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# SUBCHAPTER C—AIR PROGRAMS (CONTINUED)

## PART 53—AMBIENT AIR MONITORING REFERENCE AND EQUIVALENT METHODS

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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.

## 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. 1857–18571), 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 $PM_{10}$ 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.

*$NO_2$* means nitrogen dioxide.

*$NO_X$* means oxides of nitrogen and is defined as the sum of the concentrations of $NO_2$ and NO.

*$O_3$* 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 $PM_{10}$, $PM_{10C}$, $PM_{2.5}$, $PM_{10-2.5}$, 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.

*$PM_{10}$* 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.

*$PM_{10}$ sampler* means a device, associated with a manual method for measuring $PM_{10}$, designed to collect $PM_{10}$ from an ambient air sample, but lacking the ability to automatically analyze or measure the collected sample to determine the mass concentrations of $PM_{10}$ in the sampled air.

*$PM_{10C}$ sampler* means a $PM_{10}$ 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 $PM_{10}$ 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.

*$SO_2$* 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 $PM_{10}$ 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 ($SO_2$)* and Lead. For measuring $SO_2$ and lead, appendixes A–2 and G of part 50 of this chapter specify unique manual FRM for measuring those pollutants. Except as provided in §53.16, other manual methods for lead will not be considered for a reference method determination under this part.

(2) *$PM_{10}$*. A FRM for measuring $PM_{10}$ 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 $SO_2$, CO, $O_3$, or $NO_2$ must utilize the measurement principle and calibration procedure specified in the appropriate appendix to part 50 of this chapter (appendix A–1 only for $SO_2$ methods) 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, as amended at 75 FR 35597, June 22, 2010]

### §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) *$PM_{10}$*. 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 $PM_{10}$ 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 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, preventive 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]

### § 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 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 via the Internet and may be available from other sources.

[71 FR 61276, Oct. 17, 2006, as amended at 75 FR 35597, June 22, 2010]

### § 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, $PM_{10}$ sampler, $PM_{2.5}$ sampler, or $PM_{10-2.5}$ sampler offered for sale as part of an FRM or FEM shall function within the limits of the performance specifications referred to in §53.20(a), §53.30(a), §53.35, §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, $PM_{10}$ 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, $PM_{10}$ 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, $PM_{10}$ 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.

[71 FR 61276, Oct. 17, 2006, as amended at 78 FR 3281, Jan. 15, 2013; 80 FR 65460, Oct. 26, 2015]

### §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 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 section, 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.

(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 prehearing 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, including 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 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 90 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.

[62 FR 38784, July 18, 1997, as amended at 80 FR 65460, Oct. 26, 2015]

### § 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 cancellation 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 | Reference 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–2 | | | | | | |
| | | Automated | A–1 | ✓ | ✓ | | | | |
| | Equivalent | Manual | A–1 | ✓ | | ✓ | | | |
| | | Automated | A–1 | ✓ | ✓ | ✓ | | | |
| CO | Reference | Automated | C | ✓ | ✓ | | | | |
| | Equivalent | Manual | C | ✓ | | ✓ | | | |
| | | Automated | C | ✓ | ✓ | ✓ | | | |
| $O_3$ | Reference | Automated | D | ✓ | ✓ | | | | |
| | Equivalent | Manual | D | ✓ | | ✓ | | | |
| | | Automated | D | ✓ | ✓ | ✓ | | | |
| $NO_2$ | Reference | Automated | F | ✓ | ✓ | | | | |
| | Equivalent | Manual | F | ✓ | | ✓ | | | |
| | | Automated | F | ✓ | ✓ | ✓ | | | |
| Pb | Reference | Manual | G | | | | | | |
| | Equivalent | Manual | G | ✓ | | ✓ | | | |
| | | Automated | G | ✓ | | ✓ | | | |
| $PM_{10}$-Pb | Reference | Manual | Q | | | | | | |
| | Equivalent | Manual | Q | ✓ | | ✓ | | | |
| | | Automated | Q | ✓ | | ✓ | | | |
| $PM_{10}$ | Reference | Manual | J | ✓ | | | ✓ | | |
| | Equivalent | Manual | J | ✓ | | ✓ | ✓ | | |

| Pollutant | Reference or equivalent | Manual or automated | Applicable part 50 appendix | Applicable subparts of part 53 | | | | | |
|---|---|---|---|---|---|---|---|---|---|
| | | | | A | B | C | D | E | F |
| | | Automated | J | ✓ | | ✓ | ✓ | | |
| $PM_{2.5}$ | Reference | Manual | L | ✓ | | | | ✓ | |
| | Equivalent Class I | Manual | L | ✓ | | ✓ | | ✓ | |
| | Equivalent Class II | Manual | L[1] | ✓ | | ✓[2] | | ✓ | ✓[1 2] |
| | Equivalent Class III. | Automated | L[1] | ✓ | | ✓ | | ✓ | ✓[1] |
| $PM_{10-2.5}$ | Reference | Manual | L, O | ✓ | | | | ✓ | |
| | Equivalent Class I | Manual | L, O | ✓ | | ✓ | | ✓ | |
| | Equivalent Class II | Manual | L, O | ✓ | | ✓[2] | | ✓ | ✓[1 2] |
| | Equivalent Class III. | Automated | L[1], O[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.

[75 FR 35597, June 22, 2010]

## 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/pmqainf.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 for $SO_2$, CO, $O_3$, and $NO_2$

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### § 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 requirement specifications given in table B–1 to subpart B of part 53. A test analyzer representative of the candidate automated method must exhibit performance better than, or not outside, the specified limit or limits for each such performance parameter specified (except range) to satisfy the requirements of this subpart. Except as provided in paragraph (b) of this section, the measurement range of the candidate method must be the standard range specified in table B–1 to subpart B of part 53 to satisfy the requirements of this subpart.

(b) *Measurement ranges.* For a candidate method having more than one selectable measurement range, one range must be the standard range specified in table B–1 to subpart B of part 53, and a test analyzer representative of the method must pass the tests required by this subpart while operated in that range.

(i) *Higher ranges.* The tests may be repeated for one or more higher (broader) ranges (*i.e.*, ranges extending to higher

concentrations) than the standard range specified in table B–1 to subpart B of part 53, provided that the range does not extend to concentrations more than four times the upper range limit of the standard range specified in table B–1 to subpart B of part 53. For such higher ranges, only the tests for range (calibration), noise at 80% of the upper range limit, and lag, rise and fall time are required to be repeated. For the purpose of testing a higher range, the test procedure of § 53.23(e) may be abridged to include only those components needed to test lag, rise and fall time.

(ii) *Lower ranges.* The tests may be repeated for one or more lower (narrower) ranges (*i.e.*, ones extending to lower concentrations) than the standard range specified in table B–1 to subpart B of part 53. For methods for some pollutants, table B–1 to subpart B of part 53 specifies special performance limit requirements for lower ranges. If special low-range performance limit requirements are not specified in table B–1 to subpart B of part 53, then the performance limit requirements for the standard range apply. For lower ranges for any method, only the tests for range (calibration), noise at 0% of the measurement range, lower detectable limit, (and nitric oxide interference for $SO_2$ UVF methods) are required to be repeated, provided the tests for the standard range shows the applicable limit specifications are met for the other test parameters.

(iii) If the tests are conducted and passed only for the specified standard range, any FRM or FEM determination with respect to the method will be limited to that range. If the tests are passed for both the specified range and one or more higher or lower ranges, any such determination will include the additional higher or lower range(s) as well as the specified standard range. Appropriate test data shall be submitted for each range sought to be included in a FRM or FEM method determination under this paragraph (b).

(c) For each performance parameter (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 performance limit specification in table B–1 to subpart B of part 53; a value higher than or outside the specified limit or limits constitutes a failure. These 7 results for each parameter shall be interpreted as follows:

(1) Zero (0) failures: The candidate method passes the test for the performance parameter.

(2) Three (3) or more failures: The candidate method fails the test for the performance parameter.

(3) One (1) or two (2) failures: Repeat the test procedures for the performance 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: The candidate method passes the test for the performance parameter.

(ii) Three (3) or more failures: The candidate method fails the test for the performance parameter.

(d) The tests for *zero drift*, *span drift*, *lag time*, *rise time*, *fall time*, and *precision* shall be carried out in a single integrated procedure conducted at various line voltages and ambient temperatures specified in § 53.23(e). A temperature-controlled environmental test chamber large enough to contain the test analyzer is recommended for this test. The tests for *noise*, *lower detectable limit*, and *interference equivalent* shall be conducted at any ambient temperature between 20 °C and 30 °C, 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 in any 24-hour period.

(e) If necessary, all measurement response readings to be recorded shall be converted to concentration units or adjusted according to the calibration curve constructed in accordance with § 53.21(b).

(f) All recorder chart tracings (or equivalent data plots), 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 TO § 53.20: 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 to this subpart. Symbols and

abbreviations used in this subpart are listed in table B–5 of appendix A to this subpart.

### § 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. The test procedures assume that the test analyzer has a conventional analog measurement signal output that is connected 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. If the test analyzer does not have an analog signal output, or if a digital or other type of measurement data output is used for the tests, an alternative measurement data recording device (or devices) may be used for recording the test data, provided that the device is reasonably suited to the nature and purposes of the tests, and an analog representation of the analyzer measurements for each test can be plotted or otherwise generated that is reasonably similar to the analog measurement recordings that would be produced by a conventional chart recorder connected to a conventional analog signal output.

(b) *Calibration* of the test analyzer shall be carried out prior to conducting the tests described in this subpart. The calibration shall be as indicated in the manual referred to in § 53.4(b)(3) and as follows: If the chart recorder or alternative data recorder does not have below zero capability, adjust either the controls of the test analyzer or the chart or data 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 the data recorder) shall be operated with a similar offset zero. Construct and submit a calibration curve showing a plot of recorder scale readings or other measurement output readings (vertical or y-axis) against pollutant concentrations presented to the analyzer for measurement (horizontal or x-axis). If applicable, a plot of base analog output units (volts, millivolts, milliamps, *etc.*) against pollutant concentrations shall also be obtained and submitted. All such calibration plots shall consist of at least seven (7) approximately equally spaced, identifiable points, including 0 and 90 ±5 percent of the upper range limit (URL).

(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 during the tests and describe the specific 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; however, use of multiple test analyzers to accelerate testing is permissible for testing additional ranges of a multi-range candidate method.

### § 53.22 Generation of test atmospheres.

(a) Table B–2 to subpart B of part 53 specifies preferred methods for generating test atmospheres and suggested methods of verifying their concentrations. Only one means of establishing the concentration of a test atmosphere is normally required, provided that that means is adequately accurate and credible. If the method of generation can produce accurate, reproducible concentrations, verification is optional. If the method of generation is

not reproducible or reasonably quantifiable, then establishment of the concentration by some credible verification method is required.

(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 system shall be vented to insure that test atmospheres are presented to the test analyzer at very nearly atmospheric pressure. The delivery system shall be fabricated from borosilicate glass, FEP Teflon, or other material that is inert with regard to the gas or gases to be used.

(c) The output of the test atmosphere generation system shall be sufficiently stable to obtain stable response readings from the test analyzer 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 react with the test atmospheres or cause a detectable response on the test analyzer.

