Remove the flow plug and membrane and restore the sampler to the normal operating configuration.

(b) The associated leak test procedure shall require that for successful passage of this test, the difference between the two pressure measurements shall not be greater than the number of mm of Hg specified for the sampler by the manufacturer, based on the actual internal volume of the portion of the sampler under vacuum, that indicates a leak of less than 80 mL/min.

(c) Variations of the suggested technique or an alternative internal, filter bypass leak test technique may be required for samplers whose design or configuration would make the suggested technique impossible or impractical. The specific proposed internal leak test procedure, or particularly an alternative internal leak test technique proposed for a particular candidate sampler may be described and submitted to the EPA for specific individual acceptability either as part of a reference or equivalent method application under part 53 of this chapter or in writing in advance of such intended application under part 53 of this chapter.

7.4.7 Range of operational conditions. The sampler is required to operate properly and meet all requirements specified in this appendix over the following operational ranges.

7.4.7.1 Ambient temperature. -30 to =45 °C (Note: Although for practical reasons, the temperature range over which samplers are required to be tested under part 53 of this chapter is -20 to =40 °C, the sampler shall be designed to operate properly over this wider temperature range.)

7.4.7.2 Ambient relative humidity. 0 to 100 percent.

7.4.7.3 Barometric pressure range. 600 to 800 mm Hg.

7.4.8 Ambient temperature sensor. The sampler shall have capability to measure the temperature of the ambient air surrounding the sampler over the range of -30 to =45 °C, with a resolution of 0.1 °C and accuracy of ±2.0 °C, referenced as described in reference 3 in section 13.0 of this appendix, with and without maximum solar insolation.

7.4.8.1 The ambient temperature sensor shall be mounted external to the sampler enclosure and shall have a passive, naturally ventilated sun shield. The sensor shall be located such that the entire sun shield is at least 5 cm above the horizontal plane of the sampler case or enclosure (disregarding the

inlet and downtube) and external to the vertical plane of the nearest side or protuberance of the sampler case or enclosure. The maximum temperature measurement error of the ambient temperature measurement system shall be less than 1.6 °C at 1 m/s wind speed and 1000 W/m² solar radiation intensity.

7.4.8.2 The ambient temperature sensor shall be of such a design and mounted in such a way as to facilitate its convenient dismounting and immersion in a liquid for calibration and comparison to the filter temperature sensor, under section 7.4.11 of this appendix.

7.4.8.3 This ambient temperature measurement shall be updated at least every 30 seconds during both sampling and standby (non-sampling) modes of operation. A visual indication of the current (most recent) value of the ambient temperature measurement, updated at least every 30 seconds, shall be available to the sampler operator during both sampling and standby (non-sampling) modes of operation, as specified in table L-1 of section 7.4.19 of this appendix.

7.4.8.4 This ambient temperature measurement shall be used for the purpose of monitoring filter temperature deviation from ambient temperature, as required by section 7.4.11 of this appendix, and may be used for purposes of effecting filter temperature control, under section 7.4.10 of this appendix, or computation of volumetric flow rate, under sections 7.4.1 to 7.4.5 of this appendix, if appropriate.

7.4.8.5 Following the end of each sample period, the sampler shall report the maximum, minimum, and average temperature for the sample period, as specified in table L-1 of section 7.4.19 of this appendix.

7.4.9 Ambient barometric sensor. The sampler shall have capability to measure the barometric pressure of the air surrounding the sampler over a range of 600 to 800 mm Hg referenced as described in reference 3 in section 13.0 of this appendix; also see part 53, subpart E of this chapter. This barometric pressure measurement shall have a resolution of 5 mm Hg and an accuracy of ±10 mm Hg and shall be updated at least every 30 seconds. A visual indication of the value of the current (most recent) barometric pressure measurement, updated at least every 30 seconds, shall be available to the sampler operator during both sampling and standby (non-sampling) modes of operation, as specified in table L-1 of section 7.4.19 of this appendix. This barometric pressure measurement may be used for purposes of computation of volumetric flow rate, under sections 7.4.1 to 7.4.5 of this appendix, if appropriate. Following the end of a sample period, the sampler shall report the maximum, minimum, and mean barometric pressures for the sample period, as specified in table L-1 of section 7.4.19 of this appendix.

7.4.10 *Filter temperature control (sampling and post-sampling).* The sampler shall provide a means to limit the temperature rise of the sample filter (all sample filters for sequential samplers), from insolation and other sources, to no more 5 °C above the temperature of the ambient air surrounding the sampler, during both sampling and post-sampling periods of operation. The post-sampling period is the non-sampling period between the end of the active sampling period and the time of retrieval of the sample filter by the sampler operator.

7.4.11 *Filter temperature sensor(s).*

7.4.11.1 The sampler shall have the capability to monitor the temperature of the sample filter (all sample filters for sequential samplers) over the range of -30 to +45 °C during both sampling and non-sampling periods. While the exact location of this temperature sensor is not explicitly specified, the filter temperature measurement system must demonstrate agreement, within 1 °C, with a test temperature sensor located within 1 cm of the center of the filter downstream of the filter during both sampling and non-sampling modes, as specified in the filter temperature measurement test described in part 53, subpart E of this chapter. This filter temperature measurement shall have a resolution of 0.1 °C and accuracy of ±1.0 °C, referenced as described in reference 3 in section 13.0 of this appendix. This temperature sensor shall be of such a design and mounted in such a way as to facilitate its reasonably convenient dismounting and immersion in a liquid for calibration and comparison to the ambient temperature sensor under section 7.4.8 of this appendix.

7.4.11.2 The filter temperature measurement shall be updated at least every 30 seconds during both sampling and standby (non-sampling) modes of operation. A visual indication of the current (most recent) value of the filter temperature measurement, updated at least every 30 seconds, shall be available to the sampler operator during both sampling and standby (non-sampling) modes of operation, as specified in table L-1 of section 7.4.19 of this appendix.

7.4.11.3 For sequential samplers, the temperature of each filter shall be measured individually unless it can be shown, as specified in the filter temperature measurement test described in §53.57 of this chapter, that the temperature of each filter can be represented by fewer temperature sensors.

7.4.11.4 The sampler shall also provide a warning flag indicator following any occurrence in which the filter temperature (any filter temperature for sequential samplers) exceeds the ambient temperature by more than 5 °C for more than 30 consecutive minutes during either the sampling or post-sampling periods of operation, as specified in table L-1 of section 7.4.19 of this appendix, under section 10.12 of this appendix, regard-

ing sample validity when a warning flag occurs. It is further recommended (not required) that the sampler be capable of recording the maximum differential between the measured filter temperature and the ambient temperature and its time and date of occurrence during both sampling and post-sampling (non-sampling) modes of operation and providing for those data to be accessible to the sampler operator following the end of the sample period, as suggested in table L-1 of section 7.4.19 of this appendix.

7.4.12 *Clock/timer system.*

(a) The sampler shall have a programmable real-time clock timing/control system that:

(1) Is capable of maintaining local time and date, including year, month, day-of-month, hour, minute, and second to an accuracy of ±1.0 minute per month.

(2) Provides a visual indication of the current system time, including year, month, day-of-month, hour, and minute, updated at least each minute, for operator verification.

(3) Provides appropriate operator controls for setting the correct local time and date.

(4) Is capable of starting the sample collection period and sample air flow at a specific, operator-settable time and date, and stopping the sample air flow and terminating the sampler collection period 24 hours (1440 minutes) later, or at a specific, operator-settable time and date.

(b) These start and stop times shall be readily settable by the sampler operator to within ±1.0 minute. The system shall provide a visual indication of the current start and stop time settings, readable to ±1.0 minute, for verification by the operator, and the start and stop times shall also be available via the data output port, as specified in table L-1 of section 7.4.19 of this appendix. Upon execution of a programmed sample period start, the sampler shall automatically reset all sample period information and warning flag indications pertaining to a previous sample period. Refer also to section 7.4.15.4 of this appendix regarding retention of current date and time and programmed start and stop times during a temporary electrical power interruption.

7.4.13 *Sample time determination.* The sampler shall be capable of determining the elapsed sample collection time for each PM_{2.5} sample, accurate to within ±1.0 minute, measured as the time between the start of the sampling period, under section 7.4.12 of this appendix and the termination of the sample period, under section 7.4.12 of this appendix or section 7.4.4 of this appendix. This elapsed sample time shall not include periods when the sampler is inoperative due to a temporary interruption of electrical power, under section 7.4.15.4 of this appendix. In the event that the elapsed sample time determined for the sample period is not within the

range specified for the required sample period in section 3.3 of this appendix, the sampler shall set a warning flag indicator. The date and time of the start of the sample period, the value of the elapsed sample time for the sample period, and the flag indicator status shall be available to the sampler operator following the end of the sample period, as specified in table L-1 of section 7.4.19 of this appendix.

7.4.14 *Outdoor environmental enclosure.* The sampler shall have an outdoor enclosure (or enclosures) suitable to protect the filter and other non-weatherproof components of the sampler from precipitation, wind, dust, extremes of temperature and humidity; to help maintain temperature control of the filter (or filters, for sequential samplers); and to provide reasonable security for sampler components and settings.

7.4.15 *Electrical power supply.*

7.4.15.1 The sampler shall be operable and function as specified herein when operated on an electrical power supply voltage of 105 to 125 volts AC (RMS) at a frequency of 59 to 61 Hz. Optional operation as specified at additional power supply voltages and/or frequencies shall not be precluded by this requirement.

7.4.15.2 The design and construction of the sampler shall comply with all applicable National Electrical Code and Underwriters Laboratories electrical safety requirements.

7.4.15.3 The design of all electrical and electronic controls shall be such as to provide reasonable resistance to interference or malfunction from ordinary or typical levels of stray electromagnetic fields (EMF) as may be found at various monitoring sites and from typical levels of electrical transients or electronic noise as may often or occasionally be present on various electrical power lines.

7.4.15.4 In the event of temporary loss of electrical supply power to the sampler, the sampler shall not be required to sample or provide other specified functions during such loss of power, except that the internal clock/timer system shall maintain its local time and date setting within ± 1 minute per week, and the sampler shall retain all other time and programmable settings and all data required to be available to the sampler operator following each sample period for at least 7 days without electrical supply power. When electrical power is absent at the operator-set time for starting a sample period or is interrupted during a sample period, the sampler shall automatically start or resume sampling when electrical power is restored, if such restoration of power occurs before the operator-set stop time for the sample period.

7.4.15.5 The sampler shall have the capability to record and retain a record of the year, month, day-of-month, hour, and minute of the start of each power interruption of more than 1 minute duration, up to 10

such power interruptions per sample period. (More than 10 such power interruptions shall invalidate the sample, except where an exceedance is measured, under section 3.3 of this appendix.) The sampler shall provide for these power interruption data to be available to the sampler operator following the end of the sample period, as specified in table L-1 of section 7.4.19 of this appendix.

7.4.16 *Control devices and operator interface.* The sampler shall have mechanical, electrical, or electronic controls, control devices, electrical or electronic circuits as necessary to provide the timing, flow rate measurement and control, temperature control, data storage and computation, operator interface, and other functions specified. Operator-accessible controls, data displays, and interface devices shall be designed to be simple, straightforward, reliable, and easy to learn, read, and operate under field conditions. The sampler shall have provision for operator input and storage of up to 64 characters of numeric (or alphanumeric) data for purposes of site, sampler, and sample identification. This information shall be available to the sampler operator for verification and change and for output via the data output port along with other data following the end of a sample period, as specified in table L-1 of section 7.4.19 of this appendix. All data required to be available to the operator following a sample collection period or obtained during standby mode in a post-sampling period shall be retained by the sampler until reset, either manually by the operator or automatically by the sampler upon initiation of a new sample collection period.

7.4.17 *Data output port requirement.* The sampler shall have a standard RS-232C data output connection through which digital data may be exported to an external data storage or transmission device. All information which is required to be available at the end of each sample period shall be accessible through this data output connection. The information that shall be accessible through this output port is summarized in table L-1 of section 7.4.19 of this appendix. Since no specific format for the output data is provided, the sampler manufacturer or vendor shall make available to sampler purchasers appropriate computer software capable of receiving exported sampler data and correctly translating the data into a standard spreadsheet format and optionally any other formats as may be useful to sampler users. This requirement shall not preclude the sampler from offering other types of output connections in addition to the required RS-232C port.

7.4.18 *Operation/instruction manual.* The sampler shall include an associated comprehensive operation or instruction manual, as required by part 53 of this chapter, which includes detailed operating instructions on

the setup, operation, calibration, and maintenance of the sampler. This manual shall provide complete and detailed descriptions of the operational and calibration procedures prescribed for field use of the sampler and all instruments utilized as part of this reference method. The manual shall include adequate warning of potential safety hazards that may result from normal use or malfunction of the method and a description of necessary safety

precautions. The manual shall also include a clear description of all procedures pertaining to installation, operation, periodic and corrective maintenance, and troubleshooting, and shall include parts identification diagrams.

7.4.19 *Data reporting requirements.* The various information that the sampler is required to provide and how it is to be provided is summarized in the following table L-1.

TABLE L-1—SUMMARY OF INFORMATION TO BE PROVIDED BY THE SAMPLER

Information to be provided	Appendix L section reference	Availability				Format	
		Anytime ¹	End of period ²	Visual display ³	Data output ⁴	Digital reading ⁵	Units
Flow rate, 30-second maximum interval.	7.4.5.1	✓	✓	*	XX.X	L/min
Flow rate, average for the sample period.	7.4.5.2	*	✓	*	✓	XX.X	L/min
Flow rate, CV, for sample period.	7.4.5.2	*	✓	*	■	XX.X	%
Flow rate, 5-min. average out of spec. (FLAG ⁶).	7.4.5.2	✓	✓	✓	■	On/Off	
Sample volume, total.	7.4.5.2	*	✓	✓	■	XX.X	m ³
Temperature, ambient, 30-second interval.	7.4.8	✓	✓	XX.X	°C
Temperature, ambient, min., max., average for the sample period.	7.4.8	*	✓	✓	■	XX.X	°C
Baro pressure, ambient, 30-second interval.	7.4.9	✓	✓	XXX	mm Hg
Baro pressure, ambient, min., max., average for the sample period.	7.4.9	*	✓	✓	■	XXX	mm Hg
Filter temperature, 30-second interval.	7.4.11 ..	✓	✓	XX.X	°C
Filter temperature differential, 30-second interval, out of spec. (FLAG ⁶).	7.4.11 ..	*	✓	✓	■	On/Off	
Filter temperature, maximum differential from ambient, date, time of occurrence.	7.4.11 ..	*	*	*	*	X.X, YY/MM/DD HH:mm.	°C, Yr./Mon./Day Hrs. min
Date and time	7.4.12 ..	✓	✓	YY/MM/DD HH:mm.	Yr./Mon./Day Hrs. min
Sample start and stop time settings.	7.4.12 ..	✓	✓	✓	✓	YY/MM/DD HH:mm.	Yr./Mon./Day Hrs. min
Sample period start time.	7.4.12	✓	✓	■	YYYY/MM/DD HH:mm.	Yr./Mon./Day Hrs. min
Elapsed sample time.	7.4.13 ..	*	✓	✓	■	HH:mm	Hrs. min
Elapsed sample time, out of spec. (FLAG ⁶).	7.4.13	✓	✓	■	On/Off	

TABLE L-1—SUMMARY OF INFORMATION TO BE PROVIDED BY THE SAMPLER—Continued

Information to be provided	Appendix L section reference	Availability				Format	
		Anytime ¹	End of period ²	Visual display ³	Data output ⁴	Digital reading ⁵	Units
Power interruptions ≤1 min., start time of first 10.	7.4.15.5	*	✓	*	✓	1HH:mm, 2HH:mm, etc	Hrs. min
User-entered information, such as sampler and site identification.	7.4.16 ..	✓	✓	✓	✓■	As entered	

✓ Provision of this information is required.
 *Provision of this information is optional. If information related to the entire sample period is optionally provided prior to the end of the sample period, the value provided should be the value calculated for the portion of the sampler period completed up to the time the information is provided.
 ■ Indicates that this information is also required to be provided to the AIRS data bank; see §§ 58.26 and 58.35 of this chapter.

¹ Information is required to be available to the operator at any time the sampler is operating, whether sampling or not.
² Information relates to the entire sampler period and must be provided following the end of the sample period until reset manually by the operator or automatically by the sampler upon the start of a new sample period.
³ Information shall be available to the operator visually.
⁴ Information is to be available as digital data at the sampler's data output port specified in section 7.4.16 of this appendix following the end of the sample period until reset manually by the operator or automatically by the sampler upon the start of a new sample period.
⁵ Digital readings, both visual and data output, shall have not less than the number of significant digits and resolution specified.
⁶ Flag warnings may be displayed to the operator by a single-flag indicator or each flag may be displayed individually. Only a set (on) flag warning must be indicated; an off (unset) flag may be indicated by the absence of a flag warning. Sampler users should refer to section 10.12 of this appendix regarding the validity of samples for which the sampler provided an associated flag warning.

8.0 *Filter Weighing.* See reference 2 in section 13.0 of this appendix, for additional, more detailed guidance.

8.1 *Analytical balance.* The analytical balance used to weigh filters must be suitable for weighing the type and size of filters specified, under section 6.0 of this appendix, and have a readability of ±1 µg. The balance shall be calibrated as specified by the manufacturer at installation and recalibrated immediately prior to each weighing session. See reference 2 in section 13.0 of this appendix for additional guidance.

8.2 *Filter conditioning.* All sample filters used shall be conditioned immediately before both the pre- and post-sampling weighings as specified below. See reference 2 in section 13.0 of this appendix for additional guidance.

8.2.1 *Mean temperature.* 20 - 23 °C.
 8.2.2 *Temperature control.* ±2 °C over 24 hours.

8.2.3 *Mean humidity.* Generally, 30–40 percent relative humidity; however, where it can be shown that the mean ambient relative humidity during sampling is less than 30 percent, conditioning is permissible at a mean relative humidity within ±5 relative humidity percent of the mean ambient relative humidity during sampling, but not less than 20 percent.

8.2.4 *Humidity control.* ±5 relative humidity percent over 24 hours.

8.2.5 *Conditioning time.* Not less than 24 hours.

8.3 *Weighing procedure.*

8.3.1 New filters should be placed in the conditioning environment immediately upon arrival and stored there until the pre-sampling weighing. See reference 2 in section 13.0 of this appendix for additional guidance.

8.3.2 The analytical balance shall be located in the same controlled environment in which the filters are conditioned. The filters shall be weighed immediately following the conditioning period without intermediate or transient exposure to other conditions or environments.

8.3.3 Filters must be conditioned at the same conditions (humidity within ±5 relative humidity percent) before both the pre- and post-sampling weighings.

8.3.4 Both the pre- and post-sampling weighings should be carried out on the same analytical balance, using an effective technique to neutralize static charges on the filter, under reference 2 in section 13.0 of this appendix. If possible, both weighings should be carried out by the same analyst.

8.3.5 The pre-sampling (tare) weighing shall be within 30 days of the sampling period.

8.3.6 The post-sampling conditioning and weighing shall be completed within 240 hours (10 days) after the end of the sample period, unless the filter sample is maintained at 4 °C or less during the entire time between retrieval from the sampler and the start of the conditioning, in which case the period shall not exceed 30 days. Reference 2 in section 13.0 of this appendix has additional guidance on transport of cooled filters.

8.3.7 Filter blanks.

8.3.7.1 New field blank filters shall be weighed along with the pre-sampling (tare) weighing of each lot of PM_{2.5} filters. These blank filters shall be transported to the sampling site, installed in the sampler, retrieved from the sampler without sampling, and reweighed as a quality control check.

8.3.7.2 New laboratory blank filters shall be weighed along with the pre-sampling (tare) weighing of each set of PM_{2.5} filters. These laboratory blank filters should remain in the laboratory in protective containers during the field sampling and should be reweighed as a quality control check.

8.3.8 Additional guidance for proper filter weighing and related quality assurance activities is provided in reference 2 in section 13.0 of this appendix.

9.0 *Calibration.* Reference 2 in section 13.0 of this appendix contains additional guidance.

9.1 General requirements.

9.1.1 Multipoint calibration and single-point verification of the sampler's flow rate measurement device must be performed periodically to establish and maintain traceability of subsequent flow measurements to a flow rate standard.

9.1.2 An authoritative flow rate standard shall be used for calibrating or verifying the sampler's flow rate measurement device with an accuracy of ± 2 percent. The flow rate standard shall be a separate, stand-alone device designed to connect to the flow rate measurement adapter, Figure L-30 of this appendix. This flow rate standard must have its own certification and be traceable to a National Institute of Standards and Technology (NIST) primary standard for volume or flow rate. If adjustments to the sampler's flow rate measurement system calibration are to be made in conjunction with an audit of the sampler's flow measurement system, such adjustments shall be made following the audit. Reference 2 in section 13.0 of this appendix contains additional guidance.

9.1.3 The sampler's flow rate measurement device shall be re-calibrated after electromechanical maintenance or transport of the sampler.

9.2 Flow rate calibration/verification procedure.

9.2.1 PM_{2.5} samplers may employ various types of flow control and flow measurement devices. The specific procedure used for calibration or verification of the flow rate measurement device will vary depending on the type of flow rate controller and flow rate measurement employed. Calibration shall be in terms of actual ambient volumetric flow rates (Q^a), measured at the sampler's inlet downtube. The generic procedure given here serves to illustrate the general steps involved in the calibration of a PM_{2.5} sampler. The sampler operation/instruction manual required under section 7.4.18 of this appendix and the Quality Assurance Handbook in ref-

erence 2 in section 13.0 of this appendix provide more specific and detailed guidance for calibration.

9.2.2 The flow rate standard used for flow rate calibration shall have its own certification and be traceable to a NIST primary standard for volume or flow rate. A calibration relationship for the flow rate standard, e.g., an equation, curve, or family of curves relating actual flow rate (Q_a) to the flow rate indicator reading, shall be established that is accurate to within 2 percent over the expected range of ambient temperatures and pressures at which the flow rate standard may be used. The flow rate standard must be re-calibrated or re-verified at least annually.

9.2.3 The sampler flow rate measurement device shall be calibrated or verified by removing the sampler inlet and connecting the flow rate standard to the sampler's downtube in accordance with the operation/instruction manual, such that the flow rate standard accurately measures the sampler's flow rate. The sampler operator shall first carry out a sampler leak check and confirm that the sampler passes the leak test and then verify that no leaks exist between the flow rate standard and the sampler.

9.2.4 The calibration relationship between the flow rate (in actual L/min) indicated by the flow rate standard and by the sampler's flow rate measurement device shall be established or verified in accordance with the sampler operation/instruction manual. Temperature and pressure corrections to the flow rate indicated by the flow rate standard may be required for certain types of flow rate standards. Calibration of the sampler's flow rate measurement device shall consist of at least three separate flow rate measurements (multipoint calibration) evenly spaced within the range of -10 percent to +10 percent of the sampler's operational flow rate, section 7.4.1 of this appendix. Verification of the sampler's flow rate shall consist of one flow rate measurement at the sampler's operational flow rate. The sampler operation/instruction manual and reference 2 in section 13.0 of this appendix provide additional guidance.

9.2.5 If during a flow rate verification the reading of the sampler's flow rate indicator or measurement device differs by ± 4 percent or more from the flow rate measured by the flow rate standard, a new multipoint calibration shall be performed and the flow rate verification must then be repeated.

9.2.6 Following the calibration or verification, the flow rate standard shall be removed from the sampler and the sampler inlet shall be reinstalled. Then the sampler's normal operating flow rate (in L/min) shall be determined with a clean filter in place. If the flow rate indicated by the sampler differs by ± 2 percent or more from the required sampler flow rate, the sampler flow rate must be

adjusted to the required flow rate, under section 7.4.1 of this appendix.

9.3 Periodic calibration or verification of the calibration of the sampler's ambient temperature, filter temperature, and barometric pressure measurement systems is also required. Reference 3 of section 13.0 of this appendix contains additional guidance.

10.0 *PM_{2.5} Measurement Procedure.* The detailed procedure for obtaining valid PM_{2.5} measurements with each specific sampler designated as part of a reference method for PM_{2.5} under part 53 of this chapter shall be provided in the sampler-specific operation or instruction manual required by section 7.4.18 of this appendix. Supplemental guidance is provided in section 2.12 of the Quality Assurance Handbook listed in reference 2 in section 13.0 of this appendix. The generic procedure given here serves to illustrate the general steps involved in the PM_{2.5} sample collection and measurement, using a PM_{2.5} reference method sampler.

10.1 The sampler shall be set up, calibrated, and operated in accordance with the specific, detailed guidance provided in the specific sampler's operation or instruction manual and in accordance with a specific quality assurance program developed and established by the user, based on applicable supplementary guidance provided in reference 2 in section 13.0 of this appendix.

10.2 Each new sample filter shall be inspected for correct type and size and for pinholes, particles, and other imperfections. Unacceptable filters should be discarded. A unique identification number shall be assigned to each filter, and an information record shall be established for each filter. If the filter identification number is not or cannot be marked directly on the filter, alternative means, such as a number-identified storage container, must be established to maintain positive filter identification.

10.3 Each filter shall be conditioned in the conditioning environment in accordance with the requirements specified in section 8.2 of this appendix.

10.4 Following conditioning, each filter shall be weighed in accordance with the requirements specified in section 8.0 of this appendix and the presampling weight recorded with the filter identification number.

10.5 A numbered and preweighed filter shall be installed in the sampler following the instructions provided in the sampler operation or instruction manual.

10.6 The sampler shall be checked and prepared for sample collection in accordance with instructions provided in the sampler operation or instruction manual and with the specific quality assurance program established for the sampler by the user.

10.7 The sampler's timer shall be set to start the sample collection at the beginning of the desired sample period and stop the sample collection 24 hours later.

10.8 Information related to the sample collection (site location or identification number, sample date, filter identification number, and sampler model and serial number) shall be recorded and, if appropriate, entered into the sampler.

10.9 The sampler shall be allowed to collect the PM_{2.5} sample during the set 24-hour time period.

10.10 Within 96 hours of the end of the sample collection period, the filter, while still contained in the filter cassette, shall be carefully removed from the sampler, following the procedure provided in the sampler operation or instruction manual and the quality assurance program, and placed in a protective container. The protective container shall contain no loose material that could be transferred to the filter. The protective container shall hold the filter cassette securely such that the cover shall not come in contact with the filter's surfaces. Reference 2 in section 13.0 of this appendix contains additional information.

10.11 The total sample volume in actual m³ for the sampling period and the elapsed sample time shall be obtained from the sampler and recorded in accordance with the instructions provided in the sampler operation or instruction manual. All sampler warning flag indications and other information required by the local quality assurance program shall also be recorded.

10.12 All factors related to the validity or representativeness of the sample, such as sampler tampering or malfunctions, unusual meteorological conditions, construction activity, fires or dust storms, etc. shall be recorded as required by the local quality assurance program. The occurrence of a flag warning during a sample period shall not necessarily indicate an invalid sample but rather shall indicate the need for specific review of the QC data by a quality assurance officer to determine sample validity.

10.13 After retrieval from the sampler, the exposed filter containing the PM_{2.5} sample should be transported to the filter conditioning environment as soon as possible ideally to arrive at the conditioning environment within 24 hours for conditioning and subsequent weighing. During the period between filter retrieval from the sampler and the start of the conditioning, the filter shall be maintained as cool as practical and continuously protected from exposure to temperatures over 25 °C. See section 8.3.6 of this appendix regarding time limits for completing the post-sampling weighing. See reference 2 in section 13.0 of this appendix for additional guidance on transporting filter samplers to the conditioning and weighing laboratory.

10.14. The exposed filter containing the PM_{2.5} sample shall be re-conditioned in the conditioning environment in accordance

with the requirements specified in section 8.2 of this appendix.

10.15. The filter shall be reweighed immediately after conditioning in accordance with the requirements specified in section 8.0 of this appendix, and the postsampling weight shall be recorded with the filter identification number.

10.16. The $PM_{2.5}$ concentration shall be calculated as specified in section 12.0 of this appendix.

11.0 *Sampler Maintenance.* The sampler shall be maintained as described by the sampler's manufacturer in the sampler-specific operation or instruction manual required under section 7.4.18 of this appendix and in accordance with the specific quality assurance program developed and established by the user based on applicable supplementary guidance provided in reference 2 in section 13.0 of this appendix.

12.0 Calculations

12.1 (a) The $PM_{2.5}$ concentration is calculated as:

$$PM_{2.5} = (W_f - W_i)/V_a$$

where:

$PM_{2.5}$ = mass concentration of $PM_{2.5}$, $\mu\text{g}/\text{m}^3$;

W_f , W_i = final and initial weights, respectively, of the filter used to collect the $PM_{2.5}$ particle sample, μg ;

V_a = total air volume sampled in actual volume units, as provided by the sampler, m^3 .

NOTE: Total sample time must be between 1,380 and 1,500 minutes (23 and 25 hrs) for a fully valid $PM_{2.5}$ sample; however, see also section 3.3 of this appendix.

13.0 References.

1. Quality Assurance Handbook for Air Pollution Measurement Systems, Volume I, Principles. EPA/600/R-94/038a, April 1994. Available from CERL, ORD Publications, U.S. Environmental Protection Agency, 26 West Martin Luther King Drive, Cincinnati, Ohio 45268.

2. Copies of section 2.12 of the Quality Assurance Handbook for Air Pollution Measurement Systems, Volume II, Ambient Air Specific Methods, EPA/600/R-94/038b, are available from Department E (MD-77B), U.S. EPA, Research Triangle Park, NC 27711.

3. Quality Assurance Handbook for Air Pollution Measurement Systems, Volume IV: Meteorological Measurements, (Revised Edition) EPA/600/R-94/038d, March, 1995. Available from CERL, ORD Publications, U.S. Environmental Protection Agency, 26 West Martin Luther King Drive, Cincinnati, Ohio 45268.

4. Military standard specification (mil. spec.) 8625F, Type II, Class 1 as listed in Department of Defense Index of Specifications and Standards (DODISS), available from DODSSP-Customer Service, Standardization Documents Order Desk, 700 Robbins Avenue, Building 4D, Philadelphia, PA 1911-5094.