(e) The concentration of each test atmosphere used shall be quantitatively 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 feasible 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 flow rate or volume 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, photos, descriptions, 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.

**§ 53.23 Test procedures.**

(a) *Range*—(1) *Technical definition.* The nominal minimum and maximum concentrations that a method is capable of measuring.

NOTE TO § 53.23(a)(1): The nominal range is given as the lower and upper range limits in concentration units, for example, 0–0.5 parts per million (ppm).

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

NOTE TO § 53.23(a)(2): A single calibration curve for each measurement range for which an FRM or FEM designation is sought will normally suffice.

(b) *Noise*—(1) *Technical definition.* Spontaneous, short duration deviations in measurements or measurement signal output, about the mean output, that are not caused by input concentration changes. Measurement noise is determined as the standard deviation of a series of measurements of a constant concentration 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 measurement noise at each of two fixed concentrations, first using zero air and then a pollutant test gas concentration as indicated below. The noise limit specification in table B–1 to subpart B of part 53 shall apply to both of these tests.

(ii) For an analyzer with an analog signal output, connect an integrating-type digital meter (DM) suitable for the test analyzer's output and accurate to three significant digits, to determine the analyzer's measurement output signal.

NOTE TO § 53.23(b)(2): Use of a chart recorder in addition to the DM is optional.

(iii) Measure zero air with the test analyzer for 60 minutes. During this 60-minute interval, record twenty-five (25) test analyzer concentration measurements or DM readings at 2-minute intervals. (See Figure B–2 in appendix A of this subpart.)

(iv) If applicable, convert each DM test reading to concentration units (ppm) or adjust the test readings (if

necessary) by reference to the test analyzer's calibration curve as determined in §53.21(b). Label and record the test measurements or converted DM readings as $r_1, r_2, r_3 \ldots r_i \ldots r_{25}$.

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

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

Where $i$ indicates the $i$-th test measurement or DM reading in ppm.

(vi) Let $S$ at 0 ppm be identified as $S_0$; compare $S_0$ to the noise limit specification given in table B–1 to subpart B of part 53.

(vii) Repeat steps in Paragraphs (b)(2)(iii) through (v) of this section using a pollutant test atmosphere concentration of 80 ±5 percent of the URL instead of zero air, and let $S$ at 80 percent of the URL be identified as $S_{80}$. Compare $S_{80}$ to the noise limit specification given in table B–1 to subpart B of part 53.

(viii) Both $S_0$ and $S_{80}$ must be less than or equal to the table B–1 to subpart B of part 53 noise limit specification to pass the test for the noise parameter.

(c) *Lower detectable limit*—(1) *Technical definition*. The minimum pollutant concentration that produces a measurement or measurement output signal of at least 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 measurement reading in ppm as $B_Z$. (See Figure B–3 in appendix A of this subpart.)

(ii) Generate and measure a pollutant test concentration equal to the value for the lower detectable limit specified in table B–1 to subpart B of part 53.

NOTE TO §53.23(c)(2): If necessary, the test concentration may be generated or verified at a higher concentration, then quantitatively and accurately diluted with zero air to the final required test concentration.

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

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

(d) *Interference equivalent*—(1) *Technical definition*. Positive or negative measurement 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 potential interfering agent (interferent) specified in table B–3 to subpart B of part 53. In the event that there are substances likely to cause a significant interference which have not been specified in table B–3 to subpart B of part 53, these substances shall also be tested, in a manner similar to that for the specified interferents, at a concentration substantially higher than that likely to be found in the ambient air. The interference may be either positive or negative, depending on whether the test analyzer's measurement 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 an interferent test concentration not lower than the test concentration specified in table B–3 to subpart B of part 53 (or as otherwise required for unlisted interferents), and comparing the test analyzer's measurement response to the response caused by the pollutant alone. Known gas-phase reactions that might occur between a listed interferent and the pollutant are designated by footnote 3 in table B–3 to subpart B of part 53. In these cases, the interference equivalent shall be determined without mixing with 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 device based upon a chemical reaction to derive part of its specificity and which device requires periodic service or maintenance, the test analyzer shall be ''conditioned'' prior to conducting each interference test series. This requirement includes conditioning for the $NO_2$ converter in chemiluminescence $NO/NO_2/NO_X$ analyzers and for the ozone scrubber in UV-absorption ozone analyzers. Conditioning is as follows:

(A) Service or perform the indicated maintenance on the scrubber or prefilter device, as if it were due for such maintenance, as directed in the manual referred to in § 53.4(b)(3).

(B) Before testing for each potential interferent, allow the test analyzer to sample through the prefilter or scrubber device a test atmosphere containing the interferent at a concentration not lower than the value specified in table B–3 to subpart B of part 53 (or, for unlisted potential interferents, at a concentration substantially higher than likely to be found in ambient air). Sampling shall be at the normal flow rate and shall be continued for 6 continuous hours prior to the interference test series. Conditioning for all applicable interferents prior to any of the interference tests is permissible. Also permissible is simultaneous conditioning with multiple interferents, provided no interferent reactions are likely to occur in the conditioning system.

(iii) Generate three test atmosphere streams as follows:

(A) Test atmosphere *P:* Pollutant test concentration.

(B) Test atmosphere *I:* Interferent test 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 equal.

(B) The concentration of the 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 to subpart B of part 53.

(C) The concentration of the 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 not less than the value specified in table B–3 to subpart B of part 53 (or as otherwise required for unlisted potential interferents).

(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 (passive) mixing component to insure complete mixing of the gases.

(vi) Sample and measure the mixture of test atmospheres *P* and *Z* with the test analyzer. Allow for a stable measurement 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 (passive) mixing component to insure complete mixing of the gases.

(viii) Sample and measure this mixture of *P* and *I* with the test analyzer. Record the stable measurement reading, in concentration units, as $R_I$.

(ix) Calculate the interference equivalent (*IE*) test result as:

$$IE = R_I - R.$$

*IE* must be within the limits (inclusive) specified in table B–1 to subpart B of part 53 for each interferent tested to pass the interference equivalent test.

(x) Follow steps (iii) through (ix) of this section, in turn, to determine the interference equivalent for each listed interferent as well as for any other potential interferents identified.

(xi) For those potential interferents which cannot be mixed with the pollutant, as indicated by footnote (3) in table B–3 to subpart B of part 53, 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

measurement 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 within the limits (inclusive) specified in table B–1 to subpart B of part 53 for each interferent tested to pass the interference equivalent test.

(xii) Sum the *absolute value* of all the individual interference equivalent test results. This sum must be equal to or less than the total interferent limit given in table B–1 to subpart B of part 53 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 measurement response to zero pollutant concentration over 12- and 24-hour periods of continuous unadjusted operation.

(ii) *Span drift:* The percent change in measurement 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 measurement response.

(iv) *Rise time:* The time interval between initial measurement response and 95 percent of final response after a step increase in input concentration.

(v) *Fall time:* The time interval between initial measurement 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, denoted as the standard deviation expressed as a percentage of the upper range limits.[258]

(2) Tests for these performance parameters shall be accomplished over a period of seven (7) or fifteen (15) test days. During this time, the line voltage supplied to the test analyzer and the ambient temperature surrounding the analyzer shall be changed from day to day, as required in paragraph (e)(4) of this section. One test result for each performance parameter shall be obtained each test day, for seven (7) or fifteen (15) test days, as determined from the test results of the first seven days. The tests for each test day are performed in a single integrated procedure.

(3) The 24-hour test day may begin at any clock hour. The first approximately 12 hours of each test day are required for testing 12-hour zero drift. Tests for the other parameters shall be conducted any time during the remaining 12 hours.

(4) Table B–4 to subpart B of part 53 specifies the line voltage and room temperature to be used for each test day. The applicant may elect to specify a wider temperature range (minimum and maximum temperatures) than the range specified in table B–4 to subpart B of part 53 and to conduct these tests over that wider temperature range in lieu of the specified temperature range. If the test results show that all test parameters of this section § 53.23(e) are passed over this wider temperature range, a subsequent FRM or FEM designation for the candidate method based in part on this test shall indicate approval for operation of the method over such wider temperature range. The line voltage and temperature shall be changed to the specified values (or to the alternative, wider temperature values, if applicable) 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, if necessary) test results have been obtained. (The final block may contain fewer than three test days.) Test days need not be contiguous days, but during any idle time between tests or test days, the test analyzer must operate continuously and measurements must be recorded continuously at a low chart speed (or

[258] $NO_2$ precision in Table B–1 is also changed to percent to agree with the calculation specified in 53.23(e)(10)(vi).

equivalent data recording) and included with the test data. 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 test block, manual adjustments to the electronics, gas, or reagent flows or periodic maintenance shall not be permitted. Automatic adjustments that 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 test 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 TO § 53.23(e)(7): If necessary, the beginning of the test days succeeding such maintenance or adjustment may be delayed as required to complete the service or adjustment operation.

(8) All measurement response readings to be recorded shall be converted to concentration units or adjusted (if necessary) 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 sampled and measured long enough to cause a change in measurement response of at least 10% of full scale. Identify all readings and other pertinent data on the strip chart (or equivalent test data record). (See Figure B–1 to subpart B of part 53 illustrating the pattern of the required readings.)

(9) *Test procedure*. (i) Arrange to generate pollutant test atmospheres as follows. Test atmospheres $A_0$, $A_{20}$, and $A_{80}$ shall be maintained consistent during the tests and reproducible from test day to test day.

| 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. |
| $A_{80}$ | 80 ±5 of the upper range limit. |
| $A_{90}$ | 90 ±5 of the upper range limit. |

(ii) For steps within paragraphs (e)(9)(xxv) through (e)(9)(xxxi) of this section, a chart speed of at least 10 centimeters per hour (or equivalent resolution for a digital representation) shall be used to clearly show changes in measurement responses. The actual chart speed, chart speed changes, and time checks shall be clearly marked on the chart.

(iii) *Test day 0*. Allow sufficient time for the test analyzer to warm up and stabilize at a line voltage of 115 volts and a room temperature of 25 °C. Adjust the zero baseline to 5 percent of chart (see § 53.21(b)) and recalibrate, if necessary. No further adjustments shall be made to the analyzer until the end of the tests on the third, sixth, ninth, or twelfth test day.

(iv) Measure test atmosphere $A_0$ until a stable measurement reading is obtained and record this reading (in ppm) as $Z'_n$, where $n = 0$ (see Figure B–4 in appendix A of this subpart).

(v) [Reserved]

(vi) Measure test atmosphere $A_{80}$. Allow for a stable measurement reading and record it as $S'_n$, where $n = 0$.

(vii) The above readings for $Z'_0$ and $S'_0$ 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 to subpart B of part 53 (or to the corresponding alternative temperature if a wider temperature range is being tested).

(ix) Measure test atmosphere $A_0$ continuously for at least twelve (12) continuous hours during each test day.

(x) After the 12-hour zero drift test (step ix) is complete, sample test atmosphere $A_0$. A stable reading is not required.

(xi) Measure test atmosphere $A_{20}$ and record the stable reading (in ppm) as $P_1$. (See Figure B–4 in appendix A.)

(xii) Sample test atmosphere $A_{30}$; a stable reading is not required.

(xiii) Measure test atmosphere $A_{20}$ and record the stable reading as $P_2$.

(xiv) Sample test atmosphere $A_0$; a stable reading is not required.

(xv) Measure test atmosphere $A_{20}$ and record the stable reading as $P_3$.

(xvi) Sample test atmosphere $A_{30}$; a stable reading is not required.

(xvii) Measure test atmosphere $A_{20}$ and record the stable reading as $P_4$.

(xviii) Sample test atmosphere $A_0$; a stable reading is not required.

(xix) Measure test atmosphere $A_{20}$ and record the stable reading as $P_5$.

(xx) Sample test atmosphere $A_{30}$; a stable reading is not required.

(xxi) Measure test atmosphere $A_{20}$ and record the stable reading as $P_6$.

(xxii) Measure test atmosphere $A_{80}$ and record the stable reading as $P_7$.

(xxiii) Sample test atmosphere $A_{90}$; a stable reading is not required.

(xxiv) Measure test atmosphere $A_{80}$ and record the stable reading as $P_8$. Increase the chart speed to at least 10 centimeters per hour.

(xxv) Measure test atmosphere $A_0$. Record the stable reading as $L_1$.

(xxvi) Quickly switch the test analyzer to measure test atmosphere $A_{80}$ and mark the recorder chart to show, or otherwise record, the exact time when the switch occurred.

(xxvii) Measure test atmosphere $A_{80}$ and record the stable reading as $P_9$.

(xxviii) Sample test atmosphere $A_{90}$; a stable reading is not required.

(xxix) Measure test atmosphere $A_{80}$ and record the stable reading as $P_{10}$.

(xxx) Measure test atmosphere $A_0$ and record the stable reading as $L_2$.

(xxxi) Measure test atmosphere $A_{80}$ and record the stable reading as $P_{11}$.

(xxxii) Sample test atmosphere $A_{90}$; a stable reading is not required.

(xxxiii) Measure test atmosphere $A_{80}$ and record the stable reading as $P_{12}$.

(xxxiv) Repeat steps within paragraphs (e)(9)(viii) through (e)(9)(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 $A_0$ and $A_{80}$. Allow for a stable reading on each, and record the readings as $Z'_n$ and $S'_n$, respectively, where $n$ = the test day number (3, 6, 9, or 12). These readings must be made at least 4 hours prior to the start of the next test day.

(10) Determine the results of each day's tests as follows. Mark the recorder chart to show readings and determinations.

(i) *Zero drift.* (A) Determine the 12-hour zero drift by examining the strip chart pertaining to the 12-hour continuous zero air test. Determine the minimum ($C_{min.}$) and maximum ($C_{max.}$) measurement readings (in ppm) during this period of 12 consecutive hours, extrapolating the calibration curve to negative concentration units if necessary. Calculate the 12-hour zero drift (12*ZD*) as $12ZD = C_{max.} - C_{min.}$ (See Figure B–5 in appendix A.)

(B) Calculate the 24-hour zero drift (24*ZD*) 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 test 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 12*ZD* and 24*ZD* to the zero drift limit specifications in table B–1 to subpart B of part 53. Both 12*ZD* and 24*ZD* must be within the specified limits (inclusive) to pass the test for zero drift.

(ii) *Span drift.*

(A) Calculate the span drift (*SD*) as:

$$SD_n = \frac{S_n - S_{n-1}}{S_{n-1}} \times 100\%$$

or if a span adjustment was made on the previous test day,

$$SD_n = \frac{S_n - S'_{n-1}}{S'_{n-1}} \times 100\%$$

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 measurement reading on the $n$-th test day.

(B) *SD* must be within the span drift limits (inclusive) specified in table B–1 to subpart B of part 53 to pass the test for span drift.

(iii) *Lag time*. Determine, from the strip chart (or alternative test data record), the elapsed time in minutes between the change in test concentration (or mark) made in step (xxvi) and the first observable (two times the noise level) measurement response. This time must be equal to or less than the lag time limit specified in table B–1 to subpart B of part 53 to pass the test for lag time.

(iv) *Rise time*. Calculate 95 percent of measurement reading $P_9$ and determine, from the recorder chart (or alternative test data record), the elapsed time between the first observable (two times noise level) measurement 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 limit specified in table B–1 to subpart B of part 53 to pass the test for rise time.

(v) *Fall time*. Calculate five percent of ($P_{10}$ − $L_2$) and determine, from the strip chart (or alternative test record), the elapsed time in minutes between the first observable decrease in measurement response following reading $P_{10}$ and a response equal to $L_2$ + five percent of ($P_{10}$ − $L_2$). This time must be equal to or less than the fall time limit specification in table B–1 to subpart B of part 53 to pass the test for fall time.

(vi) *Precision*. Calculate precision (both $P_{20}$ and $P_{80}$) for each test day as follows:

(A)

$$P_{20} = \frac{1}{URL}\sqrt{\frac{1}{5}\left[\sum_{i=1}^{6} P_i^2 - \frac{1}{6}\left(\sum_{i=1}^{6} P_i\right)^2\right]} \times 100\%$$

(B)

$$P_{80} = \frac{1}{URL}\sqrt{\frac{1}{5}\left[\sum_{i=7}^{12} P_i^2 - \frac{1}{6}\left(\sum_{i=7}^{12} P_i\right)^2\right]} \times 100\%$$

(C) Both $P_{20}$ and $P_{80}$ must be equal to or less than the precision limits specified in table B–1 to subpart B of part 53 to pass the test for precision.

[76 FR 54326, Aug. 31, 2011, as amended at 80 FR 65460, Oct. 26, 2015]

FIGURE B–1 TO SUBPART B OF PART 53—EXAMPLE

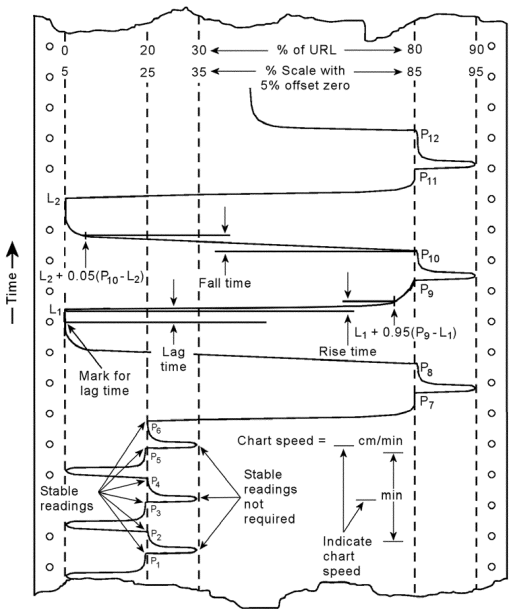



Figure B-1 to Subpart B of Part 53--Example showing the nature of the tracing obtained during the test sequence for 24-hour drift, lag time, rise time, fall time, and precision. The time scale has been greatly compressed.

TABLE B–1 TO SUBPART B OF PART 53—PERFORMANCE LIMIT SPECIFICATIONS FOR AUTOMATED METHODS

| Performance parameter | Units [1] | $SO_2$ | | $O_3$ | | CO | | $NO_2$ (Std. range) | Definitions and test procedures |
|---|---|---|---|---|---|---|---|---|---|
| | | Std. range [3] | Lower range [2 3] | Std. range [3] | Lower range [2 3] | Std. range [3] | Lower range [2 3] | | |
| 1. Range | ppm | 0–0.5 | <0.5 | 0–0.5 | <0.5 | 0–50 | <50 | 0–0.5 | Sec. 53.23(a) |
| 2. Noise | ppm | 0.001 | 0.0005 | 0.0025 | 0.001 | 0.2 | 0.1 | 0.005 | Sec. 53.23(b) |
| 3. Lower detectable limit. | ppm | 0.002 | 0.001 | 0.005 | 0.002 | 0.4 | 0.2 | 0.010 | Sec. 53.23(c) |
| 4. Interference equivalent | | | | | | | | | |
| Each interferent | ppm | ±0.005 | [4] ±0.005 | ±0.005 | ±0.005 | ±1.0 | ±0.5 | ±0.02 | Sec. 53.23(d) |
| Total, all interferents. | ppm | – | – | – | – | – | – | 0.04 | Sec. 53.23(d) |
| 5. Zero drift, 12 and 24 hour. | ppm | ±0.004 | ±0.002 | ±0.004 | ±0.002 | ±0.5 | ±0.3 | ±0.02 | Sec. 53.23(e) |
| 6. Span drift, 24 hour | | | | | | | | | |
| 20% of upper range limit. | Percent | – | – | – | – | – | – | ±20.0 | Sec. 53.23(e) |
| 80% of upper range limit. | Percent | ±3.0 | ±3.0 | ±3.0 | ±3.0 | ±2.0 | ±2.0 | ±5.0 | Sec. 53.23(e) |
| 7. Lag time | Minutes | 2 | 2 | 2 | 2 | 2.0 | 2.0 | 20 | Sec. 53.23(e) |
| 8. Rise time | Minutes | 2 | 2 | 2 | 2 | 2.0 | 2.0 | 15 | Sec. 53.23(e) |
| 9. Fall time | Minutes | 2 | 2 | 2 | 2 | 2.0 | 2.0 | 15 | Sec. 53.23(e) |
| 10. Precision | | | | | | | | | |

| Performance parameter | Units [1] | $SO_2$ | | $O_3$ | | CO | | $NO_2$ (Std. range) | Definitions and test procedures |
|---|---|---|---|---|---|---|---|---|---|
| | | Std. range [3] | Lower range [2 3] | Std. range [3] | Lower range [2 3] | Std. range [3] | Lower range [2 3] | | |
| 20% of upper range limit. | | – | – | – | – | – | – | | Sec. 53.23(e) |
| | Percent [5] | 2 | 2 | 2 | 2 | 1.0 | 1.0 | 4 | Sec. 53.23(e) |
| 80% of upper range limit. | | – | – | – | – | – | – | | Sec. 53.23(e) |
| | Percent [5] | 2 | 2 | 2 | 2 | 1.0 | 1.0 | 6 | Sec. 53.23(e) Sec. 53.23(e) |

[1] To convert from parts per million (ppm) to $\mu g/m^3$ at 25 °C and 760 mm Hg, multiply by M/0.02447, where M is the molecular weight of the gas. Percent means percent of the upper measurement range limit.

[2] Tests for interference equivalent and lag time do not need to be repeated for any lower range provided the test for the standard range shows that the lower range specification (if applicable) is met for each of these test parameters.

[3] For candidate analyzers having automatic or adaptive time constants or smoothing filters, describe their functional nature, and describe and conduct suitable tests to demonstrate their function aspects and verify that performances for calibration, noise, lag, rise, fall times, and precision are within specifications under all applicable conditions. For candidate analyzers with operator-selectable time constants or smoothing filters, conduct calibration, noise, lag, rise, fall times, and precision tests at the highest and lowest settings that are to be included in the FRM or FEM designation.

[4] For nitric oxide interference for the $SO_2$ UVF method, interference equivalent is ±0.0003 ppm for the lower range.

[5] Standard deviation expressed as percent of the URL.