14.0 *Figures L-1 through L-30 to Appendix L.*

FIGURE L-1. PM2.5 SAMPLER, ASSEMBLY

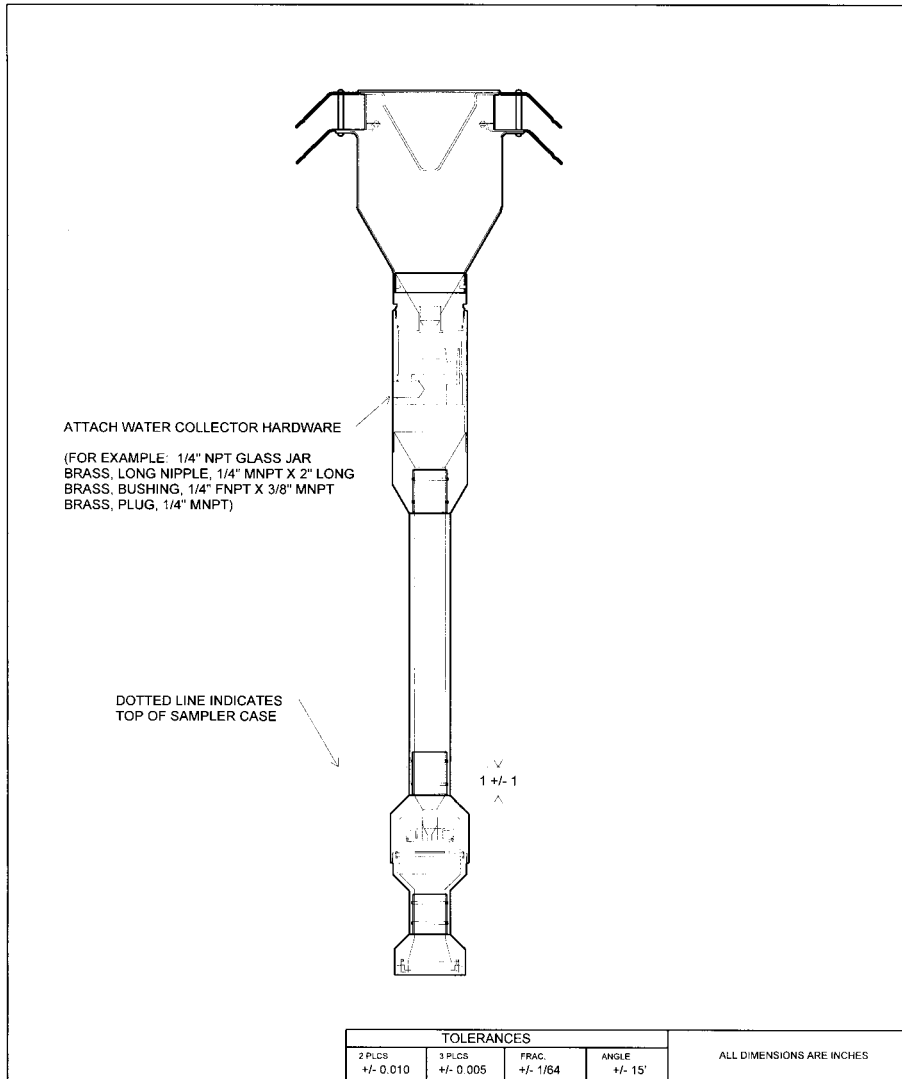


FIGURE L-2. 10-MICRON INLET, ASSEMBLY

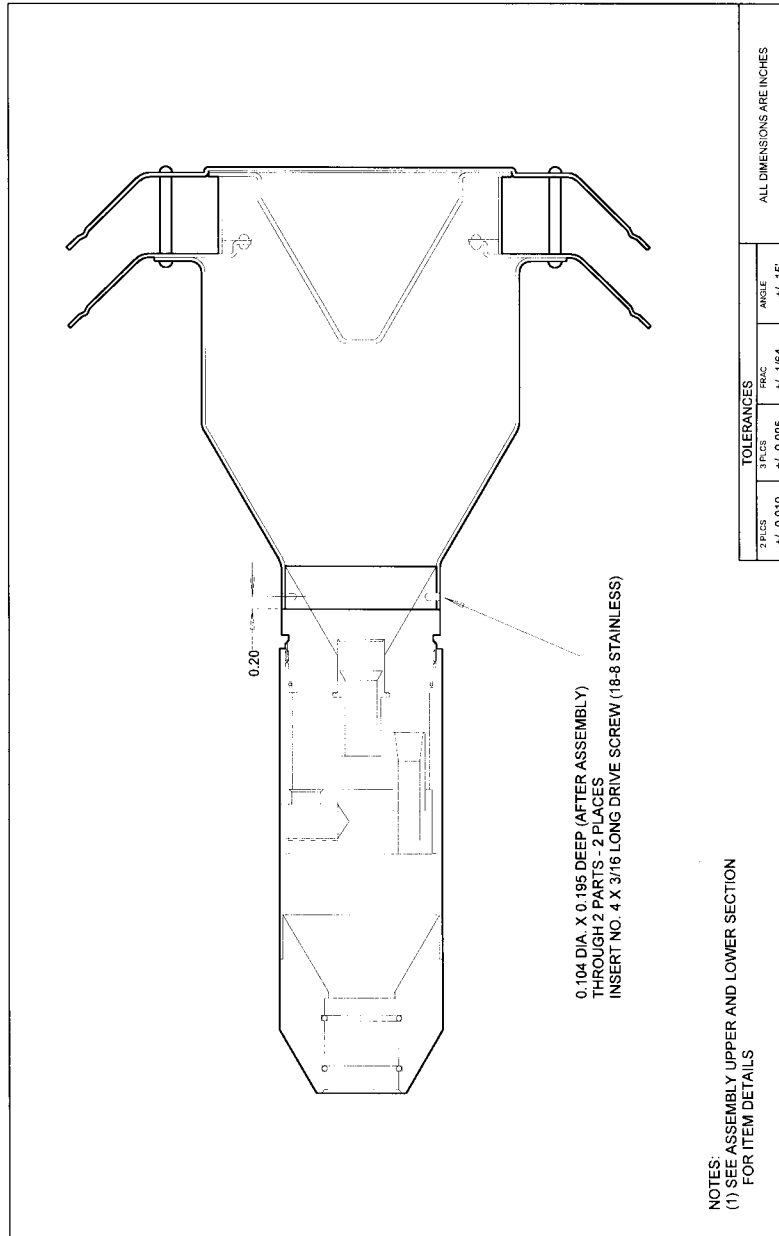


FIGURE L-3. 10-MICRON ASSEMBLY, UPPER SECTION

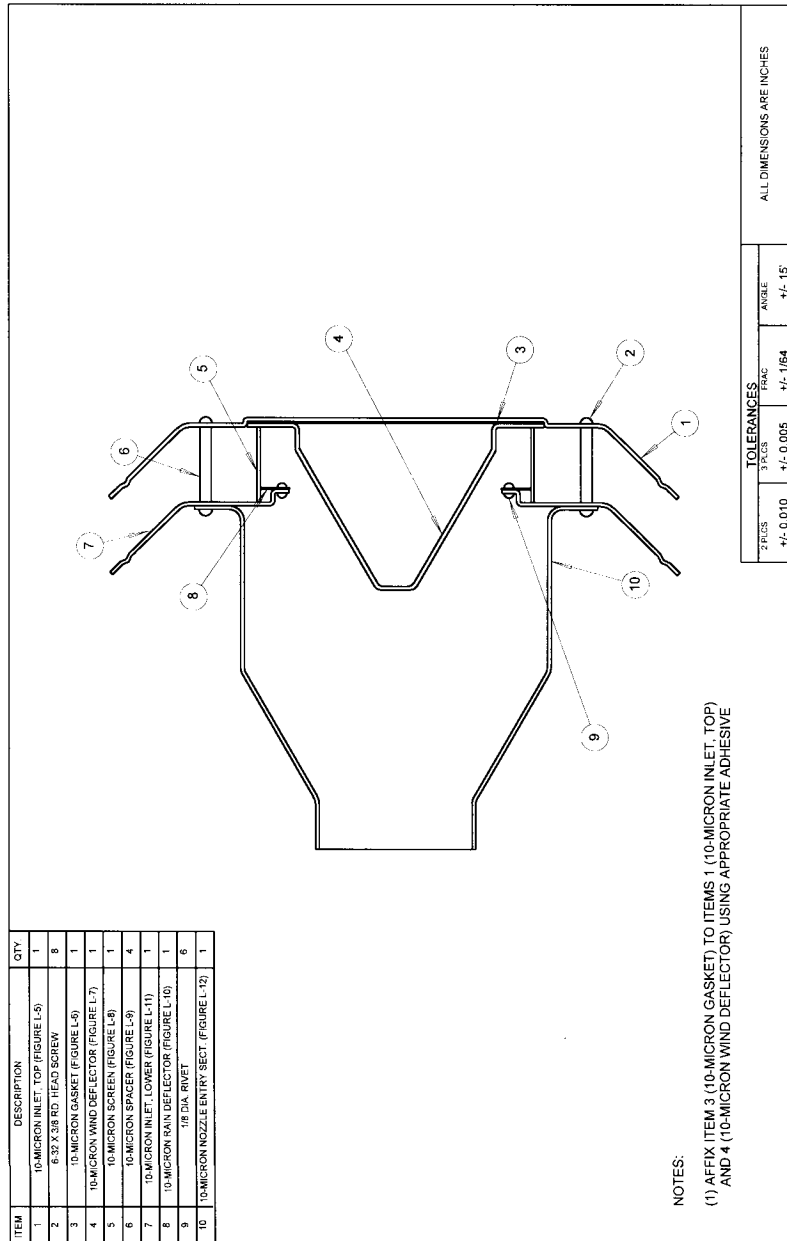


FIGURE L-4. 10-MICRON ASSEMBLY, LOWER SECTION

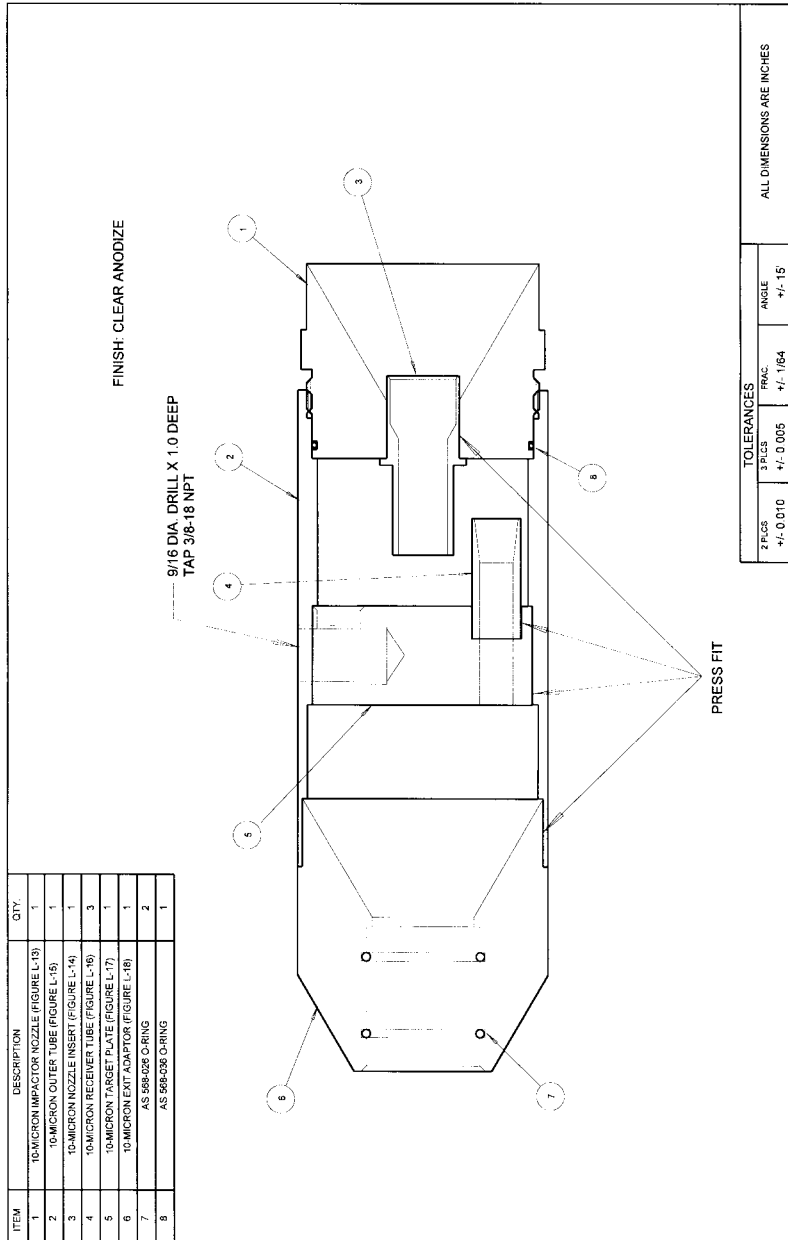


FIGURE L-5. 10-MICRON INLET, TOP

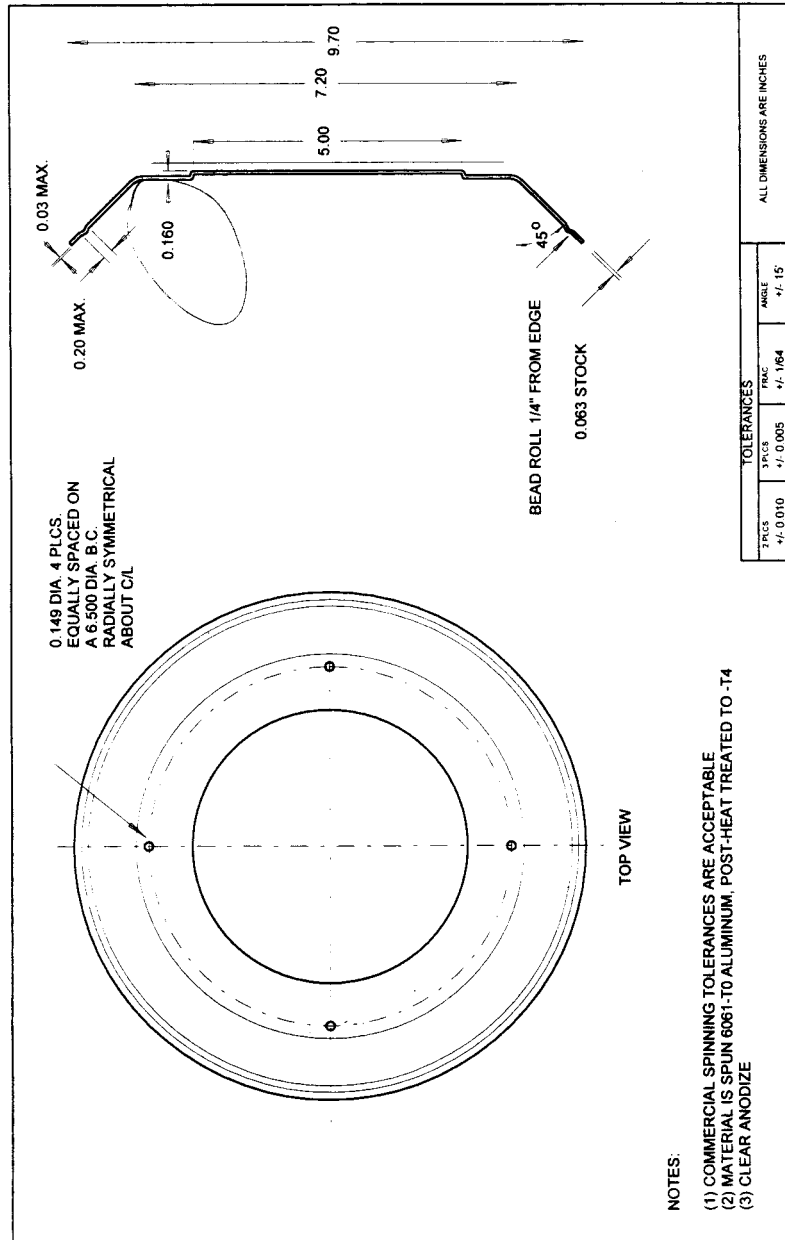


FIGURE L-6. 10-MICRON GASKET

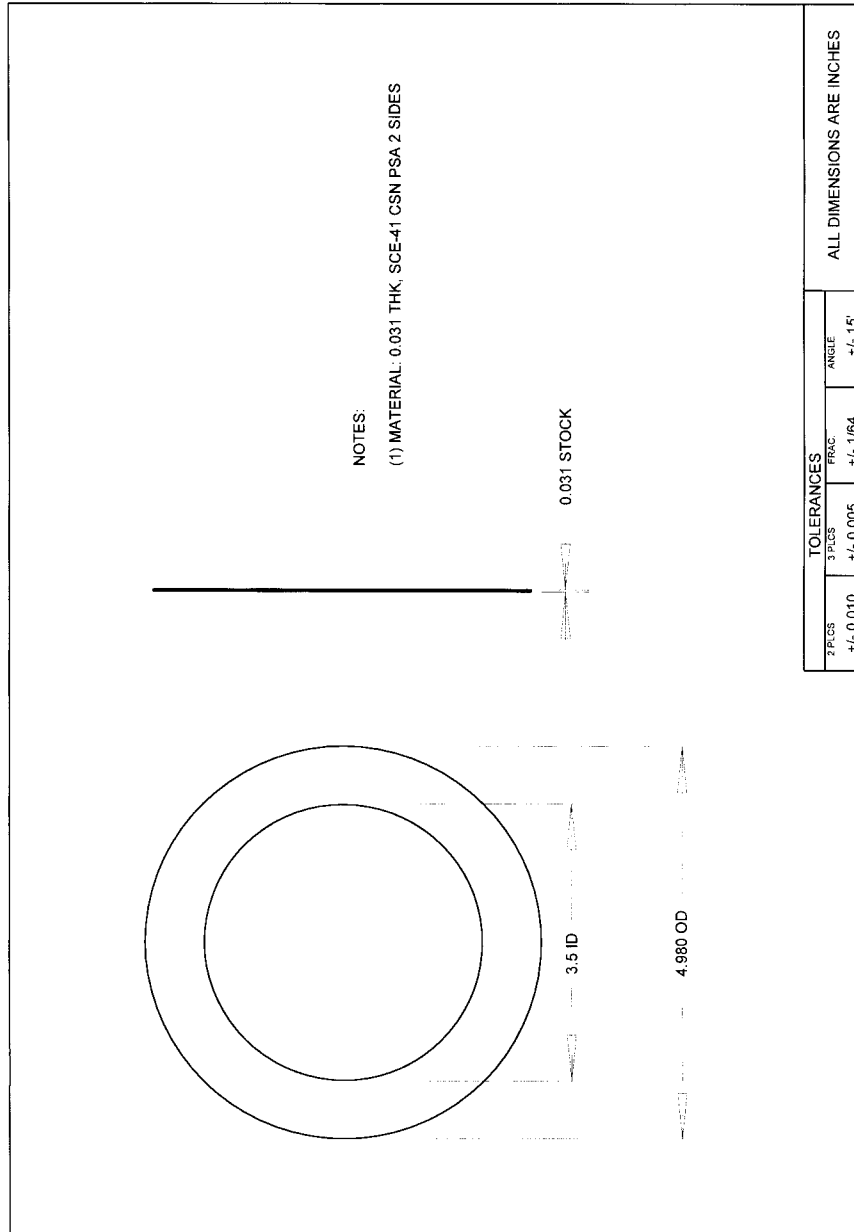


FIGURE L-7. 10-MICRON WIND DEFLECTOR

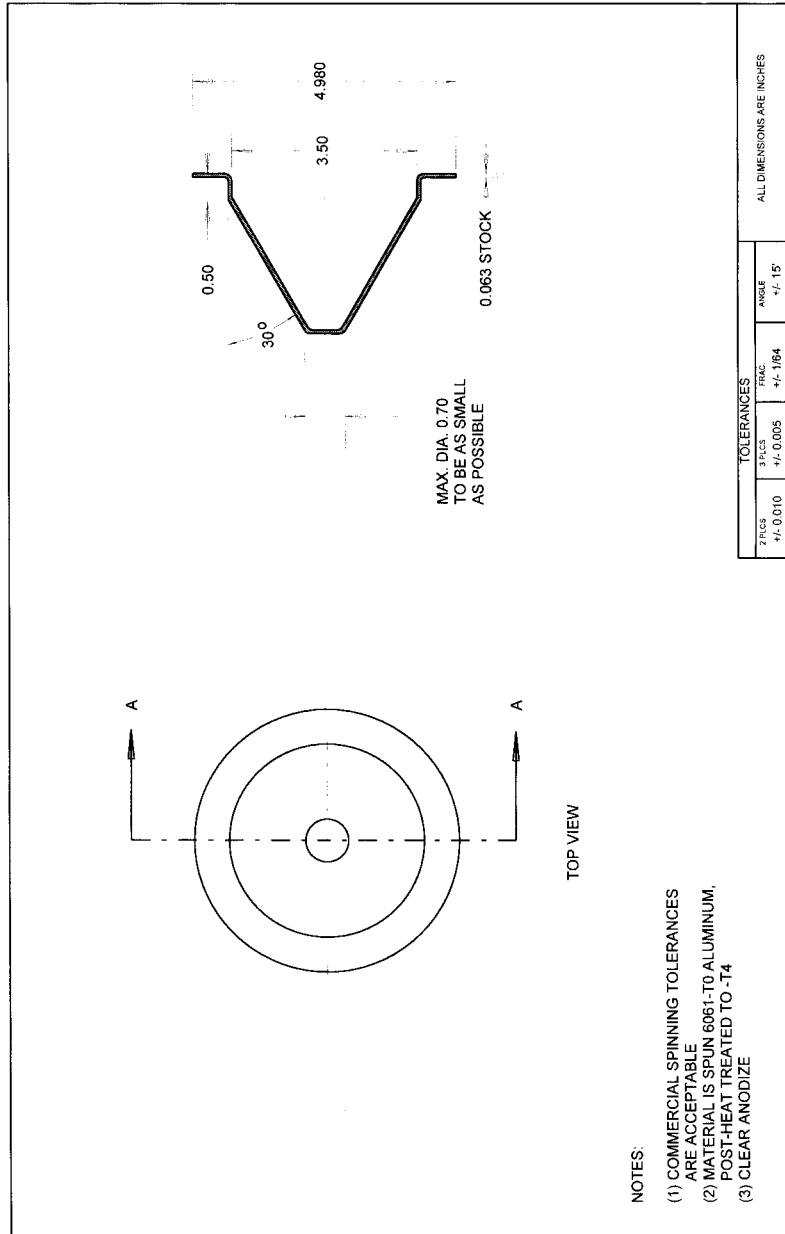


FIGURE L-8. 10-MICRON SCREEN

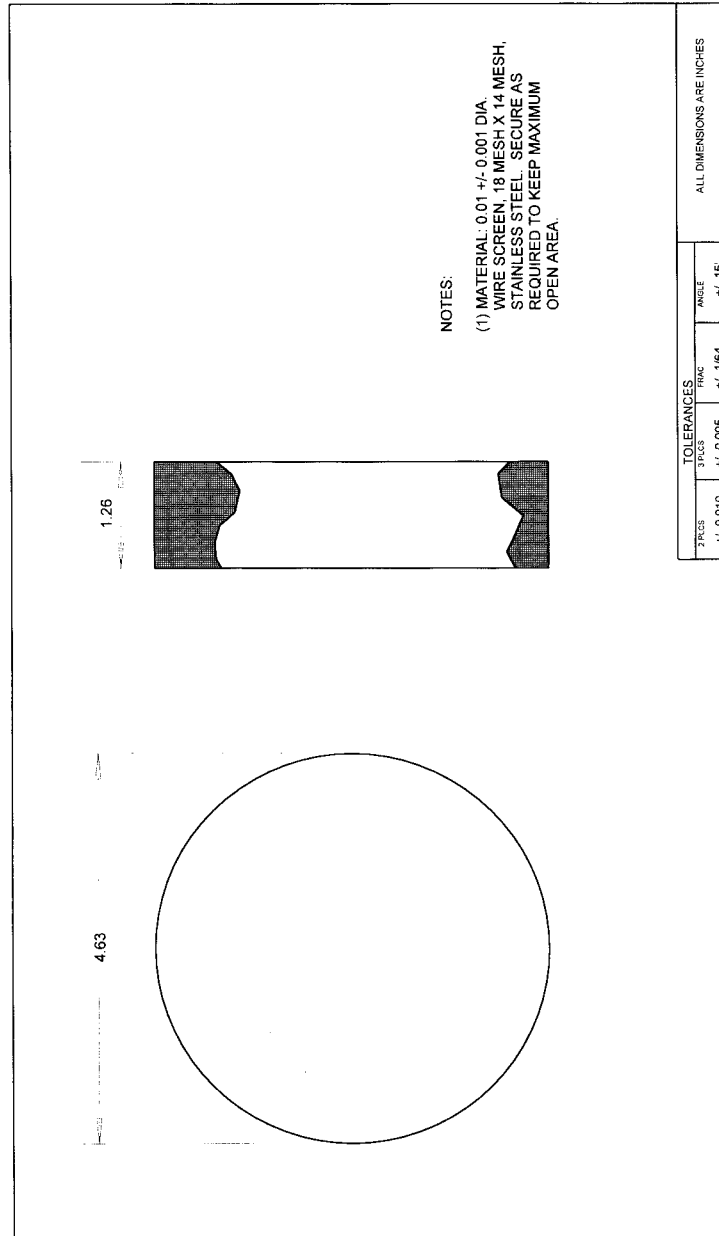


FIGURE L-9. 10-MICRON SPACER

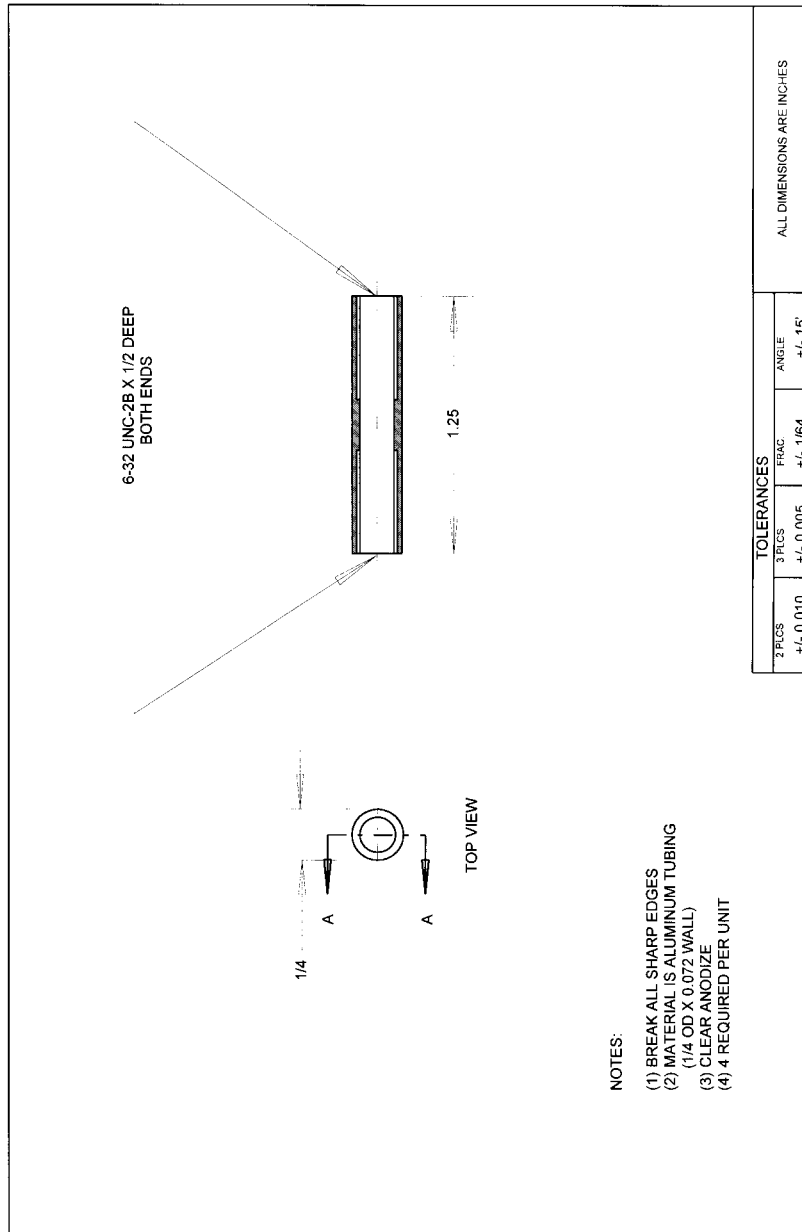


FIGURE L-10. 10-MICRON RAIN DEFLECTOR

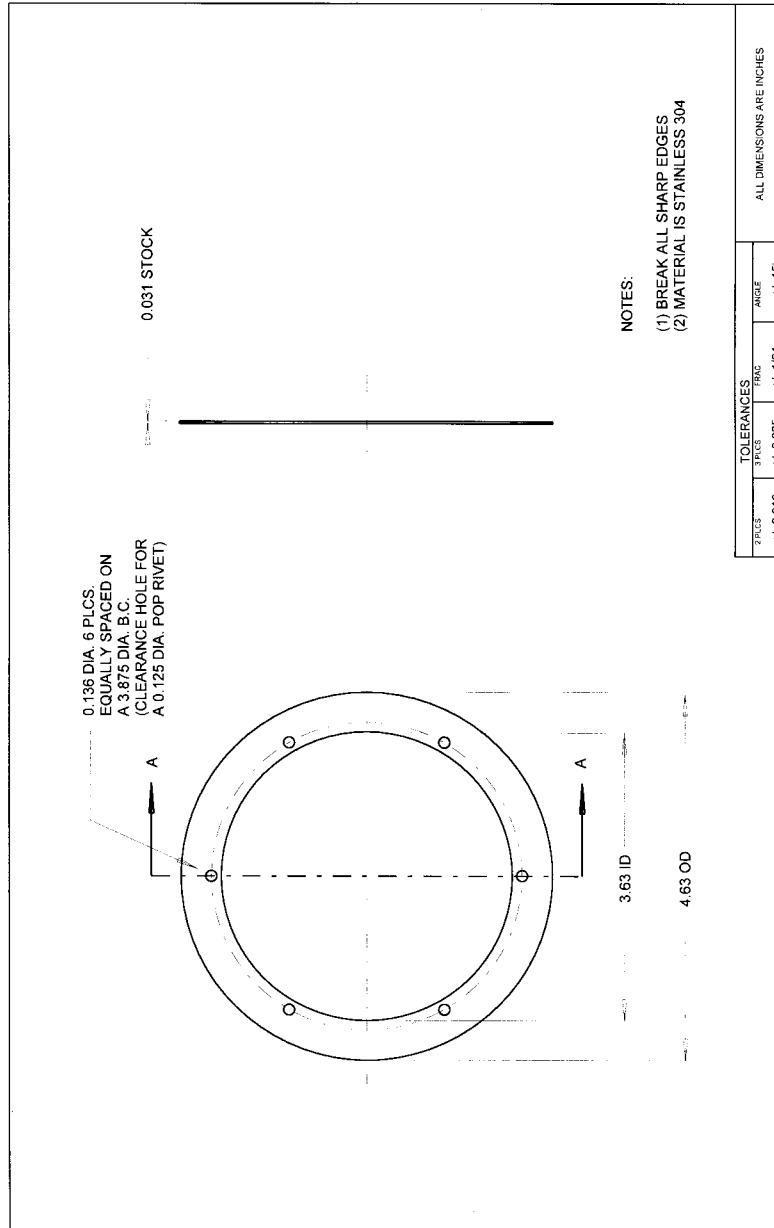


FIGURE L-11. 10-MICRON INLET, LOWER

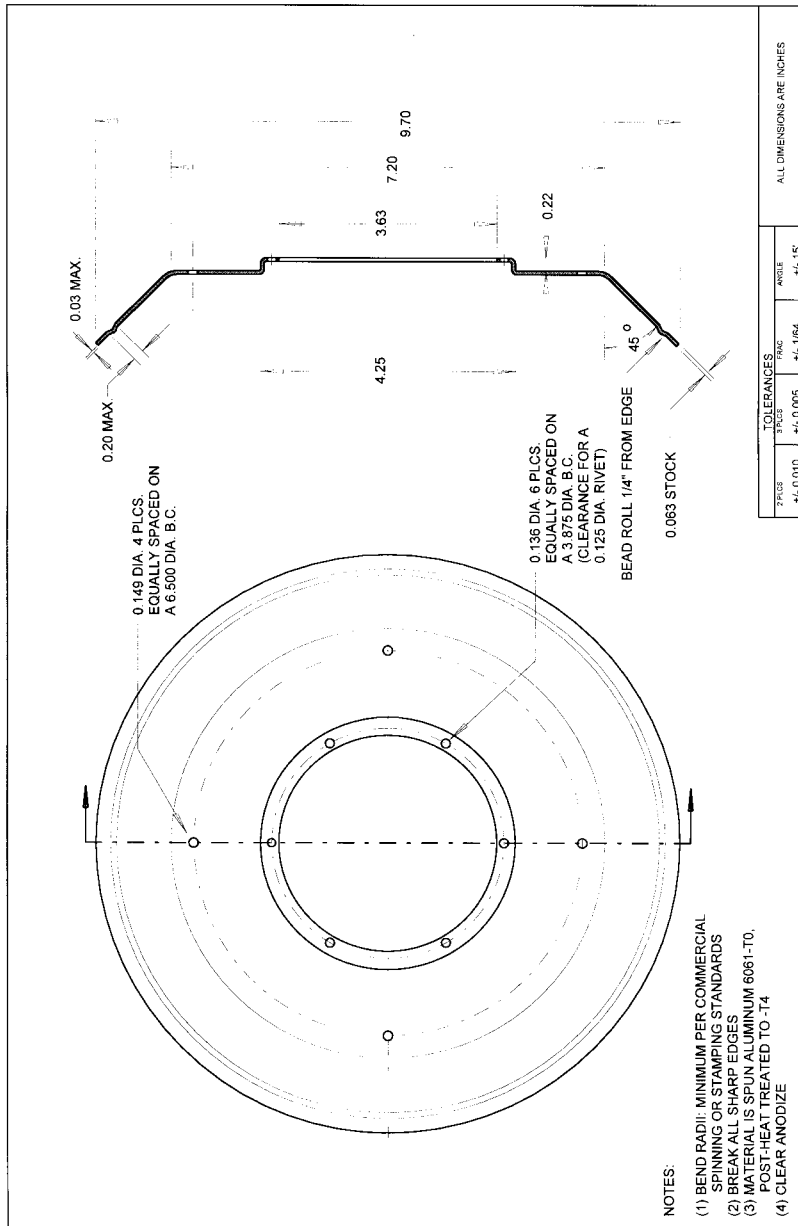


FIGURE L-13. 10-MICRON IMPACTOR NOZZLE

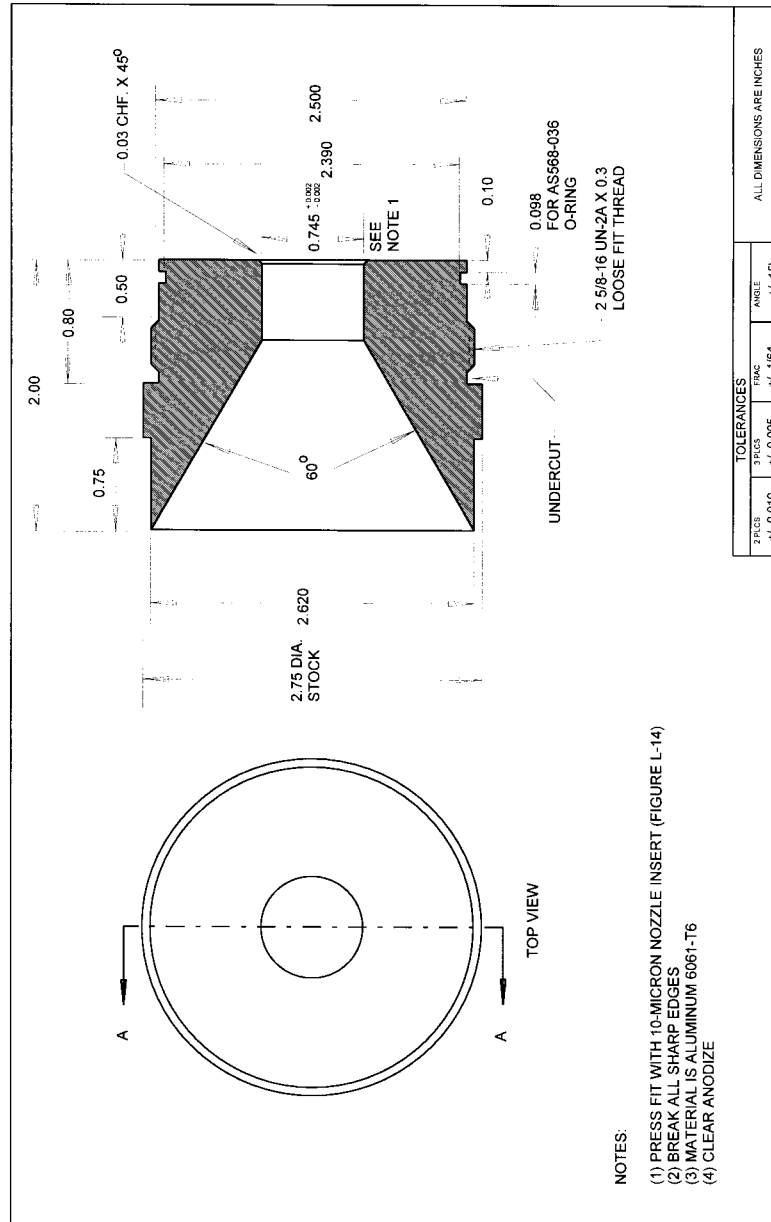


FIGURE L-14. 10-MICRON NOZZLE INSERT

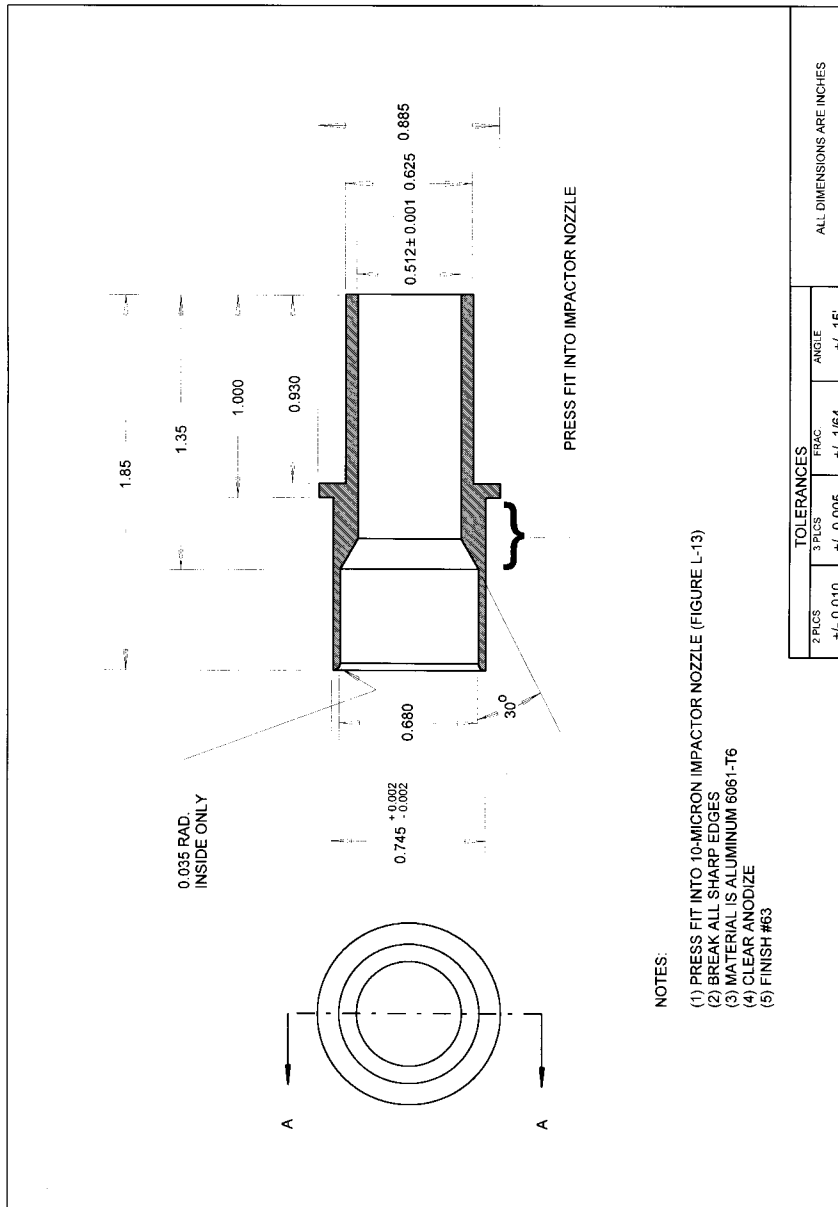


FIGURE L-15. 10-MICRON OUTER TUBE

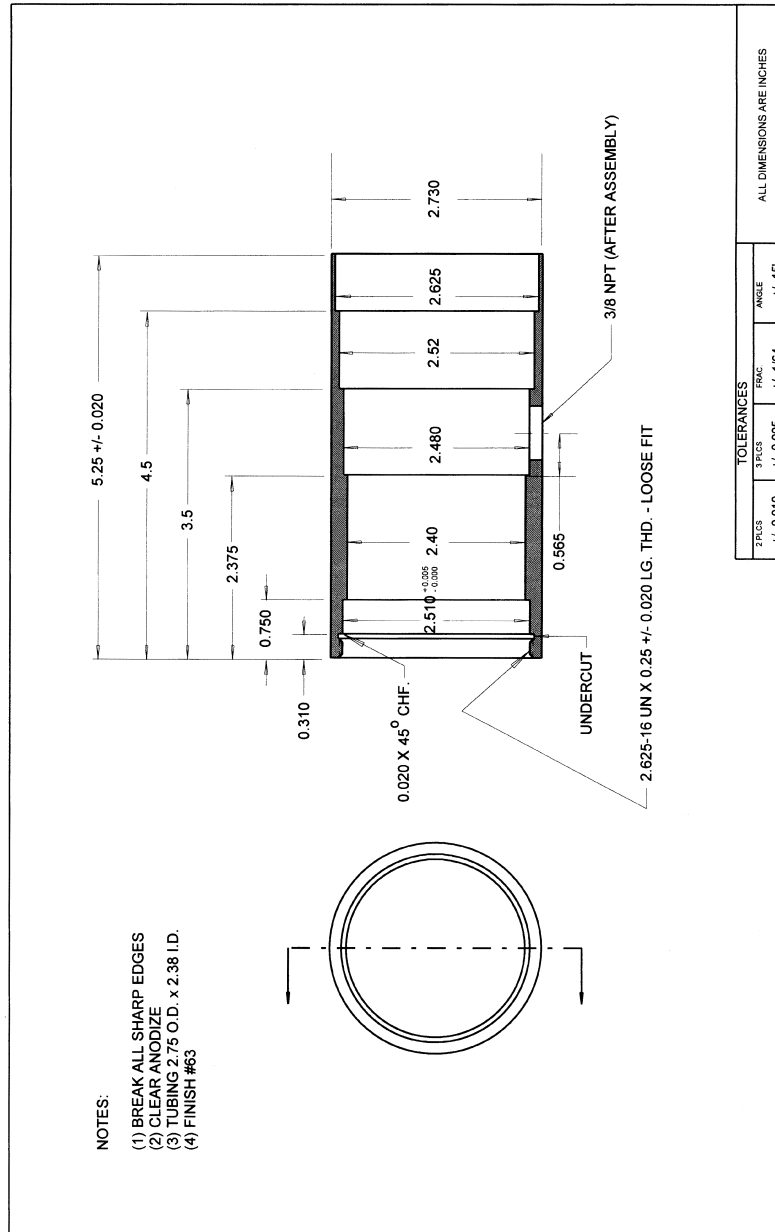
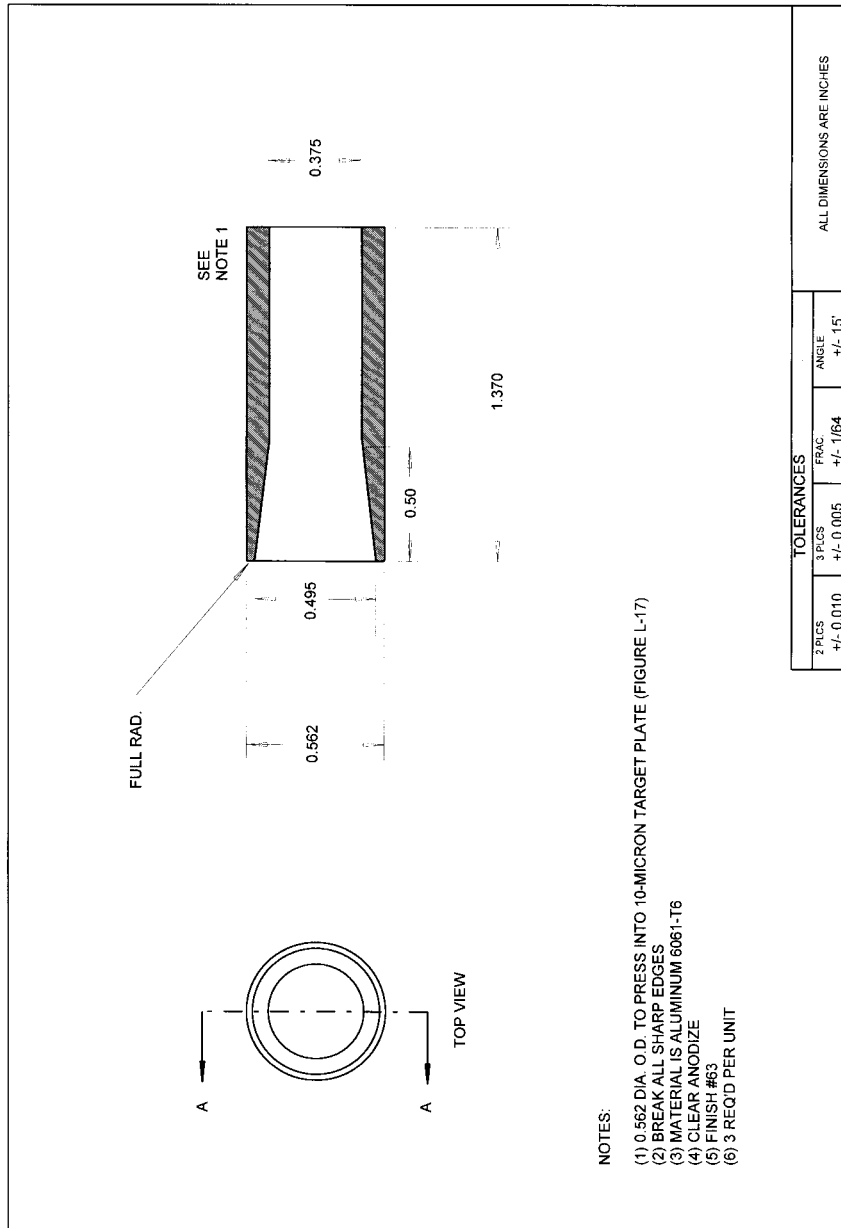


FIGURE L-16. 10-MICRON RECEIVER TUBE



NOTES:

- (1) 0.562 DIA. O.D. TO PRESS INTO 10-MICRON TARGET PLATE (FIGURE L-17)
- (2) BREAK ALL SHARP EDGES
- (3) MATERIAL IS ALUMINUM 6061-T6
- (4) CLEAR ANODIZE
- (5) FINISH #63
- (6) 3 REQ'D PER UNIT