[80 FR 65460, Oct. 26, 2015]

## Table B–2 to Subpart B of Part 53—Test Atmospheres

| Test gas | Generation | Verification |
|---|---|---|
| Ammonia | Permeation device. Similar to system described in references 1 and 2. | Indophenol method, reference 3. |
| Carbon dioxide | Cylinder of zero air or nitrogen containing $CO_2$ as required to obtain the concentration specified in table B–3. | Use NIST-certified standards whenever possible. If NIST standards are not available, obtain 2 standards from independent sources which agree within 2 percent, or obtain one standard and submit it to an independent laboratory for analysis, which must agree within 2 percent of the supplier's nominal analysis. |
| Carbon monoxide | Cylinder of zero air or nitrogen containing CO as required to obtain the concentration specified in table B–3. | Use an FRM CO analyzer as described in reference 8. |
| Ethane | Cylinder of zero air or nitrogen containing ethane as required to obtain the concentration specified in table B–3. | Gas chromatography, ASTM D2820, reference 10. Use NIST-traceable gaseous methane or propane standards for calibration. |
| Ethylene | Cylinder of pre-purified nitrogen containing ethylene as required to obtain the concentration specified in table B–3. | Do. |
| Hydrogen chloride | Cylinder [1] of pre-purified nitrogen containing approximately 100 ppm of gaseous HCl. Dilute with zero air to concentration specified in table B–3. | Collect samples in bubbler containing distilled water and analyze by the mercuric thiocyanate method, ASTM (D612), p. 29, reference 4. |
| Hydrogen sulfide | Permeation device system described in references 1 and 2. | Tentative method of analysis for $H_2S$ content of the atmosphere, p. 426, reference 5. |
| Methane | Cylinder of zero air containing methane as required to obtain the concentration specified in table B–3. | Gas chromatography ASTM D2820, reference 10. Use NIST-traceable methane standards for calibration. |
| Nitric oxide | Cylinder [1] of pre-purified nitrogen containing approximately 100 ppm 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 ...<br>2. Permeation device, similar to system described in reference 6. | 1. Use an FRM $NO_2$ analyzer calibrated with a gravimetrically calibrated permeation device.<br>2. Use an FRM $NO_2$ analyzer calibrated by gas-phase titration as described in reference 6. |
| Ozone | Calibrated ozone generator as described in reference 9. | Use an FEM ozone analyzer calibrated as described in reference 9. |
| Sulfur dioxide | 1. Permeation device as described in references 1 and 2.<br>2. Dynamic dilution of a cylinder containing approximately 100 ppm $SO_2$ as described in Reference 7. | Use an $SO_2$ FRM or FEM analyzer as described in reference 7. |
| Water | Pass zero air through distilled water at a fixed known temperature between 20° and 30 °C such that the air stream becomes saturated. Dilute with zero air to concentration specified in table B–3. | Measure relative humidity by means of a dew-point indicator, calibrated electrolytic or piezo electric hygrometer, or wet/dry bulb thermometer. |

| Test gas | Generation | Verification |
|---|---|---|
| Xylene ..... | Cylinder of pre-purified nitrogen containing 100 ppm xylene. Dilute with zero air to concentration specified in table B–3. | Use NIST-certified standards whenever possible. If NIST standards are not available, obtain 2 standards from independent sources which agree within 2 percent, or obtain one standard and submit it to an independent laboratory for analysis, which must agree within 2 percent of the supplier's nominal analysis. |
| Zero air ..... | 1. Ambient air purified by appropriate scrubbers or other devices such that it is free of contaminants likely to cause a detectable response on the analyzer.<br>2. Cylinder of compressed zero air certified by the supplier or an independent laboratory to be free of contaminants likely to cause a detectable response on the analyzer. | |

[1] Use stainless steel pressure regulator dedicated to the pollutant measured.

Reference 1. O'Keefe, A. E., and Ortaman, G. C. ''Primary Standards for Trace Gas Analysis,'' *Anal. Chem.* 38, 760 (1966).

Reference 2. Scaringelli, F. P., A. E. . Rosenberg, E*, and Bell, J. P., ''Primary Standards for Trace Gas Analysis.'' *Anal. Chem.* 42, 871 (1970).

Reference 3. ''Tentative Method of Analysis for Ammonia in the Atmosphere (Indophenol Method)'', *Health Lab Sciences,* vol. 10, No. 2, 115–118, April 1973.

Reference 4. 1973 Annual Book of ASTM Standards, American Society for Testing and Materials, 1916 Race St., Philadelphia, PA.

Reference 5. *Methods for Air Sampling and Analysis,* Intersociety Committee, 1972, American Public Health Association, 1015.

Reference 6. 40 CFR 50 Appendix F, ''Measurement Principle and Calibration Principle for the Measurement of Nitrogen Dioxide in the Atmosphere (Gas Phase Chemiluminescence).''

Reference 7. 40 CFR 50 Appendix A–1, ''Measurement Principle and Calibration Procedure for the Measurement of Sulfur Dioxide in the Atmosphere (Ultraviolet Fluorscence).''

Reference 8. 40 CFR 50 Appendix C, ''Measurement Principle and Calibration Procedure for the Measurement of Carbon Monoxide in the Atmosphere (Non-Dispersive Infrared Photometry)''.

Reference 9. 40 CFR 50 Appendix D, ''Measurement Principle and Calibration Procedure for the Measurement of Ozone in the Atmosphere''.

Reference 10. ''Standard Test Method for C, through C5 Hydrocarbons in the Atmosphere by Gas Chromatography'', D 2820, 1987 Annual Book of Aston Standards, vol 11.03, American Society for Testing and Materials, 1916 Race St., Philadelphia, PA 19103.

TABLE B–3 TO SUBPART B OF PART 53—INTERFERENT TEST CONCENTRATION,[1] PARTS PER MILLION

| Pollutant | Analyzer type | Hydrochloric acid | Ammonia | Hydrogen sulfide | Sulfur dioxide | Nitrogen dioxide | Nitric oxide | Carbon dioxide | Ethylene | Ozone | Mxylene | Water vapor | Carbon monoxide | Methane | Ethane | Naphthalene |
|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|
| $SO_2$ | Ultraviolet fluorescence | ..... | ..... | [5]0.1 | [4]0.14 | 0.5 | 0.5 | ..... | ..... | 0.5 | 0.2 | 20,000 | ..... | ..... | ..... | [6]0.05 |
| $SO_2$ | Flame photometric | ..... | ..... | 0.01 | [4]0.14 | ..... | ..... | 750 | ..... | ..... | ..... | [3]20,000 | 50 | ..... | ..... | |
| $SO_2$ | Gas chromatography | ..... | ..... | 0.1 | [4]0.14 | ..... | ..... | 750 | ..... | ..... | ..... | [3]20,000 | 50 | ..... | ..... | |
| $SO_2$ | Spectrophotometric-wet chemical (pararosanaline). | 0.2 | 0.1 | 0.1 | [4]0.14 | 0.5 | ..... | 750 | ..... | 0.5 | ..... | ..... | ..... | ..... | ..... | |
| $SO_2$ | Electrochemical | 0.2 | 0.1 | 0.1 | [4]0.14 | 0.5 | 0.5 | ..... | 0.2 | 0.5 | ..... | [3]20,000 | ..... | ..... | ..... | |
| $SO_2$ | Conductivity | 0.2 | 0.1 | ..... | [4]0.14 | 0.5 | ..... | 750 | ..... | ..... | ..... | ..... | ..... | ..... | ..... | |
| $SO_2$ | Spectrophotometric-gas phase, including DOAS | ..... | ..... | ..... | [4]0.14 | 0.5 | ..... | ..... | ..... | 0.5 | 0.2 | ..... | ..... | ..... | ..... | |
| $O_3$ | Chemiluminescent | ..... | ..... | [3]0.1 | ..... | ..... | ..... | 750 | ..... | [4]0.08 | ..... | [3]20,000 | ..... | ..... | ..... | |
| $O_3$ | Electrochemical | ..... | [3]0.1 | ..... | 0.5 | 0.5 | ..... | ..... | ..... | [4]0.08 | ..... | ..... | ..... | ..... | ..... | |
| $O_3$ | Spectrophotometric-wet chemical (potassium iodide). | ..... | [3]0.1 | ..... | 0.5 | 0.5 | [3]0.5 | ..... | ..... | [4]0.08 | ..... | ..... | ..... | ..... | ..... | |
| $O_3$ | Spectrophotometric-gas phase, including ultraviolet absorption and DOAS). | ..... | ..... | ..... | 0.5 | 0.5 | 0.5 | ..... | ..... | [4]0.08 | 0.02 | 20,000 | ..... | ..... | ..... | |
| CO | Non-dispersive Infrared | ..... | ..... | ..... | ..... | ..... | ..... | 750 | ..... | ..... | ..... | 20,000 | [4]10 | ..... | ..... | |
| CO | Gas chromatography with flame ionization detector. | ..... | ..... | ..... | ..... | ..... | ..... | ..... | ..... | ..... | ..... | 20,000 | [4]10 | ..... | 0.5 | |
| CO | Electrochemical | ..... | ..... | ..... | ..... | ..... | 0.5 | ..... | 0.2 | ..... | ..... | 20,000 | [4]10 | ..... | ..... | |
| CO | Catalytic combustion-thermal detection | ..... | 0.1 | ..... | ..... | ..... | ..... | 750 | 0.2 | ..... | ..... | 20,000 | [4]10 | 5.0 | 0.5 | |
| CO | IR fluorescence | ..... | ..... | ..... | ..... | ..... | ..... | 750 | ..... | ..... | ..... | 20,000 | [4]10 | ..... | 0.5 | |
| CO | Mercury replacement-UV photometric | ..... | ..... | ..... | ..... | ..... | ..... | ..... | 0.2 | ..... | ..... | ..... | [4]10 | ..... | 0.5 | |
| $NO_2$ | Chemiluminescent | ..... | [3]0.1 | ..... | 0.5 | [4]0.1 | 0.5 | ..... | ..... | ..... | ..... | 20,000 | ..... | ..... | ..... | |
| $NO_2$ | Spectrophotometric-wet chemical (azo-dye reaction). | ..... | ..... | ..... | 0.5 | [4]0.1 | 0.5 | 750 | ..... | 0.5 | ..... | ..... | ..... | ..... | ..... | |
| $NO_2$ | Electrochemical | 0.2 | [3]0.1 | ..... | 0.5 | [4]0.1 | 0.5 | 750 | ..... | 0.5 | ..... | 20,000 | 50 | ..... | ..... | |
| $NO_2$ | Spectrophotometric-gas phase | ..... | [3]0.1 | ..... | 0.5 | [4]0.1 | 0.5 | ..... | ..... | 0.5 | ..... | 20,000 | 50 | ..... | ..... | |

[1] Concentrations of interferent listed must be prepared and controlled to ±10 percent of the stated value.
[2] Analyzer types not listed will be considered by the Administrator as special cases.
[3] Do not mix with the pollutant.
[4] Concentration of pollutant used for test. These pollutant concentrations must be prepared to ±10 percent of the stated value.
[5] If candidate method utilizes an elevated-temperature scrubber for removal of aromatic hydrocarbons, perform this interference test.
[6] If naphthalene test concentration cannot be accurately quantified, remove the scrubber, use a test concentration that causes a full scale response, reattach the scrubber, and evaluate response for interference.

TABLE B–4 TO SUBPART B OF PART 53—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. |
| 13 | 125 | 20 | |
| 14 | 105 | 20 | |
| 15 | 125 | 30 | |

[1] Voltage specified shall be controlled to ±1 volt.
[2] Temperatures shall be controlled to ±1 °C.

TABLE B–5 TO SUBPART B OF PART 53—SYMBOLS AND ABBREVIATIONS

$B_L$—Analyzer reading at the specified *LDL* test concentration for the *LDL* test.
$B_Z$—analyzer reading at 0 concentration for the *LDL* test.
*DM*—Digital meter.
$C_{max}$—Maximum analyzer reading during the 12*ZD* test period.
$C_{min}$—Minimum analyzer reading during the 12*ZD* test period.
*i*—Subscript indicating the *i*-th quantity in a series.
*IE*—Interference equivalent.
$L_1$—First analyzer zero reading for the 24*ZD* test.
$L_2$—Second analyzer zero reading for the 24*ZD* test.
*n*—Subscript indicating the test day number.
*P*—Analyzer reading for the span drift and precision tests.
$P_i$—The *i*-th analyzer reading for the span drift and precision tests.
$P_{20}$—Precision at 20 percent of URL.
$P_{80}$—Precision at 80 percent of URL.
ppb—Parts per billion of pollutant gas (usually in air), by volume.
ppm—Parts per million of pollutant gas (usually in air), by volume.
*R*—Analyzer reading of pollutant alone for the *IE* test.
$R_1$—Analyzer reading with interferent added for the *IE* test.
$r_i$—the *i*-th analyzer or DM reading for the noise test.
*S*—Standard deviation of the noise test readings.
$S_0$—Noise value (*S*) measured at 0 concentration.
$S_{80}$—Noise value (*S*) measured at 80 percent of the URL.
$S_n$—Average of $P_7 \ldots P_{12}$ for the n-th test day of the SD test.
$S'_n$—Adjusted span reading on the *n*-th test day.
SD—Span drift
*URL*—Upper range limit of the analyzer's measurement range.
*Z*—Average of $L_1$ and $L_2$ readings for the 24ZD test.
$Z_n$—Average of $L_1$ and $L_2$ readings on the *n*-th test day for the 24ZD test.
$Z'_n$—Adjusted analyzer zero reading on the *n*-the test day for the 24ZD test.
*ZD*—Zero drift.
12*ZD*—12-hour zero drift.
24*ZD*—24-hour zero drift.

APPENDIX A TO SUBPART B OF PART 53—OPTIONAL FORMS FOR REPORTING TEST RESULTS

**NOISE TEST DATA**

Applicant _____ Date _____

Analyzer _____ Pollutant _____

Range _____ Test No. _____

| 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 | | | | | |
| STD. DEVIATION | | $S_0$ = | | $S_{80}$ = | |

Figure B-2. Form for noise test data (see §53.23(b)).

**LDL and INTERFERENCE TEST DATA**

Applicant _____ Date _____

Analyzer _____ Pollutant _____

| TEST PARAMETER | | READING or CALCULATION | TEST NUMBER 1 | 2 | 3 | 4 | 5 | 6 | 7 | 8 | 9 | 10 | 11 | 12 | 13 | 14 | 15 |
|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|
| LOWER DETECTABLE LIMIT | | $B_Z$ | | | | | | | | | | | | | | | |
| | | $B_L$ | | | | | | | | | | | | | | | |
| | | $LDL = B_L$ □ $B_Z$ | | | | | | | | | | | | | | | |
| INTER-FERENCE EQUIV-ALENT | 1 | $R_1$ | | | | | | | | | | | | | | | |
| | | $R_{I1}$ | | | | | | | | | | | | | | | |
| | | $IE = R_{I1} - R_1$ | | | | | | | | | | | | | | | |
| | 2 | $R_2$ | | | | | | | | | | | | | | | |
| | | $R_{I2}$ | | | | | | | | | | | | | | | |
| | | $IE = R_{I2} - R_2$ | | | | | | | | | | | | | | | |
| | 3* | $R_3$ | | | | | | | | | | | | | | | |
| | | $R_{I3}$ | | | | | | | | | | | | | | | |
| | | $IE = R_{I3} - R_3$ | | | | | | | | | | | | | | | |
| | 4* | $R_4$ | | | | | | | | | | | | | | | |
| | | $R_{I4}$ | | | | | | | | | | | | | | | |
| | | $IE = R_{I4} - R_4$ | | | | | | | | | | | | | | | |
| | 5* | $R_5$ | | | | | | | | | | | | | | | |
| | | $R_{I5}$ | | | | | | | | | | | | | | | |
| | | $IE = R_{I5} - R_5$ | | | | | | | | | | | | | | | |
| | TOTAL* | $\sum_{i=1}^{n} \lvert IE_i \rvert$ | | | | | | | | | | | | | | | |

*If required.

Figure B-3. Form for test data and calculations for lower detectable limit (LDL) and interference equivalent (IE) (see § 53.23(c) and (d)).

**DRIFT AND PRECISION TEST DATA**

Applicant _____ Date _____

Analyzer _____ Pollutant _____

| | ANALYZER READING, ppm | | | | | | | | | | | | | | | |
|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|
| TEST DAY | 0 | 1 | 2 | 3 | 4 | 5 | 6 | 7 | 8 | 9 | 10 | 11 | 12 | 13 | 14 | 15 |
| DATE | | | | | | | | | | | | | | | | |
| $P_1$ | | | | | | | | | | | | | | | | |
| $P_2$ | | | | | | | | | | | | | | | | |
| $P_3$ | | | | | | | | | | | | | | | | |
| $P_4$ | | | | | | | | | | | | | | | | |
| $P_5$ | | | | | | | | | | | | | | | | |
| $P_6$ | | | | | | | | | | | | | | | | |
| $P_7$ | | | | | | | | | | | | | | | | |
| $P_8$ | | | | | | | | | | | | | | | | |
| $P_9$ | | | | | | | | | | | | | | | | |
| $P_{10}$ | | | | | | | | | | | | | | | | |
| $P_{11}$ | | | | | | | | | | | | | | | | |
| $P_{12}$ | | | | | | | | | | | | | | | | |
| $S_n = \frac{1}{6}\sum_{i=7}^{12} P_i$ | | | | | | | | | | | | | | | | |
| $L_1$ | | | | | | | | | | | | | | | | |
| $L_2$ | | | | | | | | | | | | | | | | |
| $Z'_n$ | | | | | | | | | | | | | | | | |
| $S'_n$ | | | | | | | | | | | | | | | | |
| $C_{max}$ | | | | | | | | | | | | | | | | |
| $C_{min}$ | | | | | | | | | | | | | | | | |

Figure B-4. Form for drift and precision test data (see § 53.23(e)).

**CALCULATION OF ZERO DRIFT, SPAN DRIFT, AND PRECISION**

Applicant _____ Date _____

Analyzer _____ Pollutant _____

| TEST PARAMETER | | CALCULATION | TEST DAY (n) | | | | | | | | | | | | | | |
|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|
| | | | 1 | 2 | 3 | 4 | 5 | 6 | 7 | 8 | 9 | 10 | 11 | 12 | 13 | 14 | 15 |
| ZERO DRIFT | 12 HOUR | $12ZD = C_{max} - C_{min}$ | | | | | | | | | | | | | | | |
| | 24 HOUR | $Z = (L_1 + L_2)/2$ | | | | | | | | | | | | | | | |
| | | $24ZD = Z_n - Z_{n-1}$ | | | | | | | | | | | | | | | |
| | | $24ZD = Z'_n - Z'_{n-1}$ | | | | | | | | | | | | | | | |
| SPAN DRIFT | 24 HOUR | $S_n = \frac{1}{6}\sum_{i=7}^{12} P_i$ | | | | | | | | | | | | | | | |
| | | $SD_n = \frac{S_n - S_{n-1}}{S_{n-1}} \times 100\%$ | | | | | | | | | | | | | | | |
| | | $SD_n = \frac{S_n - S'_{n-1}}{S'_{n-1}} \times 100\%$ | | | | | | | | | | | | | | | |
| PREC-ISION | 20% URL ($P_{20}$) | $P_{20}$ = STANDARD DEVIATION of ($P_1 \ldots P_6$) | | | | | | | | | | | | | | | |
| | 80% URL ($P_{80}$) | $P_{80}$ = STANDARD DEVIATION of ($P_7 \ldots P_{12}$) | | | | | | | | | | | | | | | |

Figure B-5. Form for calculating zero drift, span drift, and precision (§ 53.23(e)).

**TEST DATA SUMMARY**

Applicant _____ Analyst _____

Analyzer _____ Pollutant _____

Range _____ Other information _____
Test dates _____

| Performance Parameter | | Table B-1 Spec. | Test Number (first set) | | | | | | | Test Number (second set) | | | | | | | | Number of Failures | Pass or Fail |
|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|
| | | | 1 | 2 | 3 | 4 | 5 | 6 | 7 | 8 | 9 | 10 | 11 | 12 | 13 | 14 | 15 | | |
| **Noise**, ppm | 0% URL | | | | | | | | | | | | | | | | | | |
| | 80% URL | | | | | | | | | | | | | | | | | | |
| **LDL** (> 2 x 0% noise) | | | | | | | | | | | | | | | | | | | |
| **Interference Equivalent**, ppm | IE1 | | | | | | | | | | | | | | | | | | |
| | IE2 | | | | | | | | | | | | | | | | | | |
| | IE3 | | | | | | | | | | | | | | | | | | |
| | IE4 | | | | | | | | | | | | | | | | | | |
| | IE5 | | | | | | | | | | | | | | | | | | |
| | IE6 | | | | | | | | | | | | | | | | | | |
| | Total | | | | | | | | | | | | | | | | | | |
| **Zero Drift**, ppm | 12 hr | | | | | | | | | | | | | | | | | | |
| | 24 hr. | | | | | | | | | | | | | | | | | | |
| **Span Drift**, % | 80% URL | | | | | | | | | | | | | | | | | | |
| **Lag Time**, min | | | | | | | | | | | | | | | | | | | |
| **Rise Time**, min | | | | | | | | | | | | | | | | | | | |
| **Fall Time**, min | | | | | | | | | | | | | | | | | | | |
| **Precision**, percent | 20% URL | | | | | | | | | | | | | | | | | | |
| | 80% URL | | | | | | | | | | | | | | | | | | |

Figure B-6. Form for reporting a summary of the test results (see § 53.23).

## 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 ambient air. Minor deviations in testing requirements and acceptance requirements set forth in this subpart, in connection with any documented extenuating circumstances, may be determined by the Administrator to be acceptable, at the discretion of the Administrator.

(b) *Selection of test sites*. (1) Each test site shall be in an area which can be shown to have at least moderate concentrations of various pollutants. Each site shall be clearly identified and shall be justified as an appropriate test site with suitable supporting evidence such as a description of the surrounding

area, characterization of the sources and pollutants typical in the area, maps, population density data, vehicular traffic data, emission inventories, pollutant measurements from previous years, concurrent pollutant measurements, meteorological data, and other information useful in supporting the suitability of the site for the comparison test or tests.