FIGURE L-17. 10-MICRON TARGET PLATE

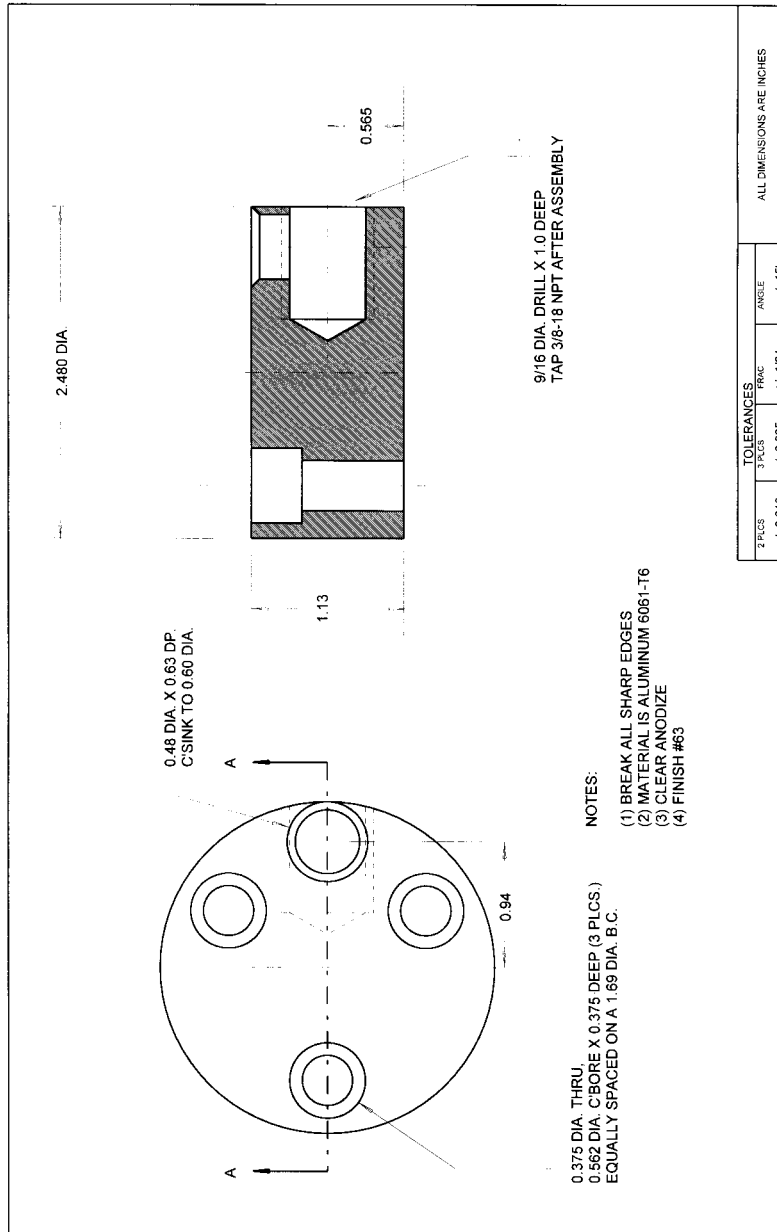


FIGURE L-18. 10-MICRON EXIT ADAPTOR

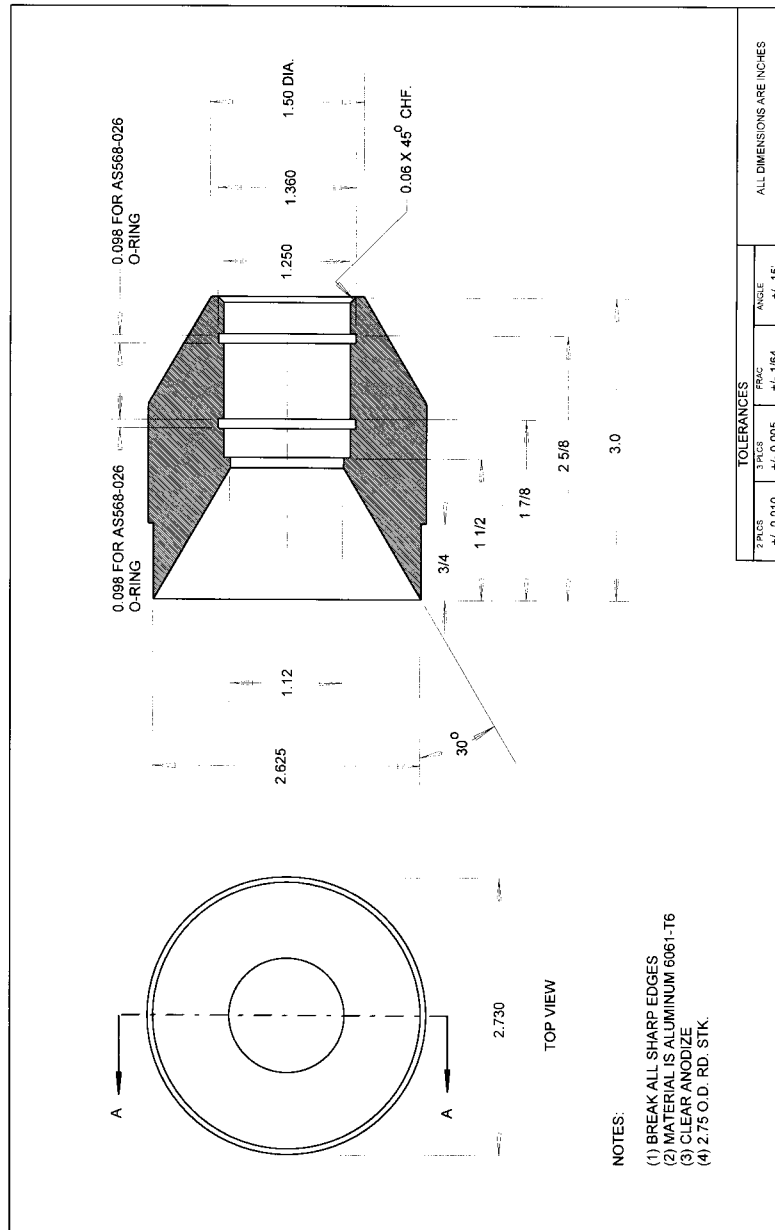


FIGURE L-19. 10-MICRON DOWN TUBE

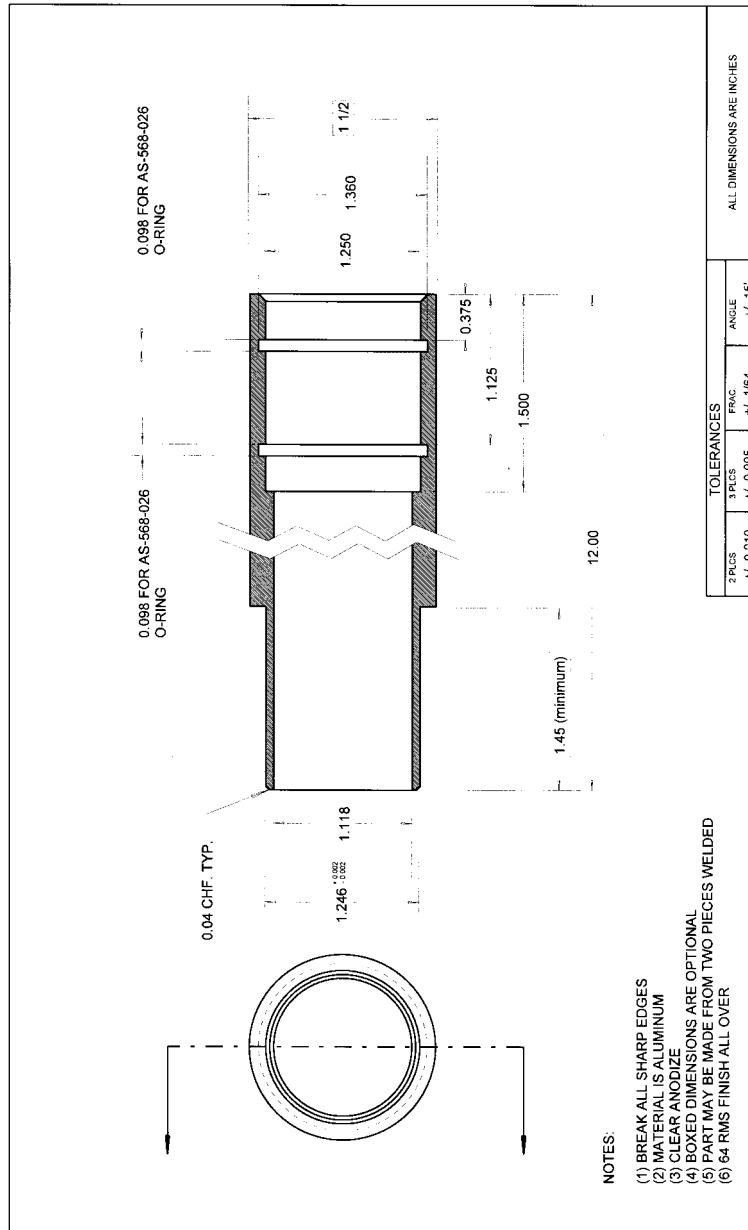


FIGURE L-20. 2.5-MICRON IMPACTOR ASSEMBLY

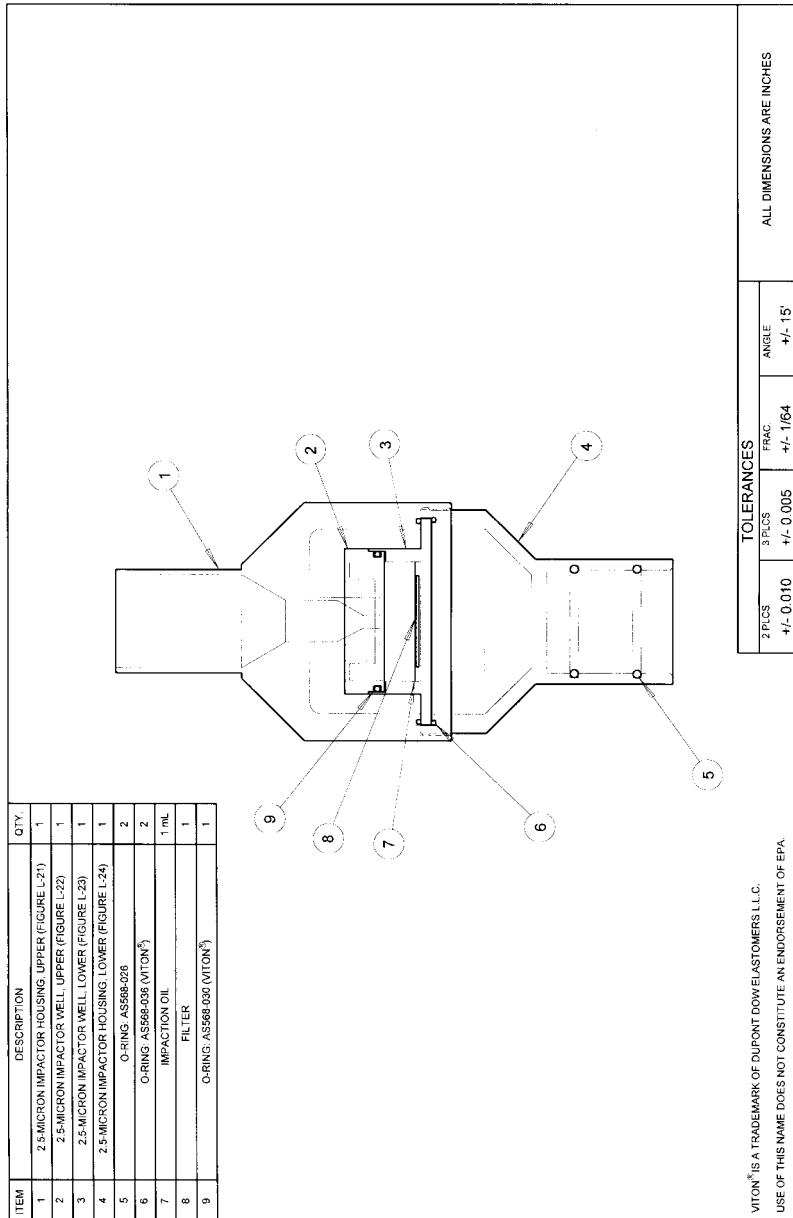


FIGURE L-21. 2.5-MICRON IMPACTOR HOUSING, UPPER

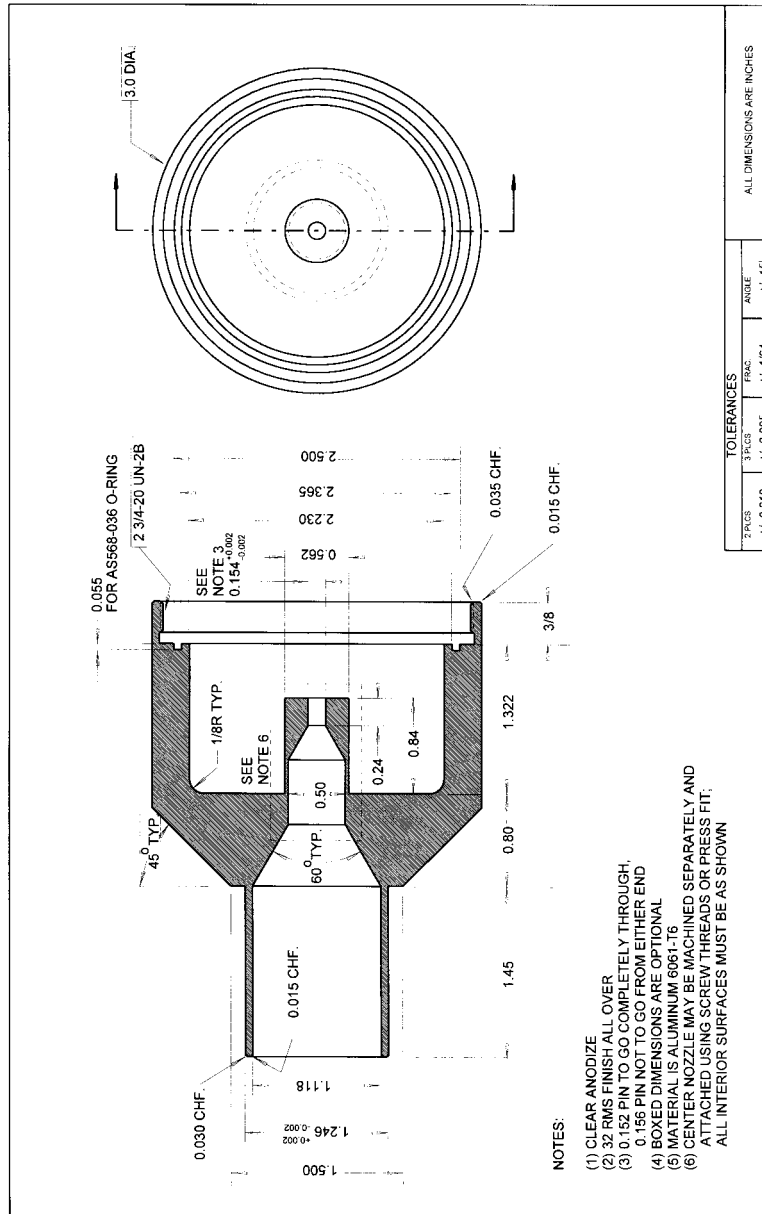


FIGURE L-22. 2.5-MICRON IMPACTOR WELL, UPPER SECTION

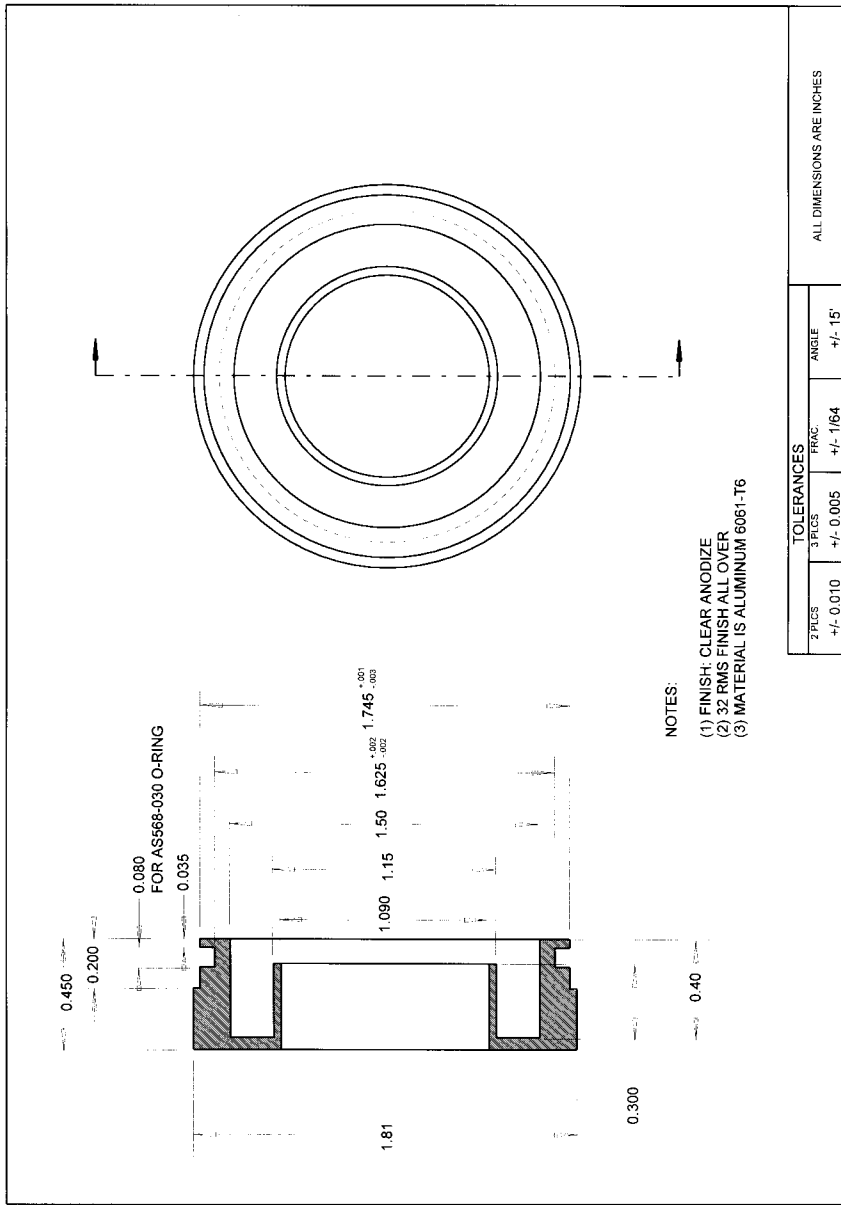


FIGURE L-23. 2.5-MICRON IMPACTOR WELL, LOWER SECTION

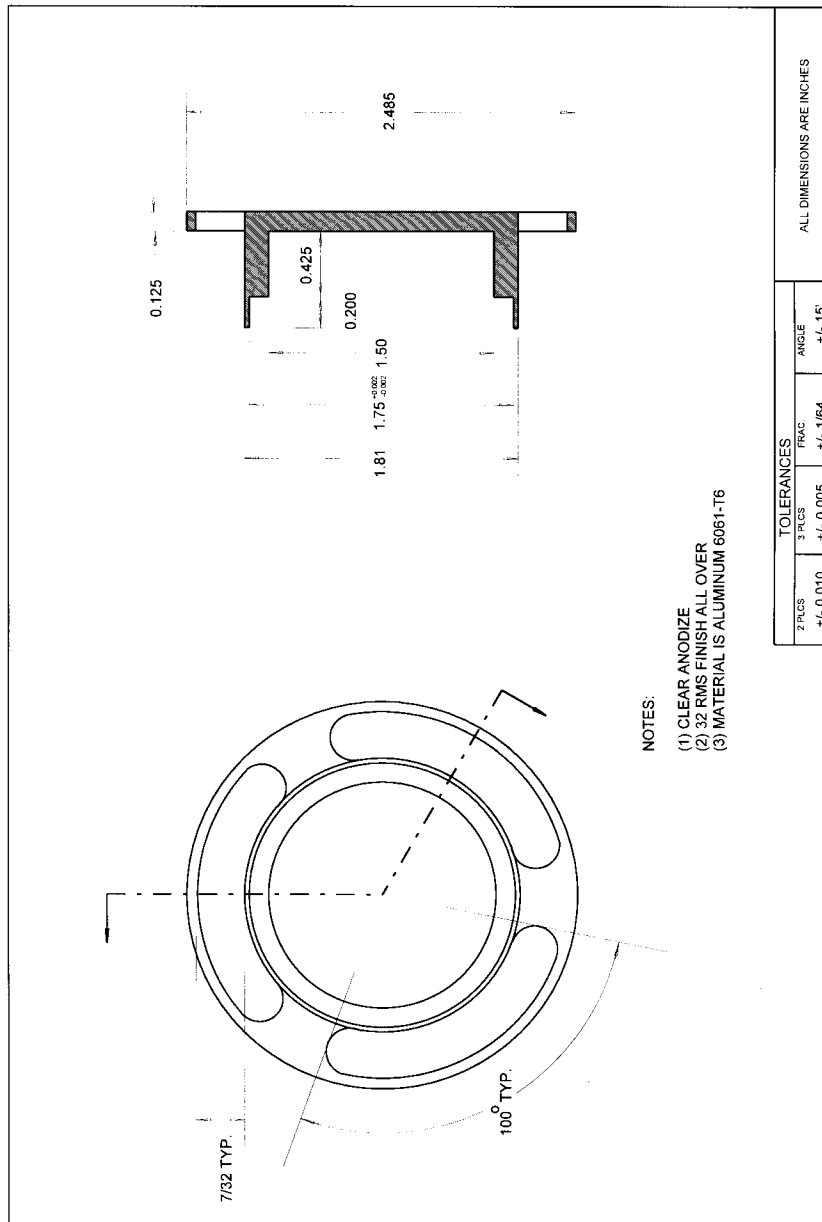


FIGURE L-24. 2.5-MICRON IMPACTOR HOUSING, LOWER

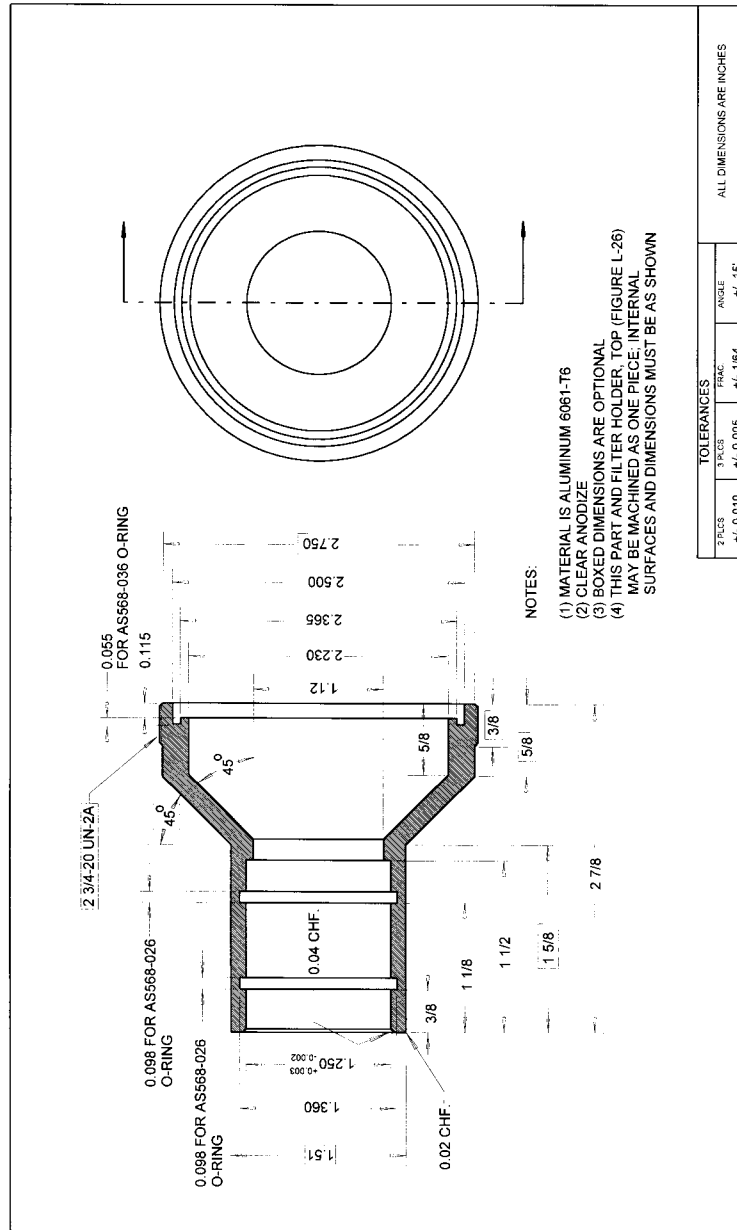


FIGURE L-25. FILTER HOLDER, ASSEMBLY

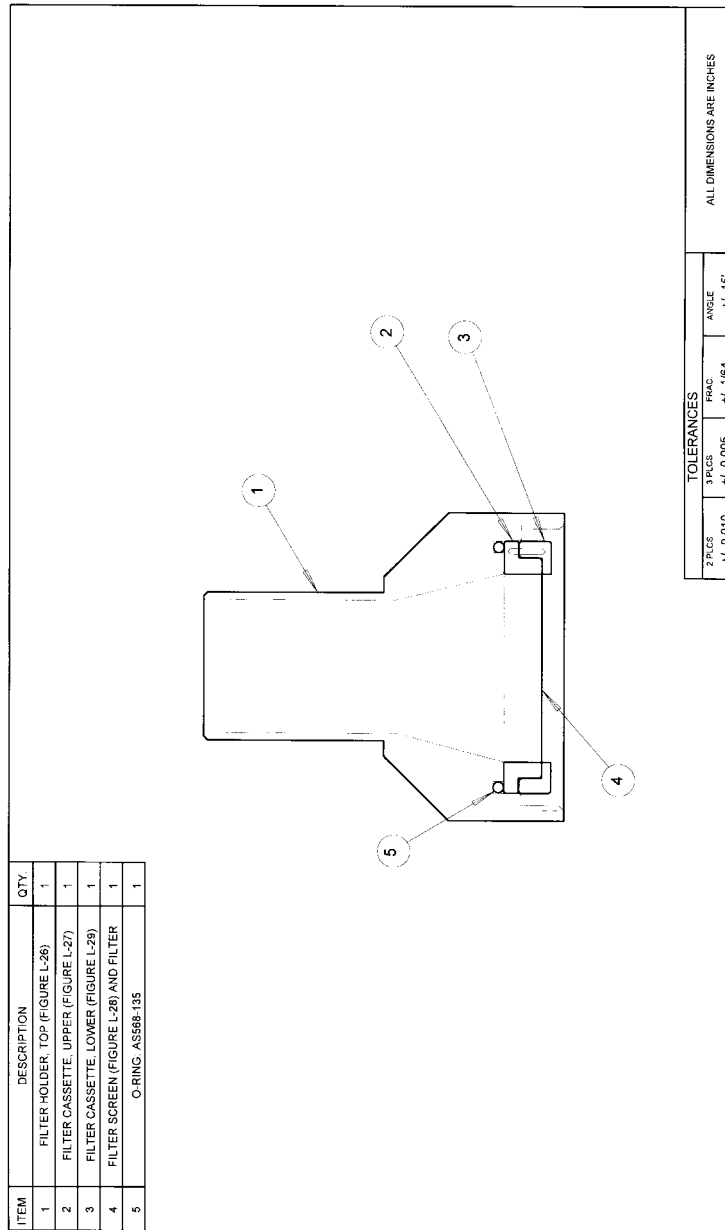


FIGURE L-26. FILTER HOLDER, TOP

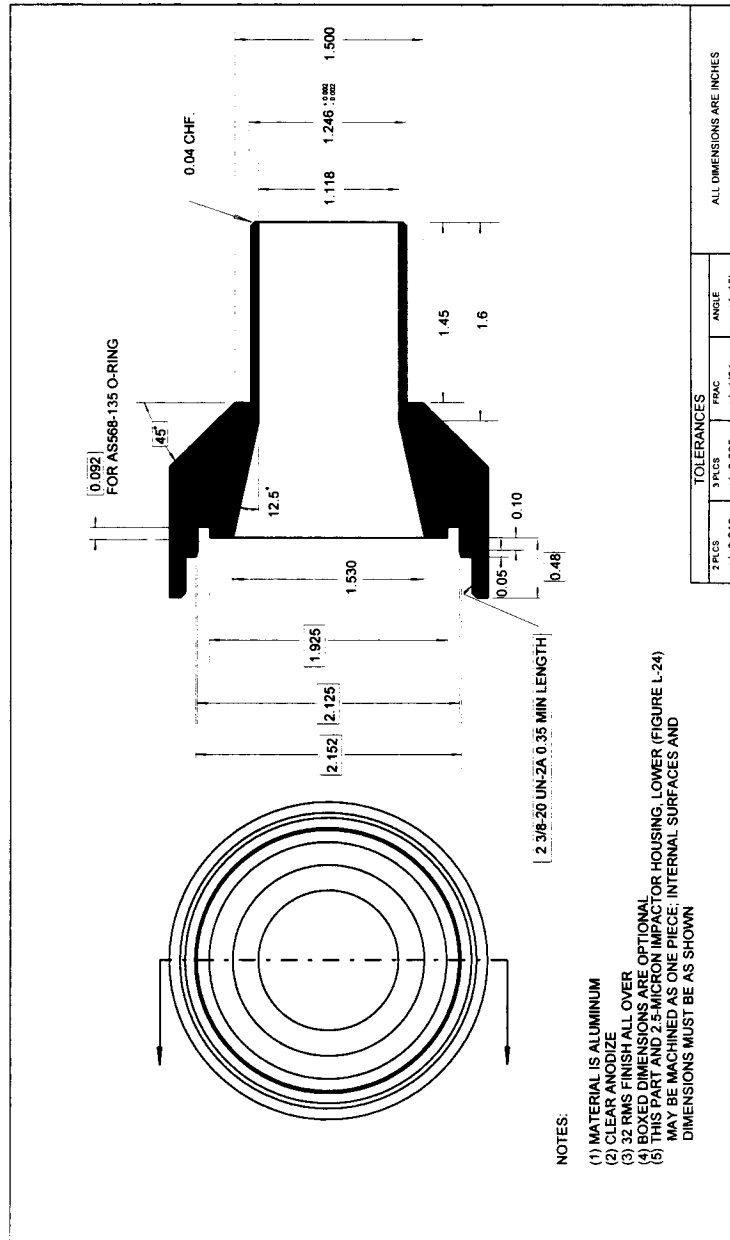


FIGURE L-27. FILTER CASSETTE, UPPER SECTION

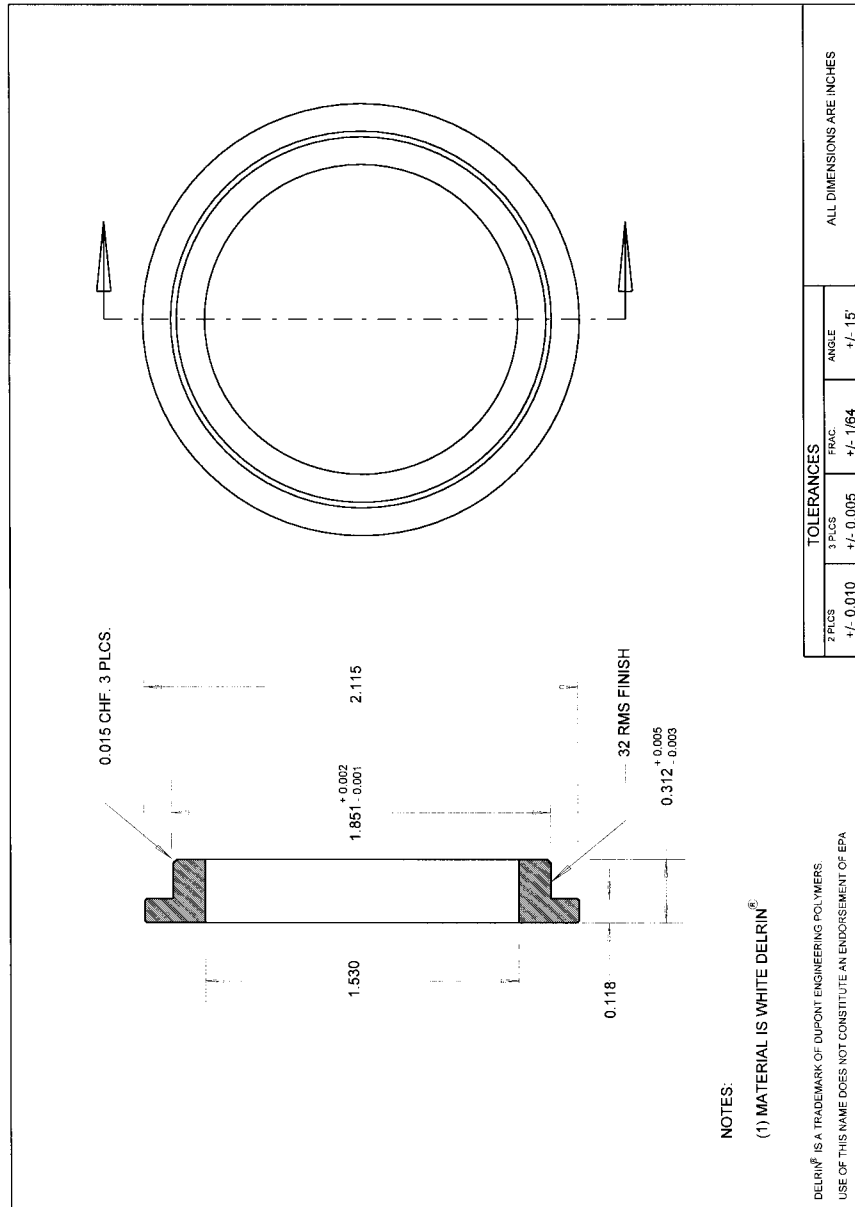
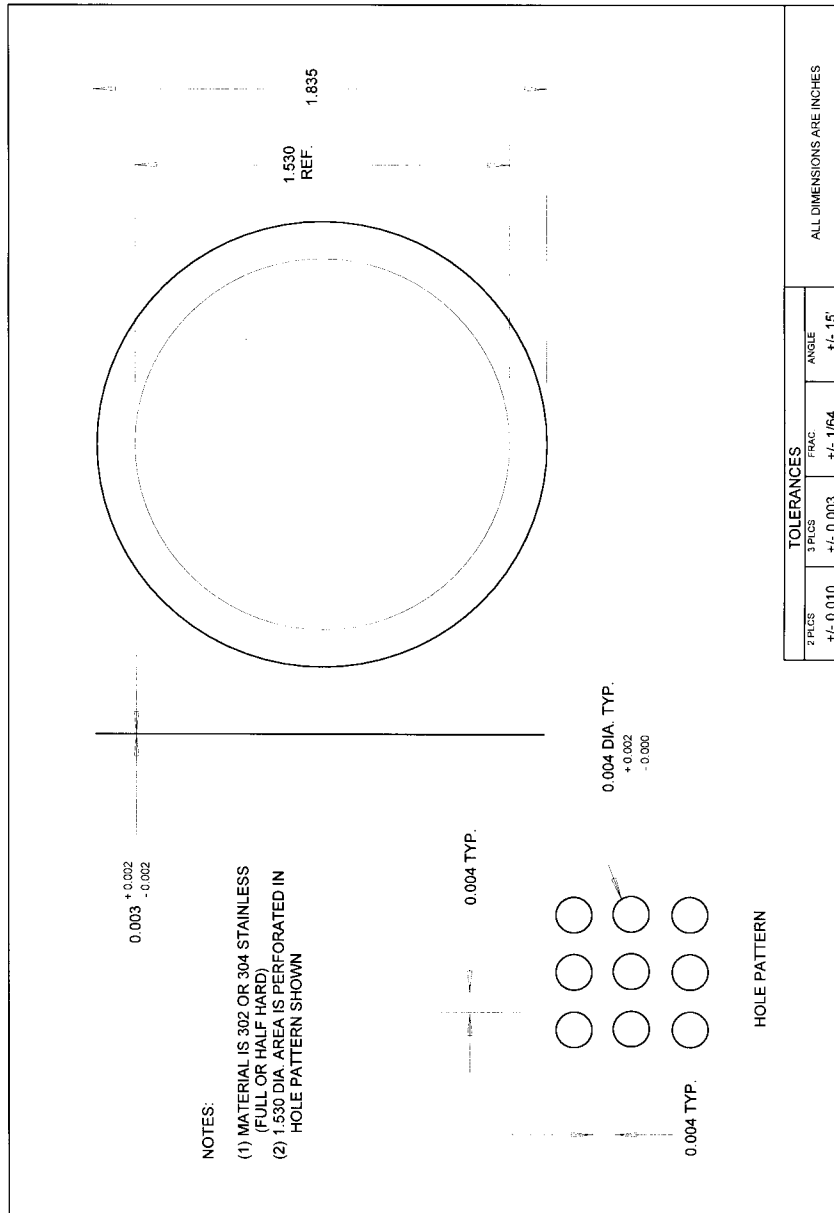


FIGURE L-28. FILTER SCREEN



NOTES:

- (1) MATERIAL IS 302 OR 304 STAINLESS (FULL OR HALF HARD)
- (2) 1.530 DIA. AREA IS PERFORATED IN HOLE PATTERN SHOWN

FIGURE L-29. FILTER CASSETTE, LOWER SECTION

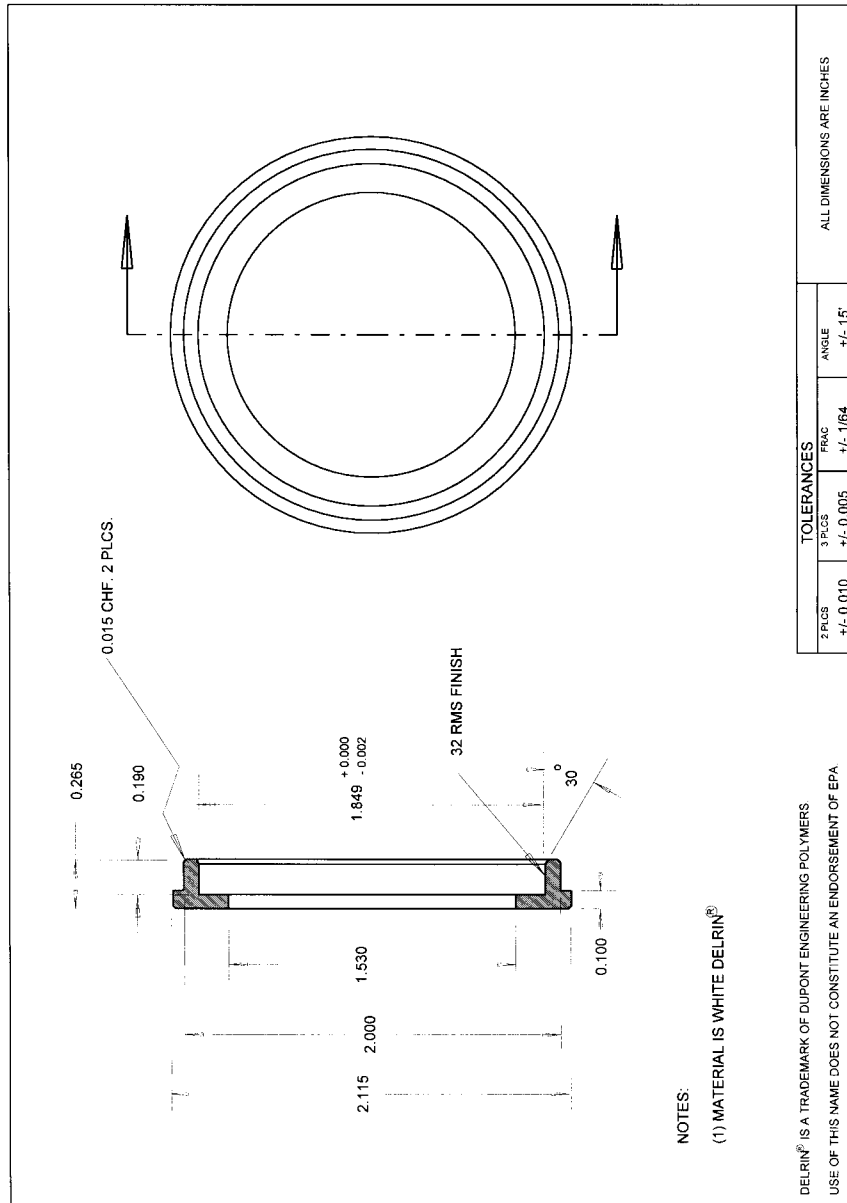
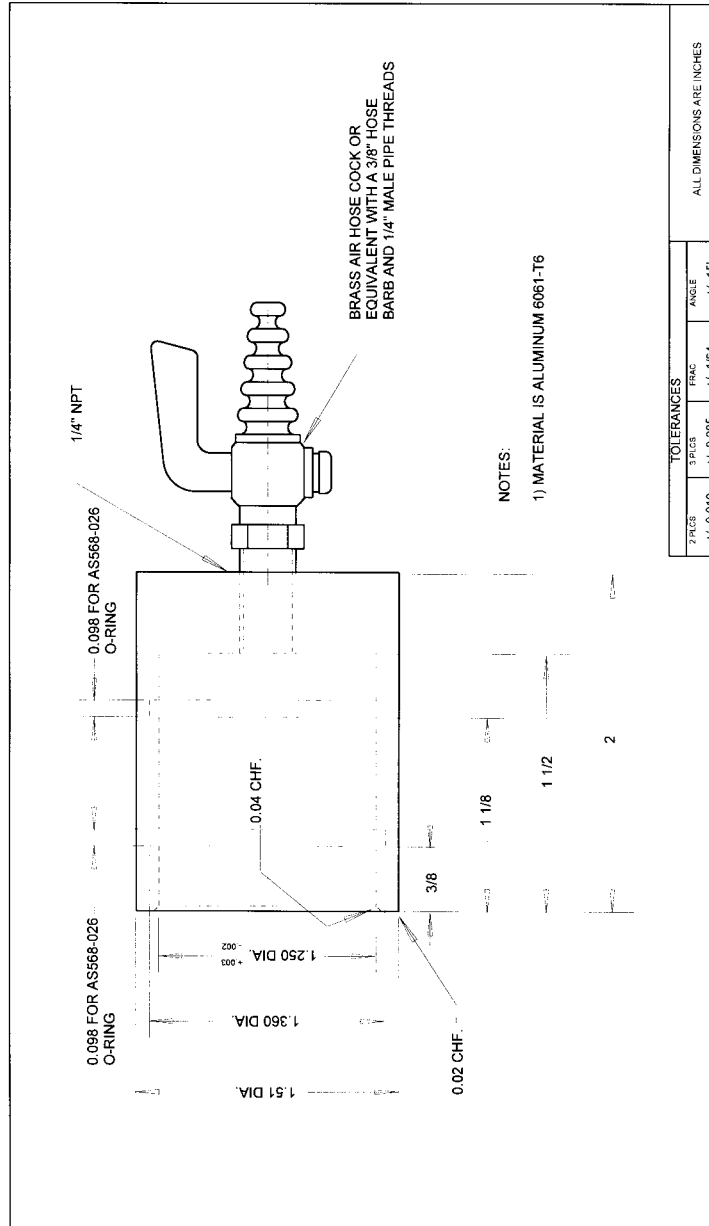


FIGURE L-30. FLOW RATE MEASUREMENT ADAPTER



[62 FR 38714, July 18, 1997, as amended at 64 FR 19719, Apr. 22, 1999]

APPENDIX M TO PART 50—REFERENCE
METHOD FOR THE DETERMINATION OF
PARTICULATE MATTER AS PM₁₀ IN
THE ATMOSPHERE

1.0 *Applicability.*

1.1 This method provides for the measurement of the mass concentration of particulate matter with an aerodynamic diameter less than or equal to a nominal 10 micrometers (PM₁₀) in ambient air over a 24-hour period for purposes of determining attainment and maintenance of the primary and secondary national ambient air quality standards for particulate matter specified in § 50.6 of this chapter. The measurement process is nondestructive, and the PM₁₀ sample can be subjected to subsequent physical or chemical analyses. Quality assurance procedures and guidance are provided in part 58, Appendices A and B of this chapter and in references 1 and 2 of section 12.0 of this appendix.

2.0 *Principle.*

2.1 An air sampler draws ambient air at a constant flow rate into a specially shaped inlet where the suspended particulate matter is inertially separated into one or more size fractions within the PM₁₀ size range. Each size fraction in the PM₁₀ size range is then collected on a separate filter over the specified sampling period. The particle size discrimination characteristics (sampling effectiveness and 50 percent cutpoint) of the sampler inlet are prescribed as performance specifications in part 53 of this chapter.

2.2 Each filter is weighed (after moisture equilibration) before and after use to determine the net weight (mass) gain due to collected PM₁₀. The total volume of air sampled, measured at the actual ambient temperature and pressure, is determined from the measured flow rate and the sampling time. The mass concentration of PM₁₀ in the ambient air is computed as the total mass of collected particles in the PM₁₀ size range divided by the volume of air sampled, and is expressed in micrograms per actual cubic meter (µg/m³).

2.3 A method based on this principle will be considered a reference method only if the associated sampler meets the requirements specified in this appendix and the requirements in part 53 of this chapter, and the method has been designated as a reference method in accordance with part 53 of this chapter.

3.0 *Range.*

3.1 The lower limit of the mass concentration range is determined by the repeatability of filter tare weights, assuming the nominal air sample volume for the sampler. For samplers having an automatic filter-changing mechanism, there may be no upper limit.

For samplers that do not have an automatic filter-changing mechanism, the upper limit is determined by the filter mass loading beyond which the sampler no longer maintains the operating flow rate within specified limits due to increased pressure drop across the loaded filter. This upper limit cannot be specified precisely because it is a complex function of the ambient particle size distribution and type, humidity, filter type, and perhaps other factors. Nevertheless, all samplers should be capable of measuring 24-hour PM₁₀ mass concentrations of at least 300 µg/m³ while maintaining the operating flow rate within the specified limits.

4.0 *Precision.*

4.1 The precision of PM₁₀ samplers must be 5 µg/m³ for PM₁₀ concentrations below 80 µg/m³ and 7 percent for PM₁₀ concentrations above 80 µg/m³, as required by part 53 of this chapter, which prescribes a test procedure that determines the variation in the PM₁₀ concentration measurements of identical samplers under typical sampling conditions. Continual assessment of precision via collocated samplers is required by part 58 of this chapter for PM₁₀ samplers used in certain monitoring networks.

5.0 *Accuracy.*

5.1 Because the size of the particles making up ambient particulate matter varies over a wide range and the concentration of particles varies with particle size, it is difficult to define the absolute accuracy of PM₁₀ samplers. Part 53 of this chapter provides a specification for the sampling effectiveness of PM₁₀ samplers. This specification requires that the expected mass concentration calculated for a candidate PM₁₀ sampler, when sampling a specified particle size distribution, be within ±10 percent of that calculated for an ideal sampler whose sampling effectiveness is explicitly specified. Also, the particle size for 50 percent sampling effectiveness is required to be 10±0.5 micrometers. Other specifications related to accuracy apply to flow measurement and calibration, filter media, analytical (weighing) procedures, and artifact. The flow rate accuracy of PM₁₀ samplers used in certain monitoring networks is required by part 58 of this chapter to be assessed periodically via flow rate audits.

6.0 *Potential Sources of Error.*

6.1 *Volatile Particles.* Volatile particles collected on filters are often lost during shipment and/or storage of the filters prior to the post-sampling weighing³. Although shipment or storage of loaded filters is sometimes unavoidable, filters should be reweighed as soon as practical to minimize these losses.

6.2 *Artifacts.* Positive errors in PM₁₀ concentration measurements may result from retention of gaseous species on filters^{4,5}. Such errors include the retention of sulfur dioxide and nitric acid. Retention of sulfur

dioxide on filters, followed by oxidation to sulfate, is referred to as artifact sulfate formation, a phenomenon which increases with increasing filter alkalinity⁶. Little or no artifact sulfate formation should occur using filters that meet the alkalinity specification in section 7.2.4 of this appendix. Artifact nitrate formation, resulting primarily from retention of nitric acid, occurs to varying degrees on many filter types, including glass fiber, cellulose ester, and many quartz fiber filters^{5, 7, 8, 9, 10}. Loss of true atmospheric particulate nitrate during or following sampling may also occur due to dissociation or chemical reaction. This phenomenon has been observed on Teflon[®] filters⁸ and inferred for quartz fiber filters^{11, 12}. The magnitude of nitrate artifact errors in PM₁₀ mass concentration measurements will vary with location and ambient temperature; however, for most sampling locations, these errors are expected to be small.

6.3 Humidity. The effects of ambient humidity on the sample are unavoidable. The filter equilibration procedure in section 9.0 of this appendix is designed to minimize the effects of moisture on the filter medium.

6.4 Filter Handling. Careful handling of filters between presampling and postsampling weighings is necessary to avoid errors due to damaged filters or loss of collected particles from the filters. Use of a filter cartridge or cassette may reduce the magnitude of these errors. Filters must also meet the integrity specification in section 7.2.3 of this appendix.

6.5 Flow Rate Variation. Variations in the sampler's operating flow rate may alter the particle size discrimination characteristics of the sampler inlet. The magnitude of this error will depend on the sensitivity of the inlet to variations in flow rate and on the particle distribution in the atmosphere during the sampling period. The use of a flow control device, under section 7.1.3 of this appendix, is required to minimize this error.

6.6 Air Volume Determination. Errors in the air volume determination may result from errors in the flow rate and/or sampling time measurements. The flow control device serves to minimize errors in the flow rate determination, and an elapsed time meter, under section 7.1.5 of this appendix, is required to minimize the error in the sampling time measurement.

7.0 Apparatus.

7.1 PM₁₀ Sampler.

7.1.1 The sampler shall be designed to:

(a) Draw the air sample into the sampler inlet and through the particle collection filter at a uniform face velocity.

(b) Hold and seal the filter in a horizontal position so that sample air is drawn downward through the filter.

(c) Allow the filter to be installed and removed conveniently.

(d) Protect the filter and sampler from precipitation and prevent insects and other debris from being sampled.

(e) Minimize air leaks that would cause error in the measurement of the air volume passing through the filter.

(f) Discharge exhaust air at a sufficient distance from the sampler inlet to minimize the sampling of exhaust air.

(g) Minimize the collection of dust from the supporting surface.

7.1.2 The sampler shall have a sample air inlet system that, when operated within a specified flow rate range, provides particle size discrimination characteristics meeting all of the applicable performance specifications prescribed in part 53 of this chapter. The sampler inlet shall show no significant wind direction dependence. The latter requirement can generally be satisfied by an inlet shape that is circularly symmetrical about a vertical axis.

7.1.3 The sampler shall have a flow control device capable of maintaining the sampler's operating flow rate within the flow rate limits specified for the sampler inlet over normal variations in line voltage and filter pressure drop.

7.1.4 The sampler shall provide a means to measure the total flow rate during the sampling period. A continuous flow recorder is recommended but not required. The flow measurement device shall be accurate to ± 2 percent.

7.1.5 A timing/control device capable of starting and stopping the sampler shall be used to obtain a sample collection period of 24 ± 1 hr (1,440 ± 60 min). An elapsed time meter, accurate to within ± 15 minutes, shall be used to measure sampling time. This meter is optional for samplers with continuous flow recorders if the sampling time measurement obtained by means of the recorder meets the ± 15 minute accuracy specification.

7.1.6 The sampler shall have an associated operation or instruction manual as required by part 53 of this chapter which includes detailed instructions on the calibration, operation, and maintenance of the sampler.

7.2 Filters.

7.2.1 **Filter Medium.** No commercially available filter medium is ideal in all respects for all samplers. The user's goals in sampling determine the relative importance of various filter characteristics, e.g., cost, ease of handling, physical and chemical characteristics, etc., and, consequently, determine the choice among acceptable filters. Furthermore, certain types of filters may not be suitable for use with some samplers, particularly under heavy loading conditions (high mass concentrations), because of high or rapid increase in the filter flow resistance that would exceed the capability of the sampler's flow control device. However, samplers equipped with automatic filter-changing

mechanisms may allow use of these types of filters. The specifications given below are minimum requirements to ensure acceptability of the filter medium for measurement of PM₁₀ mass concentrations. Other filter evaluation criteria should be considered to meet individual sampling and analysis objectives.

7.2.2 Collection Efficiency. ≤ 99 percent, as measured by the DOP test (ASTM-2986) with 0.3 μm particles at the sampler's operating face velocity.

7.2.3 Integrity. $\pm 5 \mu\text{g}/\text{m}^3$ (assuming sampler's nominal 24-hour air sample volume). Integrity is measured as the PM₁₀ concentration equivalent corresponding to the average difference between the initial and the final weights of a random sample of test filters that are weighed and handled under actual or simulated sampling conditions, but have no air sample passed through them, i.e., filter blanks. As a minimum, the test procedure must include initial equilibration and weighing, installation on an inoperative sampler, removal from the sampler, and final equilibration and weighing.

7.2.4 Alkalinity. < 25 microequivalents/gram of filter, as measured by the procedure given in reference 13 of section 12.0 of this appendix following at least two months storage in a clean environment (free from contamination by acidic gases) at room temperature and humidity.

7.3 Flow Rate Transfer Standard. The flow rate transfer standard must be suitable for the sampler's operating flow rate and must be calibrated against a primary flow or volume standard that is traceable to the National Institute of Standard and Technology (NIST). The flow rate transfer standard must be capable of measuring the sampler's operating flow rate with an accuracy of ± 2 percent.

7.4 Filter Conditioning Environment.

7.4.1 Temperature range. 15 to 30 C.

7.4.2 Temperature control. ± 3 C.

7.4.3 Humidity range. 20% to 45% RH.

7.4.4 Humidity control. $\pm 5\%$ RH.

7.5 Analytical Balance. The analytical balance must be suitable for weighing the type and size of filters required by the sampler. The range and sensitivity required will depend on the filter tare weights and mass loadings. Typically, an analytical balance with a sensitivity of 0.1 mg is required for high volume samplers (flow rates $> 0.5 \text{ m}^3/\text{min}$). Lower volume samplers (flow rates $< 0.5 \text{ m}^3/\text{min}$) will require a more sensitive balance.

8.0 Calibration.

8.1 General Requirements.

8.1.1 Calibration of the sampler's flow measurement device is required to establish traceability of subsequent flow measurements to a primary standard. A flow rate transfer standard calibrated against a primary flow or volume standard shall be used

to calibrate or verify the accuracy of the sampler's flow measurement device.

8.1.2 Particle size discrimination by inertial separation requires that specific air velocities be maintained in the sampler's air inlet system. Therefore, the flow rate through the sampler's inlet must be maintained throughout the sampling period within the design flow rate range specified by the manufacturer. Design flow rates are specified as actual volumetric flow rates, measured at existing conditions of temperature and pressure (Q_a).

8.2 Flow Rate Calibration Procedure.

8.2.1 PM₁₀ samplers employ various types of flow control and flow measurement devices. The specific procedure used for flow rate calibration or verification will vary depending on the type of flow controller and flow rate indicator employed. Calibration is in terms of actual volumetric flow rates (Q_a) to meet the requirements of section 8.1 of this appendix. The general procedure given here serves to illustrate the steps involved in the calibration. Consult the sampler manufacturer's instruction manual and reference 2 of section 12.0 of this appendix for specific guidance on calibration. Reference 14 of section 12.0 of this appendix provides additional information on various other measures of flow rate and their interrelationships.

8.2.2 Calibrate the flow rate transfer standard against a primary flow or volume standard traceable to NIST. Establish a calibration relationship, e.g., an equation or family of curves, such that traceability to the primary standard is accurate to within 2 percent over the expected range of ambient conditions, i.e., temperatures and pressures, under which the transfer standard will be used. Recalibrate the transfer standard periodically.

8.2.3 Following the sampler manufacturer's instruction manual, remove the sampler inlet and connect the flow rate transfer standard to the sampler such that the transfer standard accurately measures the sampler's flow rate. Make sure there are no leaks between the transfer standard and the sampler.

8.2.4 Choose a minimum of three flow rates (actual m^3/min), spaced over the acceptable flow rate range specified for the inlet, under section 7.1.2 of the appendix, that can be obtained by suitable adjustment of the sampler flow rate. In accordance with the sampler manufacturer's instruction manual, obtain or verify the calibration relationship between the flow rate (actual m^3/min) as indicated by the transfer standard and the sampler's flow indicator response. Record the ambient temperature and barometric pressure. Temperature and pressure corrections to subsequent flow indicator readings may be required for certain types of flow measurement devices. When such corrections are necessary, correction on an individual or

daily basis is preferable. However, seasonal average temperature and average barometric pressure for the sampling site may be incorporated into the sampler calibration to avoid daily corrections. Consult the sampler manufacturer's instruction manual and reference 2 in section 12.0 of this appendix for additional guidance.

8.2.5 Following calibration, verify that the sampler is operating at its design flow rate (actual m³/min) with a clean filter in place.

8.2.6 Replace the sampler inlet.

9.0 *Procedure.*

9.1 The sampler shall be operated in accordance with the specific guidance provided in the sampler manufacturer's instruction manual and in reference 2 in section 12.0 of this appendix. The general procedure given here assumes that the sampler's flow rate calibration is based on flow rates at ambient conditions (Q_a) and serves to illustrate the steps involved in the operation of a PM₁₀ sampler.

9.2 Inspect each filter for pinholes, particles, and other imperfections. Establish a filter information record and assign an identification number to each filter.

9.3 Equilibrate each filter in the conditioning environment (see 7.4) for at least 24 hours.

9.4 Following equilibration, weigh each filter and record the presampling weight with the filter identification number.

9.5 Install a preweighed filter in the sampler following the instructions provided in the sampler manufacturer's instruction manual.

9.6 (a) Turn on the sampler and allow it to establish run-temperature conditions. Record the flow indicator reading and, if needed, the ambient temperature and barometric pressure. Determine the sampler flow rate (actual m³/min) in accordance with the instructions provided in the sampler manufacturer's instruction manual.

(b) Note: No onsite temperature or pressure measurements are necessary if the sampler's flow indicator does not require temperature or pressure corrections or if seasonal average temperature and average barometric pressure for the sampling site are incorporated into the sampler calibration, under section 8.2.4 of this appendix. If individual or daily temperature and pressure corrections are required, ambient temperature and barometric pressure can be obtained by on-site measurements or from a nearby weather station. Barometric pressure readings obtained from airports must be station pressure, not corrected to sea level, and may need to be corrected for differences in elevation between the sampling site and the airport.

9.7 If the flow rate is outside the acceptable range specified by the manufacturer, check for leaks, and if necessary, adjust the

flow rate to the specified setpoint. Stop the sampler.

9.8 Set the timer to start and stop the sampler at appropriate times. Set the elapsed time meter to zero or record the initial meter reading.

9.9 Record the sample information (site location or identification number, sample date, filter identification number, and sampler model and serial number).

9.10 Sample for 24±1 hours.

9.11 Determine and record the average flow rate (\bar{Q}_a) in actual m³/min for the sampling period in accordance with the instructions provided in the sampler manufacturer's instruction manual. Record the elapsed time meter final reading and, if needed, the average ambient temperature and barometric pressure for the sampling period, in note following section 9.6 of this appendix.

9.12 Carefully remove the filter from the sampler, following the sampler manufacturer's instruction manual. Touch only the outer edges of the filter.

9.13 Place the filter in a protective holder or container, e.g., petri dish, glassine envelope, or manila folder.

9.14 Record any factors such as meteorological conditions, construction activity, fires or dust storms, etc., that might be pertinent to the measurement on the filter information record.

9.15 Transport the exposed sample filter to the filter conditioning environment as soon as possible for equilibration and subsequent weighing.

9.16 Equilibrate the exposed filter in the conditioning environment for at least 24 hours under the same temperature and humidity conditions used for presampling filter equilibration (see section 9.3 of this appendix).

9.17 Immediately after equilibration, reweigh the filter and record the postsampling weight with the filter identification number.

10.0 *Sampler Maintenance.*

10.1 The PM₁₀ sampler shall be maintained in strict accordance with the maintenance procedures specified in the sampler manufacturer's instruction manual.

11.0 *Calculations.*

11.1 Calculate the total volume of air sampled as:

$$V = Q_a t$$

where:

V = total air sampled, at ambient temperature and pressure, m³;

Q_a = average sample flow rate at ambient temperature and pressure, m³/min; and

t = sampling time, min.

11.2 (a) Calculate the PM₁₀ concentration as:

$$PM_{10} = (W_f - W_i) \times 10^6 / V$$

where:

PM₁₀ = mass concentration of PM₁₀, µg/m³;
 W_f, W_i = final and initial weights of filter collecting PM₁₀ particles, g; and
 10⁶ = conversion of g to µg.

(b) Note: If more than one size fraction in the PM₁₀ size range is collected by the sampler, the sum of the net weight gain by each collection filter [Σ(W_f - W_i)] is used to calculate the PM₁₀ mass concentration.

12.0 References.

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APPENDIX N TO PART 50—INTERPRETATION OF THE NATIONAL AMBIENT AIR QUALITY STANDARDS FOR PARTICULATE MATTER

1.0 General.

(a) This appendix explains the data handling conventions and computations necessary for determining when the annual and 24-hour primary and secondary national ambient air quality standards for PM specified in §50.7 of this chapter are met. Particulate matter is measured in the ambient air as PM₁₀ and PM_{2.5} (particles with an aerodynamic diameter less than or equal to a nominal 10 and 2.5 micrometers, respectively) by a reference method based on appendix M of this part for PM₁₀ and on appendix L of this part for PM_{2.5}, as applicable, and designated in accordance with part 53 of this chapter, or by an equivalent method designated in accordance with part 53 of this chapter. Data handling and computation procedures to be used in making comparisons between reported PM₁₀ and PM_{2.5} concentrations and the levels of the PM standards are specified in the following sections.

(b) Data resulting from uncontrollable or natural events, for example structural fires or high winds, may require special consideration. In some cases, it may be appropriate to exclude these data because they could result in inappropriate values to compare with the levels of the PM standards. In other cases, it may be more appropriate to retain the data for comparison with the level of the PM standards and then allow the EPA to formulate the appropriate regulatory response. Whether to exclude, retain, or make adjustments to the data affected by uncontrollable or natural events is subject to the approval of the appropriate Regional Administrator.

(c) The terms used in this appendix are defined as follows:

Average and *mean* refer to an arithmetic mean.

Daily value for PM refers to the 24-hour average concentration of PM calculated or measured from midnight to midnight (local time) for PM₁₀ or PM_{2.5}.

Designated monitors are those monitoring sites designated in a State PM Monitoring Network Description for spatial averaging in areas opting for spatial averaging in accordance with part 58 of this chapter.

98th percentile (used for PM_{2.5}) means the daily value out of a year of monitoring data below which 98 percent of all values in the group fall.

99th percentile (used for PM₁₀) means the daily value out of a year of monitoring data below which 99 percent of all values in the group fall.

Year refers to a calendar year.

(d) Sections 2.1 and 2.5 of this appendix contain data handling instructions for the option of using a spatially averaged network of monitors for the annual standard. If spatial averaging is not considered for an area, then the spatial average is equivalent to the annual average of a single site and is treated accordingly in subsequent calculations. For example, paragraph (a)(3) of section 2.1 of this appendix could be eliminated since the spatial average would be equivalent to the annual average.

2.0 Comparisons with the PM_{2.5} Standards.

2.1 Annual PM_{2.5} Standard.

(a) The annual PM_{2.5} standard is met when the 3-year average of the spatially averaged annual means is less than or equal to 15.0 µg/m³. The 3-year average of the spatially averaged annual means is determined by averaging quarterly means at each monitor to obtain the annual mean PM_{2.5} concentrations at each monitor, then averaging across all designated monitors, and finally averaging for 3 consecutive years. The steps can be summarized as follows:

- (1) Average 24-hour measurements to obtain quarterly means at each monitor.
- (2) Average quarterly means to obtain annual means at each monitor.
- (3) Average across designated monitoring sites to obtain an annual spatial mean for an area (this can be one site in which case the spatial mean is equal to the annual mean).
- (4) Average 3 years of annual spatial means to obtain a 3-year average of spatially averaged annual means.

(b) In the case of spatial averaging, 3 years of spatial averages are required to demonstrate that the standard has been met. Designated sites with less than 3 years of data shall be included in spatial averages for those years that data completeness requirements are met. For the annual PM_{2.5} standard, a year meets data completeness requirements when at least 75 percent of the scheduled sampling days for each quarter have valid data. However, years with high concentrations and more than a minimal amount of data (at least 11 samples in each quarter) shall not be ignored just because they are comprised of quarters with less than complete data. Thus, in computing annual spatially averaged means, years containing quarters with at least 11 samples but less than 75 percent data completeness shall be included in the computation if the resulting spatially averaged annual mean concentration (rounded according to the conven-

tions of section 2.3 of this appendix) is greater than the level of the standard.

(c) Situations may arise in which there are compelling reasons to retain years containing quarters which do not meet the data completeness requirement of 75 percent or the minimum number of 11 samples. The use of less than complete data is subject to the approval of the appropriate Regional Administrator.

(d) The equations for calculating the 3-year average annual mean of the PM_{2.5} standard are given in section 2.5 of this appendix.

2.2 24-Hour PM_{2.5} Standard.

(a) The 24-hour PM_{2.5} standard is met when the 3-year average of the 98th percentile values at each monitoring site is less than or equal to 65 µg/m³. This comparison shall be based on 3 consecutive, complete years of air quality data. A year meets data completeness requirements when at least 75 percent of the scheduled sampling days for each quarter have valid data. However, years with high concentrations shall not be ignored just because they are comprised of quarters with less than complete data. Thus, in computing the 3-year average 98th percentile value, years containing quarters with less than 75 percent data completeness shall be included in the computation if the annual 98th percentile value (rounded according to the conventions of section 2.3 of this appendix) is greater than the level of the standard.

(b) Situations may arise in which there are compelling reasons to retain years containing quarters which do not meet the data completeness requirement. The use of less than complete data is subject to the approval of the appropriate Regional Administrator.

(c) The equations for calculating the 3-year average of the annual 98th percentile values is given in section 2.6 of this appendix.

2.3 *Rounding Conventions.* For the purposes of comparing calculated values to the applicable level of the standard, it is necessary to round the final results of the calculations described in sections 2.5 and 2.6 of this appendix. For the annual PM_{2.5} standard, the 3-year average of the spatially averaged annual means shall be rounded to the nearest 0.1 µg/m³ (decimals 0.05 and greater are rounded up to the next 0.1, and any decimal lower than 0.05 is rounded down to the nearest 0.1). For the 24-hour PM_{2.5} standard, the 3-year average of the annual 98th percentile values shall be rounded to the nearest 1 µg/m³ (decimals 0.5 and greater are rounded up to nearest whole number, and any decimal lower than 0.5 is rounded down to the nearest whole number).

2.4 *Monitoring Considerations.*

(a) Section 58.13 of this chapter specifies the required minimum frequency of sampling

for PM_{2.5}. Exceptions to the specified sampling frequencies, such as a reduced frequency during a season of expected low concentrations, are subject to the approval of the appropriate Regional Administrator. Section 58.14 of 40 CFR part 58 and section 2.8 of appendix D of 40 CFR part 58, specify which monitors are eligible for making comparisons with the PM standards. In determining a spatial mean using two or more monitoring sites operating in a given year, the annual mean for an individual site may be included in the spatial mean if and only if the mean for that site meets the criterion specified in §2.8 of appendix D of 40 CFR part 58. In the event data from an otherwise eligible site is excluded from being averaged with data from other sites on the basis of this criterion, then the 3-year mean from that site shall be compared directly to the annual standard.

(b) For the annual PM_{2.5} standard, when designated monitors are located at the same site and are reporting PM_{2.5} values for the same time periods, and when spatial averaging has been chosen, their concentrations shall be averaged before an area-wide spatial average is calculated. Such monitors will then be considered as one monitor.

2.5 Equations for the Annual PM_{2.5} Standard.

(a) An annual mean value for PM_{2.5} is determined by first averaging the daily values of a calendar quarter:

Equation 1

$$\bar{x}_{q,y,s} = \frac{1}{n_q} \sum_{i=1}^{n_q} x_{i,q,y,s}$$

where:

$\bar{x}_{q,y,s}$ = the mean for quarter q of year y for site s;

n_q = the number of monitored values in the quarter; and

$x_{i,q,y,s}$ = the *i*th value in quarter q for year y for site s.

(b) The following equation is then to be used for calculation of the annual mean:

Equation 2

$$\bar{x}_{y,s} = \frac{1}{4} \sum_{q=1}^4 \bar{x}_{q,y,s}$$

where:

$\bar{x}_{y,s}$ = the annual mean concentration for year y (y = 1, 2, or 3) and for site s; and

$\bar{x}_{q,y,s}$ = the mean for quarter q of year y for site s.

(c)(1) The spatially averaged annual mean for year y is computed by first calculating the annual mean for each site designated to be included in a spatial average, $\bar{x}_{y,s}$, and

then computing the average of these values across sites:

Equation 3

$$\bar{x}_y = \frac{1}{n_s} \sum_{s=1}^{n_s} \bar{x}_{y,s}$$

where:

\bar{x}_y = the spatially averaged mean for year y;

$\bar{x}_{y,s}$ = the annual mean for year y and site s; and

n_s = the number of sites designated to be averaged.

(2) In the event that an area designated for spatial averaging has two or more sites at the same location producing data for the same time periods, the sites are averaged together before using Equation 3 by:

Equation 4

$$\bar{x}_{y,s^*} = \frac{1}{n_c} \sum_{s=1}^{n_c} \bar{x}_{y,s}$$

where:

\bar{x}_{y,s^*} = the annual mean for year y for the sites at the same location (which will now be considered one site);

n_c = the number of sites at the same location designated to be included in the spatial average; and

$\bar{x}_{y,s}$ = the annual mean for year y and site s.

(d) The 3-year average of the spatially averaged annual means is calculated by using the following equation:

Equation 5

$$\bar{x} = \frac{1}{3} \sum_{y=1}^3 \bar{x}_y$$

where:

\bar{x} = the 3-year average of the spatially averaged annual means; and

\bar{x}_y = the spatially averaged annual mean for year y.

Example 1—Area Designated for Spatial Averaging That Meets the Primary Annual PM_{2.5} Standard.

a. In an area designated for spatial averaging, four designated monitors recorded data in at least 1 year of a particular 3-year period. Using Equations 1 and 2, the annual means for PM_{2.5} at each site are calculated for each year. The following table can be created from the results. Data completeness percentages for the quarter with the fewest number of samples are also shown.

TABLE 1—RESULTS FROM EQUATIONS 1 AND 2

		Site 11	Site 12	Site 13	Site 14	Spatial mean
Year 1	Annual mean (µg/m ³)	12.7	12.7
	% data completeness	80	0	0	0
Year 2	Annual mean (µg/m ³)	12.6	17.5	15.2	15.05
	% data completeness	90	63	38	0
Year 3	Annual mean (µg/m ³)	12.5	18.5	14.1	16.9	15.50
	% data completeness	90	80	85	50
3-year mean	14.42

b. The data from these sites are averaged in the order described in section 2.1 of this appendix. Note that the annual mean from site #3 in year 2 and the annual mean from site #4 in year 3 do not meet the 75 percent data completeness criteria. Assuming the 38 percent data completeness represents a quarter with fewer than 11 samples, site #3 in year 2 does not meet the minimum data completeness requirement of 11 samples in each quarter. The site is therefore excluded from the calculation of the spatial mean for year 2. However, since the spatial mean for year 3 is above the level of the standard and the minimum data requirement of 11 samples in each quarter has been met, the annual mean from site #4 in year 3 is included in the cal-

ulation of the spatial mean for year 3 and in the calculation of the 3-year average. The 3-year average is rounded to 14.4 µg/m³, indicating that this area meets the annual PM_{2.5} standard.

Example 2—Area With Two Monitors at the Same Location That Meets the Primary Annual PM_{2.5} Standard.

a. In an area designated for spatial averaging, six designated monitors, with two monitors at the same location (#5 and #6), recorded data in a particular 3-year period. Using Equations 1 and 2, the annual means for PM_{2.5} are calculated for each year. The following table can be created from the results.

TABLE 2—RESULTS FROM EQUATIONS 1 AND 2

Annual mean (µg/m ³)	Site 11	Site 12	Site 13	Site 14	Site 15	Site 16	Average of 15 and 16	Spatial mean
Year 1	12.9	9.9	12.6	11.1	14.5	14.6	14.55	12.21
Year 2	14.5	13.3	12.2	10.9	16.1	16.0	16.05	13.39
Year 3	14.4	12.4	11.5	9.7	12.3	12.1	12.20	12.04
3-Year mean	12.55

b. The annual means for sites #5 and #6 are averaged together using Equation 4 before the spatial average is calculated using Equation 3 since they are in the same location. The 3-year mean is rounded to 12.6 µg/m³, indicating that this area meets the annual PM_{2.5} standard.

Example 3—Area With a Single Monitor That Meets the Primary Annual PM_{2.5} Standard.

a. Given data from a single monitor in an area, the calculations are as follows. Using Equations 1 and 2, the annual means for PM_{2.5} are calculated for each year. If the annual means are 10.28, 17.38, and 12.25 µg/m³, then the 3-year mean is:

$$\bar{x} = (1/3) \times (10.28 + 17.38 + 12.25) = 13.303 \text{ } \mu\text{g}/\text{m}^3.$$

b. This value is rounded to 13.3, indicating that this area meets the annual PM_{2.5} standard.

2.6 Equations for the 24-Hour PM_{2.5} Standard.

(a) When the data for a particular site and year meet the data completeness requirements in section 2.2 of this appendix, calculation of the 98th percentile is accom-

plished by the following steps. All the daily values from a particular site and year comprise a series of values (x₁, x₂, x₃, ..., x_n), that can be sorted into a series where each number is equal to or larger than the preceding number (x_[1], x_[2], x_[3], ..., x_[n]). In this case, x_[1] is the smallest number and x_[n] is the largest value. The 98th percentile is found from the

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sorted series of daily values which is ordered from the lowest to the highest number. Compute $(0.98) \times (n)$ as the number “i.d”, where “i” is the integer part of the result and “d” is the decimal part of the result. The 98th percentile value for year y, $P_{0.98, y}$, is given by Equation 6:

Equation 6

$$P_{0.98,y} = X_{[i+1]}$$

where:

$P_{0.98,y}$ = 98th percentile for year y;
 $X_{[i+1]}$ = the (i+1)th number in the ordered series of numbers; and
 i = the integer part of the product of 0.98 and n.

(b) The 3-year average 98th percentile is then calculated by averaging the annual 98th percentiles:

Equation 7

$$P_{0.98} = \frac{\sum_{y=1}^3 P_{0.98,y}}{3}$$

(c) The 3-year average 98th percentile is rounded according to the conventions in section 2.3 of this appendix before a comparison with the standard is made.

Example 4—Ambient Monitoring Site With Every-Day Sampling That Meets the Primary 24-Hour PM_{2.5} Standard.

a. In each year of a particular 3 year period, varying numbers of daily PM_{2.5} values (e.g., 281, 304, and 296) out of a possible 365 values were recorded at a particular site with the following ranked values (in $\mu\text{g}/\text{m}^3$):

TABLE 3—ORDERED MONITORING DATA FOR 3 YEARS

Year 1		Year 2		Year 3	
j rank	X _j value	j rank	X _j value	j rank	X _j value
275	57.9	296	54.3	290	66.0
276	59.0	297	57.1	291	68.4
277	62.2	298	63.0	292	69.8

b. Using Equation 6, the 98th percentile values for each year are calculated as follows:

$$0.98 \times 281 = 275.38 \Rightarrow i + 1 = 276 \Rightarrow P_{0.98,1} = X_{[276]} = 59.0 \mu\text{g} / \text{m}^3$$

$$0.98 \times 304 = 297.92 \Rightarrow i + 1 = 298 \Rightarrow P_{0.98,2} = X_{[298]} = 63.0 \mu\text{g} / \text{m}^3$$

$$0.98 \times 296 = 290.07 \Rightarrow i + 1 = 291 \Rightarrow P_{0.98,3} = X_{[291]} = 68.4 \mu\text{g} / \text{m}^3$$

c.1. Using Equation 7, the 3-year average 98th percentile is calculated as follows:

$$P_{0.98} = \frac{59.0 + 63.0 + 68.4}{3} = 63.46 \mu\text{g} / \text{m}^3, \text{ which rounds to } 63 \mu\text{g} / \text{m}^3.$$

2. Therefore, this site meets the 24-hour PM_{2.5} standard.

3.0 *Comparisons with the PM₁₀ Standards.*

3.1 *Annual PM₁₀ Standard.*

(a) The annual PM₁₀ standard is met when the 3-year average of the annual mean PM₁₀ concentrations at each monitoring site is less than or equal to 50 $\mu\text{g}/\text{m}^3$. The 3-year av-

erage of the annual means is determined by averaging quarterly means to obtain annual mean PM₁₀ concentrations for 3 consecutive, complete years at each monitoring site. The steps can be summarized as follows:

(1) Average 24-hour measurements to obtain a quarterly mean.

(2) Average quarterly means to obtain an annual mean.

(3) Average annual means to obtain a 3-year mean.

(b) For the annual PM₁₀ standard, a year meets data completeness requirements when at least 75 percent of the scheduled sampling days for each quarter have valid data. However, years with high concentrations and more than a minimal amount of data (at least 11 samples in each quarter) shall not be ignored just because they are comprised of quarters with less than complete data. Thus, in computing the 3-year average annual mean concentration, years containing quarters with at least 11 samples but less than 75 percent data completeness shall be included in the computation if the annual mean concentration (rounded according to the conventions of section 2.3 of this appendix) is greater than the level of the standard.

(c) Situations may arise in which there are compelling reasons to retain years containing quarters which do not meet the data completeness requirement of 75 percent or the minimum number of 11 samples. The use of less than complete data is subject to the approval of the appropriate Regional Administrator.

(d) The equations for calculating the 3-year average annual mean of the PM₁₀ standard are given in section 3.5 of this appendix.

3.2 24-Hour PM₁₀ Standard.

(a) The 24-hour PM₁₀ standard is met when the 3-year average of the annual 99th percentile values at each monitoring site is less than or equal to 150 µg/m³. This comparison shall be based on 3 consecutive, complete years of air quality data. A year meets data completeness requirements when at least 75 percent of the scheduled sampling days for each quarter have valid data. However, years with high concentrations shall not be ignored just because they are comprised of quarters with less than complete data. Thus, in computing the 3-year average of the annual 99th percentile values, years containing quarters with less than 75 percent data completeness shall be included in the computation if the annual 99th percentile value (rounded according to the conventions of section 2.3 of this appendix) is greater than the level of the standard.

(b) Situations may arise in which there are compelling reasons to retain years containing quarters which do not meet the data completeness requirement. The use of less than complete data is subject to the approval of the appropriate Regional Administrator.

(c) The equation for calculating the 3-year average of the annual 99th percentile values is given in section 2.6 of this appendix.

3.3 *Rounding Conventions.* For the annual PM₁₀ standard, the 3-year average of the annual PM₁₀ means shall be rounded to the nearest 1 µg/m³ (decimals 0.5 and greater are

rounded up to the next whole number, and any decimal less than 0.5 is rounded down to the nearest whole number). For the 24-hour PM₁₀ standard, the 3-year average of the annual 99th percentile values of PM₁₀ shall be rounded to the nearest 10 µg/m³ (155 µg/m³ and greater would be rounded to 160 µg/m³ and 154 µg/m³ and less would be rounded to 150 µg/m³).

3.4 *Monitoring Considerations.* Section 58.13 of this chapter specifies the required minimum frequency of sampling for PM₁₀. Exceptions to the specified sampling frequencies, such as a reduced frequency during a season of expected low concentrations, are subject to the approval of the appropriate Regional Administrator. For making comparisons with the PM₁₀ NAAQS, all sites meeting applicable requirements in part 58 of this chapter would be used.

3.5 Equations for the Annual PM₁₀ Standard.

(a) An annual arithmetic mean value for PM₁₀ is determined by first averaging the 24-hour values of a calendar quarter using the following equation:

Equation 8

$$\bar{x}_{q,y} = \frac{1}{n_q} \sum_{i=1}^{n_q} x_{i,q,y}$$

where:

$\bar{x}_{q,y}$ = the mean for quarter q of year y;

n_q = the number of monitored values in the quarter; and

$x_{i,q,y}$ = the ith value in quarter q for year y.

(b) The following equation is then to be used for calculation of the annual mean:

Equation 9

$$\bar{x}_y = \frac{1}{4} \sum_{q=1}^4 \bar{x}_{q,y}$$

where:

\bar{x}_y = the annual mean concentration for year y. (y=1, 2, or 3); and

$\bar{x}_{q,y}$ = the mean for a quarter q of year y.

(c) The 3-year average of the annual means is calculated by using the following equation:

Equation 10

$$\bar{x} = \frac{1}{3} \sum_{y=1}^3 \bar{x}_y$$

where:

\bar{x} = the 3-year average of the annual means; and

\bar{x}_y = the annual mean for calendar year y.

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Example 5—Ambient Monitoring Site That Does Not Meet the Annual PM₁₀ Standard.

a. Given data from a PM₁₀ monitor and using Equations 8 and 9, the annual means

for PM₁₀ are calculated for each year. If the annual means are 52.42, 82.17, and 63.23 µg/m³, then the 3-year average annual mean is:

$$\bar{x} = (1/3) \times (52.42 + 82.17 + 63.23) = 65.94, \text{ which is rounded to } 66 \mu\text{g} / \text{m}^3.$$

b. Therefore, this site does not meet the annual PM₁₀ standard.

3.6 Equation for the 24-Hour PM₁₀ Standard.

(a) When the data for a particular site and year meet the data completeness requirements in section 3.2 of this appendix, calculation of the 99th percentile is accomplished by the following steps. All the daily values from a particular site and year comprise a series of values (x₁, x₂, x₃, ..., x_n) that can be sorted into a series where each number is equal to or larger than the preceding number (x_[1], x_[2], x_[3], ..., x_[n]). In this case, x_[1] is the smallest number and x_[n] is the largest value. The 99th percentile is found from the sorted series of daily values which is ordered from the lowest to the highest number. Compute (0.99) × (n) as the number “i.d”, where “i” is the integer part of the result and “d” is the decimal part of the result. The 99th percentile value for year y, P_{0.99,y}, is given by Equation 11:

Equation 11

$$P_{0.99,y} = X_{[i+1]}$$

where:

P_{0.99,y} = the 99th percentile for year y;

x_[i+1] = the (i+1)th number in the ordered series of numbers; and

i = the integer part of the product of 0.99 and n.

(b) The 3-year average 99th percentile value is then calculated by averaging the annual 99th percentiles:

Equation 12

$$P_{0.99} = \frac{\sum_{y=1}^3 P_{0.99,y}}{3}$$

(c) The 3-year average 99th percentile is rounded according to the conventions in section 3.3 of this appendix before a comparison with the standard is made.

Example 6—Ambient Monitoring Site With Sampling Every Sixth Day That Meets the Primary 24-Hour PM₁₀ Standard.

a. In each year of a particular 3 year period, varying numbers of PM₁₀ daily values (e.g., 110, 98, and 100) out of a possible 121 daily values were recorded at a particular site with the following ranked values (in µg/m³):

TABLE 4—ORDERED MONITORING DATA FOR 3 YEARS

Year 1		Year 2		Year 3	
j rank	X _j value	j rank	X _j value	j rank	X _j value
108	120	96	143	98	140
109	128	97	148	99	144
110	130	98	150	100	147

b. Using Equation 11, the 99th percentile values for each year are calculated as follows:

$$0.99 \times 110 = 108.9 \Rightarrow i + 1 = 109 \Rightarrow P_{0.99,1} = X_{[109]} = 128 \mu\text{g} / \text{m}^3$$

$$0.99 \times 98 = 97.02 \Rightarrow i + 1 = 98 \Rightarrow P_{0.99,2} = X_{[98]} = 150 \mu\text{g} / \text{m}^3$$

$$0.99 \times 100 = 99 \Rightarrow i + 1 = 100 \Rightarrow P_{0.99,3} = X_{[100]} = 147 \mu\text{g} / \text{m}^3$$

c. 1. Using Equation 12, the 3-year average 99th percentile is calculated as follows:

$$\frac{128 + 50 + 147}{3} = 141.7 \mu\text{g} / \text{m}^3 \text{ rounds to } 140 \mu\text{g} / \text{m}^3.$$

2. Therefore, this site meets the 24-hour PM₁₀ standard.

[62 FR 38755, July 18, 1997]

PART 51—REQUIREMENTS FOR PREPARATION, ADOPTION, AND SUBMITTAL OF IMPLEMENTATION PLANS

Subparts A–E [Reserved]

Subpart F—Procedural Requirements

Sec.

- 51.100 Definitions.
- 51.101 Stipulations.
- 51.102 Public hearings.
- 51.103 Submission of plans, preliminary review of plans.
- 51.104 Revisions.
- 51.105 Approval of plans.

Subpart G—Control Strategy

- 51.110 Attainment and maintenance of national standards.
- 51.111 Description of control measures.
- 51.112 Demonstration of adequacy.
- 51.113 [Reserved]
- 51.114 Emissions data and projections.
- 51.115 Air quality data and projections.
- 51.116 Data availability.
- 51.117 Additional provisions for lead.
- 51.118 Stack height provisions.
- 51.119 Intermittent control systems.
- 51.120 Requirements for State Implementation Plan revisions relating to new motor vehicles.
- 51.121 Findings and requirements for submission of State implementation plan revisions relating to emissions of oxides of nitrogen.
- 51.122 Emissions reporting requirements for SIP revisions relating to budgets for NO_x emissions.

Subpart H—Prevention of Air Pollution Emergency Episodes

- 51.150 Classification of regions for episode plans.
- 51.151 Significant harm levels.
- 51.152 Contingency plans.
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Subpart I—Review of New Sources and Modifications

- 51.160 Legally enforceable procedures.
- 51.161 Public availability of information.
- 51.162 Identification of responsible agency.
- 51.163 Administrative procedures.
- 51.164 Stack height procedures.
- 51.165 Permit requirements.
- 51.166 Prevention of significant deterioration of air quality.

Subpart J—Ambient Air Quality Surveillance

- 51.190 Ambient air quality monitoring requirements.

Subpart K—Source Surveillance

- 51.210 General.
- 51.211 Emission reports and recordkeeping.
- 51.212 Testing, inspection, enforcement, and complaints.
- 51.213 Transportation control measures.
- 51.214 Continuous emission monitoring.

Subpart L—Legal Authority

- 51.230 Requirements for all plans.
- 51.231 Identification of legal authority.
- 51.232 Assignment of legal authority to local agencies.

Subpart M—Intergovernmental Consultation

AGENCY DESIGNATION

- 51.240 General plan requirements.
- 51.241 Nonattainment areas for carbon monoxide and ozone.
- 51.242 [Reserved]

Appendix A

Test Procedures for Determination of Instrument Performance Specifications

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Pollutant	Ref. or equivalent	Manual or automated	Applicable part 50 appendix	Applicable subparts of part 53					
				A	B	C	D	E	F
	Equivalent Class III	Automated	L ¹ , O ^{1,2}	✓	✓	✓ ¹	✓ ¹		

¹ Some requirements may apply, based on the nature of each particular candidate method, as determined by the Administrator.

² Alternative Class III requirements may be substituted.

[71 FR 61276, Oct. 17, 2006]

APPENDIX A TO SUBPART A OF PART 53—
REFERENCES

(1) American National Standard Quality Systems—Model for Quality Assurance in Design, Development, Production, Installation, and Servicing, ANSI/ISO/ASQC Q9001-1994. Available from American Society for Quality, P.O. Box 3005, Milwaukee, WI 53202 (<http://qualitypress.asq.org>).

(2) American National Standard Quality Systems for Environmental Data and Technology Programs—Requirements with guidance for use, ANSI/ASQC E4-2004. Available from American Society for Quality P.O. Box 3005, Milwaukee, WI 53202 (<http://qualitypress.asq.org>).

(3) Dimensioning and Tolerancing, ASME Y14.5M-1994. Available from the American Society of Mechanical Engineers, 345 East 47th Street, New York, NY 10017.

(4) Mathematical Definition of Dimensioning and Tolerancing Principles, ASME Y14.5.1M-1994. Available from the American Society of Mechanical Engineers, 345 East 47th Street, New York, NY 10017.

(5) ISO 10012, Quality Assurance Requirements for Measuring Equipment-Part 1: Meteorological confirmation system for measuring equipment):1992(E). Available from American Society for Quality Control, 611 East Wisconsin Avenue, Milwaukee, WI 53202.

(6) Quality Assurance Guidance Document 2.12. Monitoring PM_{2.5} in Ambient Air Using Designated Reference or Class I Equivalent Methods. U.S. EPA, National Exposure Research Laboratory, Research Triangle Park, NC, November 1998 or later edition. Currently available at <http://www.epa.gov/ttn/amtic/pmqaainf.html>.

[62 FR 38784, July 18, 1997, as amended at 71 FR 61278, Oct. 17, 2006]

Subpart B—Procedures for Testing Performance Characteristics of Automated Methods SO₂, CO, O₃, and NO₂

§ 53.20 General provisions.

(a) The test procedures given in this subpart shall be used to test the performance of candidate automated methods against the performance spec-

ifications given in table B-1. A test analyzer representative of the candidate automated method must exhibit performance better than, or equal to, the specified value for each such specification (except Range) to satisfy the requirements of this subpart. Except as provided in paragraph (b) of this section, the range of the candidate method must be the range specified in table B-1 to satisfy the requirements of this subpart.

(b) For a candidate method having more than one selectable range, one range must be that specified in table B-1 and a test analyzer representative of the method must pass the tests required by this subpart while operated in that range. The tests may be repeated for a broader range (i.e., one extending to higher concentrations) than that specified in table B-1 provided that the range does not extend to concentrations more than two times the upper range limit specified in table B-1. If the application is for a reference method determination, the tests may be repeated for a narrower range (one extending to lower concentrations) than that specified in table B-1.

If the tests are conducted or passed only for the specified range, any reference or equivalent method determination with respect to the method will be limited to that range. If the tests are passed for both the specified range and a broader range (or ranges), any such determination will include the broader range(s) as well as the specified range, provided that the tests required by subpart C of this part (if applicable) are met for the broader range(s). If the tests are passed for both the specified range and a narrower range, a reference method determination for the method will include the narrower range as well as the specified range. Appropriate test data shall be submitted for each range sought to be included in a reference or equivalent

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method determination under this paragraph (b).

(c) For each performance specification (except Range), the test procedure shall be initially repeated seven (7) times to yield 7 test results. Each result shall be compared with the corresponding specification in table B-1; a value higher than or outside that specified constitutes a failure. These 7 results for each parameter shall be interpreted as follows:

(1) Zero (0) failures: Candidate method passes the performance parameter.

(2) Three (3) or more failures: Candidate method fails the performance parameter.

(3) One (1) or two (2) failures: Repeat the test procedures for the parameter eight (8) additional times yielding a total of fifteen (15) test results. The combined total of 15 test results shall then be interpreted as follows:

(i) One (1) or two (2) failures: Candidate method passes the performance parameter.

(ii) Three (3) or more failures: Candidate method fails the performance parameter.

TABLE B-1—PERFORMANCE SPECIFICATIONS FOR AUTOMATED METHODS

Performance parameter	Units ¹	Sulfur dioxide	Photochemical oxidants	Carbon monoxide	Nitrogen dioxide	Definitions and test procedures
1. Range	Parts per million ...	0-0.5	0-0.5	0-50	0-0.5	Sec. 53.23(a).
2. Noisedo005	.005	.50	.005	Sec. 53.23(b).
3. Lower detectable limit	Parts per million01	.01	1.0	.01	Sec. 53.23(c).
4. Interference equivalentdododododo	Sec. 53.23(d).
Each interferant	Parts per million ...	±.02	±.02	±1.0	±0.02	
Total interferantdo06	.06	1.5	.04	
5. Zero drift, 12 and 24 hourdo	±.02	±.02	±1.0	±.02	Sec. 52.23(e).
6. Span drift, 24 hourdododododo	Do.
20 percent of upper range limit	Percent	±20.0	±20.0	±10.0	±20.0	
80 percent of upper range limitdo	±5.0	±5.0	±2.5	±5.0	
7. Lag time	Minutes	20	20	10	20	Do.
8. Rise timedo	15	15	5	15	Do.
9. Fall timedo	15	15	5	15	Do.
10. Precisiondododododo	Do.
20 percent of upper range limit	Parts per million01	.01	.5	.02	
80 percent of upper range limitdo015	.01	.5	.03	

¹ To convert from parts per million to µg/m³ at 25 °C and 760 mm Hg, multiply by M/0.02447, where M is the molecular weight of the gas.

(d) The tests for *zero drift*, *span drift*, *lag time*, *rise time*, *fall time*, and *precision* shall be combined into a single sequential procedure to be conducted at various line voltages and ambient temperatures specified in § 53.23(e). The tests for *noise*, *lower detectable limit*, and *interference equivalents* shall be made at any temperature between 20 °C. and 30 °C. and at any normal line voltage between 105 and 125 volts, and shall be conducted such that not more than three (3) test results for each parameter are obtained per 24 hours.

(e) All response readings to be recorded shall first be converted to concentration units according to the calibration curve constructed in accordance with § 53.21(b).

(f) All recorder chart tracings, records, test data and other documentation obtained from or pertinent

to these tests shall be identified, dated, signed by the analyst performing the test, and submitted.

NOTE: Suggested formats for reporting the test results and calculations are provided in Figures B-2, B-3, B-4, B-5, and B-6 in appendix A. Symbols and abbreviations used in this subpart are listed in table B-5, appendix A.

[40 FR 7049, Feb. 18, 1975, as amended at 40 FR 18168, Apr. 25, 1975; 41 FR 52694, Dec. 1, 1976]

§ 53.21 Test conditions.

(a) *Set-up and start-up* of the test analyzer shall be in strict accordance with the operating instructions specified in the manual referred to in § 53.4(b)(3). Allow adequate warm-up or stabilization time as indicated in the operating instructions before beginning the tests.

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'Keefe, A. E., and Ortman, 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 $r_1, r_2, r_3 \dots r_i \dots r_{25}$.

(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

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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.

(d) *Interference equivalent*—(1) *Technical definition*. Positive or negative response caused by a substance other than the one being measured.

(2) *Test procedure*. The test analyzer shall be tested for all substances likely to cause a detectable response. The test analyzer shall be challenged, in turn, with each interfering agent specified in table B-3. In the event that there are substances likely to cause a significant interference which have not been specified in table B-3, these substances shall be tested at a concentration substantially higher than that normally found in the ambient air. The

interference may be either positive or negative, depending on whether the test analyzer's response is increased or decreased by the presence of the interferent. Interference equivalents shall be determined by mixing each interferent, one at a time, with the pollutant at the concentrations specified in table B-3, and comparing the test analyzer's response to the response caused by the pollutant alone. Known gas-phase reactions that might occur between an interferent and the pollutant are designated by footnote 3 in table B-3. In these cases, the interference equivalent shall be determined in the absence of the pollutant.

(i) Allow sufficient time for warm-up and stabilization of the test analyzer.

(ii) For a candidate method using a prefilter or scrubber based upon a chemical reaction to derive part of its specificity, and which requires periodic service or maintenance, the test analyzer shall be "conditioned" prior to each interference test as follows:

TABLE B-3—INTERFERANT TEST CONCENTRATION,¹ PARTS PER MILLION

Pollutant	Analyzer type ²	Hydrochloric acid	Ammonia	Hydrogen sulfide	Sulfur dioxide	Nitrogen dioxide	Nitric oxide	Carbon dioxide	Ethylene	Ozone	M-xylene	Water vapor	Carbon monoxide	Methane	Ethane
SO ₂	Flame photometric (FPD)			0.1	1.0,14			750				320,000	50		
SO ₂	Gas chromatography (FPD)			.1	4.14			750				320,000	50		
SO ₂	Spectrophotometric-wet chemical (pararosaniline reaction)	0.2	3 0.1	.1	4.14	0.5		750		0.5					
SO ₂	Electrochemical	.2	3 1	.1	4.14	.5	0.5		0.2	.5		320,000			
SO ₂	Conductivity	.2	3 1		4.14	.5		750							
SO ₂	Spectrophotometric-gas phase				4.14	.5	.5				0.2				
O ₃	Chemiluminescent			3 1				750		4.08		320,000			
O ₃	Electrochemical			3 1	.5	.5				4.08		320,000			
O ₃	Spectrophotometric-wet chemical (potassium iodide reaction)			3 1	.5	.5	3.5			4.08					
O ₃	Spectrophotometric-gas phase				.5	.5	3.5			4.08					
CO	Infrared							750				20,000	4 10		0.5
CO	Gas chromatography with flame ionization detector											20,000	4 10		
CO	Electrochemical						.5		.2			20,000	4 10	5.0	.5
CO	Catalytic combustion-thermal detection			.1				750	.2			20,000	4 10		
CO	IR fluorescence							750				20,000	4 10		.5
CO	Mercury replacement UV photometric								.2				4 10		.5
NO ₂	Chemiluminescent			3 1	.5	4.1	.5					20,000			
NO ₂	Spectrophotometric-wet chemical (azo-dye reaction)				.5	4.1	.5	750		.5					
NO ₂	Electrochemical	0.2	3 1		.5	4.1	.5	750		.5		20,000	50		
NO ₂	Spectrophotometric-gas phase		3 1		.5	4.1	.5			.5		20,000	50		

¹ Concentrations of interferant listed must be prepared and controlled to ±10 percent of the state value.

² Analyzer types not listed will be considered by the administrator as special cases.

³ Do not mix with pollutant.

⁴ Concentration of pollutant used for test. These pollutant concentrations must be prepared to ±10 percent of the stated value.

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(A) Service or perform the indicated maintenance on the scrubber or prefilter as directed in the manual referred to in § 53.4(b)(3).

(B) Before testing for each interferent, allow the test analyzer to sample through the scrubber a test atmosphere containing the interferent at a concentration equal to the value specified in table B-3. Sampling shall be at the normal flow rate and shall be continued for 6 continuous hours prior to testing.

(iii) Generate three test atmosphere streams as follows:

(A) Test atmosphere *P*: Pollutant concentration.

(B) Test atmosphere *I*: Interference concentration.

(C) Test atmosphere *Z*: Zero air.

(iv) Adjust the individual flow rates and the pollutant or interferent generators for the three test atmospheres as follows:

(A) The flow rates of test atmospheres *I* and *Z* shall be identical.

(B) The concentration of pollutant in test atmosphere *P* shall be adjusted such that when *P* is mixed (diluted) with either test atmosphere *I* or *Z*, the resulting concentration of pollutant shall be as specified in table B-3.

(C) The concentration of interferent in test atmosphere *I* shall be adjusted such that when *I* is mixed (diluted) with test atmosphere *P*, the resulting concentration of interferent shall be equal to the value specified in table B-3.

(D) To minimize concentration errors due to flow rate differences between *I* and *Z*, it is recommended that, when possible, the flow rate of *P* be from 10 to 20 times larger than the flow rates of *I* and *Z*.

(v) Mix test atmospheres *P* and *Z* by passing the total flow of both atmospheres through a mixing flask.

(vi) Sample and measure the mixture of test atmospheres *P* and *Z* with the test analyzer. Allow for a stable reading, and record the reading, in concentration units, as *R* (see Figure B-3).

(vii) Mix test atmospheres *P* and *I* by passing the total flow of both atmospheres through a mixing flask.

(viii) Sample and measure this mixture. Record the stable reading, in concentration units, as *R_I*.

(ix) Calculate the interference equivalent (*IE*) as:

$$IE = R_I - R$$

IE must be equal to or less than the specification given in table B-1 for each interferent to pass the test.

(x) Follow steps (iii) through (ix) of this section, in turn, to determine the interference equivalent for each interferent.

(xi) For those interferents which cannot be mixed with the pollutant, as indicated by footnote (3) in table B-3, adjust the concentration of test atmosphere *I* to the specified value without being mixed or diluted by the pollutant test atmosphere. Determine *IE* as follows:

(A) Sample and measure test atmosphere *Z* (zero air). Allow for a stable reading and record the reading, in concentration units, as *R*.

(B) Sample and measure the interferent test atmosphere *I*. If the test analyzer is not capable of negative readings, adjust the analyzer (not the recorder) to give an offset zero. Record the stable reading in concentration units as *R_I*, extrapolating the calibration curve, if necessary, to represent negative readings.

(C) Calculate $IE = R_I - R$. *IE* must be equal to or less than the specification in table B-1 to pass the test.

(xii) Sum the absolute value of all the individual interference equivalents. This sum must be equal to or less than the total interferent specification given in table B-1 to pass the test.

(e) *Zero drift, span drift, lag time, rise time, fall time, and precision*—(1) *Technical definitions*—(i) *Zero drift*: The change in response to zero pollutant concentration, over 12- and 24-hour periods of continuous unadjusted operation.

(ii) *Span drift*: The percent change in response to an up-scale pollutant concentration over a 24-hour period of continuous unadjusted operation.

(iii) *Lag time*: The time interval between a step change in input concentration and the first observable corresponding change in response.

(iv) *Rise time*: The time interval between initial response and 95 percent of final response after a step increase in input concentration.

(v) *Fall time:* The time interval between initial response and 95 percent of final response after a step decrease in input concentration.

(vi) *Precision:* Variation about the mean of repeated measurements of the same pollutant concentration, expressed as one standard deviation about the mean.

(2) Tests for these performance parameters shall be accomplished over a period of seven (7) or more days. During this time, the line voltage supplied to the test analyzer and the ambient temperature surrounding the analyzer shall be varied from day to day. One test result for each performance parameter shall be obtained each test day, for seven (7) or fifteen (15) test days as necessary. The tests are performed sequentially in a single procedure.

(3) The 24-hour test day may begin at any clock hour. The first 12 hours out of each test day are required for testing 12-hour zero drift. Tests for the other parameters shall be conducted during the remaining 12 hours.

(4) Table B-4 specifies the line voltage and room temperature to be used for each test day. The line voltage and temperature shall be changed to the specified values at the start of each test day (i.e., at the start of the 12-hour zero test). Initial adjustments (day zero) shall be made at a line voltage of 115 volts (rms) and a room temperature of 25 °C.

(5) The tests shall be conducted in blocks consisting of 3 test days each until 7 or 15 test results have been obtained. (The final block may contain fewer than three test days.) If a test is interrupted by an occurrence other

than a malfunction of the test analyzer, only the block during which the interruption occurred shall be repeated.

(6) During each block, manual adjustments to the electronics, gas, or reagent flows or periodic maintenance shall not be permitted. Automatic adjustments which the test analyzer performs by itself are permitted at any time.

(7) At least 4 hours prior to the start of the first test day of each block, the test analyzer may be adjusted and/or serviced according to the periodic maintenance procedures specified in the manual referred to in §53.4(b)(3). If a new block is to immediately follow a previous block, such adjustments or servicing may be done immediately after completion of the day's tests for the last day of the previous block and at the voltage and temperature specified for that day, but only on test days 3, 6, 9, and 12.

NOTE: If necessary, the beginning of the test days succeeding such maintenance or adjustment may be delayed as necessary to complete the service or adjustment operation.

(8) All response readings to be recorded shall first be converted to concentration units according to the calibration curve. Whenever a test atmosphere is to be measured but a stable reading is not required, the test atmosphere shall be measured long enough to cause a change in response of at least 10% of full scale. Identify all readings and other pertinent data on the strip chart. (See Figure B-1 illustrating the pattern of the required readings.)

TABLE B-4—LINE VOLTAGE AND ROOM TEMPERATURE TEST CONDITIONS

Test day	Line voltage, ¹ rms	Room temperature, ² °C	Comments
0	115	25	Initial set-up and adjustments.
1	125	20	
2	105	20	
3	125	30	Adjustments and/or periodic maintenance permitted at end of tests.
4	105	30	
5	125	20	
6	105	20	Adjustments and/or periodic maintenance permitted at end of tests.
7	125	30	Examine test results to ascertain if further testing is required.
8	105	30	
9	125	20	Adjustments and/or periodic maintenance permitted at end of tests.
10	105	20	
11	125	30	
12	105	30	Adjustments and/or periodic maintenance permitted at end of tests.

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TABLE B-4—LINE VOLTAGE AND ROOM TEMPERATURE TEST CONDITIONS—Continued

Test day	Line voltage, ¹ rms	Room temperature, ² °C	Comments
13	125	20	
14	105	20	
15	125	30	

¹ Voltage specified shall be controlled to ±1 volt.
² Temperature specified shall be controlled to ±1 °C.

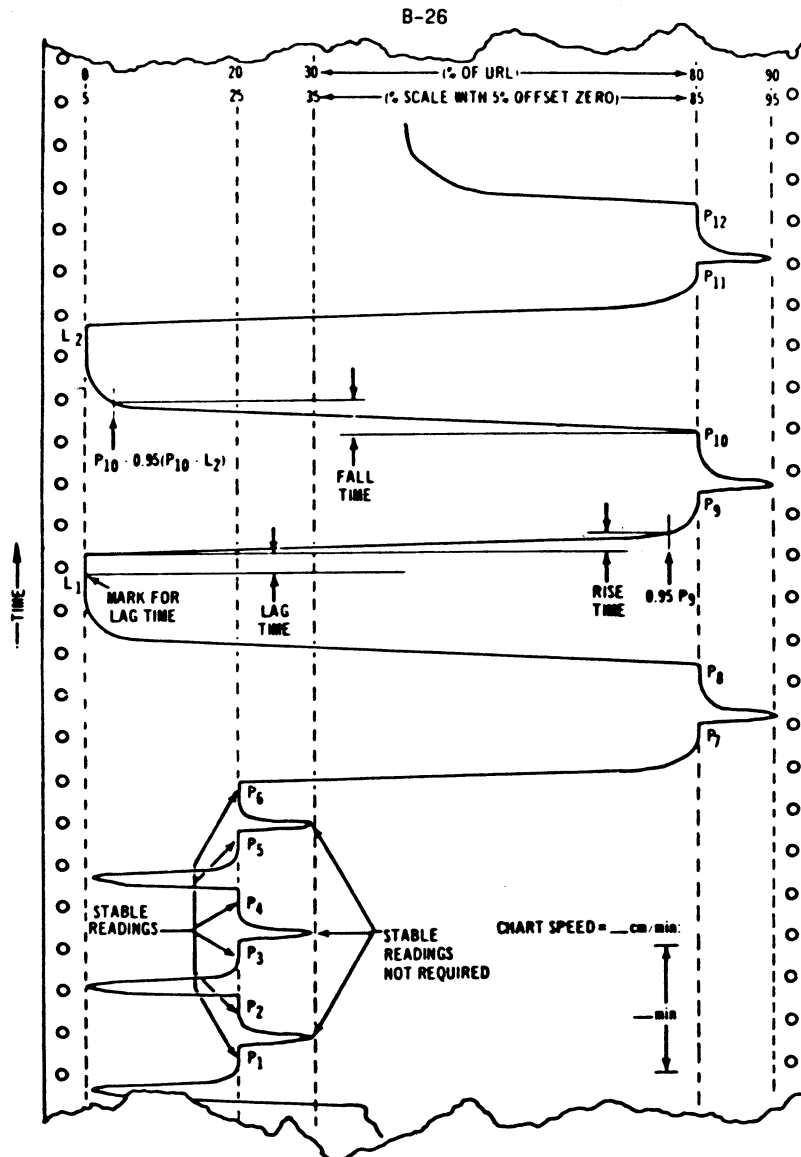


Figure B-1. Example showing the nature of the tracing obtained during the test for drift, lag time, rise time, fall time, and precision. The time scale has been greatly compressed.

(9) Test procedure. (i) Arrange to generate pollutant test atmospheres as follows:

Test atmosphere	Pollutant concentration (percent)
A ₀	Zero air.
A ₂₀	20±5 of the upper range limit.
A ₃₀	30±5 of the upper range limit.

Test atmosphere	Pollutant concentration (percent)
A ₈₀	80±5 of the upper range limit.
A ₉₀	90±5 of the upper range limit.

Test atmospheres A₀, A₂₀, and A₈₀ shall be consistent during the tests and from day to day.

(ii) For steps (xxv) through (xxxi) of this section, a chart speed of at least 10 centimeters per hour shall be used. The actual chart speed, chart speed changes, and time checks shall be clearly marked on the chart.

(iii) Allow sufficient time for test analyzer to warm up and stabilize at a line voltage of 115 volts and a room temperature of 25 °C. Recalibrate, if necessary, and adjust the zero baseline to 5 percent of chart. No further adjustments shall be made to the analyzer until the end of the tests on the third day.

(iv) Measure test atmosphere A₀ until a stable reading is obtained, and record this reading (in ppm) as Z_n, where n = 0 (see Figure B-4 in appendix A).

(v) Measure test atmosphere A₂₀. Allow for a stable reading and record it as M_n, where n = 0.

(vi) Measure test atmosphere A₈₀. Allow for a stable reading and record it as S_n, where n = 0.

(vii) The above readings for Z₀, M₀, and S₀ should be taken at least four (4) hours prior to the beginning of test day 1.

(viii) At the beginning of each test day, adjust the line voltage and room temperature to the values given in table B-4.

(ix) Measure test atmosphere A₀ continuously for at least twelve (12) continuous hours during each test day.

(x) After the 12-hour zero drift test (step ix), sample test atmosphere A₀. A stable reading is not required.

(xi) Measure test atmosphere A₂₀ and record the stable reading (in ppm) as P₁. (See Figure B-4 in appendix A.)

(xii) Sample test atmosphere A₃₀; a stable reading is not required.

(xiii) Measure test atmosphere A₂₀ and record the stable reading as P₂.

(xiv) Sample test atmosphere A₀; a stable reading is not required.

(xv) Measure test atmosphere A₂₀ and record the stable reading as P₃.

(xvi) Sample test atmosphere A₃₀; a stable reading is not required.

(xvii) Measure test atmosphere A₂₀ and record the stable reading as P₄.

(xviii) Sample test atmosphere A₀; a stable reading is not required.

(xix) Measure test atmosphere A₂₀ and record the stable reading as P₅.

(xx) Sample test atmosphere A₃₀; a stable reading is not required.

(xxi) Measure test atmosphere A₂₀ and record the stable reading as P₆.

(xxii) Measure test atmosphere A₃₀ and record the stable reading as P₇.

(xxiii) Sample test atmosphere A₉₀; a stable reading is not required.

(xxiv) Measure test atmosphere A₈₀ and record the stable reading as P₈. Increase chart speed to at least 10 centimeters per hour.

(xxv) Measure test atmosphere A₀. Record the stable reading as L₁.

(xxvi) Quickly switch the test analyzer to measure test atmosphere A₈₀ and mark the recorder chart to show the exact time when the switch occurred.

(xxvii) Measure test atmosphere A₉₀ and record the stable reading as P₈₀.

(xxviii) Sample test atmosphere A₉₀; a stable reading is not required.

(xxix) Measure test atmosphere A₈₀ and record the stable reading as P₁₀.

(xxx) Measure test atmosphere A₀ and record the stable reading as L₂.

(xxxi) Measure test atmosphere A₈₀ and record the stable reading as P₁₁.

(xxxii) Sample test atmosphere A₉₀; a stable reading is not required.

(xxxiii) Measure test atmosphere A₈₀ and record the stable reading as P₁₂.

(xxxiv) Repeat steps (viii) through (xxxiii) of this section, each test day.

(xxxv) If zero and span adjustments are made after the readings are taken on test days 3, 6, 9, or 12, complete all adjustments; then measure test atmospheres A₀, A₈₀, and A₂₀. Allow for a stable reading on each, and record the readings as Z_nS_n, and M_n respectively, where n = the test day number.

(10) Determine the results of each day's tests as follows. Mark the recorder chart to show readings and determinations.

(i) *Zero drift.* (A) 12-hour. Examine the strip chart pertaining to the 12-

hour continuous zero air test. Determine the minimum (Cmin.) and maximum (Cmax.) readings (in p/m) during this period of 12 consecutive hours, extrapolating the calibration curve to negative concentration units if necessary. Determine the 12-hour zero drift (12ZD) as $12ZD = C^{max.} - C^{min.}$. (See Figure B-5 in appendix A.)

(B) Calculate the 24-hour zero drift (24ZD) for the n -th test day as $24ZD_n = Z_n - Z_{n-1}$, or $24ZD_n = Z_n - Z'_{n-1}$ if zero adjustment was made on the previous day, where $Z_n = \frac{1}{2}(L_1 + L_2)$ for L_1 and L_2 taken on the n -th test day.

(C) Compare 12ZD and 24ZD to the zero drift specification in table B-1. Both 12ZD and 24ZD must be equal to or less than the specified value to pass the test for zero drift.

(ii) *Span drift.* (A) Span drift at 20 percent of URL (MSD)

$$MSD_n = \frac{M_n - M_{n-1}}{M_{n-1}} \times 100\%$$

$$MSD_n = \frac{M_n - M'_{n-1}}{M'_{n-1}} \times 100\%$$

If span adjustment was made on the previous day, where

$$M_n = \frac{1}{6} \sum_{i=1}^6 P_i$$

n indicates the n -th test day, and i indicates the i -th reading on the n -th day.

(B) Span drift at 80 percent of URL (USD):

$$USD_n = \frac{S_n - S_{n-1}}{S_{n-1}} \times 100\%$$

or

$$USD_n = \frac{S_n - S'_{n-1}}{S'_{n-1}} \times 100\%$$

If span adjustment was made on the previous day, where

$$S_n = \frac{1}{6} \sum_{i=7}^{12} P_i$$

n indicates the n -th test day, and i indicates the i -th reading on the n -th test day.

(C) Both USD and MSD must be equal to or less than the respective specifications given in table B-1 to pass the test for span draft.

(iii) *Lag time.* Determine, from the strip chart, the elapsed time in minutes between the mark made in step (xxvi) and the first observable (two times the noise level) response. This time must be equal to or less than the time specified in table B-1 to pass the test for lag time.

(iv) *Rise time.* Calculate 95 percent of reading P_9 and determine from the recorder chart, the elapsed time between the first observable (two times noise level) response and a response equal to 95 percent of the P_9 reading. This time must be equal to or less than the rise time specified in table B-1 to pass the test for rise time.

(v) *Fall time.* Calculate five percent of ($P_{10} - L_2$) and determine, from the strip chart, the elapsed time in minutes between the first observable decrease in response following reading P_{10} and a response equal to five percent of ($P_{10} - L_2$). This time must be equal to or less than the fall time specification in table B-1 to pass the test for fall time.

(vi) *Precision.* Calculate precision (P_{20} and P_{80}) for each day's test as follows:

(A)

$$P_{30} = \sqrt{\frac{1}{5} \left[\sum_{i=1}^6 P_i^2 - \frac{1}{6} \left(\sum_{i=1}^6 P_i \right)^2 \right]}$$

(B)

$$P_{30} = \sqrt{\frac{1}{5} \left[\sum_{i=7}^{12} P_i^2 - \frac{1}{6} \left(\sum_{i=7}^{12} P_i \right)^2 \right]}$$

(C) Both P_{20} and P_{80} must be equal to or less than the specification given in table B-1 to pass the test for precision.

[40 FR 7049, Feb. 18, 1975, as amended at 41 FR 52694, Dec. 1, 1976]

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**APPENDIX A TO SUBPART B OF PART 53—
OPTIONAL FORMS FOR REPORTING
TEST RESULTS**

**TABLE B-5—SYMBOLS AND ABBREVIATIONS—
Continued**

TABLE B-5—SYMBOLS AND ABBREVIATIONS

B_L	Analyzer reading at specified <i>LDL</i> concentration.	P_{20}	Precision at 20 percent of <i>URL</i> .
B_0	Analyzer reading at 0 concentration for <i>LDL</i> test.	P_{80}	Precision at 80 percent of <i>URL</i> .
<i>DM</i>	Digital meter.	<i>R</i>	Analyzer reading of pollutant alone for <i>IE</i> test.
C_{max}	Maximum analyzer reading during 12ZD test.	R_I	Analyzer reading with interferent added for <i>IE</i> test.
C_{min}	Minimum analyzer reading during 12ZD test.	r_i	The <i>i</i> -th <i>DM</i> reading for noise test.
<i>i</i>	Subscript indicating the <i>i</i> -th quantity in a series.	<i>S</i>	Standard deviation of noise readings.
<i>IE</i>	Interference equivalent.	S_0	Noise value (<i>S</i>) measured at 0 concentration.
L_1	First analyzer zero reading for 24ZD test.	S_{80}	Noise value (<i>S</i>) measured at 80 percent of <i>URL</i> .
L_2	Second analyzer zero reading for 24ZD test.	S_n	Average of $P_7 \dots P_{12}$ for the <i>n</i> -th test day.
M_n	Average of $P_1 \dots P_6$ for the <i>n</i> -th test day.	S'_n	Adjusted span reading at 80 percent of <i>URL</i> on the <i>n</i> -th test day.
M'_n	Adjusted span reading at 20 percent of <i>URL</i> on the <i>n</i> -th test day.	<i>URL</i>	Upper range limit.
<i>MSD</i>	Span drift at 20 percent of <i>URL</i> .	<i>USD</i>	Span drift at 80 percent of <i>URL</i> .
<i>n</i>	Subscript indicating the test day number.	<i>Z</i>	Average of L_1 and L_2 .
<i>P</i>	Analyzer reading for precision test.	Z_n	Average of L_1 and L_2 on the <i>n</i> -th test day.
P_i	The <i>i</i> -th analyzer reading for precision test.	Z'_n	Adjusted zero reading on the <i>n</i> -th test day.
		<i>ZD</i>	Zero drift.
		12ZD	12-hour zero drift.
		24ZD	24-hour zero drift.

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Applicant _____ Date _____

Test No. _____

Analyzer _____ Range _____

READING NUMBER (i)	TIME	0% of URL		80% of URL	
		DM READING	r_i , ppm	DM READING	r_i , ppm
1					
2					
3					
4					
5					
6					
7					
8					
9					
10					
11					
12					
13					
14					
15					
16					
17					
18					
19					
20					
21					
22					
23					
24					
25					
$\sum_{i=1}^{25} r_i$					
$\sum_{i=1}^{25} r_i^2$					
s			$s_0 =$		$s_{80} =$

Figure B-2. Form for noise data.

Applicant _____		Range _____														
Analyzer _____																
TEST PARAMETER	READING OR CALCULATION	1	2	3	4	5	6	7	8	9	10	11	12	13	14	15
LOWER DETECTABLE LIMIT	B_2															
	B_L															
	$LDL = B_L - B_2$															
INTERFERENCE EQUIVALENT	R_1															
	R_{11}															
	$IE_1 = R_{11} \cdot R_1$															
	R_2															
	R_{12}															
	$IE_2 = R_{12} \cdot R_2$															
	R_3															
	R_{13}															
	$IE_3 = R_{13} \cdot R_3$															
	R_4															
	R_{14}															
	$IE_4 = R_{14} \cdot R_4$															
	R_5															
	R_{15}															
	$IE_5 = R_{15} \cdot R_5$															
TOTAL	$IE_T = \sum_{i=1}^5 IE_i$															

Figure B-3. Form for data and calculations for lower detectable limit and interference equivalent.

Applicant _____
 Analyzer _____ Range _____

TEST DAY (m)	DATE	ANALYZER READING, ppm	0	1	2	3	4	5	6	7	8	9	10	11	12	13	14	15
P ₁																		
P ₂																		
P ₃																		
P ₄																		
P ₅																		
P ₆																		
$\sum_{i=1}^6 P_i^2$																		
P ₇																		
P ₈																		
P ₉																		
P ₁₀																		
P ₁₁																		
P ₁₂																		
$\sum_{i=7}^{12} P_i^2$																		
L ₁																		
L ₂																		
Z _i																		
M _{ij}																		
S _i																		
C _{max}																		
C _{min}																		

Figure B-4. Form recording data for drift and precision.