(2) If approval of one or more proposed test sites is desired prior to conducting the tests, a written request for approval of the test site or sites must be submitted to the address given in § 53.4. The request should include information identifying the type of candidate method and one or more specific proposed test sites along with a justification for each proposed specific site as described in paragraph (b)(1) of this section. The EPA will evaluate each proposed site and approve the site, disapprove the site, or request more information about the site. Any such pre-test approval of a test site by the EPA shall indicate only that the site meets the applicable test site requirements for the candidate method type; it shall not indicate, suggest, or imply that test data obtained at the site will necessarily meet any of the applicable data acceptance requirements. The Administrator may exercise discretion in selecting a different site (or sites) for any additional tests the Administrator decides to conduct.

(c) *Test atmosphere.* Ambient air sampled at an appropriate test site or sites shall be used for these tests. Simultaneous concentration measurements shall be made in each of the concentration ranges specified in tables C–1, C–3, or C–4 of this subpart, as appropriate.

(d) *Sampling or sample collection.* All test concentration measurements or samples shall be taken in such a way that both the candidate method and the reference method obtain air samples that are alike or as nearly identical as practical.

(e) *Operation.* Set-up and start-up of the test analyzer(s), test sampler(s), and reference method analyzers or samplers shall be in strict accordance with the applicable operation manual(s).

(f) *Calibration.* The reference method shall be calibrated according to the appropriate appendix to part 50 of this chapter (if it is a manual method) or according to the applicable operation manual(s) (if it is an automated method). A candidate method (or portion thereof) shall be calibrated according to the applicable operation manual(s), if such calibration is a part of the method.

(g) *Submission of test data and other information.* All recorder charts, calibration data, records, test results, procedural descriptions and details, and other documentation obtained from (or pertinent to) these tests shall be identified, dated, signed by the analyst performing the test, and submitted. For candidate methods for $PM_{2.5}$ and $PM_{10-2.5}$, all submitted information must meet the requirements of the ANSI/ASQC E4 Standard, sections 6 (reference 1 of appendix A of this subpart).

### § 53.31 [Reserved]

### § 53.32 Test procedures for methods for $SO_2$, CO, $O_3$, and $NO_2$.

(a) *Comparability.* Comparability is shown for $SO_2$, CO, $O_3$, and $NO_2$ methods when the differences between:

(1) Measurements made by a candidate manual method or by a test analyzer representative of a candidate automated method, and;

(2) Measurements made simultaneously by a reference method are less than or equal to the values for maximum discrepancy specified in table C–1 of this subpart.

(b) *Test measurements.* All test measurements are to be made at the same test site. If necessary, the concentration of pollutant in the sampled ambient air may be augmented with artificially generated pollutant to facilitate measurements in the specified ranges, as described under paragraph (f)(4) of this section.

(c) *Requirements for measurements or samples.* All test measurements made or test samples collected by means of a sample manifold as specified in paragraph (f)(4) of this section shall be at a room temperature between 20° and 30 °C, and at a line voltage between 105 and 125 volts. All methods shall be calibrated as specified in § 53.30(f) prior to initiation of the tests.

(d) *Set-up and start-up.* (1) Set-up and start-up of the test analyzer, test sampler(s), and reference method shall be in strict accordance with the applicable operation manual(s). If the test analyzer does not have an integral strip chart or digital data recorder, connect the analyzer output to a suitable strip chart or digital data recorder. This recorder shall have a chart width of at least 25 centimeters, a response time of 1 second or less, a deadband of not more than 0.25 percent of full scale, and capability of either reading measurements at least 5 percent below zero or offsetting the zero by at least 5 percent. Digital data shall be recorded at appropriate time intervals such that trend plots similar to a strip chart recording may be constructed with a similar or suitable level of detail.

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

(3) Allow adequate warmup or stabilization time as indicated in the applicable operation manual(s) before beginning the tests.

(e) *Range.* (1) Except as provided in paragraph (e)(2) of this section, each method shall be operated in the range specified for the reference method in the appropriate appendix to part 50 of this chapter (for manual reference methods), or specified in table B–1 of subpart B of this part (for automated reference methods).

(2) For a candidate method having more than one selectable range, one range must be that specified in table B–1 of subpart B of this part, and a test analyzer representative of the method must pass the tests required by this subpart while operated on that range. The tests may be repeated for one or more broader ranges (*i.e.*, ones extending to higher concentrations) than the one specified in table B–1 of subpart B of this part, provided that such a range does not extend to concentrations more than four times the upper range limit specified in table B–1 of subpart B of this part and that the test analyzer has passed the tests required by subpart B of this part (if applicable) for the broader range. If the tests required by this subpart are conducted or passed only for the range specified in table B–1 of subpart B of this part, any 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. Appropriate test data shall be submitted for each range sought to be included in such a determination.

(f) *Operation of automated methods.* (1) Once the test analyzer has been set up and calibrated and tests started, manual adjustment or normal periodic maintenance, as specified in the manual referred to in §53.4(b)(3), 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 manual adjustments were made and describe the operations performed.

(2) All test measurements shall be made with the same test analyzer; use of multiple test analyzers is not permitted. The test analyzer shall be operated continuously during the entire series of test measurements.

(3) If a test analyzer should malfunction during any of these tests, the entire set of measurements shall be repeated, and a detailed explanation of the malfunction, remedial action taken, and whether recalibration was necessary (along with all pertinent records and charts) shall be submitted.

(4) Ambient air shall be sampled from a common intake and distribution manifold designed to deliver homogenous air samples to both methods. Precautions shall be taken in the design and construction of this manifold to minimize the removal of particulate matter and trace gases, and to insure that identical samples reach the two methods. If necessary, the concentration of pollutant in the sampled ambient air may be augmented with artificially generated pollutant. However, at all times the air sample measured by the candidate and reference methods under test shall consist of not less than 80 percent ambient air by volume.

Schematic drawings, physical illustrations, descriptions, and complete details of the manifold system and the augmentation system (if used) shall be submitted.

(g) *Tests*. (1) Conduct the first set of simultaneous measurements with the candidate and reference methods:

(i) Table C–1 of this subpart specifies the type (1-or 24-hour) and number of measurements to be made in each of the three test concentration ranges.

(ii) The pollutant concentration must fall within the specified range as measured by the reference method.

(iii) The measurements shall be made in the sequence specified in table C–2 of this subpart.

(2) For each pair of measurements, determine the difference (discrepancy) between the candidate method measurement and reference method measurement. A discrepancy which exceeds the discrepancy specified in table C–1 of this subpart constitutes a failure. Figure C–1 of this subpart contains a suggested format for reporting the test results.

(3) The results of the first set of measurements shall be interpreted as follows:

(i) Zero failures: The candidate method passes the test for comparability.

(ii) Three or more failures: The candidate method fails the test for comparability.

(iii) One or two failures: Conduct a second set of simultaneous measurements as specified in table C–1 of this subpart. The results of the combined total of first-set and second-set measurements shall be interpreted as follows:

(A) One or two failures: The candidate method passes the test for comparability.

(B) Three or more failures: The candidate method fails the test for comparability.

(iv) For $SO_2$, the 1-hour and 24-hour measurements shall be interpreted separately, and the candidate method must pass the tests for both 1- and 24-hour measurements to pass the test for comparability.

(4) A 1-hour measurement consists of the integral of the instantaneous concentration over a 60-minute continuous period divided by the time period. Integration of the instantaneous concentration may be performed by any appropriate means such as chemical, electronic, mechanical, visual judgment, or by calculating the mean of not less than 12 equally-spaced instantaneous readings. Appropriate allowances or corrections shall be made in cases where significant errors could occur due to characteristic lag time or rise/fall time differences between the candidate and reference methods. Details of the means of integration and any corrections shall be submitted.

(5) A 24-hour measurement consists of the integral of the instantaneous concentration over a 24-hour continuous period divided by the time period. This integration may be performed by any appropriate means such as chemical, electronic, mechanical, or by calculating the mean of twenty-four (24) sequential 1-hour measurements.

(6) For $O_3$ and CO, no more than six 1-hour measurements shall be made per day. For $SO_2$, no more than four 1-hour measurements or one 24-hour measurement shall be made per day. One-hour measurements may be made concurrently with 24-hour measurements if appropriate.

(7) For applicable methods, control or calibration checks may be performed once per day without adjusting the test analyzer or method. These checks may be used as a basis for a linear interpolation-type correction to be applied to the measurements to correct for drift. If such a correction is used, it shall be applied to all measurements made with the method, and the correction procedure shall become a part of the method.

[62 FR 38784, July 18, 1997, as amended at 75 FR 35601, June 22, 2010; 80 FR 65466, Oct. 26, 2015]

### § 53.33 Test Procedure for Methods for Lead (Pb).

(a) *General*. The reference method for Pb in TSP includes two parts, the reference method for high-volume sampling of TSP as specified in 40 CFR 50, appendix B and the analysis method for Pb in TSP as specified in 40 CFR 50, appendix G. Correspondingly, the reference method for Pb in $PM_{10}$ includes the reference method for low-volume

sampling of $PM_{10}$ in 40 CFR 50, appendix O and the analysis method of Pb in $PM_{10}$ as specified in 40 CFR 50, appendix Q. This section explains the procedures for demonstrating the equivalence of either a candidate method for Pb in TSP to the high-volume reference methods, or a candidate method for Pb in $PM_{10}$ to the low-volume reference methods.

(1) Pb in TSP—A candidate method for Pb in TSP specifies reporting of Pb concentrations in terms of standard temperature and pressure. Comparisons of candidate methods to the reference method in 40 CFR 50, appendix G must be made in a consistent manner with regard to temperature and pressure.

(2) Pb in $PM_{10}$—A candidate method for Pb in $PM_{10}$ must specify reporting of Pb concentrations in terms of local conditions of temperature and pressure, which will be compared to similarly reported concentrations from the reference method in 40 CFR 50 appendix Q.

(b) *Comparability*. Comparability is shown for Pb methods when the differences between:

(1) Measurements made by a candidate method, and

(2) Measurements made by the reference method on simultaneously collected Pb samples (or the same sample, if applicable), are less than or equal to the values specified in table C–3 of this subpart.

(c) *Test measurements*. Test measurements may be made at any number of test sites. Augmentation of pollutant concentrations is not permitted, hence an appropriate test site or sites must be selected to provide Pb concentrations in the specified range.

(d) *Collocated samplers*. The ambient air intake points of all the candidate and reference method collocated samplers shall be positioned at the same height above the ground level, and between 2 meters (1 meter for samplers with flow rates less than 200 liters per minute (L/min)) and 4 meters apart. The samplers shall be oriented in a manner that will minimize spatial and wind directional effects on sample collection.

(e) *Sample collection*. Collect simultaneous 24-hour samples of Pb at the test site or sites with both the reference and candidate methods until at least 10 sample pairs have been obtained.

(1) A candidate method for Pb in TSP which employs a sampler and sample collection procedure that are identical to the sampler and sample collection procedure specified in the reference method in 40 CFR part 50, Appendix B, but uses a different analytical procedure than specified in 40 CFR Appendix G, may be tested by analyzing pairs of filter strips taken from a single TSP reference sampler operated according to the procedures specified by that reference method.