Applicant		Range														
Analyzer		n - th TEST DAY														
TEST PARAMETER	CALCULATION	1	2	3	4	5	6	7	8	9	10	11	12	13	14	15
Zero drift	12ZD = $C_{max} - C_{min}$															
	$Z = \frac{1}{2}(L_1 + L_2)$															
24 hour	24ZD = $Z_n - Z_{n-1}$															
	24ZD _n = $Z_n - Z'_{n-1}$															
20% URL	$M_n = \frac{1}{6} \sum_{i=1}^6 P_i$															
	$MSD_n = \frac{M_n - M_{n-1}}{M_{n-1}} \times 100\%$															
	$MSD_n = \frac{M_n - M'_{n-1}}{M'_{n-1}} \times 100\%$															
Span drift	$S_n = \frac{1}{6} \sum_{i=7}^{12} P_i$															
	$USD_n = \frac{S_n - S_{n-1}}{S_{n-1}} \times 100\%$															
80% URL	$USD_n = \frac{S_n - S'_{n-1}}{S'_{n-1}} \times 100\%$															
	$P_{20} = \sqrt{\frac{1}{5} \left[\sum_{i=1}^6 P_i^2 - \frac{1}{6} \left(\sum_{i=1}^6 P_i \right)^2 \right]}$															
Precision	$P_{80} = \sqrt{\frac{1}{5} \left[\sum_{i=7}^{12} P_i^2 - \frac{1}{6} \left(\sum_{i=7}^{12} P_i \right)^2 \right]}$															

Figure B-5. Form for calculating zero drift, span drift and precision.

Applicant _____		Analyst _____															
Analyzer _____		Range _____															
PERFORMANCE PARAMETER	Table B-1 spec.	TEST										No. of test failures					
NOISE, ppm	0% URL (S ₀)	1	2	3	4	5	6	7	8	9	10	11	12	13	14	15	
	80% URL (S ₈₀)																
LDL (must be 2 × noise)																	
INTER-FERENCE EQUIVALENT, ppm	IE ₁																
	IE ₂																
	IE ₃																
	IE ₄																
	IE ₅																
TOTAL (IE _T)																	
ZERO DRIFT, ppm	12 hour (1ZZD)																
	24 hour (2AZD)																
SPAN DRIFT, %	20% URL (MSD)																
	80% URL (USD)																
LAG TIME, min																	
RISE TIME, min																	
FALL TIME, min																	
PRECISION, ppm	20% URL (P ₂₀)																
	80% URL (P ₈₀)																

^aCompare each test LDL reading with the corresponding noise measurements. LDL reading must exceed the 0% URL noise value by a factor of 2 to pass the test for LDL.

Figure B-6. Form for summary of test results.

[40 FR 7049, Feb. 18, 1975, as amended at 40 FR 18169, Apr. 25, 1975]

Subpart C—Procedures for Determining Comparability Between Candidate Methods and Reference Methods

SOURCE: 71 FR 61278, Oct. 17, 2006, unless otherwise noted.

§ 53.30 General provisions.

(a) *Determination of comparability.* The test procedures prescribed in this subpart shall be used to determine if a candidate method is comparable to a reference method when both methods measure pollutant concentrations in

Appendix B

40 CFR Part 53.1-53.23

Includes:

Subpart A—General Provisions

Subpart B—Procedures for Testing Performance Characteristics of Automated Methods SO₂,
CO, O₃, and NO₂

§ 53.1

- 53.57 Test for filter temperature control during sampling and post-sampling periods.
- 53.58 Operational field precision and blank test.
- 53.59 Aerosol transport test for Class I equivalent method samplers.
- TABLE E-1 TO SUBPART E—SUMMARY OF TEST REQUIREMENTS FOR REFERENCE AND CLASS I EQUIVALENT METHODS FOR PM_{2.5}
- TABLE E-2 TO SUBPART E—SPECTRAL ENERGY DISTRIBUTION AND PERMITTED TOLERANCE FOR CONDUCTING RADIATIVE TESTS
- FIGURE E-1 TO SUBPART E—DESIGNATION TESTING CHECKLIST
- FIGURE E-2 TO SUBPART E—PRODUCT MANUFACTURING CHECKLIST
- APPENDIX A TO SUBPART E—REFERENCES

Subpart F—Procedures for Testing Performance Characteristics of Class II Equivalent Methods for PM_{2.5}

- 53.60 General provisions.
- 53.61 Test conditions for PM_{2.5} reference method equivalency.
- 53.62 Test procedure: Full wind tunnel test.
- 53.63 Test procedure: Wind tunnel inlet aspiration test.
- 53.64 Test procedure: Static fractionator test.
- 53.65 Test procedure: Loading test.
- 53.66 Test procedure: Volatility test.
- TABLE F-1 TO SUBPART F—PERFORMANCE SPECIFICATIONS FOR PM_{2.5} CLASS II EQUIVALENT SAMPLERS
- TABLE F-2 TO SUBPART F—PARTICLE SIZES AND WIND SPEEDS FOR FULL WIND TUNNEL TEST, WIND TUNNEL INLET ASPIRATION TEST, AND STATIC CHAMBER TEST
- TABLE F-3 TO SUBPART F—CRITICAL PARAMETERS OF IDEALIZED AMBIENT PARTICLE SIZE DISTRIBUTIONS
- TABLE F-4 TO SUBPART F—ESTIMATED MASS CONCENTRATION MEASUREMENT OF PM_{2.5} FOR IDEALIZED COARSE AEROSOL SIZE DISTRIBUTION
- TABLE F-5 TO SUBPART F—ESTIMATED MASS CONCENTRATION MEASUREMENT OF PM_{2.5} FOR IDEALIZED “TYPICAL” COARSE AEROSOL SIZE DISTRIBUTION
- TABLE F-6 TO SUBPART F—ESTIMATED MASS CONCENTRATION MEASUREMENT OF PM_{2.5} FOR IDEALIZED FINE AEROSOL SIZE DISTRIBUTION
- FIGURE E-1 TO SUBPART F—DESIGNATION TESTING CHECKLIST
- APPENDIX A TO SUBPART F—REFERENCES

AUTHORITY: Sec. 301(a) of the Clean Air Act (42 U.S.C. sec. 1857g(a)), as amended by sec. 15(c)(2) of Pub. L. 91-604, 84 Stat. 1713, unless otherwise noted.

SOURCE: 40 FR 7049, Feb. 18, 1975, unless otherwise noted.

40 CFR Ch. I (7-1-07 Edition)

Subpart A—General Provisions

SOURCE: 62 FR 38784, July 18, 1997, unless otherwise noted.

§ 53.1 Definitions.

Terms used but not defined in this part shall have the meaning given them by the Act.

Act means the Clean Air Act (42 U.S.C. 1857-1857l), as amended.

Additive and multiplicative bias means the linear regression intercept and slope of a linear plot fitted to corresponding candidate and reference method mean measurement data pairs.

Administrator means the Administrator of the Environmental Protection Agency (EPA) or his or her authorized representative.

Agency means the Environmental Protection Agency.

Applicant means a person or entity who submits an application for a Federal reference method or Federal equivalent method determination under § 53.4, or a person or entity who assumes the rights and obligations of an applicant under § 53.7. Applicant may include a manufacturer, distributor, supplier, or vendor.

Automated method or analyzer means a method for measuring concentrations of an ambient air pollutant in which sample collection (if necessary), analysis, and measurement are performed automatically by an instrument.

Candidate method means a method for measuring the concentration of an air pollutant in the ambient air for which an application for a Federal reference method determination or a Federal equivalent method determination is submitted in accordance with § 53.4, or a method tested at the initiative of the Administrator in accordance with § 53.7.

Class I equivalent method means an equivalent method for PM_{2.5} or PM_{10-2.5} which is based on a sampler that is very similar to the sampler specified for reference methods in appendix L or appendix O (as applicable) of part 50 of this chapter, with only minor deviations or modifications, as determined by EPA.

Class II equivalent method means an equivalent method for PM_{2.5} or PM_{10-2.5} that utilizes a PM_{2.5} sampler or

PM_{10-2.5} sampler in which integrated PM_{2.5} samples or PM_{10-2.5} samples are obtained from the atmosphere by filtration and subjected to a subsequent filter conditioning process followed by a gravimetric mass determination, but which is not a Class I equivalent method because of substantial deviations from the design specifications of the sampler specified for reference methods in appendix L or appendix O (as applicable) of part 50 of this chapter, as determined by EPA.

Class III equivalent method means an equivalent method for PM_{2.5} or PM_{10-2.5} that is an analyzer capable of providing PM_{2.5} or PM_{10-2.5} ambient air measurements representative of one-hour or less integrated PM_{2.5} or PM_{10-2.5} concentrations as well as 24-hour measurements determined as, or equivalent to, the mean of 24 one-hour consecutive measurements.

CO means carbon monoxide.

Collocated means two or more air samplers, analyzers, or other instruments that are operated simultaneously while located side by side, separated by a distance that is large enough to preclude the air sampled by any of the devices from being affected by any of the other devices, but small enough so that all devices obtain identical or uniform ambient air samples that are equally representative of the general area in which the group of devices is located.

Federal equivalent method (FEM) means a method for measuring the concentration of an air pollutant in the ambient air that has been designated as an equivalent method in accordance with this part; it does not include a method for which an equivalent method designation has been canceled in accordance with § 53.11 or § 53.16.

Federal reference method (FRM) means a method of sampling and analyzing the ambient air for an air pollutant that is specified as a reference method in an appendix to part 50 of this chapter, or a method that has been designated as a reference method in accordance with this part; it does not include a method for which a reference method designation has been canceled in accordance with § 53.11 or § 53.16.

ISO 9001-registered facility means a manufacturing facility that is either:

(1) An International Organization for Standardization (ISO) 9001-registered manufacturing facility, registered to the ISO 9001 standard (by the Registrar Accreditation Board (RAB) of the American Society for Quality Control (ASQC) in the United States), with registration maintained continuously; or

(2) A facility that can be demonstrated, on the basis of information submitted to the EPA, to be operated according to an EPA-approved and periodically audited quality system which meets, to the extent appropriate, the same general requirements as an ISO 9001-registered facility for the design and manufacture of designated Federal reference method and Federal equivalent method samplers and monitors.

ISO-certified auditor means an auditor who is either certified by the Registrar Accreditation Board (in the United States) as being qualified to audit quality systems using the requirements of recognized standards such as ISO 9001, or who, based on information submitted to the EPA, meets the same general requirements as provided for ISO-certified auditors.

Manual method means a method for measuring concentrations of an ambient air pollutant in which sample collection, analysis, or measurement, or some combination thereof, is performed manually. A method for PM₁₀ or PM_{2.5} which utilizes a sampler that requires manual preparation, loading, and weighing of filter samples is considered a manual method even though the sampler may be capable of automatically collecting a series of sequential samples.

NO means nitrogen oxide.

NO₂ means nitrogen dioxide.

NO_x means oxides of nitrogen and is defined as the sum of the concentrations of NO₂ and NO.

O₃ means ozone.

Operated simultaneously means that two or more collocated samplers or analyzers are operated concurrently with no significant difference in the start time, stop time, and duration of the sampling or measurement period.

Pb means lead.

PM means PM₁₀, PM_{10C}, PM_{2.5}, PM_{10-2.5}, or particulate matter of unspecified size range.

PM_{2.5} means particulate matter with an aerodynamic diameter less than or equal to a nominal 2.5 micrometers as measured by a reference method based on appendix L of part 50 of this chapter and designated in accordance with part 53 of this chapter, by an equivalent method designated in accordance with part 53 of this chapter, or by an approved regional method designated in accordance with appendix C to this part.

PM₁₀ means particulate matter with an aerodynamic diameter less than or equal to a nominal 10 micrometers as measured by a reference method based on appendix J of part 50 of this chapter and designated in accordance with this part or by an equivalent method designated in accordance with this part.

PM_{10c} means particulate matter with an aerodynamic diameter less than or equal to a nominal 10 micrometers as measured by a reference method based on appendix O of part 50 of this chapter and designated in accordance with this part or by an equivalent method designated in accordance with this part.

PM_{10–2.5} means particulate matter with an aerodynamic diameter less than or equal to a nominal 10 micrometers and greater than a nominal 2.5 micrometers as measured by a reference method based on appendix O to part 50 of this chapter and designated in accordance with this part or by an equivalent method designated in accordance with this part.

PM_{2.5} sampler means a device, associated with a manual method for measuring *PM_{2.5}*, designed to collect *PM_{2.5}* from an ambient air sample, but lacking the ability to automatically analyze or measure the collected sample to determine the mass concentrations of *PM_{2.5}* in the sampled air.

PM₁₀ sampler means a device, associated with a manual method for measuring *PM₁₀*, designed to collect *PM₁₀* from an ambient air sample, but lacking the ability to automatically analyze or measure the collected sample to determine the mass concentrations of *PM₁₀* in the sampled air.

PM_{10c} sampler means a *PM₁₀* sampler that meets the special requirements for a *PM_{10c}* sampler that is part of a *PM_{10–2.5}* reference method sampler, as specified in appendix O to part 50 of

this chapter, or a *PM₁₀* sampler that is part of a *PM_{10–2.5}* sampler that has been designated as an equivalent method for *PM_{10–2.5}*.

PM_{10–2.5} sampler means a sampler, or a collocated pair of samplers, associated with a manual method for measuring *PM_{10–2.5}* and designed to collect either *PM_{10–2.5}* directly or *PM_{10c}* and *PM_{2.5}* separately and simultaneously from concurrent ambient air samples, but lacking the ability to automatically analyze or measure the collected sample(s) to determine the mass concentrations of *PM_{10–2.5}* in the sampled air.

Sequential samples for PM samplers means two or more PM samples for sequential (but not necessarily contiguous) time periods that are collected automatically by the same sampler without the need for intervening operator service.

SO₂ means sulfur dioxide.

Test analyzer means an analyzer subjected to testing as part of a candidate method in accordance with subparts B, C, D, E, or F of this part, as applicable.

Test sampler means a *PM₁₀* sampler, *PM_{2.5}* sampler, or *PM_{10–2.5}* sampler subjected to testing as part of a candidate method in accordance with subparts C, D, E, or F of this part.

Ultimate purchaser means the first person or entity who purchases a Federal reference method or a Federal equivalent method for purposes other than resale.

[71 FR 61271, Oct. 17, 2006]

§ 53.2 General requirements for a reference method determination.

The following general requirements for a Federal reference method (FRM) determination are summarized in table A–1 of this subpart.

(a) *Manual methods*—(1) *Sulfur dioxide (SO₂) and lead*. For measuring *SO₂* and lead, appendices A and G of part 50 of this chapter specify unique manual FRM for measuring these pollutants. Except as provided in § 53.16, other manual methods for *SO₂* and lead will not be considered for FRM determinations under this part.

(2) *PM₁₀*. A FRM for measuring *PM₁₀* must be a manual method that meets all requirements specified in appendix

PM_{2.5} means particulate matter with an aerodynamic diameter less than or equal to a nominal 2.5 micrometers as measured by a reference method based on appendix L of part 50 of this chapter and designated in accordance with part 53 of this chapter, by an equivalent method designated in accordance with part 53 of this chapter, or by an approved regional method designated in accordance with appendix C to this part.

PM₁₀ means particulate matter with an aerodynamic diameter less than or equal to a nominal 10 micrometers as measured by a reference method based on appendix J of part 50 of this chapter and designated in accordance with this part or by an equivalent method designated in accordance with this part.

PM_{10c} means particulate matter with an aerodynamic diameter less than or equal to a nominal 10 micrometers as measured by a reference method based on appendix O of part 50 of this chapter and designated in accordance with this part or by an equivalent method designated in accordance with this part.

PM_{10–2.5} means particulate matter with an aerodynamic diameter less than or equal to a nominal 10 micrometers and greater than a nominal 2.5 micrometers as measured by a reference method based on appendix O to part 50 of this chapter and designated in accordance with this part or by an equivalent method designated in accordance with this part.

PM_{2.5} sampler means a device, associated with a manual method for measuring *PM_{2.5}*, designed to collect *PM_{2.5}* from an ambient air sample, but lacking the ability to automatically analyze or measure the collected sample to determine the mass concentrations of *PM_{2.5}* in the sampled air.

PM₁₀ sampler means a device, associated with a manual method for measuring *PM₁₀*, designed to collect *PM₁₀* from an ambient air sample, but lacking the ability to automatically analyze or measure the collected sample to determine the mass concentrations of *PM₁₀* in the sampled air.

PM_{10c} sampler means a *PM₁₀* sampler that meets the special requirements for a *PM_{10c}* sampler that is part of a *PM_{10–2.5}* reference method sampler, as specified in appendix O to part 50 of

this chapter, or a *PM₁₀* sampler that is part of a *PM_{10–2.5}* sampler that has been designated as an equivalent method for *PM_{10–2.5}*.

PM_{10–2.5} sampler means a sampler, or a collocated pair of samplers, associated with a manual method for measuring *PM_{10–2.5}* and designed to collect either *PM_{10–2.5}* directly or *PM_{10c}* and *PM_{2.5}* separately and simultaneously from concurrent ambient air samples, but lacking the ability to automatically analyze or measure the collected sample(s) to determine the mass concentrations of *PM_{10–2.5}* in the sampled air.

Sequential samples for PM samplers means two or more PM samples for sequential (but not necessarily contiguous) time periods that are collected automatically by the same sampler without the need for intervening operator service.

SO₂ means sulfur dioxide.

Test analyzer means an analyzer subjected to testing as part of a candidate method in accordance with subparts B, C, D, E, or F of this part, as applicable.

Test sampler means a *PM₁₀* sampler, *PM_{2.5}* sampler, or *PM_{10–2.5}* sampler subjected to testing as part of a candidate method in accordance with subparts C, D, E, or F of this part.

Ultimate purchaser means the first person or entity who purchases a Federal reference method or a Federal equivalent method for purposes other than resale.

[71 FR 61271, Oct. 17, 2006]

§ 53.2 General requirements for a reference method determination.

The following general requirements for a Federal reference method (FRM) determination are summarized in table A–1 of this subpart.

(a) *Manual methods*—(1) *Sulfur dioxide (SO₂) and lead*. For measuring *SO₂* and lead, appendices A and G of part 50 of this chapter specify unique manual FRM for measuring these pollutants. Except as provided in § 53.16, other manual methods for *SO₂* and lead will not be considered for FRM determinations under this part.

(2) *PM₁₀*. A FRM for measuring *PM₁₀* must be a manual method that meets all requirements specified in appendix

J of part 50 of this chapter and must include a PM_{10} sampler that has been shown in accordance with this part to meet all requirements specified in this subpart A and subpart D of this part.

(3) *PM_{2.5}*. A FRM for measuring $PM_{2.5}$ must be a manual method that meets all requirements specified in appendix L of part 50 of this chapter and must include a $PM_{2.5}$ sampler that has been shown in accordance with this part to meet the applicable requirements specified in this subpart A and subpart E of this part. Further, FRM samplers must be manufactured in an ISO 9001-registered facility, as defined in § 53.1 and as set forth in § 53.51.

(4) *PM_{10-2.5}*. A FRM for measuring $PM_{10-2.5}$ must be a manual method that meets all requirements specified in appendix O of part 50 of this chapter and must include PM_{10C} and $PM_{2.5}$ samplers that have been shown in accordance with this part to meet the applicable requirements specified in this subpart A and subpart E of this part. Further, $PM_{10-2.5}$ FRM samplers must be manufactured in an ISO 9001-registered facility, as defined in § 53.1 and as set forth in § 53.51.

(b) *Automated methods*. An automated FRM for measuring CO, O₃, or NO₂ must utilize the measurement principle and calibration procedure specified in the appropriate appendix to part 50 of this chapter and must have been shown in accordance with this part to meet the requirements specified in this subpart A and subpart B of this part.

[71 FR 61271, Oct. 17, 2006]

§ 53.3 General requirements for an equivalent method determination.

(a) *Manual methods*. A manual Federal equivalent method (FEM) must have been shown in accordance with this part to satisfy the applicable requirements specified in this subpart A and subpart C of this part. In addition, a PM sampler associated with a manual method for PM_{10} , $PM_{2.5}$, or $PM_{10-2.5}$ must have been shown in accordance with this part to satisfy the following additional requirements, as applicable:

(1) *PM₁₀*. A PM_{10} sampler associated with a manual method for PM_{10} must satisfy the requirements of subpart D of this part.

(2) *PM_{2.5} Class I*. A $PM_{2.5}$ Class I FEM sampler must also satisfy all requirements of subpart E of this part, which shall include appropriate demonstration that each and every deviation or modification from the FRM sampler specifications does not significantly alter the performance of the sampler.

(3) *PM_{2.5} Class II*. (i) A $PM_{2.5}$ Class II FEM sampler must also satisfy the applicable requirements of subparts E and F of this part or the alternative requirements in paragraph (a)(3)(ii) of this section.

(ii) In lieu of the applicable requirements specified for Class II $PM_{2.5}$ methods in subparts C and F of this part, a Class II $PM_{2.5}$ FEM sampler may alternatively meet the applicable requirements in paragraphs (b)(3)(i) through (iii) of this section and the testing, performance, and comparability requirements specified for Class III equivalent methods for $PM_{2.5}$ in subpart C of this part.

(4) *PM_{10-2.5} Class I*. A $PM_{10-2.5}$ Class I FEM sampler must also satisfy the applicable requirements of subpart E of this part (there are no additional requirements specifically for Class I $PM_{10-2.5}$ methods in subpart C of this part).

(5) *PM_{10-2.5} Class II*. (i) A $PM_{10-2.5}$ Class II FEM sampler must also satisfy the applicable requirements of subpart C of this part and also the applicable requirements and provisions of paragraphs (b)(3)(i) through (iii) of this section, or the alternative requirements in paragraph (a)(5)(ii) of this section.

(ii) In lieu of the applicable requirements specified for Class II $PM_{10-2.5}$ methods in subpart C of this part and in paragraph (b)(3)(iii) of this section, a Class II $PM_{10-2.5}$ FEM sampler may alternatively meet the applicable requirements in paragraphs (b)(3)(i) and (ii) of this section and the testing, performance, and comparability requirements specified for Class III FEMs for $PM_{10-2.5}$ in subpart C of this part.

(6) *ISO 9001*. All designated FEMs for $PM_{2.5}$ or $PM_{10-2.5}$ must be manufactured in an ISO 9001-registered facility, as defined in § 53.1 and as set forth in § 53.51.

(b) *Automated methods*. All types of automated FEMs must have been shown in accordance with this part to

J of part 50 of this chapter and must include a PM_{10} sampler that has been shown in accordance with this part to meet all requirements specified in this subpart A and subpart D of this part.

(3) *PM_{2.5}*. A FRM for measuring $PM_{2.5}$ must be a manual method that meets all requirements specified in appendix L of part 50 of this chapter and must include a $PM_{2.5}$ sampler that has been shown in accordance with this part to meet the applicable requirements specified in this subpart A and subpart E of this part. Further, FRM samplers must be manufactured in an ISO 9001-registered facility, as defined in § 53.1 and as set forth in § 53.51.

(4) *PM_{10-2.5}*. A FRM for measuring $PM_{10-2.5}$ must be a manual method that meets all requirements specified in appendix O of part 50 of this chapter and must include PM_{10C} and $PM_{2.5}$ samplers that have been shown in accordance with this part to meet the applicable requirements specified in this subpart A and subpart E of this part. Further, $PM_{10-2.5}$ FRM samplers must be manufactured in an ISO 9001-registered facility, as defined in § 53.1 and as set forth in § 53.51.

(b) *Automated methods*. An automated FRM for measuring CO, O₃, or NO₂ must utilize the measurement principle and calibration procedure specified in the appropriate appendix to part 50 of this chapter and must have been shown in accordance with this part to meet the requirements specified in this subpart A and subpart B of this part.

[71 FR 61271, Oct. 17, 2006]

§ 53.3 General requirements for an equivalent method determination.

(a) *Manual methods*. A manual Federal equivalent method (FEM) must have been shown in accordance with this part to satisfy the applicable requirements specified in this subpart A and subpart C of this part. In addition, a PM sampler associated with a manual method for PM_{10} , $PM_{2.5}$, or $PM_{10-2.5}$ must have been shown in accordance with this part to satisfy the following additional requirements, as applicable:

(1) *PM₁₀*. A PM_{10} sampler associated with a manual method for PM_{10} must satisfy the requirements of subpart D of this part.

(2) *PM_{2.5} Class I*. A $PM_{2.5}$ Class I FEM sampler must also satisfy all requirements of subpart E of this part, which shall include appropriate demonstration that each and every deviation or modification from the FRM sampler specifications does not significantly alter the performance of the sampler.

(3) *PM_{2.5} Class II*. (i) A $PM_{2.5}$ Class II FEM sampler must also satisfy the applicable requirements of subparts E and F of this part or the alternative requirements in paragraph (a)(3)(ii) of this section.

(ii) In lieu of the applicable requirements specified for Class II $PM_{2.5}$ methods in subparts C and F of this part, a Class II $PM_{2.5}$ FEM sampler may alternatively meet the applicable requirements in paragraphs (b)(3)(i) through (iii) of this section and the testing, performance, and comparability requirements specified for Class III equivalent methods for $PM_{2.5}$ in subpart C of this part.

(4) *PM_{10-2.5} Class I*. A $PM_{10-2.5}$ Class I FEM sampler must also satisfy the applicable requirements of subpart E of this part (there are no additional requirements specifically for Class I $PM_{10-2.5}$ methods in subpart C of this part).

(5) *PM_{10-2.5} Class II*. (i) A $PM_{10-2.5}$ Class II FEM sampler must also satisfy the applicable requirements of subpart C of this part and also the applicable requirements and provisions of paragraphs (b)(3)(i) through (iii) of this section, or the alternative requirements in paragraph (a)(5)(ii) of this section.

(ii) In lieu of the applicable requirements specified for Class II $PM_{10-2.5}$ methods in subpart C of this part and in paragraph (b)(3)(iii) of this section, a Class II $PM_{10-2.5}$ FEM sampler may alternatively meet the applicable requirements in paragraphs (b)(3)(i) and (ii) of this section and the testing, performance, and comparability requirements specified for Class III FEMs for $PM_{10-2.5}$ in subpart C of this part.

(6) *ISO 9001*. All designated FEMs for $PM_{2.5}$ or $PM_{10-2.5}$ must be manufactured in an ISO 9001-registered facility, as defined in § 53.1 and as set forth in § 53.51.

(b) *Automated methods*. All types of automated FEMs must have been shown in accordance with this part to

satisfy the applicable requirements specified in this subpart A and subpart C of this part. In addition, an automated FEM must have been shown in accordance with this part to satisfy the following additional requirements, as applicable:

(1) An automated FEM for pollutants other than PM must be shown in accordance with this part to satisfy the applicable requirements specified in subpart B of this part.

(2) An automated FEM for PM₁₀ must be shown in accordance with this part to satisfy the applicable requirements of subpart D of this part.

(3) A Class III automated FEM for PM_{2.5} or PM_{10–2.5} must be shown in accordance with this part to satisfy the requirements in paragraphs (b)(3)(i) through (iii) of this section, as applicable.

(i) All pertinent requirements of 40 CFR part 50, appendix L, including sampling height, range of operational conditions, ambient temperature and pressure sensors, outdoor enclosure, electrical power supply, control devices and operator interfaces, data output port, operation/instruction manual, data output and reporting requirements, and any other requirements that would be reasonably applicable to the method, unless adequate (as determined by the Administrator) rationale can be provided to support the contention that a particular requirement does not or should not be applicable to the particular candidate method.

(ii) All pertinent tests and requirements of subpart E of this part, such as instrument manufacturing quality control; final assembly and inspection; manufacturer's audit checklists; leak checks; flow rate accuracy, measurement accuracy, and flow rate cut-off; operation following power interruptions; effect of variations in power line voltage, ambient temperature and ambient pressure; and aerosol transport; unless adequate (as determined by the Administrator) rationale can be provided to support the contention that a particular test or requirement does not or should not be applicable to the particular candidate method.

(iii) Candidate methods shall be tested for and meet any performance requirements, such as inlet aspiration,

particle size separation or selection characteristics, change in particle separation or selection characteristics due to loading or other operational conditions, or effects of surface exposure and particle volatility, determined by the Administrator to be necessary based on the nature, design, and specifics of the candidate method and the extent to which it deviates from the design and performance characteristics of the reference method. These performance requirements and the specific test(s) for them will be determined by Administrator for each specific candidate method or type of candidate method and may be similar to or based on corresponding tests and requirements set forth in subpart F of this part or may be special requirements and tests tailored by the Administrator to the specific nature, design, and operational characteristics of the candidate method. For example, a candidate method with an inlet design deviating substantially from the design of the reference method inlet would likely be subject to an inlet aspiration test similar to that set forth in § 53.63. Similarly, a candidate method having an inertial fractionation system substantially different from that of the reference method would likely be subject to a static fractionation test and a loading test similar to those set forth in §§ 53.64 and 53.65, respectively. A candidate method with more extensive or profound deviations from the design and function of the reference method may be subject to other tests, full wind-tunnel tests similar to those described in § 53.62, or to special tests adapted or developed individually to accommodate the specific type of measurement or operation of the candidate method.

(4) All designated FEM for PM_{2.5} or PM_{10–2.5} must be manufactured in an ISO 9001-registered facility, as defined in § 53.1 and as set forth in § 53.51.

[71 FR 61271, Oct. 17, 2006]

§ 53.4 Applications for reference or equivalent method determinations.

(a) Applications for FRM or FEM determinations shall be submitted in duplicate to: Director, National Exposure Research Laboratory, Reference and Equivalent Method Program (MD–D205–03), U.S. Environmental Protection

satisfy the applicable requirements specified in this subpart A and subpart C of this part. In addition, an automated FEM must have been shown in accordance with this part to satisfy the following additional requirements, as applicable:

(1) An automated FEM for pollutants other than PM must be shown in accordance with this part to satisfy the applicable requirements specified in subpart B of this part.

(2) An automated FEM for PM₁₀ must be shown in accordance with this part to satisfy the applicable requirements of subpart D of this part.

(3) A Class III automated FEM for PM_{2.5} or PM_{10–2.5} must be shown in accordance with this part to satisfy the requirements in paragraphs (b)(3)(i) through (iii) of this section, as applicable.

(i) All pertinent requirements of 40 CFR part 50, appendix L, including sampling height, range of operational conditions, ambient temperature and pressure sensors, outdoor enclosure, electrical power supply, control devices and operator interfaces, data output port, operation/instruction manual, data output and reporting requirements, and any other requirements that would be reasonably applicable to the method, unless adequate (as determined by the Administrator) rationale can be provided to support the contention that a particular requirement does not or should not be applicable to the particular candidate method.

(ii) All pertinent tests and requirements of subpart E of this part, such as instrument manufacturing quality control; final assembly and inspection; manufacturer's audit checklists; leak checks; flow rate accuracy, measurement accuracy, and flow rate cut-off; operation following power interruptions; effect of variations in power line voltage, ambient temperature and ambient pressure; and aerosol transport; unless adequate (as determined by the Administrator) rationale can be provided to support the contention that a particular test or requirement does not or should not be applicable to the particular candidate method.

(iii) Candidate methods shall be tested for and meet any performance requirements, such as inlet aspiration,

particle size separation or selection characteristics, change in particle separation or selection characteristics due to loading or other operational conditions, or effects of surface exposure and particle volatility, determined by the Administrator to be necessary based on the nature, design, and specifics of the candidate method and the extent to which it deviates from the design and performance characteristics of the reference method. These performance requirements and the specific test(s) for them will be determined by Administrator for each specific candidate method or type of candidate method and may be similar to or based on corresponding tests and requirements set forth in subpart F of this part or may be special requirements and tests tailored by the Administrator to the specific nature, design, and operational characteristics of the candidate method. For example, a candidate method with an inlet design deviating substantially from the design of the reference method inlet would likely be subject to an inlet aspiration test similar to that set forth in § 53.63. Similarly, a candidate method having an inertial fractionation system substantially different from that of the reference method would likely be subject to a static fractionation test and a loading test similar to those set forth in §§ 53.64 and 53.65, respectively. A candidate method with more extensive or profound deviations from the design and function of the reference method may be subject to other tests, full wind-tunnel tests similar to those described in § 53.62, or to special tests adapted or developed individually to accommodate the specific type of measurement or operation of the candidate method.

(4) All designated FEM for PM_{2.5} or PM_{10–2.5} must be manufactured in an ISO 9001-registered facility, as defined in § 53.1 and as set forth in § 53.51.

[71 FR 61271, Oct. 17, 2006]

§ 53.4 Applications for reference or equivalent method determinations.

(a) Applications for FRM or FEM determinations shall be submitted in duplicate to: Director, National Exposure Research Laboratory, Reference and Equivalent Method Program (MD-D205-03), U.S. Environmental Protection

Environmental Protection Agency

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Agency, Research Triangle Park, North Carolina 27711 (Commercial delivery address: 4930 Old Page Road, Durham, North Carolina 27703).

(b) Each application shall be signed by an authorized representative of the applicant, shall be marked in accordance with § 53.15 (if applicable), and shall contain the following:

(1) A clear identification of the candidate method, which will distinguish it from all other methods such that the method may be referred to unambiguously. This identification must consist of a unique series of descriptors such as title, identification number, analyte, measurement principle, manufacturer, brand, model, etc., as necessary to distinguish the method from all other methods or method variations, both within and outside the applicant's organization.

(2) A detailed description of the candidate method, including but not limited to the following: The measurement principle, manufacturer, name, model number and other forms of identification, a list of the significant components, schematic diagrams, design drawings, and a detailed description of the apparatus and measurement procedures. Drawings and descriptions pertaining to candidate methods or samplers for $PM_{2.5}$ or $PM_{10-2.5}$ must meet all applicable requirements in reference 1 of appendix A of this subpart, using appropriate graphical, nomenclature, and mathematical conventions such as those specified in references 3 and 4 of appendix A of this subpart.

(3) A copy of a comprehensive operation or instruction manual providing a complete and detailed description of the operational, maintenance, and calibration procedures prescribed for field use of the candidate method and all instruments utilized as part of that method (under § 53.9(a)).

(i) As a minimum this manual shall include:

(A) Description of the method and associated instruments.

(B) Explanation of all indicators, information displays, and controls.

(C) Complete setup and installation instructions, including any additional materials or supplies required.

(D) Details of all initial or startup checks or acceptance tests and any auxiliary equipment required.

(E) Complete operational instructions.

(F) Calibration procedures and descriptions of required calibration equipment and standards.

(G) Instructions for verification of correct or proper operation.

(H) Trouble-shooting guidance and suggested corrective actions for abnormal operation.

(I) Required or recommended routine, periodic, and preventative maintenance and maintenance schedules.

(J) Any calculations required to derive final concentration measurements.

(K) Appropriate references to any applicable appendix of part 50 of this chapter; reference 6 of appendix A of this subpart; and any other pertinent guidelines.

(ii) The manual shall also include adequate warning of potential safety hazards that may result from normal use and/or malfunction of the method and a description of necessary safety precautions. (See § 53.9(b).) However, the previous requirement shall not be interpreted to constitute or imply any warranty of safety of the method by EPA. For samplers and automated methods, the manual shall include a clear description of all procedures pertaining to installation, operation, preventative maintenance, and trouble-shooting and shall also include parts identification diagrams. The manual may be used to satisfy the requirements of paragraphs (b)(1) and (2) of this section to the extent that it includes information necessary to meet those requirements.

(4) A statement that the candidate method has been tested in accordance with the procedures described in subparts B, C, D, E, and/or F of this part, as applicable.

(5) Descriptions of test facilities and test configurations, test data, records, calculations, and test results as specified in subparts B, C, D, E, and/or F of this part, as applicable. Data must be sufficiently detailed to meet appropriate principles described in part B, sections 3.3.1 (paragraph 1) and 3.5.1 and part C, section 4.6 of reference 2 of

appendix A of this subpart; and in paragraphs 1 through 3 of section 4.8 (Records) of reference 5 of appendix A of this subpart. Salient requirements from these references include the following:

(i) The applicant shall maintain and include records of all relevant measuring equipment, including the make, type, and serial number or other identification, and most recent calibration with identification of the measurement standard or standards used and their National Institute of Standards and Technology (NIST) traceability. These records shall demonstrate the measurement capability of each item of measuring equipment used for the application and include a description and justification (if needed) of the measurement setup or configuration in which it was used for the tests. The calibration results shall be recorded and identified in sufficient detail so that the traceability of all measurements can be determined and any measurement could be reproduced under conditions close to the original conditions, if necessary, to resolve any anomalies.

(ii) Test data shall be collected according to the standards of good practice and by qualified personnel. Test anomalies or irregularities shall be documented and explained or justified. The impact and significance of the deviation on test results and conclusions shall be determined. Data collected shall correspond directly to the specified test requirement and be labeled and identified clearly so that results can be verified and evaluated against the test requirement. Calculations or data manipulations must be explained in detail so that they can be verified.

(6) A statement that the method, analyzer, or sampler tested in accordance with this part is representative of the candidate method described in the application.

(c) For candidate automated methods and candidate manual methods for PM_{10} , $PM_{2.5}$, and $PM_{10-2.5}$ the application shall also contain the following:

(1) A detailed description of the quality system that will be utilized, if the candidate method is designated as a reference or equivalent method, to ensure that all analyzers or samplers offered for sale under that designation

will have essentially the same performance characteristics as the analyzer(s) or samplers tested in accordance with this part. In addition, the quality system requirements for candidate methods for $PM_{2.5}$ and $PM_{10-2.5}$ must be described in sufficient detail, based on the elements described in section 4 of reference 1 (Quality System Requirements) of appendix A of this subpart. Further clarification is provided in the following sections of reference 2 of appendix A of this subpart: part A (Management Systems), sections 2.2 (Quality System and Description), 2.3 (Personnel Qualification and Training), 2.4 (Procurement of Items and Services), 2.5 (Documents and Records), and 2.7 (Planning); part B (Collection and Evaluation of Environmental Data), sections 3.1 (Planning and Scoping), 3.2 (Design of Data Collection Operations), and 3.5 (Assessment and Verification of Data Usability); and part C (Operation of Environmental Technology), sections 4.1 (Planning), 4.2 (Design of Systems), and 4.4 (Operation of Systems).

(2) A description of the durability characteristics of such analyzers or samplers (see §53.9(c)). For methods for $PM_{2.5}$ and $PM_{10-2.5}$ the warranty program must ensure that the required specifications (see Table A-1 to this subpart) will be met throughout the warranty period and that the applicant accepts responsibility and liability for ensuring this conformance or for resolving any nonconformities, including all necessary components of the system, regardless of the original manufacturer. The warranty program must be described in sufficient detail to meet appropriate provisions of the ANSI/ASQC and ISO 9001 standards (references 1 and 2 in appendix A of this subpart) for controlling conformance and resolving nonconformance, particularly sections 4.12, 4.13, and 4.14 of reference 1 in appendix A of this subpart.

(i) Section 4.12 in reference 1 of appendix A of this subpart requires the manufacturer to establish and maintain a system of procedures for identifying and maintaining the identification of inspection and test status throughout all phases of manufacturing to ensure that only instruments

that have passed the required inspections and tests are released for sale.

(ii) Section 4.13 in reference 1 of appendix A of this subpart requires documented procedures for control of nonconforming product, including review and acceptable alternatives for disposition; section 4.14 in reference 1 of appendix A of this subpart requires documented procedures for implementing corrective (4.14.2) and preventive (4.14.3) action to eliminate the causes of actual or potential nonconformities. In particular, section 4.14.3 requires that potential causes of nonconformities be eliminated by using information such as service reports and customer complaints to eliminate potential causes of nonconformities.

(d) For candidate reference or equivalent methods for PM_{2.5} and Class II or Class III equivalent methods for PM_{10-2.5}, the applicant, if requested by EPA, shall provide to EPA for test purposes one sampler or analyzer that is representative of the sampler or analyzer associated with the candidate method. The sampler or analyzer shall be shipped FOB destination to Director, National Exposure Research Laboratory, Reference and Equivalent Method Program (MD-D205-03), U.S. Environmental Protection Agency, 4930 Old Page Road, Durham, North Carolina 27703, scheduled to arrive concurrent with or within 30 days of the arrival of the other application materials. This analyzer or sampler may be subjected to various tests that EPA determines to be necessary or appropriate under §53.5(f), and such tests may include special tests not described in this part. If the instrument submitted under this paragraph malfunctions, becomes inoperative, or fails to perform as represented in the application before the necessary EPA testing is completed, the applicant shall be afforded an opportunity to repair or replace the device at no cost to EPA. Upon completion of EPA testing, the analyzer or sampler submitted under this paragraph shall be repacked by EPA for return shipment to the applicant, using the same packing materials used for shipping the instrument to EPA unless alternative packing is provided by the applicant. Arrangements

for, and the cost of, return shipment shall be the responsibility of the applicant. The EPA does not warrant or assume any liability for the condition of the analyzer or sampler upon return to the applicant.

[71 FR 61271, Oct. 17, 2006]

§ 53.5 Processing of applications.

After receiving an application for a FRM or FEM determination, the Administrator will, within 120 calendar days after receipt of the application, take one or more of the following actions:

(a) Send notice to the applicant, in accordance with §53.8, that the candidate method has been determined to be a reference or equivalent method.

(b) Send notice to the applicant that the application has been rejected, including a statement of reasons for rejection.

(c) Send notice to the applicant that additional information must be submitted before a determination can be made and specify the additional information that is needed (in such cases, the 120-day period shall commence upon receipt of the additional information).

(d) Send notice to the applicant that additional test data must be submitted and specify what tests are necessary and how the tests shall be interpreted (in such cases, the 120-day period shall commence upon receipt of the additional test data).

(e) Send notice to the applicant that the application has been found to be substantially deficient or incomplete and cannot be processed until additional information is submitted to complete the application and specify the general areas of substantial deficiency.

(f) Send notice to the applicant that additional tests will be conducted by the Administrator, specifying the nature of and reasons for the additional tests and the estimated time required (in such cases, the 120-day period shall commence 1 calendar day after the additional tests have been completed).

[71 FR 61271, Oct. 17, 2006]

that have passed the required inspections and tests are released for sale.

(ii) Section 4.13 in reference 1 of appendix A of this subpart requires documented procedures for control of nonconforming product, including review and acceptable alternatives for disposition; section 4.14 in reference 1 of appendix A of this subpart requires documented procedures for implementing corrective (4.14.2) and preventive (4.14.3) action to eliminate the causes of actual or potential nonconformities. In particular, section 4.14.3 requires that potential causes of nonconformities be eliminated by using information such as service reports and customer complaints to eliminate potential causes of nonconformities.

(d) For candidate reference or equivalent methods for PM_{2.5} and Class II or Class III equivalent methods for PM_{10-2.5}, the applicant, if requested by EPA, shall provide to EPA for test purposes one sampler or analyzer that is representative of the sampler or analyzer associated with the candidate method. The sampler or analyzer shall be shipped FOB destination to Director, National Exposure Research Laboratory, Reference and Equivalent Method Program (MD-D205-03), U.S. Environmental Protection Agency, 4930 Old Page Road, Durham, North Carolina 27703, scheduled to arrive concurrent with or within 30 days of the arrival of the other application materials. This analyzer or sampler may be subjected to various tests that EPA determines to be necessary or appropriate under § 53.5(f), and such tests may include special tests not described in this part. If the instrument submitted under this paragraph malfunctions, becomes inoperative, or fails to perform as represented in the application before the necessary EPA testing is completed, the applicant shall be afforded an opportunity to repair or replace the device at no cost to EPA. Upon completion of EPA testing, the analyzer or sampler submitted under this paragraph shall be repacked by EPA for return shipment to the applicant, using the same packing materials used for shipping the instrument to EPA unless alternative packing is provided by the applicant. Arrangements

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[71 FR 61271, Oct. 17, 2006]

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§ 53.6 Right to witness conduct of tests.

(a) Submission of an application for a reference or equivalent method determination shall constitute consent for the Administrator or the Administrator's authorized representative, upon presentation of appropriate credentials, to witness or observe any tests required by this part in connection with the application or in connection with any modification or intended modification of the method by the applicant.

(b) The applicant shall have the right to witness or observe any test conducted by the Administrator in connection with the application or in connection with any modification or intended modification of the method by the applicant.

(c) Any tests by either party that are to be witnessed or observed by the other party shall be conducted at a time and place mutually agreeable to both parties.

§ 53.7 Testing of methods at the initiative of the Administrator.

(a) In the absence of an application for a reference or equivalent method determination, the Administrator may conduct the tests required by this part for such a determination, may compile such other information as may be necessary in the judgment of the Administrator to make such a determination, and on the basis of the tests and information may determine that a method satisfies applicable requirements of this part.

(b) In the absence of an application requesting the Administrator to consider revising an appendix to part 50 of this chapter in accordance with § 53.16, the Administrator may conduct such tests and compile such information as may be necessary in the Administrator's judgment to make a determination under § 53.16(d) and on the basis of the tests and information make such a determination.

(c) If a method tested in accordance with this section is designated as a reference or equivalent method in accordance with § 53.8 or is specified or designated as a reference method in accordance with § 53.16, any person or entity who offers the method for sale as

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a reference or equivalent method thereafter shall assume the rights and obligations of an applicant for purposes of this part, with the exception of those pertaining to submission and processing of applications.

§ 53.8 Designation of reference and equivalent methods.

(a) A candidate method determined by the Administrator to satisfy the applicable requirements of this part shall be designated as a FRM or FEM (as applicable) by and upon publication of a notice of the designation in the FEDERAL REGISTER.

(b) Upon designation, a notice indicating that the method has been designated as a FRM or FEM shall be sent to the applicant.

(c) The Administrator will maintain a current list of methods designated as FRM or FEM in accordance with this part and will send a copy of the list to any person or group upon request. A copy of the list will be available for inspection or copying at EPA Regional Offices and may be available via the Internet or other sources.

[71 FR 61276, Oct. 17, 2006]

§ 53.9 Conditions of designation.

Designation of a candidate method as a FRM or FEM shall be conditioned to the applicant's compliance with the following requirements. Failure to comply with any of the requirements shall constitute a ground for cancellation of the designation in accordance with § 53.11.

(a) Any method offered for sale as a FRM or FEM shall be accompanied by a copy of the manual referred to in § 53.4(b)(3) when delivered to any ultimate purchaser, and an electronic copy of the manual suitable for incorporating into user-specific standard operating procedure documents shall be readily available to any users.

(b) Any method offered for sale as a FRM or FEM shall generate no unreasonable hazard to operators or to the environment during normal use or when malfunctioning.

(c) Any analyzer, PM₁₀ sampler, PM_{2.5} sampler, or PM_{10-2.5} sampler offered for sale as part of a FRM or FEM shall function within the limits of the performance specifications referred to in

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(b) The applicant shall have the right to witness or observe any test conducted by the Administrator in connection with the application or in connection with any modification or intended modification of the method by the applicant.

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(b) In the absence of an application requesting the Administrator to consider revising an appendix to part 50 of this chapter in accordance with § 53.16, the Administrator may conduct such tests and compile such information as may be necessary in the Administrator's judgment to make a determination under § 53.16(d) and on the basis of the tests and information make such a determination.

(c) If a method tested in accordance with this section is designated as a reference or equivalent method in accordance with § 53.8 or is specified or designated as a reference method in accordance with § 53.16, any person or entity who offers the method for sale as

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(b) Any method offered for sale as a FRM or FEM shall generate no unreasonable hazard to operators or to the environment during normal use or when malfunctioning.

(c) Any analyzer, PM₁₀ sampler, PM_{2.5} sampler, or PM_{10-2.5} sampler offered for sale as part of a FRM or FEM shall function within the limits of the performance specifications referred to in

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(b) Any method offered for sale as a FRM or FEM shall generate no unreasonable hazard to operators or to the environment during normal use or when malfunctioning.

(c) Any analyzer, PM₁₀ sampler, PM_{2.5} sampler, or PM_{10-2.5} sampler offered for sale as part of a FRM or FEM shall function within the limits of the performance specifications referred to in

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(c) If a method tested in accordance with this section is designated as a reference or equivalent method in accordance with § 53.8 or is specified or designated as a reference method in accordance with § 53.16, any person or entity who offers the method for sale as

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(b) Any method offered for sale as a FRM or FEM shall generate no unreasonable hazard to operators or to the environment during normal use or when malfunctioning.

(c) Any analyzer, PM₁₀ sampler, PM_{2.5} sampler, or PM_{10-2.5} sampler offered for sale as part of a FRM or FEM shall function within the limits of the performance specifications referred to in

§ 53.20(a), § 53.30(a), § 53.50, or § 53.60, as applicable, for at least 1 year after delivery and acceptance when maintained and operated in accordance with the manual referred to in § 53.4(b)(3).

(d) Any analyzer, PM₁₀ sampler, PM_{2.5} sampler, or PM_{10-2.5} sampler offered for sale as a FRM or FEM shall bear a prominent, permanently affixed label or sticker indicating that the analyzer or sampler has been designated by EPA as a FRM or FEM (as applicable) in accordance with this part and displaying any designated method identification number that may be assigned by EPA.

(e) If an analyzer is offered for sale as a FRM or FEM and has one or more selectable ranges, the label or sticker required by paragraph (d) of this section shall be placed in close proximity to the range selector and shall indicate clearly which range or ranges have been designated as parts of the FRM or FEM.

(f) An applicant who offers analyzers, PM₁₀ samplers, PM_{2.5} samplers, or PM_{10-2.5} samplers for sale as FRM or FEMs shall maintain an accurate and current list of the names and mailing addresses of all ultimate purchasers of such analyzers or samplers. For a period of 7 years after publication of the FRM or FEM designation applicable to such an analyzer or sampler, the applicant shall notify all ultimate purchasers of the analyzer or sampler within 30 days if the designation has been canceled in accordance with § 53.11 or § 53.16 or if adjustment of the analyzer or sampler is necessary under § 53.11(b).

(g) If an applicant modifies an analyzer, PM₁₀ sampler, PM_{2.5} sampler, or PM_{10-2.5} sampler that has been designated as a FRM or FEM, the applicant shall not sell the modified analyzer or sampler as a reference or equivalent method nor attach a label or sticker to the modified analyzer or sampler under paragraph (d) or (e) of this section until the applicant has received notice under § 53.14(c) that the existing designation or a new designation will apply to the modified analyzer or sampler or has applied for and received notice under § 53.8(b) of a new FRM or FEM determination for the modified analyzer or sampler.

(h) An applicant who has offered PM_{2.5} or PM_{10-2.5} samplers or analyzers for sale as part of a FRM or FEM may continue to do so only so long as the facility in which the samplers or analyzers are manufactured continues to be an ISO 9001-registered facility, as set forth in subpart E of this part. In the event that the ISO 9001 registration for the facility is withdrawn, suspended, or otherwise becomes inapplicable, either permanently or for some specified time interval, such that the facility is no longer an ISO 9001-registered facility, the applicant shall notify EPA within 30 days of the date the facility becomes other than an ISO 9001-registered facility, and upon such notification, EPA shall issue a preliminary finding and notification of possible cancellation of the FRM or FEM designation under § 53.11.

(i) An applicant who has offered PM_{2.5} or PM_{10-2.5} samplers or analyzers for sale as part of a FRM or FEM may continue to do so only so long as updates of the Product Manufacturing Checklist set forth in subpart E of this part are submitted annually. In the event that an annual Checklist update is not received by EPA within 12 months of the date of the last such submitted Checklist or Checklist update, EPA shall notify the applicant within 30 days that the Checklist update has not been received and shall, within 30 days from the issuance of such notification, issue a preliminary finding and notification of possible cancellation of the reference or equivalent method designation under § 53.11.

[71 FR 61276, Oct. 17, 2006]

§ 53.10 Appeal from rejection of application.

Any applicant whose application for a reference or equivalent method determination has been rejected may appeal the Administrator's decision by taking one or more of the following actions:

(a) The applicant may submit new or additional information in support of the application.

(b) The applicant may request that the Administrator reconsider the data and information already submitted.

(c) The applicant may request that any test conducted by the Administrator that was a material factor in the

§ 53.20(a), § 53.30(a), § 53.50, or § 53.60, as applicable, for at least 1 year after delivery and acceptance when maintained and operated in accordance with the manual referred to in § 53.4(b)(3).

(d) Any analyzer, PM₁₀ sampler, PM_{2.5} sampler, or PM_{10-2.5} sampler offered for sale as a FRM or FEM shall bear a prominent, permanently affixed label or sticker indicating that the analyzer or sampler has been designated by EPA as a FRM or FEM (as applicable) in accordance with this part and displaying any designated method identification number that may be assigned by EPA.

(e) If an analyzer is offered for sale as a FRM or FEM and has one or more selectable ranges, the label or sticker required by paragraph (d) of this section shall be placed in close proximity to the range selector and shall indicate clearly which range or ranges have been designated as parts of the FRM or FEM.

(f) An applicant who offers analyzers, PM₁₀ samplers, PM_{2.5} samplers, or PM_{10-2.5} samplers for sale as FRM or FEMs shall maintain an accurate and current list of the names and mailing addresses of all ultimate purchasers of such analyzers or samplers. For a period of 7 years after publication of the FRM or FEM designation applicable to such an analyzer or sampler, the applicant shall notify all ultimate purchasers of the analyzer or sampler within 30 days if the designation has been canceled in accordance with § 53.11 or § 53.16 or if adjustment of the analyzer or sampler is necessary under § 53.11(b).

(g) If an applicant modifies an analyzer, PM₁₀ sampler, PM_{2.5} sampler, or PM_{10-2.5} sampler that has been designated as a FRM or FEM, the applicant shall not sell the modified analyzer or sampler as a reference or equivalent method nor attach a label or sticker to the modified analyzer or sampler under paragraph (d) or (e) of this section until the applicant has received notice under § 53.14(c) that the existing designation or a new designation will apply to the modified analyzer or sampler or has applied for and received notice under § 53.8(b) of a new FRM or FEM determination for the modified analyzer or sampler.

(h) An applicant who has offered PM_{2.5} or PM_{10-2.5} samplers or analyzers for sale as part of a FRM or FEM may continue to do so only so long as the facility in which the samplers or analyzers are manufactured continues to be an ISO 9001-registered facility, as set forth in subpart E of this part. In the event that the ISO 9001 registration for the facility is withdrawn, suspended, or otherwise becomes inapplicable, either permanently or for some specified time interval, such that the facility is no longer an ISO 9001-registered facility, the applicant shall notify EPA within 30 days of the date the facility becomes other than an ISO 9001-registered facility, and upon such notification, EPA shall issue a preliminary finding and notification of possible cancellation of the FRM or FEM designation under § 53.11.

(i) An applicant who has offered PM_{2.5} or PM_{10-2.5} samplers or analyzers for sale as part of a FRM or FEM may continue to do so only so long as updates of the Product Manufacturing Checklist set forth in subpart E of this part are submitted annually. In the event that an annual Checklist update is not received by EPA within 12 months of the date of the last such submitted Checklist or Checklist update, EPA shall notify the applicant within 30 days that the Checklist update has not been received and shall, within 30 days from the issuance of such notification, issue a preliminary finding and notification of possible cancellation of the reference or equivalent method designation under § 53.11.

[71 FR 61276, Oct. 17, 2006]

§ 53.10 Appeal from rejection of application.

Any applicant whose application for a reference or equivalent method determination has been rejected may appeal the Administrator's decision by taking one or more of the following actions:

(a) The applicant may submit new or additional information in support of the application.

(b) The applicant may request that the Administrator reconsider the data and information already submitted.

(c) The applicant may request that any test conducted by the Administrator that was a material factor in the

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decision to reject the application be repeated.

§ 53.11 Cancellation of reference or equivalent method designation.

(a) *Preliminary finding.* If the Administrator makes a preliminary finding on the basis of any available information that a representative sample of a method designated as a reference or equivalent method and offered for sale as such does not fully satisfy the requirements of this part or that there is any violation of the requirements set forth in § 53.9, the Administrator may initiate proceedings to cancel the designation in accordance with the following procedures.

(b) *Notification and opportunity to demonstrate or achieve compliance.* (1) After making a preliminary finding in accordance with paragraph (a) of this section, the Administrator will send notice of the preliminary finding to the applicant, together with a statement of the facts and reasons on which the preliminary finding is based, and will publish notice of the preliminary finding in the FEDERAL REGISTER.

(2) The applicant will be afforded an opportunity to demonstrate or to achieve compliance with the requirements of this part within 60 days after publication of notice in accordance with paragraph (b)(1) of this section or within such further period as the Administrator may allow, by demonstrating to the satisfaction of the Administrator that the method in question satisfies the requirements of this part, by commencing a program to make any adjustments that are necessary to bring the method into compliance, or by taking such action as may be necessary to cure any violation of the requirements of § 53.9. If adjustments are necessary to bring the method into compliance, all such adjustments shall be made within a reasonable time as determined by the Administrator. If the applicant demonstrates or achieves compliance in accordance with this paragraph (b)(2), the Administrator will publish notice of such demonstration or achievement in the FEDERAL REGISTER.

(c) *Request for hearing.* Within 60 days after publication of a notice in accordance with paragraph (b)(1) of this sec-

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tion, the applicant or any interested person may request a hearing as provided in § 53.12.

(d) *Notice of cancellation.* If, at the end of the period referred to in paragraph (b)(2) of this section, the Administrator determines that the reference or equivalent method designation should be canceled, a notice of cancellation will be published in the FEDERAL REGISTER and the designation will be deleted from the list maintained under § 53.8(c). If a hearing has been requested and granted in accordance with § 53.12, action under this paragraph (d) will be taken only after completion of proceedings (including any administrative review) conducted in accordance with § 53.13 and only if the decision of the Administrator reached in such proceedings is that the designation in question should be canceled.

§ 53.12 Request for hearing on cancellation.

Within 60 days after publication of a notice in accordance with § 53.11(b)(1), the applicant or any interested person may request a hearing on the Administrator's action. If, after reviewing the request and supporting data, the Administrator finds that the request raises a substantial issue of fact, a hearing will be granted in accordance with § 53.13 with respect to such issue. The request shall be in writing, signed by an authorized representative of the applicant or interested person, and shall include a statement specifying:

(a) Any objections to the Administrator's action.

(b) Data or other information in support of such objections.

§ 53.13 Hearings.

(a)(1) After granting a request for a hearing under § 53.12, the Administrator will designate a presiding officer for the hearing.

(2) If a time and place for the hearing have not been fixed by the Administrator, the hearing will be held as soon as practicable at a time and place fixed by the presiding officer, except that the hearing shall in no case be held sooner than 30 days after publication of a notice of hearing in the FEDERAL REGISTER.

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§ 53.11 Cancellation of reference or equivalent method designation.

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(b) *Notification and opportunity to demonstrate or achieve compliance.* (1) After making a preliminary finding in accordance with paragraph (a) of this section, the Administrator will send notice of the preliminary finding to the applicant, together with a statement of the facts and reasons on which the preliminary finding is based, and will publish notice of the preliminary finding in the FEDERAL REGISTER.

(2) The applicant will be afforded an opportunity to demonstrate or to achieve compliance with the requirements of this part within 60 days after publication of notice in accordance with paragraph (b)(1) of this section or within such further period as the Administrator may allow, by demonstrating to the satisfaction of the Administrator that the method in question satisfies the requirements of this part, by commencing a program to make any adjustments that are necessary to bring the method into compliance, or by taking such action as may be necessary to cure any violation of the requirements of § 53.9. If adjustments are necessary to bring the method into compliance, all such adjustments shall be made within a reasonable time as determined by the Administrator. If the applicant demonstrates or achieves compliance in accordance with this paragraph (b)(2), the Administrator will publish notice of such demonstration or achievement in the FEDERAL REGISTER.

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(b) *Notification and opportunity to demonstrate or achieve compliance.* (1) After making a preliminary finding in accordance with paragraph (a) of this section, the Administrator will send notice of the preliminary finding to the applicant, together with a statement of the facts and reasons on which the preliminary finding is based, and will publish notice of the preliminary finding in the FEDERAL REGISTER.

(2) The applicant will be afforded an opportunity to demonstrate or to achieve compliance with the requirements of this part within 60 days after publication of notice in accordance with paragraph (b)(1) of this section or within such further period as the Administrator may allow, by demonstrating to the satisfaction of the Administrator that the method in question satisfies the requirements of this part, by commencing a program to make any adjustments that are necessary to bring the method into compliance, or by taking such action as may be necessary to cure any violation of the requirements of § 53.9. If adjustments are necessary to bring the method into compliance, all such adjustments shall be made within a reasonable time as determined by the Administrator. If the applicant demonstrates or achieves compliance in accordance with this paragraph (b)(2), the Administrator will publish notice of such demonstration or achievement in the FEDERAL REGISTER.

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(b) *Notification and opportunity to demonstrate or achieve compliance.* (1) After making a preliminary finding in accordance with paragraph (a) of this section, the Administrator will send notice of the preliminary finding to the applicant, together with a statement of the facts and reasons on which the preliminary finding is based, and will publish notice of the preliminary finding in the FEDERAL REGISTER.

(2) The applicant will be afforded an opportunity to demonstrate or to achieve compliance with the requirements of this part within 60 days after publication of notice in accordance with paragraph (b)(1) of this section or within such further period as the Administrator may allow, by demonstrating to the satisfaction of the Administrator that the method in question satisfies the requirements of this part, by commencing a program to make any adjustments that are necessary to bring the method into compliance, or by taking such action as may be necessary to cure any violation of the requirements of § 53.9. If adjustments are necessary to bring the method into compliance, all such adjustments shall be made within a reasonable time as determined by the Administrator. If the applicant demonstrates or achieves compliance in accordance with this paragraph (b)(2), the Administrator will publish notice of such demonstration or achievement in the FEDERAL REGISTER.

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Within 60 days after publication of a notice in accordance with § 53.11(b)(1), the applicant or any interested person may request a hearing on the Administrator's action. If, after reviewing the request and supporting data, the Administrator finds that the request raises a substantial issue of fact, a hearing will be granted in accordance with § 53.13 with respect to such issue. The request shall be in writing, signed by an authorized representative of the applicant or interested person, and shall include a statement specifying:

(a) Any objections to the Administrator's action.

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§ 53.13 Hearings.

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(2) If a time and place for the hearing have not been fixed by the Administrator, the hearing will be held as soon as practicable at a time and place fixed by the presiding officer, except that the hearing shall in no case be held sooner than 30 days after publication of a notice of hearing in the FEDERAL REGISTER.

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(3) For purposes of the hearing, the parties shall include EPA, the applicant or interested person(s) who requested the hearing, and any person permitted to intervene in accordance with paragraph (c) of this section.

(4) The Deputy General Counsel or the Deputy General Counsel's representative will represent EPA in any hearing under this section.

(5) Each party other than EPA may be represented by counsel or by any other duly authorized representative.

(b)(1) Upon appointment, the presiding officer will establish a hearing file. The file shall contain copies of the notices issued by the Administrator pursuant to § 53.11(b)(1), together with any accompanying material, the request for a hearing and supporting data submitted therewith, the notice of hearing published in accordance with paragraph (a)(2) of this section, and correspondence and other material data relevant to the hearing.

(2) The hearing file shall be available for inspection by the parties or their representatives at the office of the presiding officer, except to the extent that it contains information identified in accordance with § 53.15.

(c) The presiding officer may permit any interested person to intervene in the hearing upon such a showing of interest as the presiding officer may require; provided that permission to intervene may be denied in the interest of expediting the hearing where it appears that the interests of the person seeking to intervene will be adequately represented by another party (or by other parties), including EPA.

(d)(1) The presiding officer, upon the request of any party or at the officer's discretion, may arrange for a pre-hearing conference at a time and place specified by the officer to consider the following:

- (i) Simplification of the issues.
- (ii) Stipulations, admissions of fact, and the introduction of documents.
- (iii) Limitation of the number of expert witnesses.
- (iv) Possibility of agreement on disposing of all or any of the issues in dispute.
- (v) Such other matters as may aid in the disposition of the hearing, includ-

ing such additional tests as may be agreed upon by the parties.

(2) The results of the conference shall be reduced to writing by the presiding officer and made part of the record.

(e)(1) Hearings shall be conducted by the presiding officer in an informal but orderly and expeditious manner. The parties may offer oral or written evidence, subject to exclusion by the presiding officer of irrelevant, immaterial, or repetitious evidence.

(2) Witnesses shall be placed under oath.

(3) Any witness may be examined or cross-examined by the presiding officer, the parties, or their representatives. The presiding officer may, at his/her discretion, limit cross-examination to relevant and material issues.

(4) Hearings shall be reported verbatim. Copies of transcripts of proceedings may be purchased from the reporter.

(5) All written statements, charts, tabulations, and data offered in evidence at the hearing shall, upon a showing satisfactory to the presiding officer of their authenticity, relevancy, and materiality, be received in evidence and shall constitute part of the record.

(6) Oral argument shall be permitted. The presiding officer may limit oral presentations to relevant and material issues and designate the amount of time allowed for oral argument.

(f)(1) The presiding officer shall make an initial decision which shall include written findings and conclusions and the reasons therefore on all the material issues of fact, law, or discretion presented on the record. The findings, conclusions, and written decision shall be provided to the parties and made part of the record. The initial decision shall become the decision of the Administrator without further proceedings unless there is an appeal to, or review on motion of, the Administrator within 30 calendar days after the initial decision is filed.

(2) On appeal from or review of the initial decision, the Administrator will have all the powers consistent with making the initial decision, including the discretion to require or allow briefs, oral argument, the taking of additional evidence or the remanding to

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the presiding officer for additional proceedings. The decision by the Administrator will include written findings and conclusions and the reasons or basis therefore on all the material issues of fact, law, or discretion presented on the appeal or considered in the review.

§ 53.14 Modification of a reference or equivalent method.

(a) An applicant who offers a method for sale as a reference or equivalent method shall report to the EPA Administrator prior to implementation any intended modification of the method, including but not limited to modifications of design or construction or of operational and maintenance procedures specified in the operation manual (see § 53.9(g)). The report shall be signed by an authorized representative of the applicant, marked in accordance with § 53.15 (if applicable), and addressed as specified in § 53.4(a).

(b) A report submitted under paragraph (a) of this section shall include:

(1) A description, in such detail as may be appropriate, of the intended modification.

(2) A brief statement of the applicant's belief that the modification will, will not, or may affect the performance characteristics of the method.

(3) A brief statement of the probable effect if the applicant believes the modification will or may affect the performance characteristics of the method.

(4) Such further information, including test data, as may be necessary to explain and support any statement required by paragraphs (b)(2) and (b)(3) of this section.

(c) Within 30 calendar days after receiving a report under paragraph (a) of this section, the Administrator will take one or more of the following actions:

(1) Notify the applicant that the designation will continue to apply to the method if the modification is implemented.

(2) Send notice to the applicant that a new designation will apply to the method (as modified) if the modification is implemented, submit notice of the determination for publication in the FEDERAL REGISTER, and revise or

supplement the list referred to in § 53.8(c) to reflect the determination.

(3) Send notice to the applicant that the designation will not apply to the method (as modified) if the modification is implemented and submit notice of the determination for publication in the FEDERAL REGISTER.

(4) Send notice to the applicant that additional information must be submitted before a determination can be made and specify the additional information that is needed (in such cases, the 30-day period shall commence upon receipt of the additional information).

(5) Send notice to the applicant that additional tests are necessary and specify what tests are necessary and how they shall be interpreted (in such cases, the 30-day period shall commence upon receipt of the additional test data).

(6) Send notice to the applicant that additional tests will be conducted by the Administrator and specify the reasons for and the nature of the additional tests (in such cases, the 30-day period shall commence 1 calendar day after the additional tests are completed).

(d) An applicant who has received a notice under paragraph (c)(3) of this section may appeal the Administrator's action as follows:

(1) The applicant may submit new or additional information pertinent to the intended modification.

(2) The applicant may request the Administrator to reconsider data and information already submitted.

(3) The applicant may request that the Administrator repeat any test conducted that was a material factor in the Administrator's determination. A representative of the applicant may be present during the performance of any such retest.

§ 53.15 Trade secrets and confidential or privileged information.

Any information submitted under this part that is claimed to be a trade secret or confidential or privileged information shall be marked or otherwise clearly identified as such in the submittal. Information so identified will be treated in accordance with part 2 of this chapter (concerning public information).

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the presiding officer for additional proceedings. The decision by the Administrator will include written findings and conclusions and the reasons or basis therefore on all the material issues of fact, law, or discretion presented on the appeal or considered in the review.

§ 53.14 Modification of a reference or equivalent method.

(a) An applicant who offers a method for sale as a reference or equivalent method shall report to the EPA Administrator prior to implementation any intended modification of the method, including but not limited to modifications of design or construction or of operational and maintenance procedures specified in the operation manual (see § 53.9(g)). The report shall be signed by an authorized representative of the applicant, marked in accordance with § 53.15 (if applicable), and addressed as specified in § 53.4(a).

(b) A report submitted under paragraph (a) of this section shall include:

(1) A description, in such detail as may be appropriate, of the intended modification.

(2) A brief statement of the applicant's belief that the modification will, will not, or may affect the performance characteristics of the method.

(3) A brief statement of the probable effect if the applicant believes the modification will or may affect the performance characteristics of the method.

(4) Such further information, including test data, as may be necessary to explain and support any statement required by paragraphs (b)(2) and (b)(3) of this section.

(c) Within 30 calendar days after receiving a report under paragraph (a) of this section, the Administrator will take one or more of the following actions:

(1) Notify the applicant that the designation will continue to apply to the method if the modification is implemented.

(2) Send notice to the applicant that a new designation will apply to the method (as modified) if the modification is implemented, submit notice of the determination for publication in the FEDERAL REGISTER, and revise or

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supplement the list referred to in § 53.8(c) to reflect the determination.

(3) Send notice to the applicant that the designation will not apply to the method (as modified) if the modification is implemented and submit notice of the determination for publication in the FEDERAL REGISTER.

(4) Send notice to the applicant that additional information must be submitted before a determination can be made and specify the additional information that is needed (in such cases, the 30-day period shall commence upon receipt of the additional information).

(5) Send notice to the applicant that additional tests are necessary and specify what tests are necessary and how they shall be interpreted (in such cases, the 30-day period shall commence upon receipt of the additional test data).

(6) Send notice to the applicant that additional tests will be conducted by the Administrator and specify the reasons for and the nature of the additional tests (in such cases, the 30-day period shall commence 1 calendar day after the additional tests are completed).

(d) An applicant who has received a notice under paragraph (c)(3) of this section may appeal the Administrator's action as follows:

(1) The applicant may submit new or additional information pertinent to the intended modification.

(2) The applicant may request the Administrator to reconsider data and information already submitted.

(3) The applicant may request that the Administrator repeat any test conducted that was a material factor in the Administrator's determination. A representative of the applicant may be present during the performance of any such retest.

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Any information submitted under this part that is claimed to be a trade secret or confidential or privileged information shall be marked or otherwise clearly identified as such in the submittal. Information so identified will be treated in accordance with part 2 of this chapter (concerning public information).

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§ 53.14 Modification of a reference or equivalent method.

(a) An applicant who offers a method for sale as a reference or equivalent method shall report to the EPA Administrator prior to implementation any intended modification of the method, including but not limited to modifications of design or construction or of operational and maintenance procedures specified in the operation manual (see § 53.9(g)). The report shall be signed by an authorized representative of the applicant, marked in accordance with § 53.15 (if applicable), and addressed as specified in § 53.4(a).

(b) A report submitted under paragraph (a) of this section shall include:

(1) A description, in such detail as may be appropriate, of the intended modification.

(2) A brief statement of the applicant's belief that the modification will, will not, or may affect the performance characteristics of the method.

(3) A brief statement of the probable effect if the applicant believes the modification will or may affect the performance characteristics of the method.

(4) Such further information, including test data, as may be necessary to explain and support any statement required by paragraphs (b)(2) and (b)(3) of this section.

(c) Within 30 calendar days after receiving a report under paragraph (a) of this section, the Administrator will take one or more of the following actions:

(1) Notify the applicant that the designation will continue to apply to the method if the modification is implemented.

(2) Send notice to the applicant that a new designation will apply to the method (as modified) if the modification is implemented, submit notice of the determination for publication in the FEDERAL REGISTER, and revise or

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supplement the list referred to in § 53.8(c) to reflect the determination.

(3) Send notice to the applicant that the designation will not apply to the method (as modified) if the modification is implemented and submit notice of the determination for publication in the FEDERAL REGISTER.

(4) Send notice to the applicant that additional information must be submitted before a determination can be made and specify the additional information that is needed (in such cases, the 30-day period shall commence upon receipt of the additional information).

(5) Send notice to the applicant that additional tests are necessary and specify what tests are necessary and how they shall be interpreted (in such cases, the 30-day period shall commence upon receipt of the additional test data).

(6) Send notice to the applicant that additional tests will be conducted by the Administrator and specify the reasons for and the nature of the additional tests (in such cases, the 30-day period shall commence 1 calendar day after the additional tests are completed).

(d) An applicant who has received a notice under paragraph (c)(3) of this section may appeal the Administrator's action as follows:

(1) The applicant may submit new or additional information pertinent to the intended modification.

(2) The applicant may request the Administrator to reconsider data and information already submitted.

(3) The applicant may request that the Administrator repeat any test conducted that was a material factor in the Administrator's determination. A representative of the applicant may be present during the performance of any such retest.

§ 53.15 Trade secrets and confidential or privileged information.

Any information submitted under this part that is claimed to be a trade secret or confidential or privileged information shall be marked or otherwise clearly identified as such in the submittal. Information so identified will be treated in accordance with part 2 of this chapter (concerning public information).

§ 53.16 Supersession of reference methods.

(a) This section prescribes procedures and criteria applicable to requests that the Administrator specify a new reference method, or a new measurement principle and calibration procedure on which reference methods shall be based, by revision of the appropriate appendix to part 50 of this chapter. Such action will ordinarily be taken only if the Administrator determines that a candidate method or a variation thereof is substantially superior to the existing reference method(s).

(b) In exercising discretion under this section, the Administrator will consider:

(1) The benefits, in terms of the requirements and purposes of the Act, that would result from specifying a new reference method or a new measurement principle and calibration procedure.

(2) The potential economic consequences of such action for State and local control agencies.

(3) Any disruption of State and local air quality monitoring programs that might result from such action.

(c) An applicant who wishes the Administrator to consider revising an appendix to part 50 of this chapter on the ground that the applicant's candidate method is substantially superior to the existing reference method(s) shall submit an application for a reference or equivalent method determination in accordance with § 53.4 and shall indicate therein that such consideration is desired. The application shall include, in addition to the information required by § 53.4, data and any other information supporting the applicant's claim that the candidate method is substantially superior to the existing reference method(s).

(d) After receiving an application under paragraph (c) of this section, the Administrator will publish notice of its receipt in the FEDERAL REGISTER and, within 120 calendar days after receipt of the application, take one of the following actions:

(1) Determine that it is appropriate to propose a revision of the appendix to part 50 of this chapter in question and send notice of the determination to the applicant.

(2) Determine that it is inappropriate to propose a revision of the appendix to part 50 of this chapter in question, determine whether the candidate method is a reference or equivalent method, and send notice of the determinations, including a statement of reasons for the determination not to propose a revision, to the applicant.

(3) Send notice to the applicant that additional information must be submitted before a determination can be made and specify the additional information that is needed (in such cases, the 120-day period shall commence upon receipt of the additional information).

(4) Send notice to the applicant that additional tests are necessary, specifying what tests are necessary and how the test shall be interpreted (in such cases, the 120-day period shall commence upon receipt of the additional test data).

(5) Send notice to the applicant that additional tests will be conducted by the Administrator, specifying the nature of and reasons for the additional tests and the estimated time required (in such cases, the 120-day period shall commence 1 calendar day after the additional tests have been completed).

(e)(1)(i) After making a determination under paragraph (d)(1) of this section, the Administrator will publish a notice of proposed rulemaking in the FEDERAL REGISTER. The notice of proposed rulemaking will indicate that the Administrator proposes:

(A) To revise the appendix to part 50 of this chapter in question.

(B) Where the appendix specifies a measurement principle and calibration procedure, to cancel reference method designations based on the appendix.

(C) To cancel equivalent method designations based on the existing reference method(s).

(ii) The notice of proposed rulemaking will include the terms or substance of the proposed revision, will indicate what period(s) of time the Administrator proposes to allow for replacement of existing methods under section 2.3 of appendix C to part 58 of this chapter, and will solicit public comments on the proposal with particular reference to the considerations

set forth in paragraphs (a) and (b) of this section.

(2)(i) If, after consideration of comments received, the Administrator determines that the appendix to part 50 in question should be revised, the Administrator will, by publication in the FEDERAL REGISTER:

(A) Promulgate the proposed revision, with such modifications as may be appropriate in view of comments received.

(B) Where the appendix to part 50 (prior to revision) specifies a measurement principle and calibration procedure, cancel reference method designations based on the appendix.

(C) Cancel equivalent method designations based on the existing reference method(s).

(D) Specify the period(s) that will be allowed for replacement of existing methods under section 2.3 of appendix C to part 58 of this chapter, with such modifications from the proposed period(s) as may be appropriate in view of comments received.

(3) Canceled designations will be deleted from the list maintained under §53.8(c). The requirements and procedures for cancellation set forth in §53.11 shall be inapplicable to cancella-

tion of reference or equivalent method designations under this section.

(4) If the appendix to part 50 of this chapter in question is revised to specify a new measurement principle and calibration procedure on which the applicant's candidate method is based, the Administrator will take appropriate action under §53.5 to determine whether the candidate method is a reference method.

(5) Upon taking action under paragraph (e)(2) of this section, the Administrator will send notice of the action to all applicants for whose methods reference and equivalent method designations are canceled by such action.

(f) An applicant who has received notice of a determination under paragraph (d)(2) of this section may appeal the determination by taking one or more of the following actions:

(1) The applicant may submit new or additional information in support of the application.

(2) The applicant may request that the Administrator reconsider the data and information already submitted.

(3) The applicant may request that any test conducted by the Administrator that was a material factor in making the determination be repeated.

TABLE A-1 TO SUBPART A OF PART 53—SUMMARY OF APPLICABLE REQUIREMENTS FOR REFERENCE AND EQUIVALENT METHODS FOR AIR MONITORING OF CRITERIA POLLUTANTS

Pollutant	Ref. or equivalent	Manual or automated	Applicable part 50 appendix	Applicable subparts of part 53					
				A	B	C	D	E	F
SO ₂	Reference	Manual	A						
	Equivalent	Manual	✓	✓					
CO	Reference	Automated	C	✓	✓				
	Equivalent	Manual	✓	✓					
O ₃	Reference	Automated	D	✓	✓				
	Equivalent	Manual	✓	✓					
NO ₂	Reference	Automated	F	✓	✓				
	Equivalent	Manual	✓	✓					
Pb	Reference	Automated	G	✓	✓				
	Equivalent	Manual	✓	✓					
PM ₁₀	Reference	Manual	J	✓	✓				
	Equivalent	Manual	✓	✓					
PM _{2.5}	Reference	Automated	L	✓	✓				
	Equivalent Class I	Manual	L	✓	✓				
PM _{10-2.5}	Equivalent Class II	Manual	L ¹	✓	✓ ²	✓	✓ ^{1,2}		
	Equivalent Class III	Automated	L ¹	✓	✓ ¹	✓ ¹	✓ ¹		
	Reference	Manual	O ²	✓	✓				
	Equivalent Class I	Manual	O ₂	✓	✓				
Equivalent Class II	Manual	O ²	✓	✓ ²	✓ ¹	✓ ^{1,2}			

Pollutant	Ref. or equivalent	Manual or automated	Applicable part 50 appendix	Applicable subparts of part 53					
				A	B	C	D	E	F
	Equivalent Class III	Automated	L ¹ , O ^{1,2}	✓	✓	✓ ¹	✓ ¹		

¹ Some requirements may apply, based on the nature of each particular candidate method, as determined by the Administrator.

² Alternative Class III requirements may be substituted.

[71 FR 61276, Oct. 17, 2006]

APPENDIX A TO SUBPART A OF PART 53—
REFERENCES

(1) American National Standard Quality Systems—Model for Quality Assurance in Design, Development, Production, Installation, and Servicing, ANSI/ISO/ASQC Q9001-1994. Available from American Society for Quality, P.O. Box 3005, Milwaukee, WI 53202 (<http://qualitypress.asq.org>).

(2) American National Standard Quality Systems for Environmental Data and Technology Programs—Requirements with guidance for use, ANSI/ASQC E4-2004. Available from American Society for Quality P.O. Box 3005, Milwaukee, WI 53202 (<http://qualitypress.asq.org>).

(3) Dimensioning and Tolerancing, ASME Y14.5M-1994. Available from the American Society of Mechanical Engineers, 345 East 47th Street, New York, NY 10017.

(4) Mathematical Definition of Dimensioning and Tolerancing Principles, ASME Y14.5.1M-1994. Available from the American Society of Mechanical Engineers, 345 East 47th Street, New York, NY 10017.

(5) ISO 10012, Quality Assurance Requirements for Measuring Equipment-Part 1: Meteorological confirmation system for measuring equipment:1992(E). Available from American Society for Quality Control, 611 East Wisconsin Avenue, Milwaukee, WI 53202.

(6) Quality Assurance Guidance Document 2.12. Monitoring PM_{2.5} in Ambient Air Using Designated Reference or Class I Equivalent Methods. U.S. EPA, National Exposure Research Laboratory, Research Triangle Park, NC, November 1998 or later edition. Currently available at <http://www.epa.gov/ttn/amtic/pmqaainf.html>.

[62 FR 38784, July 18, 1997, as amended at 71 FR 61278, Oct. 17, 2006]

Subpart B—Procedures for Testing Performance Characteristics of Automated Methods SO₂, CO, O₃, and NO₂

§ 53.20 General provisions.

(a) The test procedures given in this subpart shall be used to test the performance of candidate automated methods against the performance spec-

ifications given in table B-1. A test analyzer representative of the candidate automated method must exhibit performance better than, or equal to, the specified value for each such specification (except Range) to satisfy the requirements of this subpart. Except as provided in paragraph (b) of this section, the range of the candidate method must be the range specified in table B-1 to satisfy the requirements of this subpart.

(b) For a candidate method having more than one selectable range, one range must be that specified in table B-1 and a test analyzer representative of the method must pass the tests required by this subpart while operated in that range. The tests may be repeated for a broader range (i.e., one extending to higher concentrations) than that specified in table B-1 provided that the range does not extend to concentrations more than two times the upper range limit specified in table B-1. If the application is for a reference method determination, the tests may be repeated for a narrower range (one extending to lower concentrations) than that specified in table B-1.

If the tests are conducted or passed only for the specified range, any reference or equivalent method determination with respect to the method will be limited to that range. If the tests are passed for both the specified range and a broader range (or ranges), any such determination will include the broader range(s) as well as the specified range, provided that the tests required by subpart C of this part (if applicable) are met for the broader range(s). If the tests are passed for both the specified range and a narrower range, a reference method determination for the method will include the narrower range as well as the specified range. Appropriate test data shall be submitted for each range sought to be included in a reference or equivalent

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method determination under this paragraph (b).

(c) For each performance specification (except Range), the test procedure shall be initially repeated seven (7) times to yield 7 test results. Each result shall be compared with the corresponding specification in table B-1; a value higher than or outside that specified constitutes a failure. These 7 results for each parameter shall be interpreted as follows:

(1) Zero (0) failures: Candidate method passes the performance parameter.

(2) Three (3) or more failures: Candidate method fails the performance parameter.

(3) One (1) or two (2) failures: Repeat the test procedures for the parameter eight (8) additional times yielding a total of fifteen (15) test results. The combined total of 15 test results shall then be interpreted as follows:

(i) One (1) or two (2) failures: Candidate method passes the performance parameter.

(ii) Three (3) or more failures: Candidate method fails the performance parameter.

TABLE B-1—PERFORMANCE SPECIFICATIONS FOR AUTOMATED METHODS

Performance parameter	Units ¹	Sulfur dioxide	Photochemical oxidants	Carbon monoxide	Nitrogen dioxide	Definitions and test procedures
1. Range	Parts per million ...	0-0.5	0-0.5	0-50	0-0.5	Sec. 53.23(a).
2. Noisedo005	.005	.50	.005	Sec. 53.23(b).
3. Lower detectable limit	Parts per million01	.01	1.0	.01	Sec. 53.23(c).
4. Interference equivalentdododododo	Sec. 53.23(d).
Each interferant	Parts per million ...	±.02	±.02	±1.0	±0.02	
Total interferantdo06	.06	1.5	.04	
5. Zero drift, 12 and 24 hourdo	±.02	±.02	±1.0	±.02	Sec. 52.23(e).
6. Span drift, 24 hourdododododo	Do.
20 percent of upper range limit	Percent	±20.0	±20.0	±10.0	±20.0	
80 percent of upper range limitdo	±5.0	±5.0	±2.5	±5.0	
7. Lag time	Minutes	20	20	10	20	Do.
8. Rise timedo	15	15	5	15	Do.
9. Fall timedo	15	15	5	15	Do.
10. Precisiondododododo	Do.
20 percent of upper range limit	Parts per million01	.01	.5	.02	
80 percent of upper range limitdo015	.01	.5	.03	

¹ To convert from parts per million to µg/m³ at 25 °C and 760 mm Hg, multiply by M/0.02447, where M is the molecular weight of the gas.

(d) The tests for *zero drift*, *span drift*, *lag time*, *rise time*, *fall time*, and *precision* shall be combined into a single sequential procedure to be conducted at various line voltages and ambient temperatures specified in § 53.23(e). The tests for *noise*, *lower detectable limit*, and *interference equivalents* shall be made at any temperature between 20 °C. and 30 °C. and at any normal line voltage between 105 and 125 volts, and shall be conducted such that not more than three (3) test results for each parameter are obtained per 24 hours.

(e) All response readings to be recorded shall first be converted to concentration units according to the calibration curve constructed in accordance with § 53.21(b).

(f) All recorder chart tracings, records, test data and other documentation obtained from or pertinent

to these tests shall be identified, dated, signed by the analyst performing the test, and submitted.

NOTE: Suggested formats for reporting the test results and calculations are provided in Figures B-2, B-3, B-4, B-5, and B-6 in appendix A. Symbols and abbreviations used in this subpart are listed in table B-5, appendix A.

[40 FR 7049, Feb. 18, 1975, as amended at 40 FR 18168, Apr. 25, 1975; 41 FR 52694, Dec. 1, 1976]

§ 53.21 Test conditions.

(a) *Set-up and start-up* of the test analyzer shall be in strict accordance with the operating instructions specified in the manual referred to in § 53.4(b)(3). Allow adequate warm-up or stabilization time as indicated in the operating instructions before beginning the tests.

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method determination under this paragraph (b).

(c) For each performance specification (except Range), the test procedure shall be initially repeated seven (7) times to yield 7 test results. Each result shall be compared with the corresponding specification in table B-1; a value higher than or outside that specified constitutes a failure. These 7 results for each parameter shall be interpreted as follows:

(1) Zero (0) failures: Candidate method passes the performance parameter.

(2) Three (3) or more failures: Candidate method fails the performance parameter.

(3) One (1) or two (2) failures: Repeat the test procedures for the parameter eight (8) additional times yielding a total of fifteen (15) test results. The combined total of 15 test results shall then be interpreted as follows:

(i) One (1) or two (2) failures: Candidate method passes the performance parameter.

(ii) Three (3) or more failures: Candidate method fails the performance parameter.

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1. Range	Parts per million ...	0-0.5	0-0.5	0-50	0-0.5	Sec. 53.23(a).
2. Noisedo005	.005	.50	.005	Sec. 53.23(b).
3. Lower detectable limit	Parts per million01	.01	1.0	.01	Sec. 53.23(c).
4. Interference equivalentdododododo	Sec. 53.23(d).
Each interferant	Parts per million ...	±.02	±.02	±1.0	±0.02	
Total interferantdo06	.06	1.5	.04	
5. Zero drift, 12 and 24 hourdo	±.02	±.02	±1.0	±.02	Sec. 52.23(e).
6. Span drift, 24 hourdododododo	Do.
20 percent of upper range limit	Percent	±20.0	±20.0	±10.0	±20.0	
80 percent of upper range limitdo	±5.0	±5.0	±2.5	±5.0	
7. Lag time	Minutes	20	20	10	20	Do.
8. Rise timedo	15	15	5	15	Do.
9. Fall timedo	15	15	5	15	Do.
10. Precisiondododododo	Do.
20 percent of upper range limit	Parts per million01	.01	.5	.02	
80 percent of upper range limitdo015	.01	.5	.03	

¹ To convert from parts per million to µg/m³ at 25 °C and 760 mm Hg, multiply by M/0.02447, where M is the molecular weight of the gas.

(d) The tests for *zero drift*, *span drift*, *lag time*, *rise time*, *fall time*, and *precision* shall be combined into a single sequential procedure to be conducted at various line voltages and ambient temperatures specified in § 53.23(e). The tests for *noise*, *lower detectable limit*, and *interference equivalents* shall be made at any temperature between 20 °C. and 30 °C. and at any normal line voltage between 105 and 125 volts, and shall be conducted such that not more than three (3) test results for each parameter are obtained per 24 hours.

(e) All response readings to be recorded shall first be converted to concentration units according to the calibration curve constructed in accordance with § 53.21(b).

(f) All recorder chart tracings, records, test data and other documentation obtained from or pertinent

to these tests shall be identified, dated, signed by the analyst performing the test, and submitted.

NOTE: Suggested formats for reporting the test results and calculations are provided in Figures B-2, B-3, B-4, B-5, and B-6 in appendix A. Symbols and abbreviations used in this subpart are listed in table B-5, appendix A.

[40 FR 7049, Feb. 18, 1975, as amended at 40 FR 18168, Apr. 25, 1975; 41 FR 52694, Dec. 1, 1976]

§ 53.21 Test conditions.

(a) *Set-up and start-up* of the test analyzer shall be in strict accordance with the operating instructions specified in the manual referred to in § 53.4(b)(3). Allow adequate warm-up or stabilization time as indicated in the operating instructions before beginning the tests.

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.

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.

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(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'Keefe, A. E., and Ortman, 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 $r_1, r_2, r_3 \dots r_i \dots r_{25}$.

(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

(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 $r_1, r_2, r_3 \dots r_i \dots r_{25}$.

(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

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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.

(d) *Interference equivalent*—(1) *Technical definition*. Positive or negative response caused by a substance other than the one being measured.

(2) *Test procedure*. The test analyzer shall be tested for all substances likely to cause a detectable response. The test analyzer shall be challenged, in turn, with each interfering agent specified in table B-3. In the event that there are substances likely to cause a significant interference which have not been specified in table B-3, these substances shall be tested at a concentration substantially higher than that normally found in the ambient air. The

interference may be either positive or negative, depending on whether the test analyzer's response is increased or decreased by the presence of the interferent. Interference equivalents shall be determined by mixing each interferent, one at a time, with the pollutant at the concentrations specified in table B-3, and comparing the test analyzer's response to the response caused by the pollutant alone. Known gas-phase reactions that might occur between an interferent and the pollutant are designated by footnote 3 in table B-3. In these cases, the interference equivalent shall be determined in the absence of the pollutant.

(i) Allow sufficient time for warm-up and stabilization of the test analyzer.

(ii) For a candidate method using a prefilter or scrubber based upon a chemical reaction to derive part of its specificity, and which requires periodic service or maintenance, the test analyzer shall be "conditioned" prior to each interference test as follows:

TABLE B-3—INTERFERANT TEST CONCENTRATION,¹ PARTS PER MILLION

Pollutant	Analyzer type ²	Hydrochloric acid	Ammonia	Hydrogen sulfide	Sulfur dioxide	Nitrogen dioxide	Nitric oxide	Carbon dioxide	Ethylene	Ozone	M-xylene	Water vapor	Carbon monoxide	Methane	Ethane
SO ₂	Flame photometric (FPD)			0.1	1.0,14			750				320,000	50		
SO ₂	Gas chromatography (FPD)			.1	4.14			750				320,000	50		
SO ₂	Spectrophotometric-wet chemical (pararosaniline reaction)	0.2	3 0.1	.1	4.14	0.5		750		0.5					
SO ₂	Electrochemical	.2	3 1	.1	4.14	.5	0.5		0.2	.5		320,000			
SO ₂	Conductivity	.2	3 1		4.14	.5		750							
SO ₂	Spectrophotometric-gas phase				4.14	.5	.5				0.2				
O ₃	Chemiluminescent			3 1				750		4.08		320,000			
O ₃	Electrochemical			3 1	.5	.5				4.08		320,000			
O ₃	Spectrophotometric-wet chemical (potassium iodide reaction)			3 1	.5	.5	3.5			4.08					
O ₃	Spectrophotometric-gas phase				.5	.5	3.5			4.08					
CO	Infrared							750				20,000	4 10		0.5
CO	Gas chromatography with flame ionization detector											20,000	4 10		
CO	Electrochemical						.5		.2			20,000	4 10	5.0	.5
CO	Catalytic combustion-thermal detection			.1				750	.2			20,000	4 10		
CO	IR fluorescence							750				20,000	4 10		.5
CO	Mercury replacement UV photometric								.2				4 10		.5
NO ₂	Chemiluminescent			3 1	.5	4.1	.5					20,000			
NO ₂	Spectrophotometric-wet chemical (azo-dye reaction)				.5	4.1	.5	750		.5					
NO ₂	Electrochemical	0.2	3 1		.5	4.1	.5	750		.5		20,000	50		
NO ₂	Spectrophotometric-gas phase		3 1		.5	4.1	.5			.5		20,000	50		

¹ Concentrations of interferant listed must be prepared and controlled to ±10 percent of the state value.

² Analyzer types not listed will be considered by the administrator as special cases.

³ Do not mix with pollutant.

⁴ Concentration of pollutant used for test. These pollutant concentrations must be prepared to ±10 percent of the stated value.

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(A) Service or perform the indicated maintenance on the scrubber or prefilter as directed in the manual referred to in § 53.4(b)(3).

(B) Before testing for each interferent, allow the test analyzer to sample through the scrubber a test atmosphere containing the interferent at a concentration equal to the value specified in table B-3. Sampling shall be at the normal flow rate and shall be continued for 6 continuous hours prior to testing.

(iii) Generate three test atmosphere streams as follows:

(A) Test atmosphere *P*: Pollutant concentration.

(B) Test atmosphere *I*: Interference concentration.

(C) Test atmosphere *Z*: Zero air.

(iv) Adjust the individual flow rates and the pollutant or interferent generators for the three test atmospheres as follows:

(A) The flow rates of test atmospheres *I* and *Z* shall be identical.

(B) The concentration of pollutant in test atmosphere *P* shall be adjusted such that when *P* is mixed (diluted) with either test atmosphere *I* or *Z*, the resulting concentration of pollutant shall be as specified in table B-3.

(C) The concentration of interferent in test atmosphere *I* shall be adjusted such that when *I* is mixed (diluted) with test atmosphere *P*, the resulting concentration of interferent shall be equal to the value specified in table B-3.

(D) To minimize concentration errors due to flow rate differences between *I* and *Z*, it is recommended that, when possible, the flow rate of *P* be from 10 to 20 times larger than the flow rates of *I* and *Z*.

(v) Mix test atmospheres *P* and *Z* by passing the total flow of both atmospheres through a mixing flask.

(vi) Sample and measure the mixture of test atmospheres *P* and *Z* with the test analyzer. Allow for a stable reading, and record the reading, in concentration units, as *R* (see Figure B-3).

(vii) Mix test atmospheres *P* and *I* by passing the total flow of both atmospheres through a mixing flask.

(viii) Sample and measure this mixture. Record the stable reading, in concentration units, as *R_I*.

(ix) Calculate the interference equivalent (*IE*) as:

$$IE = R_I - R$$

IE must be equal to or less than the specification given in table B-1 for each interferent to pass the test.

(x) Follow steps (iii) through (ix) of this section, in turn, to determine the interference equivalent for each interferent.

(xi) For those interferents which cannot be mixed with the pollutant, as indicated by footnote (3) in table B-3, adjust the concentration of test atmosphere *I* to the specified value without being mixed or diluted by the pollutant test atmosphere. Determine *IE* as follows:

(A) Sample and measure test atmosphere *Z* (zero air). Allow for a stable reading and record the reading, in concentration units, as *R*.

(B) Sample and measure the interferent test atmosphere *I*. If the test analyzer is not capable of negative readings, adjust the analyzer (not the recorder) to give an offset zero. Record the stable reading in concentration units as *R_I*, extrapolating the calibration curve, if necessary, to represent negative readings.

(C) Calculate $IE = R_I - R$. *IE* must be equal to or less than the specification in table B-1 to pass the test.

(xii) Sum the absolute value of all the individual interference equivalents. This sum must be equal to or less than the total interferent specification given in table B-1 to pass the test.

(e) *Zero drift, span drift, lag time, rise time, fall time, and precision*—(1) *Technical definitions*—(i) *Zero drift*: The change in response to zero pollutant concentration, over 12- and 24-hour periods of continuous unadjusted operation.

(ii) *Span drift*: The percent change in response to an up-scale pollutant concentration over a 24-hour period of continuous unadjusted operation.

(iii) *Lag time*: The time interval between a step change in input concentration and the first observable corresponding change in response.

(iv) *Rise time*: The time interval between initial response and 95 percent of final response after a step increase in input concentration.

(v) *Fall time:* The time interval between initial response and 95 percent of final response after a step decrease in input concentration.

(vi) *Precision:* Variation about the mean of repeated measurements of the same pollutant concentration, expressed as one standard deviation about the mean.

(2) Tests for these performance parameters shall be accomplished over a period of seven (7) or more days. During this time, the line voltage supplied to the test analyzer and the ambient temperature surrounding the analyzer shall be varied from day to day. One test result for each performance parameter shall be obtained each test day, for seven (7) or fifteen (15) test days as necessary. The tests are performed sequentially in a single procedure.

(3) The 24-hour test day may begin at any clock hour. The first 12 hours out of each test day are required for testing 12-hour zero drift. Tests for the other parameters shall be conducted during the remaining 12 hours.

(4) Table B-4 specifies the line voltage and room temperature to be used for each test day. The line voltage and temperature shall be changed to the specified values at the start of each test day (i.e., at the start of the 12-hour zero test). Initial adjustments (day zero) shall be made at a line voltage of 115 volts (rms) and a room temperature of 25 °C.

(5) The tests shall be conducted in blocks consisting of 3 test days each until 7 or 15 test results have been obtained. (The final block may contain fewer than three test days.) If a test is interrupted by an occurrence other

than a malfunction of the test analyzer, only the block during which the interruption occurred shall be repeated.

(6) During each block, manual adjustments to the electronics, gas, or reagent flows or periodic maintenance shall not be permitted. Automatic adjustments which the test analyzer performs by itself are permitted at any time.

(7) At least 4 hours prior to the start of the first test day of each block, the test analyzer may be adjusted and/or serviced according to the periodic maintenance procedures specified in the manual referred to in §53.4(b)(3). If a new block is to immediately follow a previous block, such adjustments or servicing may be done immediately after completion of the day's tests for the last day of the previous block and at the voltage and temperature specified for that day, but only on test days 3, 6, 9, and 12.

NOTE: If necessary, the beginning of the test days succeeding such maintenance or adjustment may be delayed as necessary to complete the service or adjustment operation.

(8) All response readings to be recorded shall first be converted to concentration units according to the calibration curve. Whenever a test atmosphere is to be measured but a stable reading is not required, the test atmosphere shall be measured long enough to cause a change in response of at least 10% of full scale. Identify all readings and other pertinent data on the strip chart. (See Figure B-1 illustrating the pattern of the required readings.)