(2) A candidate method for Pb in $PM_{10}$ which employs a sampler and sample collection procedure that are identical to the sampler and sample collection procedure specified in the reference method in 40 CFR part 50, Appendix O, but uses a different analytical procedure than specified in 40 CFR Appendix Q, requires the use of two $PM_{10}$ reference samplers because a single 46.2-mm filter from a reference sampler may not be divided prior to analysis. It is possible to analyze a 46.2-mm filter first with the non-destructive X-ray Fluorescence (XRF) FRM and subsequently extract the filter for other analytical techniques. If the filter is subject to XRF with subsequent extraction for other analyses, then a single $PM_{10}$ reference sampler may be used for sample collection.

(3) A candidate method for Pb in TSP or Pb in $PM_{10}$ which employs a direct reading (e.g., continuous or semi-continuous sampling) method that uses the same sampling inlet and flow rate as the FRM and the same or different analytical procedure may be tested. The direct measurements are then aggregated to 24-hour equivalent concentrations for comparison with the FRM. For determining precision in section (k), two collocated direct reading devices must be used.

(f) *Audit samples*. Three audit samples must be obtained from the address given in §53.4(a). For Pb in TSP collected by the high-volume sampling method, the audit samples are ¾ × 8-inch glass fiber strips containing known amounts of Pb in micrograms per strip (μg/strip) equivalent to the following nominal percentages of the

National Ambient Air Quality Standard (NAAQS): 30%, 100%, and 250%. For Pb in $PM_{10}$ collected by the low-volume sampling method, the audit samples are 46.2-mm polytetrafluorethylene (PTFE) filters containing known amounts of Pb in micrograms per filter (µg/filter) equivalent to the same percentages of the NAAQS: 30%, 100%, and 250%. The true amount of Pb (Tqi), in total µg/strip (for TSP) or total µg/filter (for $PM_{10}$), will be provided for each audit sample.

(g) *Filter analysis.* (1) For both the reference method samples (e) and the audit samples (f), analyze each filter or filter extract three times in accordance with the reference method analytical procedure. This applies to both the Pb in TSP and Pb in $PM_{10}$ methods. The analysis of replicates should not be performed sequentially, i.e., a single sample should not be analyzed three times in sequence. Calculate the indicated Pb concentrations for the reference method samples in micrograms per cubic meter (µg/m$^3$) for each analysis of each filter. Calculate the indicated total Pb amount for the audit samples in µg/strip for each analysis of each strip or µg/filter for each analysis of each audit filter. Label these test results as $R_{1A}$, $R_{1B}$, $R_{1C}$, $R_{2A}$, $R_{2B}$, etc., $Q_{1A}$, $Q_{1B}$, $Q_{1C}$, etc., where R denotes results from the reference method samples; Q denotes results from the audit samples; 1, 2, 3 indicate the filter number, and A, B, C indicate the first, second, and third analysis of each filter, respectively.

(2) For the candidate method samples, analyze each sample filter or filter extract three times and calculate, in accordance with the candidate method, the indicated Pb concentration in µg/m$^3$ for each analysis of each filter. The analysis of replicates should not be performed sequentially. Label these test results as $C_{1A}$, $C_{1B}$, $C_{2C}$, etc., where C denotes results from the candidate method. For candidate methods which provide a direct reading or measurement of Pb concentrations without a separable procedure, $C_{1A} = C_{1B} = C_{1C}$, $C_{2A} = C_{2B} = C_{2C}$, etc.

(h) *Average Pb concentration.* For the reference method, calculate the average Pb concentration for each filter by averaging the concentrations calculated from the three analyses as described in (g)(1) using equation 1 of this section:

Equation 1

$$R_{iave} = \frac{(R_{iA} + R_{iB} + R_{iC})}{3}$$

Where, i is the filter number.

(i) *Analytical Bias.* (1) For the audit samples, calculate the average Pb concentration for each strip or filter analyzed by the reference method by averaging the concentrations calculated from the three analyses as described in (g)(1) using equation 2 of this section:

Equation 2

$$Q_{iave} = \frac{(Q_{iA} + Q_{iB} + Q_{iC})}{3}$$

Where, i is audit sample number.

(2) Calculate the percent difference ($D_q$) between the average Pb concentration for each audit sample and the true Pb concentration ($T_q$) using equation 3 of this section:

Equation 3

$$D_{qi} = \frac{Q_{iave} - T_{qi}}{T_{qi}} \times 100$$

(3) If any difference value ($D_{qi}$) exceeds ±5 percent, the bias of the reference method analytical procedure is out-of-control. Corrective action must be taken to determine the source of the error(s) (e.g., calibration standard discrepancies, extraction problems, etc.) and the reference method and audit sample determinations must be repeated according to paragraph (g) of this section, or the entire test procedure (starting with paragraph (e) of this section) must be repeated.

(j) *Acceptable filter pairs.* Disregard all filter pairs for which the Pb concentration, as determined in paragraph (h) of this section by the average of the three reference method determinations, falls outside the range of 30% to 250% of the Pb NAAQS level in µg/m$^3$ for Pb in both TSP and $PM_{10}$. All remaining filter pairs must be subjected to the tests for precision and comparability in paragraphs (k) and (l) of this section. At

least five filter pairs must be within the specified concentration range for the tests to be valid.

(k) *Test for precision*. (1) Calculate the precision (P) of the analysis (in percent) for each filter and for each method, as the maximum minus the minimum divided by the average of the three concentration values, using equation 4 or equation 5 of this section:

Equation 4

$$P_{Ri} = \frac{R_{i\max} - R_{i\min}}{R_{iave}} \times 100$$

or

Equation 5

$$P_{Ci} = \frac{C_{i\max} - C_{i\min}}{C_{iave}} \times 100$$

Where, i indicates the filter number.

(2) If a direct reading candidate method is tested, the precision is determined from collocated devices using equation 5 above.

(3) If any reference method precision value ($P_{Ri}$) exceeds 15 percent, the precision of the reference method analytical procedure is out-of-control. Corrective action must be taken to determine the source(s) of imprecision, and the reference method determinations must be repeated according to paragraph (g) of this section, or the entire test procedure (starting with paragraph (e) of this section) must be repeated.

(4) If any candidate method precision value ($P_{Ci}$) exceeds 15 percent, the candidate method fails the precision test.

(5) The candidate method passes this test if all precision values (*i.e.*, all $P_{Ri}$'s and all $P_{Ci}$'s) are less than 15 percent.

(l) *Test for comparability*. (1) For each filter or analytical sample pair, calculate all nine possible percent differences (D) between the reference and candidate methods, using all nine possible combinations of the three determinations (A, B, and C) for each method using equation 6 of this section:

Equation 6

$$D_{in} = \frac{C_{ij} - R_{jk}}{R_{jk}} \times 100$$

Where, i is the filter number, and n numbers from 1 to 9 for the nine possible difference combinations for the three determinations for each method (j = A, B, C, candidate; k = A, B, C, reference).

(2) If none of the percent differences (D) exceeds ±20 percent, the candidate method passes the test for comparability.

(3) If one or more of the percent differences (D) exceed ±20 percent, the candidate method fails the test for comparability.

(4) The candidate method must pass both the precision test (paragraph (k) of this section) and the comparability test (paragraph (l) of this section) to qualify for designation as an equivalent method.

(m) *Method Detection Limit (MDL)*. Calculate the estimated MDL using the guidance provided in 40 CFR, Part 136 Appendix B. It is essential that all sample processing steps of the analytical method be included in the determination of the method detection limit. Take a minimum of seven blank filters from each lot to be used and calculate the detection limit by processing each through the entire candidate analytical method. Make all computations according to the defined method with the final results in μg/m³. The MDL of the candidate method must be equal to, or less than 5% of the level of the Pb NAAQS.

[73 FR 67057, Nov. 12, 2008]

### §53.34 Test procedure for methods for $PM_{10}$ and Class I methods for $PM_{2.5}$.

(a) *Comparability*. Comparability is shown for $PM_{10}$ methods and for Class I methods for $PM_{2.5}$ when the relationship between:

(1) Measurements made by a candidate method, and

(2) Measurements made by a corresponding reference method on simultaneously collected samples (or the same sample, if applicable) at each of one or more test sites (as required) is

such that the linear regression parameters (slope, intercept, and correlation coefficient) describing the relationship meet the requirements specified in table C–4 of this subpart.

(b) *Methods for $PM_{10}$.* Test measurements must be made, or derived from particulate samples collected, at not less than two test sites, each of which must be located in a geographical area characterized by ambient particulate matter that is significantly different in nature and composition from that at the other test site(s). Augmentation of pollutant concentrations is not permitted, hence appropriate test sites must be selected to provide the minimum number of test $PM_{10}$ concentrations in the ranges specified in table C–4 of this subpart. The tests at the two sites may be conducted in different calendar seasons, if appropriate, to provide $PM_{10}$ concentrations in the specified ranges.

(c) *$PM_{10}$ methods employing the same sampling procedure as the reference method but a different analytical method.* Candidate methods for $PM_{10}$ which employ a sampler and sample collection procedure that are identical to the sampler and sample collection procedure specified in the reference method, but use a different analytical procedure, may be tested by analyzing common samples. The common samples shall be collected according to the sample collection procedure specified by the reference method and shall be analyzed in accordance with the analytical procedures of both the candidate method and the reference method.

(d) *Methods for $PM_{2.5}$.* Augmentation of pollutant concentrations is not permitted, hence appropriate test sites must be selected to provide the minimum number of test measurement sets to meet the requirements for $PM_{2.5}$ concentrations in the ranges specified in table C–4 of this subpart. Only one test site is required, and the site need only meet the $PM_{2.5}$ ambient concentration levels required by table C–4 of this subpart and the requirements of § 53.30(b) of this subpart. A total of 10 valid measurement sets is required.

(e) *Collocated measurements.* (1) Set up three reference method samplers collocated with three candidate method samplers or analyzers at each of the number of test sites specified in table C–4 of this subpart.

(2) The ambient air intake points of all the candidate and reference method collocated samplers or analyzers shall be positioned at the same height above the ground level, and between 2 meters (1 meter for samplers or analyzers with flow rates less than 200 L/min) and 4 meters apart. The samplers shall be oriented in a manner that will minimize spatial and wind directional effects on sample collection.

(3) At each site, obtain as many sets of simultaneous $PM_{10}$ or $PM_{2.5}$ measurements as necessary (see table C–4 of this subpart), each set consisting of three reference method and three candidate method measurements, all obtained simultaneously.

(4) Candidate $PM_{10}$ method measurements shall be nominal 24-hour (±1 hour) integrated measurements or shall be averaged to obtain the mean concentration for a nominal 24-hour period. $PM_{2.5}$ measurements may be either nominal 24-or 48-hour integrated measurements. All collocated measurements in a measurement set must cover the same nominal 24-or 48-hour time period.

(5) For samplers, retrieve the samples promptly after sample collection and analyze each sample according to the reference method or candidate method, as appropriate, and determine the $PM_{10}$ or $PM_{2.5}$ concentration in $\mu g/m^3$. If the conditions of paragraph (c) of this section apply, collect sample sets only with the three reference method samplers. Guidance for quality assurance procedures for $PM_{2.5}$ methods is found in ''Quality Assurance Document 2.12'' (reference (2) in appendix A to this subpart).