TABLE B-4—LINE VOLTAGE AND ROOM TEMPERATURE TEST CONDITIONS

Test day	Line voltage, ¹ rms	Room temperature, ² °C	Comments
0	115	25	Initial set-up and adjustments.
1	125	20	
2	105	20	
3	125	30	Adjustments and/or periodic maintenance permitted at end of tests.
4	105	30	
5	125	20	
6	105	20	Adjustments and/or periodic maintenance permitted at end of tests.
7	125	30	Examine test results to ascertain if further testing is required.
8	105	30	
9	125	20	Adjustments and/or periodic maintenance permitted at end of tests.
10	105	20	
11	125	30	
12	105	30	Adjustments and/or periodic maintenance permitted at end of tests.

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TABLE B-4—LINE VOLTAGE AND ROOM TEMPERATURE TEST CONDITIONS—Continued

Test day	Line voltage, ¹ rms	Room temperature, ² °C	Comments
13	125	20	
14	105	20	
15	125	30	

¹ Voltage specified shall be controlled to ±1 volt.
² Temperature specified shall be controlled to ±1 °C.

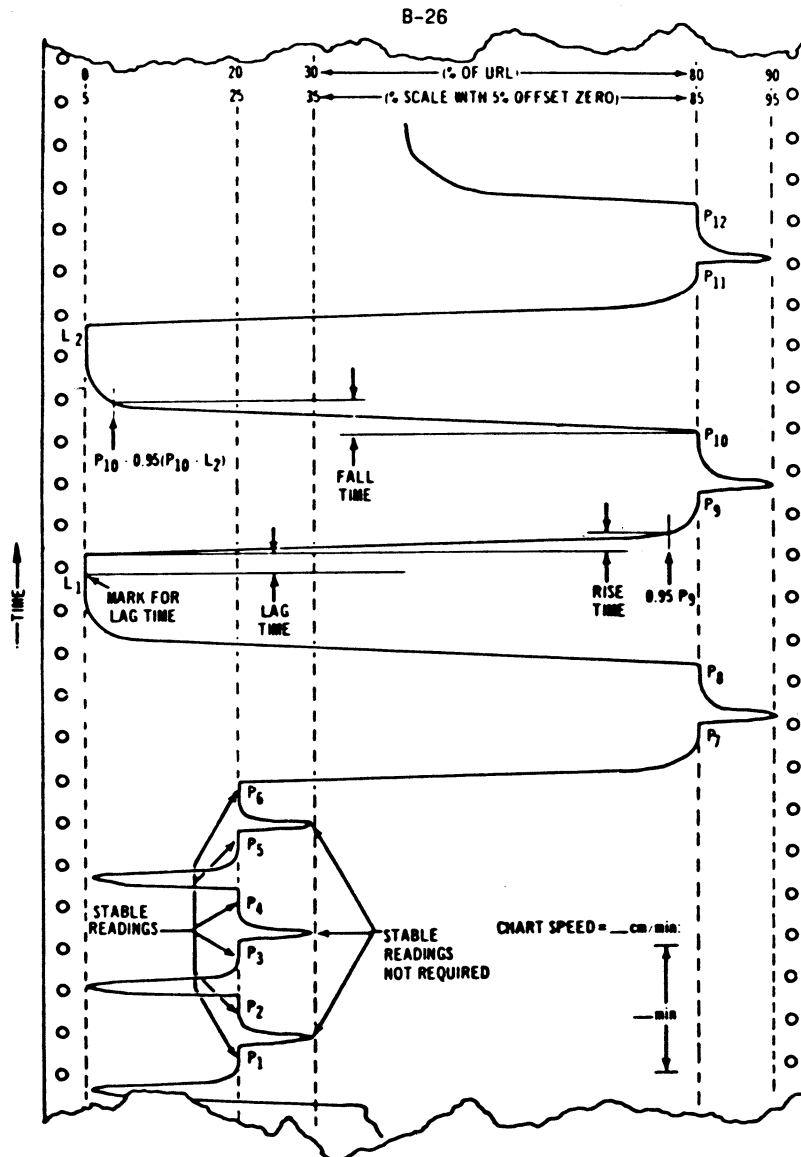


Figure B-1. Example showing the nature of the tracing obtained during the test for drift, lag time, rise time, fall time, and precision. The time scale has been greatly compressed.

(9) Test procedure. (i) Arrange to generate pollutant test atmospheres as follows:

Test atmosphere	Pollutant concentration (percent)
A ₀	Zero air.
A ₂₀	20±5 of the upper range limit.
A ₃₀	30±5 of the upper range limit.

Test atmosphere	Pollutant concentration (percent)
A ₈₀	80±5 of the upper range limit.
A ₉₀	90±5 of the upper range limit.

Test atmospheres A₀, A₂₀, and A₈₀ shall be consistent during the tests and from day to day.

(ii) For steps (xxv) through (xxxi) of this section, a chart speed of at least 10 centimeters per hour shall be used. The actual chart speed, chart speed changes, and time checks shall be clearly marked on the chart.

(iii) Allow sufficient time for test analyzer to warm up and stabilize at a line voltage of 115 volts and a room temperature of 25 °C. Recalibrate, if necessary, and adjust the zero baseline to 5 percent of chart. No further adjustments shall be made to the analyzer until the end of the tests on the third day.

(iv) Measure test atmosphere A₀ until a stable reading is obtained, and record this reading (in ppm) as Z_n, where n = 0 (see Figure B-4 in appendix A).

(v) Measure test atmosphere A₂₀. Allow for a stable reading and record it as M_n, where n = 0.

(vi) Measure test atmosphere A₈₀. Allow for a stable reading and record it as S_n, where n = 0.

(vii) The above readings for Z₀, M₀, and S₀ should be taken at least four (4) hours prior to the beginning of test day 1.

(viii) At the beginning of each test day, adjust the line voltage and room temperature to the values given in table B-4.

(ix) Measure test atmosphere A₀ continuously for at least twelve (12) continuous hours during each test day.

(x) After the 12-hour zero drift test (step ix), sample test atmosphere A₀. A stable reading is not required.

(xi) Measure test atmosphere A₂₀ and record the stable reading (in ppm) as P₁. (See Figure B-4 in appendix A.)

(xii) Sample test atmosphere A₃₀; a stable reading is not required.

(xiii) Measure test atmosphere A₂₀ and record the stable reading as P₂.

(xiv) Sample test atmosphere A₀; a stable reading is not required.

(xv) Measure test atmosphere A₂₀ and record the stable reading as P₃.

(xvi) Sample test atmosphere A₃₀; a stable reading is not required.

(xvii) Measure test atmosphere A₂₀ and record the stable reading as P₄.

(xviii) Sample test atmosphere A₀; a stable reading is not required.

(xix) Measure test atmosphere A₂₀ and record the stable reading as P₅.

(xx) Sample test atmosphere A₃₀; a stable reading is not required.

(xxi) Measure test atmosphere A₂₀ and record the stable reading as P₆.

(xxii) Measure test atmosphere A₃₀ and record the stable reading as P₇.

(xxiii) Sample test atmosphere A₉₀; a stable reading is not required.

(xxiv) Measure test atmosphere A₈₀ and record the stable reading as P₈. Increase chart speed to at least 10 centimeters per hour.

(xxv) Measure test atmosphere A₀. Record the stable reading as L₁.

(xxvi) Quickly switch the test analyzer to measure test atmosphere A₈₀ and mark the recorder chart to show the exact time when the switch occurred.

(xxvii) Measure test atmosphere A₉₀ and record the stable reading as P₈₀.

(xxviii) Sample test atmosphere A₉₀; a stable reading is not required.

(xxix) Measure test atmosphere A₈₀ and record the stable reading as P₁₀.

(xxx) Measure test atmosphere A₀ and record the stable reading as L₂.

(xxxi) Measure test atmosphere A₈₀ and record the stable reading as P₁₁.

(xxxii) Sample test atmosphere A₉₀; a stable reading is not required.

(xxxiii) Measure test atmosphere A₈₀ and record the stable reading as P₁₂.

(xxxiv) Repeat steps (viii) through (xxxiii) of this section, each test day.

(xxxv) If zero and span adjustments are made after the readings are taken on test days 3, 6, 9, or 12, complete all adjustments; then measure test atmospheres A₀, A₈₀, and A₂₀. Allow for a stable reading on each, and record the readings as Z_nS_n, and M_n respectively, where n = the test day number.

(10) Determine the results of each day's tests as follows. Mark the recorder chart to show readings and determinations.

(i) *Zero drift.* (A) 12-hour. Examine the strip chart pertaining to the 12-

hour continuous zero air test. Determine the minimum (Cmin.) and maximum (Cmax.) readings (in p/m) during this period of 12 consecutive hours, extrapolating the calibration curve to negative concentration units if necessary. Determine the 12-hour zero drift (12ZD) as $12ZD = C^{max.} - C^{min.}$. (See Figure B-5 in appendix A.)

(B) Calculate the 24-hour zero drift (24ZD) for the n -th test day as $24ZD_n = Z_n - Z_{n-1}$, or $24ZD_n = Z_n - Z'_{n-1}$ if zero adjustment was made on the previous day, where $Z_n = \frac{1}{2}(L_1 + L_2)$ for L_1 and L_2 taken on the n -th test day.

(C) Compare 12ZD and 24ZD to the zero drift specification in table B-1. Both 12ZD and 24ZD must be equal to or less than the specified value to pass the test for zero drift.

(ii) *Span drift.* (A) Span drift at 20 percent of URL (MSD)

$$MSD_n = \frac{M_n - M_{n-1}}{M_{n-1}} \times 100\%$$

$$MSD_n = \frac{M_n - M'_{n-1}}{M'_{n-1}} \times 100\%$$

If span adjustment was made on the previous day, where

$$M_n = \frac{1}{6} \sum_{i=1}^6 P_i$$

n indicates the n -th test day, and i indicates the i -th reading on the n -th day.

(B) Span drift at 80 percent of URL (USD):

$$USD_n = \frac{S_n - S_{n-1}}{S_{n-1}} \times 100\%$$

or

$$USD_n = \frac{S_n - S'_{n-1}}{S'_{n-1}} \times 100\%$$

If span adjustment was made on the previous day, where

$$S_n = \frac{1}{6} \sum_{i=7}^{12} P_i$$

n indicates the n -th test day, and i indicates the i -th reading on the n -th test day.

(C) Both USD and MSD must be equal to or less than the respective specifications given in table B-1 to pass the test for span draft.

(iii) *Lag time.* Determine, from the strip chart, the elapsed time in minutes between the mark made in step (xxvi) and the first observable (two times the noise level) response. This time must be equal to or less than the time specified in table B-1 to pass the test for lag time.

(iv) *Rise time.* Calculate 95 percent of reading P_9 and determine from the recorder chart, the elapsed time between the first observable (two times noise level) response and a response equal to 95 percent of the P_9 reading. This time must be equal to or less than the rise time specified in table B-1 to pass the test for rise time.

(v) *Fall time.* Calculate five percent of ($P_{10} - L_2$) and determine, from the strip chart, the elapsed time in minutes between the first observable decrease in response following reading P_{10} and a response equal to five percent of ($P_{10} - L_2$). This time must be equal to or less than the fall time specification in table B-1 to pass the test for fall time.

(vi) *Precision.* Calculate precision (P_{20} and P_{80}) for each day's test as follows:

(A)

$$P_{30} = \sqrt{\frac{1}{5} \left[\sum_{i=1}^6 P_i^2 - \frac{1}{6} \left(\sum_{i=1}^6 P_i \right)^2 \right]}$$

(B)

$$P_{30} = \sqrt{\frac{1}{5} \left[\sum_{i=7}^{12} P_i^2 - \frac{1}{6} \left(\sum_{i=7}^{12} P_i \right)^2 \right]}$$

(C) Both P_{20} and P_{80} must be equal to or less than the specification given in table B-1 to pass the test for precision.

[40 FR 7049, Feb. 18, 1975, as amended at 41 FR 52694, Dec. 1, 1976]

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**APPENDIX A TO SUBPART B OF PART 53—
OPTIONAL FORMS FOR REPORTING
TEST RESULTS**

**TABLE B-5—SYMBOLS AND ABBREVIATIONS—
Continued**

TABLE B-5—SYMBOLS AND ABBREVIATIONS

B_L	Analyzer reading at specified <i>LDL</i> concentration.	P_{20}	Precision at 20 percent of <i>URL</i> .
B_0	Analyzer reading at 0 concentration for <i>LDL</i> test.	P_{80}	Precision at 80 percent of <i>URL</i> .
<i>DM</i>	Digital meter.	R	Analyzer reading of pollutant alone for <i>IE</i> test.
C_{max}	Maximum analyzer reading during 12ZD test.	R_I	Analyzer reading with interferent added for <i>IE</i> test.
C_{min}	Minimum analyzer reading during 12ZD test.	r_i	The <i>i</i> -th <i>DM</i> reading for noise test.
<i>i</i>	Subscript indicating the <i>i</i> -th quantity in a series.	S	Standard deviation of noise readings.
<i>IE</i>	Interference equivalent.	S_0	Noise value (<i>S</i>) measured at 0 concentration.
L_1	First analyzer zero reading for 24ZD test.	S_{80}	Noise value (<i>S</i>) measured at 80 percent of <i>URL</i> .
L_2	Second analyzer zero reading for 24ZD test.	S_n	Average of $P_7 \dots P_{12}$ for the <i>n</i> -th test day.
M_n	Average of $P_1 \dots P_6$ for the <i>n</i> -th test day.	S'_n	Adjusted span reading at 80 percent of <i>URL</i> on the <i>n</i> -th test day.
M'_n	Adjusted span reading at 20 percent of <i>URL</i> on the <i>n</i> -th test day.	<i>URL</i>	Upper range limit.
<i>MSD</i>	Span drift at 20 percent of <i>URL</i> .	<i>USD</i>	Span drift at 80 percent of <i>URL</i> .
<i>n</i>	Subscript indicating the test day number.	Z	Average of L_1 and L_2 .
<i>P</i>	Analyzer reading for precision test.	Z_n	Average of L_1 and L_2 on the <i>n</i> -th test day.
P_i	The <i>i</i> -th analyzer reading for precision test.	Z'_n	Adjusted zero reading on the <i>n</i> -th test day.
		<i>ZD</i>	Zero drift.
		12ZD	12-hour zero drift.
		24ZD	24-hour zero drift.

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Applicant _____ Date _____

Test No. _____

Analyzer _____ Range _____

READING NUMBER (i)	TIME	0% of URL		80% of URL	
		DM READING	r_i , ppm	DM READING	r_i , ppm
1					
2					
3					
4					
5					
6					
7					
8					
9					
10					
11					
12					
13					
14					
15					
16					
17					
18					
19					
20					
21					
22					
23					
24					
25					
$\sum_{i=1}^{25} r_i$					
$\sum_{i=1}^{25} r_i^2$					
s			$s_0 =$		$s_{80} =$

Figure B-2. Form for noise data.

Applicant _____		Range _____														
Analyzer _____																
TEST PARAMETER	READING OR CALCULATION	1	2	3	4	5	6	7	8	9	10	11	12	13	14	15
LOWER DETECTABLE LIMIT	B_2															
	B_L															
	$LDL = B_L - B_2$															
INTERFERENCE EQUIVALENT	R_1															
	R_{11}															
	$IE_1 = R_{11} \cdot R_1$															
	R_2															
	R_{12}															
2	$IE_2 = R_{12} \cdot R_2$															
	R_3															
	R_{13}															
3	$IE_3 = R_{13} \cdot R_3$															
	R_4															
	R_{14}															
4	$IE_4 = R_{14} \cdot R_4$															
	R_5															
	R_{15}															
5	$IE_5 = R_{15} \cdot R_5$															
	TOTAL	$IE_T = \sum_{i=1}^n IE_i$														

Figure B-3. Form for data and calculations for lower detectable limit and interference equivalent.

Applicant _____
 Analyzer _____ Range _____

TEST DAY (m)	DATE	ANALYZER READING, ppm	0	1	2	3	4	5	6	7	8	9	10	11	12	13	14	15
P ₁																		
P ₂																		
P ₃																		
P ₄																		
P ₅																		
P ₆																		
$\sum_{i=1}^6 P_i^2$																		
P ₇																		
P ₈																		
P ₉																		
P ₁₀																		
P ₁₁																		
P ₁₂																		
$\sum_{i=7}^{12} P_i^2$																		
L ₁																		
L ₂																		
Z _i																		
M _i																		
S _i																		
C _{max}																		
C _{min}																		

Figure B-4. Form recording data for drift and precision.

Applicant		Range														
Analyzer		n - th TEST DAY														
TEST PARAMETER	CALCULATION	1	2	3	4	5	6	7	8	9	10	11	12	13	14	15
Zero drift	12ZD = $C_{max} - C_{min}$															
	$Z = \frac{1}{2}(L_1 + L_2)$															
24 hour	24ZD = $Z_n - Z_{n-1}$															
	24ZD _n = $Z_n - Z'_{n-1}$															
20% URL	$M_n = \frac{1}{6} \sum_{i=1}^6 P_i$															
	$MSD_n = \frac{M_n - M_{n-1}}{M_{n-1}} \times 100\%$															
	$MSD_n = \frac{M_n - M'_{n-1}}{M'_{n-1}} \times 100\%$															
Span drift	$S_n = \frac{1}{6} \sum_{i=7}^{12} P_i$															
	$USD_n = \frac{S_n - S_{n-1}}{S_{n-1}} \times 100\%$															
80% URL	$USD_n = \frac{S_n - S'_{n-1}}{S'_{n-1}} \times 100\%$															
	$P_{20} = \sqrt{\frac{1}{5} \left[\sum_{i=1}^6 P_i^2 - \frac{1}{6} \left(\sum_{i=1}^6 P_i \right)^2 \right]}$															
Precision	$P_{80} = \sqrt{\frac{1}{5} \left[\sum_{i=7}^{12} P_i^2 - \frac{1}{6} \left(\sum_{i=7}^{12} P_i \right)^2 \right]}$															

Figure B-5. Form for calculating zero drift, span drift and precision.

Applicant _____		Analyst _____															
Analyzer _____		Range _____															
PERFORMANCE PARAMETER	Table B-1 spec.	TEST										No. of test failures					
NOISE, ppm	0% URL (S ₀)	1	2	3	4	5	6	7	8	9	10	11	12	13	14	15	
	80% URL (S ₈₀)																
LDL (must be 2 × noise)																	
INTER-FERENCE EQUIVALENT, ppm	IE ₁																
	IE ₂																
	IE ₃																
	IE ₄																
	IE ₅																
TOTAL (IE _T)																	
ZERO DRIFT, ppm	12 hour (1ZZD)																
	24 hour (2ZZD)																
SPAN DRIFT, %	20% URL (MSD)																
	80% URL (USD)																
LAG TIME, min																	
RISE TIME, min																	
FALL TIME, min																	
PRECISION, ppm	20% URL (P ₂₀)																
	80% URL (P ₈₀)																

^aCompare each test LDL reading with the corresponding noise measurements. LDL reading must exceed the 0% URL noise value by a factor of 2 to pass the test for LDL.

Figure B-6. Form for summary of test results.

[40 FR 7049, Feb. 18, 1975, as amended at 40 FR 18169, Apr. 25, 1975]

Subpart C—Procedures for Determining Comparability Between Candidate Methods and Reference Methods

SOURCE: 71 FR 61278, Oct. 17, 2006, unless otherwise noted.

§ 53.30 General provisions.

(a) *Determination of comparability.* The test procedures prescribed in this subpart shall be used to determine if a candidate method is comparable to a reference method when both methods measure pollutant concentrations in

Appendix C

Glossary and Acronyms



Glossary and Acronyms

Glossary

A

Abscissa. The X-axis on a graph.

Absolute Filter. A filter or filter medium of ultra-high collection efficiency for very small particles (submicrometer size), so that essentially all particles of interest or of concern are collected. Commonly, the efficiency is 99.95% or higher for a standard aerosol of 0.3 μm diameter.

Absolute Pressure. A pressure scale that starts with a value of zero for an absolute vacuum.

Absolute Temperature. A temperature scale that starts with zero at absolute zero temperature.

Absolute Zero Temperature. The temperature at which molecular kinetic energy is negligible.

Absorption. The transfer of molecules from the bulk of the gas to a liquid surface, followed by diffusion of these molecules to the bulk of the liquid.

ACFM. The acronym for actual cubic feet per minute, which is the actual gas flow rate expressed in the American Engineering system of units. ACFM is a measure of the volume of gas that passes a given point during a one-minute period.

Accuracy. A measure of the closeness of an individual measurement or the average of a number of measurements to the true value. Accuracy includes a combination of random error (precision) and systematic error (bias) components that are due to sampling and analytical operations; the EPA recommends using the terms "*precision*" and "*bias*" rather than "accuracy," to convey the information usually associated with accuracy (see Bias).

Acute Exposure. One dose (or exposure) or multiple doses (or exposures) occurring within a short time relative to the life of a person or other organism (e.g., approximately 24 hours or less for humans).

Adsorbs. The adhesion of a substance to the surface of a solid or liquid.

Adsorption. The process whereby vapor phase compounds in the gas stream pass through a bed or layer of highly porous material (adsorbent). The vapor phase compounds diffuse to the surface of the adsorbent and are retained due to weak attractive forces.

Aerodynamic Diameter (a.d.). The diameter of a unit density sphere having the same terminal settling velocity as the particle in question. Operationally, the size of a particle as measured by an inertial device.

Aerodynamic Diameter. The diameter of a spherical particle having a density of 1 gm/cm³ that has the same inertial properties (i.e. settling velocity) in the gas as the particle of interest.

Aerosol. Suspended solid or liquid particles in the atmosphere.

Air at EPA Standard Conditions. Air at 25°C and 760 mm Hg (29.92 in. Hg).

Air Pollution. The presence of unwanted material in the air. The term "unwanted material" here refers to material concentrations present for a sufficient time and under such circumstances as to interfere significantly with comfort, health, or welfare of persons, or with the full use and enjoyment of property.

Air Toxic. Any air pollutant that causes or may cause cancer, respiratory, cardiovascular, or developmental effects, reproductive dysfunctions, neurological disorders, heritable gene mutations, or other serious or irreversible chronic or acute health effects in humans. See "Hazardous Air Pollutant."

Ambient Measurement. A measurement (usually of the concentration of a chemical or pollutant) taken in an ambient medium, normally with the intent of relating the measured value to the exposure of an organism that contacts that medium.

Anion. An atom or molecule with a negative charge.

Approved Regional Method (ARM). A continuous PM_{2.5} method that has been approved specifically within a state or local air monitoring network for purposes of comparison to EPA's National Ambient Air Quality Standards (NAAQS) and to meet other monitoring objectives.

Aqueous Liquid. A liquid whose main component is water, but which can also contain significant concentrations of other dissolved solids and liquids (usually <15% by weight) and/or low-to-moderate levels of suspended solids (usually <2% by weight).

Area Source (in the context of the Clean Air Act, Air Toxics Provisions). Any stationary source that falls below a major source threshold of emissions (see "Major Source,"), such as a dry cleaner or gas station. The term generally excludes motor vehicles (both road and nonroad).

Aspirator. Any apparatus, such as a squeeze bulb, fan, pump, or venturi, that produces a movement of a fluid by suction.

Atmosphere, The. The whole mass of air surrounding the earth, composed largely of oxygen and nitrogen.

Atmosphere, An. A specific gaseous mass, occurring either naturally or artificially, containing any number of constituents and in any proportion.

Attainment Area. An area that meets the air quality standard for a criteria pollutant (under NAAQS).

Audit (quality). A systematic and independent examination to determine whether quality activities and related results comply with planned arrangements, and whether these arrangements are implemented effectively and are suitable to achieve objectives.

B

Background Levels. Two types of background levels may exist for chemical substances: (a) naturally occurring levels, or ambient concentrations of substances present in the environment without human influence; and (b) anthropogenic levels, or concentrations of substances present in the environment due to human-made, non-site sources (e.g., automobiles, industries) [IRIS, 1999: Glossary of IRIS Terms].

Baghouse. This term is often used interchangeably with the term “filtration systems.” However, it is applicable only to pulse jet, cartridge, reverse air, and shaker-type filtration systems. The term “baghouse” does not have any clear meaning for HEPA filtration systems.

Barometric Pressure. The total pressure exerted by the atmosphere. This term is synonymous with “atmospheric pressure.”

Bias. The systematic or persistent distortion of a measurement process that causes errors in one direction (i.e., the expected sample measurement is different from the sample’s true value).

Bottom Ash. Incombustible matter resulting from combustion that does not leave as fly ash.

Brake Horsepower. The horsepower required to drive a fan. This includes the energy losses in the fan but does not include the drive losses between motor and fan.

Breathing Zone. The location in the atmosphere where persons breathe.

Brownian Diffusion. The slight deflection of very small particles in a gas stream that occurs when rapidly moving gas molecules strike them.

BTU (British Thermal Unit). The quantity of heat that must be transferred to a one pound mass of water to raise the temperature by 1°F.

C

Calibration. The process of comparing a standard or instrument with one of greater accuracy (small uncertainty) to obtain quantitative estimates of the actual values of the standard being calibrated, the deviation of the actual value from a nominal value, or the difference between the value indicated by an instrument and the actual value.

Calibration Standards. Devices that are specifically designed to be placed in a monitoring location and can be used to calibrate air monitoring instruments. These devices are commercially available from a number of vendors. These units usually are permeation devices or mass flow calibrators (MFC). The flow rates of these devices are verified by the transfer standard on a set schedule.

Cancer. A disease of heritable, somatic mutations affecting cell growth and differentiation, characterized by abnormal, uncontrolled growth of cells.

Carbon Bed Adsorber. An air pollution control system that is used to collect and concentrate organic compounds on an activated carbon adsorbent.

Cascade Impactor. A sampling device used to determine the particle size distribution. Particles are separated and deposited on a series of stages that correspond to different aerodynamic diameters.

Catalyst. A substance, usually present in small amounts compared to the reactants, that speeds up the chemical reaction rate without being consumed in the process.

Catalytic Oxidizer. An air pollution control device that uses a catalyst to accelerate the oxidation reaction at lower temperatures than possible in gas phase thermal oxidation.

Certification. A certification is the process of checking a transfer standard against a primary standard and establishing a mathematical relationship that is used to adjust the transfer standard values back to the primary standard.

Chemical Abstracts Service Registry Number (CAS No.). A unique, chemical-specific number used in identifying a substance. The registry numbers are assigned by the Chemical Abstract Service, a division of the American Chemical Society.

Chimney Effect. A phenomenon consisting of a vertical movement of a localized mass of air or other gases due to temperature/pressure differences.

Chronic Exposure. Multiple exposures occurring over an extended period of time, or a significant fraction of the animal's or the individual's lifetime.

Class I Areas. Class I areas are protected by the PSD program and include national parks, national wilderness areas, national monuments, national seashores, and other areas of special national or regional natural, recreational, scenic, or historical value.

Class II Areas. Attainment areas that are neither industrialized nor meet the specific requirements for classification as Class I areas. They are protected by the PSD program.

Class III Areas. Industrialized attainment areas. They are protected by the PSD program.

Cloud. A visible dispersion occupying a discrete portion of space, with apparent boundaries.

Coarse Particles. EPA classification of particles having aerodynamic diameters ranging from 2.5 to 10 μm (PM_{10-2.5})

Collection Efficiency. A ratio of pollutants entering a control device versus pollutants leaving the device expressed as a percent.

Collector. A device for removing and retaining contaminants from air or other gases. Usually this term is applied to cleaning devices in exhaust systems.

Collocated Samples. Two or more portions collected at the same point in time and space so as to be considered identical. These samples are also known as field replicates and should be identified as such.

Combustion. The production of heat and light energy through a chemical process, usually oxidation. Products of complete combustion include water and carbon dioxide, while incomplete combustion can yield partially oxidized organic compounds and carbon monoxide. Factors that promote complete combustion include the proper fuel-air ratio, temperature range, and adequate amount of time for the fuel and its by-products to complete the combustion reactions.

Condensable Particulate Matter. Particulate matter, contained almost entirely within the PM_{2.5} classification, that forms from condensing gases or vapors. It forms by chemical reactions as well as by physical phenomena.

Condensate. Liquid or solid matter formed by condensation from the vapor phase. In sampling, the term is applied to the components of an atmosphere that have been isolated by simple cooling.

Condensation. The process of converting a material in the gaseous phase to a liquid or solid state by decreasing temperature, increasing pressure, or both. Usually in air sampling only cooling is used.

Condenser. A simple, relatively inexpensive device that normally uses water or air to cool and condense a vapor stream.

Condensoid. The particles of a dispersion formed by condensation.

Conductivity. A measure of the ability of a material to conduct an electric charge.

Conservation of Matter. A general principle of non-nuclear reactions and processes where matter is neither created nor destroyed.

Contaminant. Unwanted material.

Count Median Size. A measurement of particle size for samples of particulate matter, consisting of that diameter of particle such that one half of the number of particles is larger and half is smaller.

Criteria Air Pollutant. One of six common air pollutants determined to be hazardous to human health and regulated under the EPA's National Ambient Air Quality Standards (NAAQS). The six criteria air pollutants are carbon monoxide, lead, nitrogen dioxide, ozone, sulfur dioxide, and particulate matter. The term "criteria pollutants" derives from the requirement that the EPA must describe the characteristics and potential health and welfare effects of these pollutants. It is on the basis of these criteria that standards are set or revised.

Cryogenic Sampling. See Sampling, Condensation.

Cyclonic Separator. A mechanical collector that uses centrifugal force to drive particles to the wall

of the device.

D

Dalton's Law of Partial Pressures. The law stating that the total pressure of a gas is the sum of the pressures exerted by each component gas.

Data Acquisition System. A strip chart recorder, analog computer, or digital computer for recording measurement data from the analyzer output.

Data Quality Objectives (DQOs). The qualitative and quantitative statements derived from the DQO Process that clarify a study's technical and quality objectives, define the appropriate type of data, and specify tolerable levels of potential decision errors that will be used as the basis for establishing the quality and quantity of data needed to support decisions.

Density. The quantity of mass contained in a specific volume.

Denuder. A device designed to remove gases from an air sampling stream by the process of molecular diffusion to a collecting surface.

Desorption. The process of using low pressure steam or hot nitrogen gas to remove compounds from an adsorbent bed.

Dew Point. The temperature at which the partial pressure of a substance (in vapor form) equals the equilibrium vapor pressure of the substance. At this temperature, a vapor begins to condense at a constant pressure.

Differential Pressure Meter. Any flow measuring device that operates by restricting air flow and measuring the pressure drop across the restriction.

Diffusion. The random motion of small particles suspended in a gas or liquid, also known as Brownian Movement. It can also be described as a process by which the molecules of two or more substances gradually mix.

Diffusivity. Measure of the extent to which very small particles are influenced by molecular collisions which cause the particles to move in a random manner across the direction of gas flow.

Dimensions. Units of measure used to express the magnitude of mass, distance, force, and time.

Dimensional Calculations. A form of mathematical calculation in which the units applying to each value are stated explicitly and are handled algebraically.

Dispersion. The most general term for a system consisting of particulate matter suspended in air or other gases.

Dispersoid. The particles of a dispersion.

Diurnal. Recurring daily. Applied to (variations in concentration of) air contaminants, diurnal indicates variations following a distinctive pattern and recurring from day to day.

Dose-response assessment. A determination of the relationship between the magnitude of an administered, applied, or internal dose and a specific biological response. Response can be expressed as measured or observed incidence, percent response in groups of subjects (or populations), or as the probability of occurrence within a population [IRIS, 1999: Glossary of IRIS Terms].

Droplet. A small liquid particle of such size and density as to fall under still conditions, but which may remain suspended under turbulent conditions.

Dry Scrubber. An air pollution control device used to remove an acid gas pollutant from a gas stream. The pollutant is collected on or in a solid or liquid material, which is injected into the gas stream. A dry scrubber produces a dry product that must be collected downstream from this control device.

Dust. A loose term applied to solid particles predominantly larger than colloidal and capable of temporary suspension in air or other gases. Dusts do not tend to flocculate except under electrostatic forces; they do not diffuse but settle under the influence of gravity. Derivation from larger masses through the application of physical force is usually implied.

Dust Fall. See Particle Fall.

Dust Loading. An engineering term for "dust concentration," usually applied to the contents of collection ducts and the emission from stacks.

E

Ecological Risk Assessment. A process that evaluates the likelihood that adverse ecological effects may occur or are occurring as a result of exposure to one or more stressors. Stressors are defined as any chemical, biological, or physical entity that can induce an adverse response on ecological individuals, populations, communities, or ecosystems.

Efficiency. The ratio of attained performance to absolute performance, commonly expressed in percent.

Efficiency, Fractional. The mean collection efficiency for specific size fractions of a contaminant. Commonly this term has been applied to the performance of air cleaning equipment toward particulate matter in various size ranges.

Ejector. A device that uses a fluid under pressure, such as steam, air, or water, to move another fluid by developing suction. Suction is developed by discharging the fluid under pressure through a venturi.

Electrostatic Attraction. An affinity or attraction between oppositely charged particles and/or

collection media.

Electrostatic Precipitator. A type of air pollution control system that uses high voltage fields to electrically charge and collect particulate matter. The charged particles approach an electrically grounded collection plate and accumulate as a dust layer, which is partially removed by mechanical rapping (hammers) on a routine basis.

Emissions. The total of substances discharged into the air from a stack, vent, or other discrete source.

Emission Mixture. The total mixture in the outside atmosphere of emission from all sources.

Emission Sampling Train. Equipment usually consisting of (1) a sampling nozzle and probe, (2) filter and impingers for collection of gaseous and/or particulate components, (3) flow meter and flow regulation devices, and (4) a vacuum pump for collecting a representative sample of a gas stream.

Entrainment. The process in which material, such as water droplets or particulate matter, is picked up and carried along by a gas or liquid stream.

Entry Loss. The loss of pressure that occurs when airflow moves into a system.

Equilibrium. A steady state condition. The amount of mass transferred in one direction is exactly balanced by the amount of mass transferred in the reverse direction.

Evaporative Cooling Tower. Equipment used to reduce the temperature of a gas stream. Fine droplets, injected into a vessel, are evaporated as they absorb heat from the gas stream.

Exposure. Contact made between a chemical, physical, or biological agent and the outer boundary of an organism. Exposure is quantified as the amount of an agent available at the exchange boundaries of the organism (e.g., skin, lungs, gut).

Exposure Assessment. An identification and evaluation of the human population exposed to a toxic agent, describing its composition and size, as well as the type magnitude, frequency, route and duration of exposure.

F

Fabric Filter. A filtration device using one or more filter bags, sheets, or panels to remove particles from a gas stream.

Fan Drive. The way in which the motor shaft is linked to the fan wheel to transmit power and control speed.

Federal Equivalent Method. A method for measuring the concentration of an air pollutant in the ambient air that has been designated as an equivalent method in accordance with 40 CFR Part 53.

Federal Reference Method. A method of sampling and analyzing the ambient air for an air pollutant that is specified as a reference method in an appendix to 40 CFR Part 50.

Fine Particles. EPA classification of particles having aerodynamic diameters greater than 0.1 micrometer and less than or equal to 2.5 micrometers.

Flocculation. Synonymous with agglomeration.

Flowmeter. An instrument for measuring the rate of flow of a fluid moving through a pipe or duct system. The instrument is calibrated to give volume or mass rate of flow.

Flow Rate (Actual). The volume of gas moving through a system or stack per unit time expressed at actual conditions of temperature and pressure.

Flow Rate (Standard). The volume of gas moving through a system or stack per unit time expressed at standard conditions of temperature and pressure.

Flow Rate (Volumetric). The volume of gas moving through a ventilation system, stack, or air pollution control system per unit time.

Fly Ash. Uncombusted particulate matter in the combustion gases resulting from the burning of coal and other material.

Fog. A loose term applied to visible aerosols in which the dispersed phase is liquid. Formation by condensation is usually implied; in meteorology, a dispersion of water or ice.

Force. An influence on a physical object that causes a change in movement and/or shape.

Freezing Out. See Sampling, Condensation.

Fugitive Emissions. Emissions that escape from industrial processes and equipment.

Fume. Properly, the solid particles generated by condensation from the gaseous state, generally after volatilization from melted substances, and often accompanied by a chemical reaction such as oxidation. Fumes flocculate and sometimes coalesce. Popularly, the term is used in reference to any of all types of contaminant, and in many laws or regulations, with the added qualification that the contaminant has some unwanted action.

G

Gas. One of the three states of aggregation of matter, having neither independent shape nor volume and tending to expand indefinitely.

Gauge Pressure. The relative pressure inside a vessel or container (the difference between the inside pressure and atmospheric pressure).

Grab Sample. See Sampling, Instantaneous.

Gravimetry/Gravimetric. A weight-based measurement.

Gravitational settling/sedimentation. A process by which suspended particles in air settle to the bottom.

H

Hazardous Air Pollutants. See Air Toxics.

Heterogeneous Nucleation. The accumulation of material from the vapor phase onto an existing particle.

High-Volume Air Sampler (Hi-Vol). A device for sampling large volumes of an atmosphere for collecting the contained particulate matter by filtration. Consists of a high-capacity blower, a filter to collect suspended particles, and a means for measuring the flow rate.

Homogeneous Nucleation. The formation of a particle from the vapor phase involving only one compound.

Hood. A shaped inlet designed to capture contaminated air and conduct it into the exhaust duct system.

Hood Capture Velocity. The air velocity at any point in front of the hood or at the hood opening necessary to overcome opposing air currents and to capture the contaminated air at that point by pulling it into the hood.

Human Health Inhalation Assessment. A process that evaluates the likelihood of an adverse impact of a chemical or group of chemicals on human health for people that are exposed through the inhalation of the chemical(s). Inhalation can be defined as drawing of air (and pollutants) into the lungs via the nasal or oral respiratory passages.

Human Health Multipathway Assessment. A process that evaluates the likelihood of an adverse impact of a chemical or group of chemicals on human health for people that are exposed through multiple exposure pathways. These pathways could include inhaling the chemical(s), eating food that the chemical(s) has deposited on, accidentally eating dirt or dust that contains the chemical(s), or skin contact with dirt or water that contains the chemical(s).

Hydrometer. An instrument for measuring the specific weight of a liquid by utilizing the principle of buoyancy.

Hygroscopic. Characterized by readily absorbing moisture.

I

Impaction. A forcible contact of particles of matter. A term often used synonymously with

impingement.

Impactor. A sampling device that employs the principle of impaction (impingement).

Impingement. The act of bringing matter forcibly in contact. When used in air sampling, refers to a process for the collection of particulate matter in which the gas being sampled is directed forcibly against a surface.

Impingement (Dry). The process of impingement carried out so that particulate matter carried in the gas stream is retained upon the surface against which the stream is directed. The collecting surface may be treated with a film of adhesive.

Impingement (Wet). The process of impingement carried out within a body of liquid, the latter serving to retain the particulate matter.

Impinger. Broadly, a sampling instrument employing impingement for the collection of particulate matter. Commonly, this term is applied to specific instruments, the “midget” and “standard” impinger.

Impinger, Midget. A specific instrument employing wet impingement, using a liquid volume of 10 ml and a gas flow of 0.1 cu. ft. per minute.

Impinger, Standard. A specific instrument employing wet impingement, using a liquid volume of 75 ml and a gas flow of 1 cu. ft. per minute (e.g. Greenberg-Smith Impinger).

Individual Exposure or Risk Assessment. A process that evaluates the likelihood of an adverse impact on human health (risk), or amount of exposure, of a chemical or group of chemicals for an actual or hypothetical person.

Inertial Impaction. The process whereby a particle moving in a gas stream strikes slowly moving or stationary obstacles or targets (e.g. liquid droplets) directly in its path. Compare with Interception.

Inhalable Particles. Particles with aerodynamic diameters of $<10 \mu\text{m}$ that are capable of being inhaled into the human lung.

Inorganic. A term that applies to compounds that contain no carbon-hydrogen bonds.

Interception. The process whereby a particle moving in a gas stream is offset slightly from directly impacting a moving or stationary obstacle or target. As the particle tries to move past the obstacle, the obstacle intercepts (and collects) the particle. Compare with Inertial Impaction.

Interference. An undesired positive or negative output caused by a substance other than the one being measured.

Isokinetic. A term describing a condition of sampling in which the flow of gas into the sampling device (at the opening or face of the inlet) has the same flow rate and direction as the gas stream

being sampled.

J

K

Kinematic Viscosity. See Viscosity (Kinematic).

L

Lognormal Distribution. A frequency distribution that is symmetric (i.e. bell shaped) when plotted on a logarithmic abscissa.

Lower Explosive Limit. The lowest concentration at which a gas or vapor is flammable or explosive at ambient conditions.

M

Major Source (in the context of the Clean Air Act, air toxics provisions). Any stationary source or group of stationary sources located within a contiguous area and under common control that emits, or has the potential to emit considering controls in the aggregate, 10 tons per year or more of any hazardous air pollutant or 25 tons per year or more of any combination of hazardous air pollutants (Section 112(a)(1)).

Manometer. An instrument used for measuring the pressure of liquids and gases. An open-tube manometer consists of a U-shaped tube that usually contains a liquid such as mercury or water. One end of the tube is open to the atmosphere and the other end of the tube is connected to the container where the pressure is to be measured.

Mass. The measure of the magnitude of a physical object that is related directly to the atoms in the object.

Mass Concentration. Concentration expressed in terms of mass of substance per unit volume of gas or liquid.

Mass Flowmeter. Device that measures the mass flow rate of air passing a point, usually using the rate of cooling or heat transfer from a heated probe.

Mass Flow Controller. A device that works on the principle of heat loss. The mass flow meter within the MFC has a small thermister that is sensitive to heat loss. A potential voltage is applied to the thermister. As the air flow increases across the thermister, the resistance of the thermister changes. This change in resistance can be measured very accurately by electronic circuitry. The mass flow circuitry can then be integrated with a controlling loop circuit that can control/monitor

the flow instantaneously. Usually, MFCs have two channels, gas and diluent or air flow. The gas portion of the unit allows for gases from compressed cylinders to be allowed in and metered. The air flow side of the unit blends down the high concentration from the compressed cylinders to the desired working concentration. The flow rate of both portions of the unit must be measured accurately. It is important when purchasing a MFC calibrator that it meet the 40 CFR 50 requirements of having an accuracy within +/- 2%.

Mass Median Particle Diameter. The particle diameter at which half the particulate mass is composed of particles larger than this diameter and half the mass is composed of particles smaller than this diameter.

Mass Transfer. A natural phenomenon in which a component travels from a region of high concentration to one of low concentration in order to minimize concentration differences in a mixture.

Master Flow-Rate Standard. A flow-rate measuring device, such as a standard orifice meter, that has been calibrated against a primary standard.

Matter. The substance of which a physical object is composed.

Maximum Achievable Control Technology (MACT). EPA standards mandated by the 1990 CAAA for the control of toxic emissions from various industries.

Mean. The arithmetic average of distribution.

Method Detection Limit (MDL). The minimum concentration of an analyte that can be reported with 95% confidence that the value is above zero, based on a standard deviation of at least seven repetitive measurements of the analyte in the matrix of concern at a concentration near the low standard.

Micrograms. A unit of measurement for mass. One microgram equals 10^{-6} grams.

Milligrams. A unit of measurement for mass. One milligram equals 10^{-3} grams.

Mist. A loose term applied to dispersions of liquid particles, the dispersion being of low concentration and the particles of large size. In meteorology, a light dispersion of water droplets of sufficient size to be falling.

Mode. The value that occurs the most frequently in a distribution.

Mole. The mass of a material equal to the combined mass of 6.023×10^{23} atoms or molecules of the material.

Mole Fraction. An expression of the number of moles of a compound divided by the total number of moles of all compounds present.

Momentum. The product of the mass of an object times its velocity.

N

Nanograms. A unit of measurement for mass. One nanogram equals 10^{-9} grams.

National Ambient Air Quality Standards (NAAQS). Enforced air quality standards established by EPA that apply to outdoor air throughout the country.

Natural Logarithm (Napierian). The exponent applied to the base number of 2.718 to equal a given value. For example, 2 is the natural logarithm that is equal to a value of 7.389. $(2.718)^2=7.389$

Negative Pressure. A relative pressure that is below atmospheric pressure.

Nucleate. To form a central part around which other parts can attach.

Non-Attainment Areas. A geographic area that does not meet one or more of the federal air quality standards (NAAQS) for the criteria pollutants.

O

Odor. That property of a substance affecting the sense of smell; any smell; scent; perfume.

Odor Concentration. The number of unit volumes that a unit volume of sample will occupy when diluted to the odor threshold.

Odor Unit. Unit volume of air at the odor threshold.

Odorant. Odorous substance.

Operating Permit (Permit). Document required by EPA under Title V for any major stationary source. Potential-to-emit limits defining a major source are determined by the air quality of the geographical region where the facility is located. A permit contains the specific information about how the facility will comply with established emission standards and guidelines set forth by EPA.

Ordinate. The Y-axis on a graph.

Organic. A term that applies to compounds that contain carbon-hydrogen bonds.

Orifice Meter. A flowmeter, employing as the measure of flow rate the difference between the pressures measured on the upstream and downstream sides of the orifice (that is, the pressure differential across the orifice) in the conveying pipe or duct.

Ozone Season. The ozone season from May 1 to September 30 (Northern Hemisphere) when ground-level ozone formation concentrations tend to be highest.

P

Partial Pressure. The fraction of the total static pressure in a gas stream caused by one of the constituents of the mixture.

Particle. A small discrete mass of solid or liquid matter.

Particle Concentration. Concentration expressed in terms of number of particles per unit volume of air or other gas. (Note: On expressing particle concentration, the method of determining the concentration should be stated.)

Particle Fall. A measurement of air contamination consisting of the mass rate at which solid particles deposit from the atmosphere. A term used in the same sense as the older terms Dust Fall and Soot Fall, but without any implication as to nature and source of the particles.

Particle Size. An expression for the size of liquid or solid particles expressed as the average or equivalent diameter.

Particle Size Distribution. The relative percentage by weight or number of each of the different size fractions of particulate matter.

Particulate. Solids or liquids existing in the form of separate particles.

Particulate Matter. Solid or liquid matter that is dispersed in a gas, or insoluble solid matter dispersed in a liquid, that gives a heterogeneous mixture.

Penetration. The ratio of the amount of pollutants leaving an air pollution control device versus the amount of pollutants entering the device.

Penetration Efficiency. The ratio of the amount of pollutants leaving an air pollution control device versus the amount of pollutants entering the device, expressed as a percent.

Permeation Devices. Calibration units that pass a known volume of air over a permeation tube. The permeation tube is a small cylinder (usually steel) that has a permeable membrane at one end. Usually the tube is filled with a liquid that permeates out through the membrane at a given rate at a very narrow temperature range. By knowing the permeation rate and the air flow rate, a NIST-traceable concentration in parts per million can be calculated.

Photochemical Reaction. Any chemical reaction that is initiated as a result of absorption of light.

Photochemical Smog. Air pollution resulting from photochemical reactions.

Physical Attrition. The act of wearing or grinding down by friction.

PM_{2.5}. EPA defines PM_{2.5} as particulate matter with a diameter of 2.5 micrometers collected with 50% efficiency by a PM_{2.5} sampling collection device. However, for convenience in this reference

material, the term $PM_{2.5}$ includes all particles having an aerodynamic diameter of less than or equal to 2.5 micrometers.

PM₁₀. EPA defines PM_{10} as particulate matter with a diameter of 10 micrometers collected with 50% efficiency by a PM_{10} sampling collection device. However, for convenience in this reference material, the term PM_{10} includes all particles having an aerodynamic diameter of less than or equal to 10 micrometers.

Pollutant Ranking Assessment. An assessment used to rank chemicals with respect to their impact on human health and the environment. Often used to select a subset of the most important chemicals on which to focus further risk assessments. For example, the 33 air toxics highlighted as priority pollutants in EPA's Urban Strategy were selected based on a number of factors, including toxicity-weighted emissions, monitoring data, past air quality modeling analysis, and a review of existing risk assessment literature.

Population Exposure or Risk Assessment. A process that evaluates the likelihood of an adverse impact on human health (risk), or amount of exposure, of a chemical or group of chemicals, for groups of actual or hypothetical people. For cancer-causing chemicals, population risk can be expressed as the number of people estimated to be exposed to specific risk levels or as the number of excess cancer cases expected to occur.

Potential-to-Emit. The total emissions that a facility would release by operating at maximum load for 24 hours per day and 365 days per year.

PPBV [or PPB(V/V)]. A unit of measure of the concentration of gases in air expressed as parts of the gas per billion (10⁹) parts of the air-gas mixture, normally both by volume.

PPMV [or PPM(V/V)]. The part per million concentration that is determined by comparing the volume of one constituent with the total volume of the substance. Gas concentrations are always expressed in a ppm(v/v) format as opposed to the ppm(w/w) format often used for liquids. Throughout APTI courses, the term ppm when applied to gases means ppm(v/v).

PPMVD. The part per million concentration that is determined by comparing the volume of one constituent with the volume of the other constituents with the exception of moisture.

PPM(W/W). The part per million concentration that is determined by comparing the mass of one constituent with the total mass of the sample. Liquid concentrations are often expressed in a ppm(w/w) format as opposed to the ppm(v/v) format used for gases. Throughout APTI courses, the term ppm when applied to liquids means ppm(w/w). Note that the abbreviation "w/w" is used despite the fact that the ppm concentration is based on a ratio of masses.

Precipitation, Electrostatic. A process consisting of the separation of particulate matter from air or other gases under the influence of an electrostatic field.

Precipitation, Meteorological. The precipitation of water from the atmosphere in the form of hail, mist, rain, sleet, and snow. Deposits of dew, fog, and frost are excluded.

Precipitation, Thermal. A process consisting of the separation of particulate matter from air and other gases under the influence of a relatively large temperature gradient extending over a short distance. In the “Thermal Precipitator” (a sampling instrument), the air or gas is drawn through a narrow chamber across which extends a heated wire, particulate matter being deposited upon the adjacent collecting surface.

Precipitation, Ultrasonic. A process consisting of the separation of particulate matter from air and other gases following agglomeration induced by an ultrasonic field.

Precipitator, Electrostatic. Apparatus employing electrostatic precipitation for the separation of particles from a gas stream. The apparatus may be designed either for sampling or for cleaning large volumes of gas.

Precision. The degree of mutual agreement between individual measurements, namely repeatability and reproducibility.

Preconcentration. The process of removing organic vapors from a gas stream and transferring them to a smaller, more concentrated gas stream.

Pressure Drop. A measure of the resistance the gas stream encounters as it flows through an air control device or other piece of equipment.

Pressure Static. The pressure of a fluid at rest, or in motion, exerted perpendicularly to the direction of flow.

Pressure, Velocity. That pressure caused by and related to the velocity of the flow of fluid; a measure of the kinetic energy of the fluid.

Pressure, Total. The pressure representing the sum of static pressure and velocity pressure at the point of measurement.

Pressure Gauge. The difference in pressure existing within a system and that of the atmosphere. Zero gauge pressure is equal to atmospheric pressure.

Primary Flow-Rate Standard. A device or means of measuring flow rate based on direct primary observations such as time and physical dimensions.

Primary Standard. This is a flow device that is certified to be directly traceable to the NIST-SRM. These devices usually provide paperwork that proves that the device is traceable. Bubble meters, volumetric burettes, and some piston devices can be considered to be primary standards. Check with the vendor for certification of a primary standard. The primary standard should remain in the central laboratory and not be moved.

Probe. A tube used for sampling or for measuring pressures at a distance from the actual collection or measuring apparatus. It is commonly used for reaching inside stacks and ducts.

Promulgate. To put a law into effect by formal public announcement.

Q

Quality. The totality of features and characteristics of a product or service that bears on its ability to meet the stated or implied needs and expectations of the user.

Quality Assurance (QA). An integrated system of management activities involving planning, implementation, assessment, reporting, and quality improvement to ensure that a process, item, or service is of the type and quality needed and expected by the client.

Quality Assurance Project Plan (QAPP). A formal document describing in comprehensive detail the necessary quality assurance (QA), quality control (QC), and other technical activities that must be implemented to ensure that the results of the work performed will satisfy the stated performance criteria. The QAPP components are divided into four classes: 1) Project Management, 2) Measurement/Data Acquisition, 3) Assessment/Oversight, and 4) Data Validation and Usability. Guidance and requirements on preparation of QAPPs can be found in EPA QA/R-5 and QA/G-5.

Quality Control (QC). The overall system of technical activities that measures the attributes and performance of a process, item, or service against defined standards to verify that they meet the stated Operational techniques and activities that are used to fulfill requirements for quality. The system of activities and checks used to ensure that measurement systems are maintained within prescribed limits, providing protection against “out of control” conditions and ensuring the results are of acceptable quality.

R

°R. Rankine is an absolute temperature scale often used in engineering. Using this scale, the freezing point of water is 492°R and the boiling point of water is 672°R.

Receptor. The entity which is exposed to the stressor (USEPA, 1997: Guidance on Cumulative Risk Assessment, Planning and Scoping).

Reference Concentration (RfC). An estimate (with uncertainty spanning perhaps an order of magnitude) of a continuous inhalation exposure to the human population (including sensitive subgroups) that is likely to be without an appreciable risk of deleterious effects during a lifetime. It can be derived from a no-observed-adverse-effect level (NOAEL), lowest-observed-adverse-effect level (LOAEL), or benchmark concentration, with uncertainty factors generally applied to reflect limitations of the data used. Generally used in EPA’s noncancer human health risk assessments.

Reference Dose (RfD). An estimate (with uncertainty spanning perhaps an order of magnitude) of a daily oral dose exposure to the human population (including sensitive subgroups) that is likely to be without an appreciable risk of deleterious effects during a lifetime. It can be derived from a NOAEL, LOAEL, or benchmark dose, with uncertainty factors generally applied to reflect limitations of the data used. Generally used in EPA’s noncancer human health risk assessments.

Relative Pressure. The difference in pressure between a point in a ventilation system or air pollution control system and the atmospheric pressure.

Relative Temperature. The difference between the measured temperature value and an arbitrarily selected value, such as the freezing point of water.

Resistivity. A measure of a particle's ability to conduct electricity (expressed in units of ohm-cm). Resistivity is very important in the efficient operation of electrostatic precipitators.

Reynolds' Number, Gas or Flow. A dimensionless number corresponding to the ratio of the fluid inertial force to the fluid viscous force in a flow system. It is used as an index for turbulence.

Reynolds' Number, Particle. A dimensionless number corresponding to the ratio of the inertial force of a particle to the viscous force of the surrounding fluid in a flow system. It is used as an index for turbulence.

Risk (in the context of human health). The probability of injury, disease, or death from exposure to a chemical agent or a mixture of chemicals. In quantitative terms, risk is expressed as values ranging from zero (representing the certainty that harm will not occur) to one (representing the certainty that harm will occur).

Risk Assessment (in the context of human health). The determination of potential adverse health effects from exposure to chemicals, including both quantitative and qualitative expressions of risk. The process of risk assessment involves four major steps: hazard identification, dose-response assessment, exposure assessment, and risk characterization.

Risk-Based Concentrations (RBCs). An estimate of a health-based air concentration of a chemical that a person or ecosystem could be exposed to that would not result in a risk of cancer or other adverse health effects above a specified level of concern. The risk-based concentration is developed from toxicological dose response values (for human health, usually selecting the most protective of both cancer toxicological dose response values and noncancer toxicological dose response values) and assumptions about exposure rates. It is a tool used to identify which chemicals may pose a threat to human or ecological receptors without conducting an exposure or risk assessment.

Risk-Based Doses (RBDs). An estimate of a health-based, media-specific concentration (e.g., soil or water) of a chemical that a person or ecosystem could be exposed to that would not result in a risk of cancer or other adverse health effects above a specified level of concern. The risk-based dose is developed from toxicological dose response values (for human health, usually selecting the most protective of both cancer toxicological dose response values and noncancer toxicological dose response values) and assumptions about exposure rates. It is a tool used to identify which chemicals may pose a threat to human or ecological receptors without conducting an exposure or risk assessment.

Risk Characterization. The final, summarizing step of a risk assessment. The risk characterization integrates information from the preceding components of the risk assessment and synthesizes an overall conclusion about risk that is complete, informative, and useful for decision makers. It conveys the risk assessor's judgment as to the nature and existence of (or lack of) human health or ecological risks.

Risk Management (in the context of human health). A decision making process that accounts for political, social, economic, and engineering implications together with risk-related information in order to develop, analyze, and compare management options and select the appropriate managerial response to a potential chronic health hazard.

Rotameter. A device, based on the principle of Stoke's law, for measuring rate of fluid flow. It consists of a tapered vertical tube having a circular cross section, and containing a flow that is free to move in a vertical path to a height dependent upon the rate of fluid flow upward through the tube.

S

Sample, Integrated. A sample obtained over a period of time with (1) the collected atmosphere being retained in a single vessel, or (2) with a separated component accumulating into a single whole. Examples are particle sampling, in which all the particles separated from the air are accumulated in one mass of fluid; the absorption of acid gas in an alkaline solution; and collection of air in a plastic bag. Such a sample does not reflect variations in concentration during the period of sampling.

Sample, Continuous. Withdrawal of a portion of the atmosphere over a period of time with continuous analysis or with separation of the desired material continuously and in a "linear" form. Examples are continuous withdrawal of the atmosphere accompanied by absorption of a component in a flowing stream of absorbent or by filtration on a moving strip or paper. Such a sample may be obtained with a considerable concentration of the contaminant, but it still indicates fluctuations in concentration that occur during the period of sampling.

Sampling. A process consisting of the withdrawal or isolation of a fractional part of a whole. In air or gas analysis, the separation of a portion of an ambient atmosphere with or without the simultaneous isolation of selected components.

Sampling, Condensation. A process consisting of the collection of one or several components of a gaseous mixture by the simple cooling of the gas stream in a device that retains the condensate.

Sampling, Continuous. Sampling without interruptions throughout an operation or for a predetermined time.

Sampling, Instantaneous. Obtaining a sample of an atmosphere in a very short period of time such that this sampling time is insignificant in comparison with the duration of the operation or the period being studied.

Sampling, Intermittent. Sampling successively for limited periods of time throughout an operation or for a predetermined period of time. The duration of sampling periods and of the intervals between are not necessarily regular and are not specified.

SCFM. The acronym for standard cubic feet per minute, which is the gas flow rate at standard conditions expressed in the American Engineering system of units.

Scrubbing Liquid. A liquid used to remove particulate or gaseous pollutants by absorption or chemical reaction through contact with the gas stream.

Series Collection. An operation involving the use of two or more collectors joined in a series.

Settling Velocity. The terminal rate of fall of a particle through a fluid as induced by gravity or other external force; the rate at which frictional drag balances the accelerating force (or the external force).

Sheave(s). Part of the drive system that supports the belt that extends from the drive shaft to the fan shaft.

Sieving. A process that occurs in fabric filters whereby the dust cake, which has accumulated on the fabric surface, acts as a filter for collecting particles.

Silica Gel. A regenerative adsorbent consisting of amorphous silica (SiO_2) with OH surface groups, making it a polar material and enhancing surface reactions.

Smog. A term derived from smoke and fog, applied to extensive atmospheric contamination by aerosols which arise partly through natural processes and partly from the activities of human subjects. Now sometimes used loosely for any contamination of air. (See also Smog, Photochemical.)

Smog, Photochemical. Air contamination caused by chemical reactions of pollutants formed primarily by the action of sunlight on oxides of nitrogen and hydrocarbons.

Smoke. Small gas-borne particles resulting from incomplete combustion, consisting predominantly of carbon and other combustible material, and present in sufficient quantity to be observable independently of the presence of other solids.

Solute. The substance distributed (i.e. dissolved or suspended) in a solvent.

Solution. A substance (usually a liquid) that dissolves a solute to form a solution.

Sorbent. A liquid or solid medium in or upon which materials are retained by absorption or adsorption.

Sorption. A process consisting of either absorption or adsorption or both.

Specific Gravity. A ratio of the density of a liquid and the density of pure water at a specific temperature.

Spirometer. A displacement gasometer consisting of an inverted bell resting upon or sealed by liquid (or other means) and capable of showing the amount of gas added to or withdrawn from the bell by the displacement (rise or fall) of the bell.

Standard. A concept that has been established by authority, custom, or agreement to serve as a model or rule in the measurement of quantity of the establishment of a practice or procedure.

Standard Conditions (EPA-defined). EPA-defined standard conditions of temperature and

pressure are 68°F (20°C) and 14.7 psia (760 mm Hg).

Standard Deviation. A measure of the dispersion of a set of numbers.

Standard Operating Procedure (SOP). A written document that details the method for an operation, analysis, or action with thoroughly prescribed techniques and steps and that is officially approved as the method for performing certain routine or repetitive tasks.

State Implementation Plan (SIP). A complex and voluminous document that contains comprehensive emission inventories, proposed control strategies, demonstration of modeling/calculation results, summaries of regulatory authority, description of monitoring programs, and enforcement procedures.

Static Pressure. A measure of the resistance to airflow through a system.

Stationary Source. Any building, structure, facility, or installation which emits or may emit any air pollutant.

Stratosphere. The atmospheric layer just above the troposphere, which starts at approximately 7.5 miles (12 km) above the Earth and rises to approximately 31.1 miles (50 km). The beneficial ozone layer resides in the stratosphere.

Stressors. Physical, chemical, or biological entities that can induce adverse effects on ecosystems or human health [EPA/OA/Office of Communications, Education, and Media Relations: Terms of Environment: Glossary, Abbreviations, and Acronyms (Revised December 1997)].

Sulfates. Inorganic salts of sulfuric acid (H₂SO₄) containing the divalent, negative ion.

Sulfites. Inorganic salts of sulfurous acid (H₂SO₃) containing the divalent, negative ion.

T

Terminal Settling Velocity. The velocity of a falling particle when the gravitational force downward is balanced by the air resistance (or drag) force upward.

Total Filterable Particulate Matter. Particulate matter of all sizes is regulated as total filterable particulate matter. This category of air pollutants was the first one that was subject to air pollution control regulations.

Total Suspended Particulates (TSP). Particulate matter collected by the high-volume sampler, usually particles of up to 100 micrometers in aerodynamic diameter

Traceability. This term is defined in 40 CFR Parts 50 and 58 as meaning, “that a local standard has been compared and certified, either directly or via not more than one intermediate standard, to a primary standard such as a National Institute of Standards and Technology Standard Reference material (NIST-SRM).”

Transfer Standard. A transfer standard is a device that is certified against a primary standard. These standards usually travel to monitoring stations. Transfer standards can be volumetric, electronic flow meters, wet test meters, pressure gauges, or pressure/flow transducers. These devices usually have a certain amount of error involved in their operation and can drift with time. Therefore they must be verified against a primary standard on a known set schedule.

Traceability to NIST. Documented procedure by which a standard is related to a more reliable standard verified by the National Institute of Standards Technology (NIST).

Troposphere. The lowest layer of the Earth's atmosphere, which rises to a height of approximately 7.5 miles (12 km). Air masses, fronts, and storms reside in this layer.

U

Ultrafine Particles. EPA classification of particles having aerodynamic diameters less than or equal to 0.1 micrometer.

Uncertainty. An allowance assigned to a measured value to take into account two major components of error: The systematic error and the random error attributed to the imprecision of the measurement process.

Unit Risk Estimate or Factor (URE or URF). The upper-bound excess lifetime cancer risk estimated to result for continuous exposure to an agent at a concentration of 1 Fg/L in water, or 1 Fg/m³ in air. The interpretation of unit risk would be as follows: if unit risk = 1.5×10^{-6} Fg/L, 1.5 excess tumors are expected to develop per 1,000,000 people if exposed daily for a lifetime to 1 Fg of the chemical in 1 liter of drinking water.

V

Van der Waal Forces. Weak attraction forces (1 to 10 Kcal/gm-mole) between molecules such as in gases and liquids.

Vapor. The gaseous phase of matter which normally exists in a liquid or solid state.

Vapor Pressure. In a closed system at a constant temperature, the pressure exerted by gaseous molecules that are in equilibrium with molecules of the same kind in the liquid or solid state.

Velocity Pressure. The pressure required to accelerate air from zero velocity to a greater velocity. It is proportional to the kinetic energy of the air stream.

Venturi Scrubber. A type of wet scrubber that is usually highly efficient but requires a large amount of energy to operate. (Wet scrubbers are air pollution control devices.) In venturi scrubbers, a scrubbing liquid is introduced into the gas stream, which then passes through a contracted area of the scrubber at a high velocity creating a high dispersion of fine droplets. These fine droplets capture the gaseous and particulate pollutants.

Verification. A verification is the process of checking one primary authority against another primary authority. This can be done by inter-comparing two primary standards against each other or an agency primary standard against another agency's primary standard or NIST standard.

Verification. Confirmation by examination and provision of objective evidence that specified requirements have been fulfilled. In design and development, verification concerns the process of examining a result of a given activity to determine conformance to the stated requirements for that activity.

Viscosity (Absolute). The resistance of a fluid to shear stress.

Viscosity (Kinematic). The value obtained when the absolute viscosity is divided by the density of the fluid.

Volume Concentration. Concentration expressed in terms of gaseous volume of substance per unit volume of air or other gas, usually expressed in percent or parts per million.

Volume Percent. Percentage of the total volume of a gas sample that is comprised of the volume of a single gaseous constituent.

W

Working Flow-Rate Standard. A flow-rate measuring device, such as a standard orifice meter, that has been calibrated against a master flow-rate standard. The working flow-rate standard is used to calibrate a flow measuring or flow rate indicating instrument.

X

Y

Z

Acronyms

ACFM	Actual Cubic Feet per Minute
AIRS	Aerometric Information Retrieval System
ADBA	AIRS Data Base Administrator
AIRMoN	Atmospheric Integrated Research Monitoring Network
ALAPCO	Association of Local Air Pollution Control Officials
AMTIC	Ambient Monitoring Technical Information Center
APTI	Air Pollution Training Institute
AQI	Air Quality Index

AQS	Air Quality (data) System
AQSSD	Air Quality Strategies and Standards Division
ARM	Approved Regional Method
AWMA	Air and Waste Management Association
BAM	Beta Attenuation Monitor
BTU	British Thermal Unit
CAA	Clean Air Act
CAC	Correlating Acceptable Continuous (monitor)
CAIR	Clean Air Interstate Rule
CASAC	Clean Air Science Advisory Committee
CBI	Confidential Business Information
CBSA	Core Based Statistical Area
CENR	Committee for Environment and Natural Resources
CEU	Continuing Education Unit
CFR	Code of Federal Regulations
CMAQ	Community Model Air Quality (system)
CO	Carbon Monoxide
CO	Contracting Officer
CRPAQS	Central Valley (California) Regional Particulate Air Quality Study
CSA	Consolidated Statistical Area
CV	Coefficient of Variance
CY	Calendar year
DC	Direct Current
DCO	Document Control Officer
DD	Division Director
DHS	Department of Homeland Security
DMC	Data Management Center
DOE	Department of Energy
DOI	Department of Interior
DQA	Data Quality Assessment
DQAO	Deputy QA Officers
DQI	Data Quality Indicator
DQOs	Data Quality Objectives
EC	Elemental Carbon
EDO	Environmental Data Operation
EMAD	Emissions, Monitoring, and Analysis Division
EPA	U.S. Environmental Protection Agency
EPAAR	EPA Acquisition Regulations
ESAT	Environmental Services Assistance Team
ESD	Emission Standards Division
ETSD	Enterprise Technology Services Division
FAR	Federal Acquisition Regulations
FEM	Federal Equivalent Method
FIPS	Federal Information Processing Standards
FLM	Federal Land Manager
FPD	Flame Photometric Detection
FRM	Federal Reference Method
FY	Fiscal Year

GAO	General Accounting Office
GC	Gas Chromatograph
GFC	Gas Filter Correlation
GIS	Geographical Information Systems
GLP	Good Laboratory Practice
HAP	Hazardous Air Pollutants
HCN	Hydrogen Cyanide
HEI	Health Effects Institute
IACET	International Association for Continuing Education and Training
IADN	Interagency Deposition Network
IAG	Interagency Agreement
IC	Ion Chromatography
IDP	Individual Development Plans
IMPROVE	Interagency Monitoring of Protected Visual Environments
IR	Infrared
IT	Information Technology
ITEP	Institute of Tribal Environmental Professionals
ITPID	Information Transfer and Program Integration Division
ITT	Information Transfer Technology
K	Thousand
LAN	Local Area Network
LDL	Lower Detectable Limit
M	Million
MACT	Maximum Achievable Control Technology
MANE-VU	Mid-Atlantic/Northeast Visibility Union
MDL	Method Detection Limit
MDN	Mercury Disposition Network
MQAG	Monitoring and Quality Assurance Group
MQOs	Measurement Quality Objectives
MPA	Monitoring Planning Area
Mo	Molybdenum
MSA	Metropolitan Statistical Area
MSR	Management System Review
N₂	Nitrogen
NAAMS	National Ambient Air Monitoring Strategy
NADP	National Atmospheric Deposition Program
NAAQS	National Ambient Air Quality Standards
NAMS	National Air Monitoring Station
NAPAP	National Acid Precipitation Assessment Program
NARSTO	North American Research Strategy for Tropospheric Ozone
NAS	National Academy of Science
NASA	National Aeronautics and Space Administration
NAU	Northern Arizona University
NCORE	National Core Monitoring Network
NDIR	Non-Dispersive Infrared
NECMSA	New England County Metropolitan Statistical Area
NESHAP	National Emission Standards for Hazardous Air Pollutants
NH₃	Ammonia

NH₄⁺	Ammonium	
NIST	National Institute of Standards and Technology	
NMHC	Non-Methane Hydrocarbons	
NMSC	National Monitoring Strategy (or Steering) Committee	
NO	Nitrogen Oxide	
NO₂	Nitrogen Dioxide	
NO_x	Nitrogen Oxides	
NO_y	Reactive Nitrogen Oxides	
NOAA	National Oceanic and Atmospheric Administration	
NPAP	National Performance Audit Program	
NPEP	National Performance Evaluation Program	
NPN	Non-Propyl Nitrate	
NPS	National Parks Service	
NSPS	New Source Performance Standard	
NTN	National Trends Network	
O₃	Ozone	
OAP	Office of Atmospheric Programs	
OAQPS	Office of Air Quality Planning and Standards	
OARM	Office of Administration and Resources Management	
OC	Organic Carbon	
OEI	Office of Environmental Information	
OIRM	Office of Information Resources Management	
OMB	Office of Management and Budget	
ORD	Office of Research and Development	
ORIA	Office of Radiation and Indoor Air	
PAMS	Photochemical Assessment Monitoring Stations	
P&A	Precision and Accuracy	
Pb	Lead	
PBT	Persistent Bioaccumulative Toxics	
PBMS	Performance Based Measurement System	
PC	Personal Computer	
PE	Performance Evaluation	
PEP	Performance Evaluation Program	
Pt	Platinum	
PM	Particulate Matter	
PM₁₀	Particulate Matter with aerodynamic diameter less than micrometers	10
PM_{2.5}	Particulate Matter with aerodynamic diameter less than micrometers	2.5
PMT	Photomultiplier Tube	
POP	Persistent Organic Pollutants	
ppb	part per billion	
ppm	part per million	
PR	Procurement Request	
PMSA	Primary Metropolitan Statistical Area	
PSD	Prevention of Signification Deterioration	
PDW	Primary Wind Direction	
QA	Quality Assurance	

QC	Quality Control
QA/QC	Quality Assurance/Quality Control
QAPP	Quality Assurance Project Plan
QMP	Quality Management Plan
RADM	Regional Acid Deposition Model
REM	Regional Equivalent Monitor
RCRA	Resource Conservation and Recovery Act
RO	EPA Regional Office
ROM	Regional Oxidant Model
RPO	Regional Planning Organization
RTP	Research Triangle Park (North Carolina)
S & T	Science and Technology
SAMWG	Standing Air Monitoring Workgroup
SCFM	Standard Cubic Feet per Minute
SCG	Source Characterization Group
SIPS	State Implementation Plans
SIRMO	Servicing Information Resources Management Officer
SLAMS	State and Local Air Monitoring Station
SLTs	State and Local Agencies and Tribes
SO₂	Sulfur Dioxide
SOP	Standard Operating Procedure
SOW	Statement or Scope Of Work
SPM	Special Purpose Monitor
SPMS	Special Purpose Monitoring Stations
SRP	Standard Reference Photometer
SS	Supersite
STAG	State and Tribal Air Grant
STAPPA	State and Territorial Air Pollution Program Administrators
STN	Speciation Trend Network
Strategy	The National Air Monitoring Strategy
SVOC	Semi-Volatile Organic Compound
SYSOP	System Operator
TAD	Technical Assistance Document
TAMS	Tribal Air Monitoring Support (Center)
TAR	Tribal Authority Rule
TBD	To Be Determined
TEOM	Tapered Element Oscillation Monitor
TIP	Tribal Implementation Plan
TNMOC	Total Non-Methane Organic Compound
TSA	Technical System Audit
TSP	Total Suspended Solids
TSP	Total Suspended Particulates
USB	Universal Serial Bus
VOC	Volatile Organic Compound
UV	Ultraviolet
WAM	Work Assignment Manager
XML	Extensible Markup Language

Appendix D

Conversion Factors and Useful Information

DRAFT

Conversion Factors and Useful Information

International Metric System – Le Systeme International d’Unites (SI Units)

Base Units of the International Metric System (SI)		
Quantity	Name of the Unit	Symbol
Length	meter	m
Mass	kilogram	kg
Time	second	s
Temperature	Kelvin	K
Electric current	ampere	A
Luminous intensity	candela	cd
Amount of substance	mole	mol

Recommended decimal multiples and submultiples with corresponding prefixes and names

Factor	Prefix	Symbol	Meaning
10^{12}	tera	T	One trillion times
10^9	giga	G	One billion times
10^6	mega	M	One million times
10^3	kilo	k	One thousand times
10^2	hecto	h	One hundred times
10	deca	da	Ten times
10^{-1}	deca	d	One tenth of
10^{-2}	centi	c	One hundredth of
10^{-3}	milli	m	One thousandth of
10^{-6}	micro		One millionth of
10^{-9}	nano	n	One billionth of
10^{-12}	pico	p	One trillionth of
10^{-15}	femto	f	One quadrillionth of
10^{-18}	atto	a	One quintillionth of

Pressure

From/to	mm Hg	in. Hg	in. H ₂ O	ft H ₂ O	atm	lb/in. ²	Kg/cm ²
mm Hg	1	0.03937	0.5553	0.04460	0.00132	0.01934	0.00136
in. Hg	25.40	1	13.60	1.133	0.03342	0.4912	0.03453
in. H ₂ O	1.868	0.07355	1	0.08333	0.00246	0.03613	0.00254
ft. H ₂ O	22.42	0.8826	12	1	0.02950	0.4335	0.03048
atm	760	29.92	406.8	33.90	1	14.70	1.033

lb/in. ²	51.71	2.036	27.67	2.301	0.06805	1	0.07031
kg/cm ²	735.6	28.96	393.7	32.81	0.9678	14.22	1

Volume

From/to	cm ³	liter	m ³	in. ³	ft ³
cm ³	1	0.001	1 x 10 ⁻³	0.06102	3.53 x 10 ⁻⁵
liter	1000	1	0.001	61.02	0.03532
m ³	1 x 10 ³	1000	1	6.10 x 10 ²	35.31
in. ³	16.39	0.01639	1.64 x 10 ⁻²	1	5.79 x 10 ⁻²
ft ³	2.83 x 10 ²	28.32	0.02832	1728	1

Temperature

$^{\circ}\text{C} = \frac{5}{9} (\text{F} - 32)$	$^{\circ}\text{F} = \frac{9}{5} (\text{C} + 32)$
$\text{K} = ^{\circ}\text{C} + 273.2$	$^{\circ}\text{R} = ^{\circ}\text{F} + 459.7$

Conversion Factors - flow

Desired units \ Given units	$\frac{\text{m}^3}{\text{sec}}$	$\frac{\text{m}^3}{\text{min}}$	$\frac{\text{m}^3}{\text{hour}}$	$\frac{\text{ft}^3}{\text{sec}}$	$\frac{\text{ft}^3}{\text{min}}$	$\frac{\text{ft}^3}{\text{hour}}$	$\frac{\text{L}}{\text{sec}}$	$\frac{\text{L}}{\text{min}}$	$\frac{\text{cm}^3}{\text{sec}}$	$\frac{\text{cm}^3}{\text{min}}$
$\frac{\text{m}^3}{\text{sec}}$	1	60	3600	35.3144	21.1887 x 10 ²	12.7132 x 10 ⁴	999.973	59.998 x 10 ³	1 x 10 ⁵	6 x 10 ⁷
$\frac{\text{m}^3}{\text{min}}$	0.0167	1	60	0.5886	35.3144	21.189 x 10 ²	16.667	999.973	16.667 x 10 ³	1 x 10 ⁴

$\frac{\text{m}^3}{\text{hour}}$	2.778×10^{-3}	16.667×10^{-3}	1	98.90×10^{-6}	0.5886	35.3144	27.777×10^{-2}	16.667	2.777×10^2	1.666×10^4
$\frac{\text{ft}^3}{\text{sec}}$	28.317×10^{-3}	1.699	101.94	1	60	3600	28.316	16.9896×10^2	2.8317×10^4	1.699×10^6
$\frac{\text{ft}^3}{\text{min}}$	4.7195×10^{-6}	28.317×10^{-3}	1.699	16.667×10^{-2}	1	60	47.193×10^{-2}	28.316	4.7195×10^3	2.8317
$\frac{\text{ft}^3}{\text{hour}}$	7.8658×10^{-6}	4.7195×10^{-4}	28.317×10^{-3}	2.778×10^{-4}	16.667×10^{-2}	1	7.866×10^{-3}	0.4719	78.658	4.7195×10^2
$\frac{\text{L}}{\text{sec}}$	1.000027×10^{-3}	6.00016×10^{-2}	3.6	35.316×10^{-3}	2.11896	127.138	1	60	1000.027	16.667
$\frac{\text{L}}{\text{min}}$	1.6667×10^{-6}	1.000027×10^{-3}	6.00016×10^{-2}	5.886×10^{-6}	35.316×10^{-3}	2.11896	1.6667×10^{-2}	1	16.667	1000.027
$\frac{\text{cm}^3}{\text{sec}}$	6×10^{-6}	6×10^{-6}	3.6×10^{-3}	3.5314×10^{-6}	2.1189×10^{-3}	1.271	9.99973×10^{-4}	5.9998×10^{-2}	1	16.667×10^{-2}
$\frac{\text{cm}^3}{\text{min}}$	1×10^{-6}	1×10^{-4}	6×10^{-6}	5.886×10^{-7}	0.3531×10^{-6}	2.11887×10^{-3}	5.9998×10^{-2}	9.99973×10^{-6}	60	1

To convert a value from a given unit to a desired unit, multiply the given value by the factor opposite the given unit and beneath the desired unit.

Conversion Factors - ppm vs. $\mu\text{g}/\text{m}^3$

Desired units \ Given units	Parts per million by volume – ppm					
	O ₃	NO ₂	SO ₂	H ₂ S	CO	HC as methane
$\mu\text{g}/\text{m}^3$	5.10×10^{-4}	5.32×10^{-4}	3.83×10^{-4}	7.19×10^{-4}	-	-
mg/m ³	-	-	-	-	0.875	1.53

Desired units \ Given units	$\mu\text{g}/\text{m}^3$				mg/m ³	
	O ₃	NO ₂	SO ₂	H ₂ S	CO	HC
ppm	1960	1880	2610	1390	1.14	0.654

To convert a value from a given unit to a desired unit, multiply the given value by the factor opposite the given units and beneath the desired unit.

Atomic weight and numbers

Name	Symbol	Atomic number	Atomic weight	Name	Symbol	Atomic number	Atomic weight
Actinium	Ac	89	-	Mercury	Hg	80	200.59
Aluminum	Al	13	26.9815	Molybdenum	Mo	42	95.94
Americium	Am	95	-	Neodymium	Nd	60	144.24
Antimony	Sb	51	121.75	Neon	Ne	10	20.183
Argon	Ar	18	39.948	Neptunium	Np	93	-
Arsenic	As	33	74.9216	Nickel	Ni	28	58.71
Astatine	At	85	-	Niobium	Nb	41	92.906
Barium	Ba	56	137.34	Nitrogen	N	7	14.0067
Berkelium	Bk	97	-	Nobelium	No	102	-
Beryllium	Be	4	9.0122	Osmium	Os	75	190.2
Bismuth	Bi	83	208.980	Oxygen	O	8	15.9994
Boron	B	5	10.811	Palladium	Pd	46	106.4
Bromine	Br	35	79.904	Phosphorus	P	15	30.9738
Cadmium	Cd	48	112.40	Platinum	Pt	78	195.09
Calcium	Ca	20	40.08	Plutonium	Pu	94	-
Californium	Cf	98	-	Polonium	Po	84	-
Carbon	C	6	12.01115	Potassium	K	19	39.102
Cerium	Ce	58	140.12	Praseodymium	Pr	59	140.907
Cesium	Cs	55	132.905	Promethium	Pm	61	-
Chlorine	Cl	17	35.453	Protactinium	Pa	91	-
Chromium	Cr	24	51.996	Radium	Ra	88	-
Cobalt	Co	27	58.9332	Radon	Rn	86	-
Copper	Cu	29	63.546	Rhenium	Re	75	186.2
Curium	Cm	96	-	Rhodium	Rh	45	102.905
Dysprosium	Dy	66	162.50	Rubidium	Rb	37	84.57
Einsteinium	Es	99	-	Ruthenium	Ru	44	101.07
Erbium	Er	68	167.26	Samarium	Sm	62	150.35
Europium	Eu	63	151.96	Scandium	Sc	21	44.956
Fermium	Fm	100	-	Selenium	Se	34	78.96
Fluorine	F	9	18.9984	Silicon	Si	14	28.086
Francium	Fr	87	-	Silver	Ag	47	107.868
Gadolinium	Gd	64	157.25	Sodium	Na	11	22.9898
Gallium	Ga	31	69.72	Strontium	Sr	38	87.62
Germanium	Ge	32	72.59	Sulfur	S	16	32.064
Gold	Au	79	196.967	Tantalum	Ta	73	180.946
Hafnium	Hf	72	178.49	Technetium	Tc	43	-
Helium	He	2	4.0026	Tellurium	Te	52	127.60
Holmium	Ho	67	164.930	Terbium	Tb	65	158.924
Hydrogen	H	1	1.00797	Thallium	Tl	81	204.37
Indium	In	49	114.82	Thorium	Th	90	232.038
Iodine	I	53	126.9044	Thulium	Tm	59	168.934
Iridium	Ir	77	196.2	Tin	Sn	50	118.69
Iron	Fe	26	55.847	Titanium	Ti	22	47.90
Krypton	Kr	36	83.80	Tungsten	W	74	183.85
Lanthanum	La	57	138.91	Uranium	U	92	238.03
Lawrencium	Lr	103	-	Vanadium	V	23	50.942
Lead	Pb	82	207.19	Xenon	Xe	54	131.30
Lithium	Li	3	6.939	Ytterbium	Yb	70	173.04
Lutetium	Lu	71	174.97	Yttrium	Y	39	88.905
Magnesium	Mg	12	24.312	Zinc	Zn	30	65.37
Manganese	Mn	25	54.9380	Zirconium	Zr	40	91.22
Mendelevium	Md	101	-				

Saturation vapor pressure over water (°C, mm Hg)*

Values for fractional degree between 50 and 89 were obtained by interpolation											
Temp. °C	0.0	0.2	0.4	0.6	0.8	Temp. °C	0.0	0.2	0.4	0.6	0.8
-15	1.436	1.414	1.390	1.368	1.345	42	61.50	62.14	62.80	63.46	64.12
-14	1.560	1.534	1.511	1.485	1.460	43	64.80	65.48	66.16	66.86	67.56
-13	1.691	1.665	1.637	1.611	1.585	44	68.26	68.97	69.69	70.41	71.14
-12	1.834	1.804	1.776	1.748	1.720						
-11	1.987	1.955	1.924	1.893	1.863	45	71.88	72.62	73.36	74.12	74.88
						46	75.65	76.43	77.21	78.00	78.80
-10	2.149	2.116	2.084	2.050	2.018	47	79.60	80.41	81.23	82.05	82.87
-9	2.326	2.289	2.254	2.219	2.184	48	83.71	84.56	85.42	86.28	87.14
-8	2.514	2.475	2.437	2.399	2.362	49	88.02	88.90	89.79	90.69	91.59
-7	2.715	2.674	2.635	2.593	2.553						
-6	2.931	2.887	2.843	2.800	2.757	50	92.51	93.5	94.4	95.3	96.3
						51	97.20	98.2	99.1	100.1	101.1
-5	3.163	3.115	3.069	3.022	2.976	52	102.09	103.1	104.1	105.1	106.2
-4	3.410	3.359	3.309	3.259	3.211	53	107.20	108.2	109.3	110.4	111.4
-3	3.673	3.620	3.567	3.514	3.461	54	112.51	113.6	114.7	115.8	116.9
-2	3.956	3.898	3.841	3.785	3.730						
-1	4.258	4.196	4.135	4.075	4.016	55	118.04	119.1	120.3	121.5	122.6
						56	123.90	125.0	126.2	127.4	128.6
-0	4.579	4.513	4.448	4.385	4.320	57	129.82	131.0	132.3	133.5	134.7
						58	136.08	137.3	138.5	139.9	141.2
0	4.579	4.647	4.715	4.785	4.855	59	142.60	143.9	145.2	146.6	148.0
1	4.926	4.998	5.070	5.144	5.219						
2	5.294	5.370	5.447	5.525	5.605	60	149.38	150.7	152.1	153.5	155.0
3	5.685	5.766	5.848	5.931	6.015	61	156.43	157.8	159.3	160.8	162.3
4	6.101	6.187	6.274	6.363	6.453	62	163.77	165.2	166.8	168.3	169.8
						63	171.38	172.9	174.5	176.1	177.7
5	6.543	6.635	6.728	6.822	6.917	64	179.31	180.9	182.5	184.2	185.8
6	7.013	7.111	7.209	7.309	7.411						
7	7.513	7.617	7.722	7.828	7.936	65	187.54	189.2	190.9	192.6	194.3
8	8.045	8.155	8.267	8.380	8.484	66	196.09	197.8	199.5	201.3	203.1
9	8.609	8.727	8.845	8.965	9.086	67	204.96	206.8	208.6	210.5	212.3
						68	214.17	216.0	218.0	219.9	221.8
10	9.209	9.333	9.458	9.585	9.714	69	223.78	225.7	227.7	229.7	231.7
11	9.844	9.976	10.109	10.244	10.380						
12	10.518	10.658	10.799	10.941	11.085	70	233.7	235.7	237.7	239.7	241.8
13	11.231	11.379	11.528	11.680	11.833	71	243.9	246.0	248.2	250.3	252.7
14	11.987	12.144	12.302	12.462	12.624	72	254.6	256.8	259.0	261.2	263.4
						73	265.7	268.0	270.2	272.6	274.8
15	12.788	12.953	13.121	13.290	13.461	74	277.2	279.4	281.8	284.2	286.6
16	13.634	13.809	13.987	14.166	14.347	75	289.1	291.5	294.0	296.4	298.8
17	14.530	14.715	14.903	15.092	15.284	76	301.4	303.8	306.4	308.9	311.4
18	15.477	15.673	15.871	16.071	16.272	77	314.1	316.6	319.2	322.0	324.6
19	16.477	16.685	16.894	17.105	17.319	78	327.3	330.0	332.8	335.6	338.2
						79	341.0	343.8	346.6	349.4	352.2
20	17.535	17.853	17.974	18.197	18.422						
21	18.650	18.880	19.113	19.349	19.587	80	355.1	358.0	361.0	363.8	366.8
22	19.827	20.070	20.312	20.565	20.815	81	369.7	372.6	375.6	378.8	381.8
23	21.068	21.324	21.583	21.845	22.110	82	384.9	388.0	391.2	394.4	397.4
24	22.377	22.648	22.922	23.198	23.476	83	400.6	403.8	407.0	410.2	413.6
						84	416.8	420.2	423.6	426.8	430.2
25	23.756	24.039	24.326	24.617	24.912						
26	25.209	25.509	25.812	26.117	26.426	85	433.6	437.0	440.4	444.0	447.5
27	26.739	27.055	27.374	27.696	28.021	86	450.9	454.4	458.0	461.6	465.2
28	28.349	28.680	29.015	29.354	29.697	87	468.7	472.4	476.0	479.8	483.4
29	30.043	30.392	30.745	31.102	31.461	88	487.1	491.0	494.7	498.5	502.2
						89	506.1	510.0	513.9	517.8	521.8
30	31.824	32.191	32.561	32.934	33.312						
31	33.695	34.082	34.471	34.864	35.261	90	525.76	529.77	533.90	537.86	541.95
32	35.663	36.068	36.477	36.891	37.308	91	546.05	550.18	554.35	558.53	562.75
33	37.729	38.155	38.584	39.018	39.457	92	566.99	571.26	575.55	579.87	584.22
34	39.898	40.344	40.796	41.251	41.710	93	588.60	593.00	597.43	601.89	606.38
						94	610.90	615.44	620.01	624.61	629.24
35	42.175	42.644	43.117	43.595	44.078	95	633.90	638.59	643.30	648.05	652.82
36	44.563	45.054	45.549	46.050	46.556	96	657.62	662.45	667.31	672.20	677.12
37	47.067	47.582	48.102	48.627	49.157	97	682.07	687.04	692.05	697.10	702.17
38	49.692	50.231	50.774	51.323	51.877	98	707.27	712.40	717.56	722.75	727.98
39	52.442	53.009	53.580	54.156	54.737	99	733.24	738.53	743.85	749.20	754.58
40	55.324	55.91	56.51	57.11	57.72	100	760.00	765.45	770.93	776.55	782.00
41	58.34	58.96	59.58	60.22	60.86	101	787.57	793.18	798.82	804.50	810.21

*Handbook of Chemistry and Physics, 45th edition. Chemical Rubber Publishing Company, 1965.

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Appendix E

Ambient Air Monitoring Quality Assurance Information and Web Addresses

The following information provides key guidance documents and reports that can be found on various sites within the Ambient Monitoring Technical Information Center (AMTIC) Website. The following identifiers are used to describe national ambient air monitoring programs:

SLAMS - State or Local Air Monitoring Stations Network
NCORE - National Core Network
PAMS - Photochemical Assessment Monitoring Stations
CSN PM2.5 - Chemical Speciation Network
NATTS - National Toxics Trends Network
SLAMS/NPAP - National Performance Audit Program
SLAM/PEP - National PM2.5 Performance Evaluation Program

Quality Assurance Information

Identifier	Title	EPA Number	Pub Date Year	URL
GUIDANCE DOCUMENTS				
CSN NATTS	Particulate Matter (PM2.5) Speciation Guidance Document NATTS Technical Assistance Document (TAD)		1999 2007	http://www.epa.gov/ttn/amtic/files/ambient/pm25/spec/specfinl.pdf http://www.epa.gov/ttn/amtic/airtox.html
NCore	NCore Technical Assistance Document (TAD)		2005	http://www.epa.gov/ttn/amtic/files/ambient/monitorstrat/precursor/tadversion4.pdf
NCore	QA Handbook for Air Pollution Measurement Systems Volume IV Meteorological Measurement Systems Technical Assistance Document (TAD) for Sampling and	EPA-454/B-08-002	2008	http://www.epa.gov/ttn/amtic/met.html
PAMS	Analysis of Ozone Precursors;	EPA/600-R-98/161	1998	http://www.epa.gov/ttn/amtic/files/ambient/pams/newtad.pdf
SLAMS	QA Handbook for Air Pollution Measurement Systems Volume II	EPA-454/R-98-004	1998	http://www.epa.gov/ttn/amtic/files/ambient/qaqc/redbook.pdf
SLAMS	Guideline on the Meaning and the Use of Precision and Bias Data Required by 40 CFR Part 58 Appendix A	EPA-545/B-07-001	2007	http://www.epa.gov/ttn/amtic/files/ambient/qaqc/P&B%20Guidance%2010.10.07%20vers1.1.pdf
SLAMS PM2.5	PM _{2.5} Quality Assurance Program Overview		1997	http://www.epa.gov/ttn/amtic/files/ambient/pm25/ga/pm25ga.pdf
QA REPORTS				
CSN NATTS	PM 2.5 Speciation Lab Audit Reports and Assessments National Air Toxics Trends Stations Quality Assurance Annual Reports and Proficiency Reports		Various Years Various Years	http://www.epa.gov/ttn/amtic/pmspec.html http://www.epa.gov/ttn/amtic/airtoxqa.html
SLAMS	2007 Quality Management Plan and Quality Assurance Project Plan Tracking Matrix as of June 25, 2007		2007	http://www.epa.gov/ttn/amtic/files/ambient/qaqc/Region%20Matrix%2006.25.07.pdf
SLAMS SLAMS-PM2.5	Annual Precision, Bias and Completeness Reports for Criteria Pollutants 3-Year and Annual QA Reports		Various Years Various Years	http://www.epa.gov/ttn/amtic/parslist.html http://www.epa.gov/ttn/amtic/anlqa.html
SLAMS-PEP	Laboratory Comparison Study of Gravimetric Laboratories Performing PM _{2.5} Filter Weighing for the PM _{2.5} Performance Evaluation Program and Tribal Air Monitoring Support		Various Years	http://www.epa.gov/ttn/amtic/pmpep.html
Methods				
CSN NATTS NCore	Speciation Field Guidance Documents Air Toxics Methods- Various Methods Calibration of Meterological Measurement -Videos		Various Years 2007 2008	http://www.epa.gov/ttn/amtic/specquid.html http://www.epa.gov/ttn/amtic/airtox.html http://www.epa.gov/ttn/amtic/met.html
SLAMS	QA Handbook Vol II (DRAFT Procedure for the "Determination of Ozone By Ultraviolet Analysis")		1998	http://www.epa.gov/ttn/amtic/files/ambient/qaqc/ozone4.pdf
SLAMS	Sec. 2.10 of QA Handbook - Draft - PM10- Dichot revised to local standard and pressure	EPA-600/4-77-027a	1997	http://www.epa.gov/ttn/amtic/files/ambient/qaqc/2-10meth.pdf
SLAMS	Sec. 2.11 of QA Handbook - Draft - PM10 Hi Vol revised to local standard and pressure		1997	http://www.epa.gov/ttn/amtic/files/ambient/qaqc/2-11meth.pdf
SLAMS	Section 2.3 -- DRAFT - Reference Method for the Determination of Nitrogen Dioxide in the Atmosphere (Chemiluminescence)		2002	http://www.epa.gov/ttn/amtic/files/ambient/pm25/ga/no2.pdf
SLAMS-NPAP	DRAFT SOP for Through-the-Probe Performance Evaluations of Ambient Air Quality Monitoring of Criteria Air Pollutants		2007	http://www.epa.gov/ttn/amtic/files/ambient/npapsop/npaptpopsop.pdf
SLAMS-NPAP	Quality Assurance Project Plan for the Audit Support Program - NPAP and NATTS		2006	http://www.epa.gov/ttn/amtic/files/ambient/qaqc/NPAPQAPPPrvsn071007onforTTP.pdf

Quality Assurance Information

Identifier	Title	EPA Number	Pub Date Year	URL
SLAMS-PEP	Method Compendium "Field Standard Operating Procedures for the PM _{2.5} Performance Evaluation Program"		2006	http://www.epa.gov/ttn/amtic/files/ambient/pm25/qa/pepfield.pdf
SLAMS-PEP	Method Compendium "PM _{2.5} Mass Weighing Laboratory Standard Operating Procedures for the Performance Evaluation Program		1998	http://www.epa.gov/ttn/amtic/files/ambient/pm25/qa/pepsop.pdf
SLAMS-PM2.5	2.12 "Monitoring PM _{2.5} in Ambient Air Using Designated Reference or Class I Equivalent Methods"		1998	http://www.epa.gov/ttn/amtic/files/ambient/pm25/qa/m212covd.pdf
IMPLEMENTATION PLANS and QAPPs				
CSN	Speciation Laboratory Standard Operating Procedures		Various Years	http://www.epa.gov/ttn/amtic/specsop.html
CSN	Quality Management Plan for the PM _{2.5} Speciation Trends Network	EPA-454/R-01-009	2001	http://www.epa.gov/ttn/amtic/files/ambient/pm25/spec/finlqmp.pdf
CSN	"Speciation Trends Network Quality Assurance Project Plan"	EPA-454/R-01-001	2001	http://www.epa.gov/ttn/amtic/files/ambient/pm25/spec/1025sqap.pdf
NATTS	Model Quality Assurance Project Plan for the National Air Toxics Trends Stations - updated version 1.1		2007	http://www.epa.gov/ttn/amtic/files/ambient/airtox/NATTS_Model_QA_PP.pdf
NATTS	Model QAPP for Local-Scale Monitoring Projects"	EPA-454/R-01-007	2006	http://www.epa.gov/ttn/amtic/files/ambient/airtox/pilotqapp.pdf
NATTS	National Air Toxics Trends Stations - Quality Management Plan Final		2005	http://www.epa.gov/ttn/amtic/files/ambient/airtox/nattsqmp.pdf
PAMS	PAMS Implementation Manual	EPA-454/B-93-051	1994	http://www.epa.gov/ttn/amtic/files/ambient/pams/b93-051a.pdf
SLAMS	Quality Assurance Project Plan for the Audit Support Program - NPAP and NATTS		2008	http://www.epa.gov/ttn/amtic/files/ambient/qaqc/NPAPQAPPrvsn071007onforTTP.pdf
SLAMS PM2.5	PM _{2.5} Model QA Project Plan (QAPP)"	EPA-454/R-98-005	1998	http://www.epa.gov/ttn/amtic/files/ambient/pm25/qa/totdoc.pdf
SLAMS PM2.5	PM2.5 FRM Network Federal Performance Evaluation Program Quality Assurance Project Plan (QAPP)		2007	http://www.epa.gov/ttn/amtic/files/ambient/pm25/qa/pepgapp_DRAFT_12-2007_cmt_vrsn.pdf
SLAMS PM2.5	PM _{2.5} Performance Evaluation Program Implementaion Plan		1998	http://www.epa.gov/ttn/amtic/files/ambient/pm25/qa/pep-ip.pdf
WHITE PAPERS/IMPORTANT MEMOS				
CSN	Current List of CSN Sites as of 07-11-2007		2007	http://www.epa.gov/ttn/amtic/specgen.html
CSN	Modification of Carbon Procedures in the Speciation Network;		2006	http://www.epa.gov/ttn/amtic/files/ambient/pm25/spec/fagcarbon.pdf
SLAMS	Overview and Frequently Asked Questions		Various Years	http://www.epa.gov/ttn/amtic/qamsmtg.html
SLAMS	QA National Meeting Presentations		Various Years	http://www.epa.gov/ttn/amtic/qanews.html
SLAMS	QA Newsletters		Various Years	http://www.epa.gov/ttn/amtic/qanews.html