(f) *Sequential samplers.* For sequential samplers, the sampler shall be configured for the maximum number of sequential samples and shall be set for automatic collection of all samples sequentially such that the test samples are collected equally, to the extent possible, among all available sequential channels or utilizing the full available sequential capability.

(g) *Calculation of reference method averages and precisions.* (1) For each of the measurement sets, calculate the

average $PM_{10}$ or $PM_{2.5}$ concentration obtained with the reference method samplers, using equation 7 of this section:

*Equation 7*

$$\bar{R}_j = \frac{\sum_{i=1}^{3} R_{i,j}}{3}$$

Where:

R = The concentration measurements from the reference methods;
i = The sampler number; and
j = The measurement set number.

(2) For each of the measurement sets, calculate the precision of the reference method $PM_{10}$ or $PM_{2.5}$ measurements as the standard deviation, $P_{Rj}$, using equation 8 of this section:

*Equation 8*

$$P_{Rj} = \sqrt{\frac{\sum_{i=1}^{3} R_{i,j}^2 - \frac{1}{3}\left(\sum_{i=1}^{3} R_{i,j}\right)^2}{2}}$$

(3) For each measurement set, also calculate the precision of the reference method $PM_{10}$ or $PM_{2.5}$ measurements as the relative standard deviation, $RP_{Rj}$, in percent, using equation 9 of this section:

*Equation 9*

$$RP_{Rj} \frac{P_{Rj}}{R_j} \times 100\%$$

(h) *Acceptability of measurement sets.* Each measurement set is acceptable and valid only if the three reference method measurements and the three candidate method measurements are obtained and are valid, $\bar{R}_j$ falls within the acceptable concentration range specified in table C–4 of this subpart, and either $P_{Rj}$ or $RP_{Rj}$ is within the corresponding limit for reference method precision specified in table C–4 of this subpart. For each site, table C–4 of this subpart specifies the minimum number of measurement sets required having $\bar{R}_j$ above and below specified concentrations for 24- or 48-hour samples. Additional measurement sets shall be obtained, as necessary, to provide the minimum number of acceptable measurement sets for each category and the minimum total number of acceptable measurement sets for each test site. If more than the minimum number of measurement sets are collected that meet the acceptability criteria, all such measurement sets shall be used to demonstrate comparability.

(i) *Candidate method average concentration measurement.* For each of the acceptable measurement sets, calculate the average $PM_{10}$ or $PM_{2.5}$ concentration measurements obtained with the candidate method samplers, using equation 10 of this section:

*Equation 10*

$$\bar{C}_j = \frac{\sum_{i=1}^{3} C_{i,j}}{3}$$

Where:

C = The concentration measurements from the candidate methods;
i = The measurement number in the set; and
j = The measurement set number.

(j) *Test for comparability.* (1) For each site, plot all of the average $PM_{10}$ or $PM_{2.5}$ measurements obtained with the candidate method ($\bar{C}_j$) against the corresponding average $PM_{10}$ or $PM_{2.5}$ measurements obtained with the reference method ($\bar{R}_j$. For each site, calculate and record the linear regression slope and intercept, and the correlation coefficient.

(2) To pass the test for comparability, the slope, intercept, and correlation coefficient calculated under paragraph (j)(1) of this section must be within the limits specified in table C–4 of this subpart for all test sites.

### §53.35 Test procedure for Class II and Class III methods for $PM_{2.5}$ and $PM_{-2.5}$.

(a) *Overview.* Class II and Class III candidate equivalent methods shall be tested for comparability of $PM_{2.5}$ or $PM_{10-2.5}$ measurements to corresponding collocated $PM_{2.5}$ or $PM_{10-2.5}$ reference method measurements at each of multiple field sites, as required.

Comparability is shown for the candidate method when simultaneous collocated measurements made by candidate and reference methods meet the comparability requirements specified in this section § 53.35 and in table C–4 of this subpart at each of the required test sites.

(b) *Test sites and seasons.* A summary of the test site and seasonal testing requirements is presented in table C–5 of this subpart.

(1) *Test sites.* Comparability testing is required at each of the applicable U.S. test sites required by this paragraph (b). Each test site must also meet the general test site requirements specified in § 53.30(b).

(i) *$PM_{2.5}$ Class II and Class III candidate methods.* Test sites should be chosen to provide representative chemical and meteorological characteristics with respect to nitrates, sulfates, organic compounds, and various levels of temperature, humidity, wind, and elevation. For Class III methods, one test site shall be selected in each of the following four general locations (A, B, C, and D). For Class II methods, two test sites, one western site (A or B) and one midwestern or eastern site (C or D), shall be selected from these locations.

(A) Test site A shall be in the Los Angeles basin or California Central Valley area in a location that is characterized by relatively high $PM_{2.5}$, nitrates, and semi-volatile organic pollutants.

(B) Test site B shall be in a western city such as Denver, Salt Lake City, or Albuquerque in an area characterized by cold weather, higher elevation, winds, and dust.

(C) Test site C shall be in a midwestern city characterized by substantial temperature variation, high nitrates, and wintertime conditions.

(D) Test site D shall be in a northeastern or mid-Atlantic city that is seasonally characterized by high sulfate concentrations and high relative humidity.

(ii) *$PM_{10-2.5}$ Class II and Class III candidate methods.* Test sites shall be chosen to provide modest to high levels of $PM_{10-2.5}$ representative of locations in proximity to urban sources of $PM_{10-2.5}$ such as high-density traffic on paved roads, industrial sources, and construction activities. For Class III methods, one test site shall be selected in each of the four following general locations (A, B, C, and D), and at least one of the test sites shall have characteristic wintertime temperatures of 0 °C or lower. For Class II methods, two test sites, one western site (A or B) and one midwestern or eastern site (C or D), shall be selected from these locations.

(A) Test site A shall be in the Los Angeles basin or the California Central Valley area in a location that is characterized by relatively high $PM_{2.5}$, nitrates, and semi-volatile organic pollutants.

(B) Test site B shall be in a western city characterized by a high ratio of $PM_{10-2.5}$ to $PM_{2.5}$, with exposure to windblown dust, such as Las Vegas or Phoenix.

(C) Test site C shall be in a midwestern city characterized by substantial temperature variation, high nitrates, and wintertime conditions.

(D) Test site D shall be in a large city east of the Mississippi River, having characteristically high sulfate concentrations and high humidity levels.

(2) *Test seasons.* (i) For $PM_{2.5}$ and $PM_{10-2.5}$ Class III candidate methods, test campaigns are required in both summer and winter seasons at test site A, in the winter season only at test sites B and C, and in the summer season only at test site D. (A total of five test campaigns is required.) The summer season shall be defined as the typically warmest three or four months of the year at the site; the winter season shall be defined as the typically coolest three or four months of the year at the site.

(ii) For Class II $PM_{2.5}$ and $PM_{10-2.5}$ candidate methods, one test campaign is required at test site A or B and a second test campaign at test site C or D (total of two test campaigns).

(3) *Test concentrations.* The test sites should be selected to provide ambient concentrations within the concentration limits specified in table C–4 of this subpart, and also to provide a wide range of test concentrations. A narrow range of test concentrations may result in a low concentration coefficient of variation statistic for the test measurements, making the test for correlation coefficient more difficult to pass

(see paragraph (h) of this section, test for comparison correlation).

(4) *Pre-approval of test sites.* The EPA recommends that the applicant seek EPA approval of each proposed test site prior to conducting test measurements at the site. To do so, the applicant should submit a request for approval as described in § 53.30(b)(2).

(c) *Collocated measurements.* (1) For each test campaign, three reference method samplers and three candidate method samplers or analyzers shall be installed and operated concurrently at each test site within each required season (if applicable), as specified in paragraph (b) of this section. All reference method samplers shall be of single-filter design (not multi-filter, sequential sample design). Each candidate method shall be setup and operated in accordance with its associated manual referred to in § 53.4(b)(3) and in accordance with applicable guidance in ''Quality Assurance Document 2.12'' (reference (2) in appendix A to this subpart). All samplers or analyzers shall be placed so that they sample or measure air representative of the surrounding area (within one kilometer) and are not unduly affected by adjacent buildings, air handling equipment, industrial operations, traffic, or other local influences. The ambient air inlet points of all samplers and analyzers shall be positioned at the same height above the ground level and between 2 meters (1 meter for instruments having sample inlet flow rates less than 200 L/min) and 4 meters apart.

(2) A minimum of 23 valid and acceptable measurement sets of $PM_{2.5}$ or $PM_{10-2.5}$ 24-hour (nominal) concurrent concentration measurements shall be obtained during each test campaign at each test site. To be considered acceptable for the test, each measurement set shall consist of at least two valid reference method measurements and at least two valid candidate method measurements, and the $PM_{2.5}$ or $PM_{10-2.5}$ measured concentration, as determined by the average of the reference method measurements, must fall within the acceptable concentration range specified in table C–4 of this subpart. Each measurement set shall include all valid measurements obtained. For each measurement set containing fewer than three reference method measurements or fewer than three candidate method measurements, an explanation and appropriate justification shall be provided to account for the missing measurement or measurements.

(3) More than 23 valid measurement sets may be obtained during a particular test campaign to provide a more advantageous range of concentrations, more representative conditions, additional higher or lower measurements, or to otherwise improve the comparison of the methods. All valid data sets obtained during each test campaign shall be submitted and shall be included in the analysis of the data.

(4) The integrated-sample reference method measurements shall be of at least 22 hours and not more than 25 hours duration. Each reference method sample shall be retrieved promptly after sample collection and analyzed according to the reference method to determine the $PM_{2.5}$ or $PM_{10-2.5}$ measured concentration in $\mu g/m^3$. Guidance and quality assurance procedures applicable to $PM_{2.5}$ or $PM_{10-2.5}$ reference methods are found in ''Quality Assurance Document 2.12'' (reference (2) in appendix A to this subpart).

(5) Candidate method measurements shall be timed or processed and averaged as appropriate to determine an equivalent mean concentration representative of the same time period as that of the concurrent integrated-sample reference method measurements, such that all measurements in a measurement set shall be representative of the same time period. In addition, hourly average concentration measurements shall be obtained from each of the Class III candidate method analyzers for each valid measurement set and submitted as part of the application records.

(6) In the following tests, all measurement sets obtained at a particular test site, from both seasonal campaigns if applicable, shall be combined and included in the test data analysis for the site. Data obtained at different test sites shall be analyzed separately. All measurements should be reported as

normally obtained, and no measurement values should be rounded or truncated prior to data analysis. In particular, no negative measurement value, if otherwise apparently valid, should be modified, adjusted, replaced, or eliminated merely because its value is negative. Calculated mean concentrations or calculated intermediate quantities should retain at least one order-of-magnitude greater resolution than the input values. All measurement data and calculations shall be recorded and submitted in accordance with § 53.30(g), including hourly test measurements obtained from Class III candidate methods.

(d) *Calculation of mean concentrations*—(1) *Reference method outlier test.* For each of the measurement sets for each test site, check each reference method measurement to see if it might be an anomalous value (outlier) as follows, where $R_{i,j}$ is the measurement of reference method sampler i on test day j. In the event that one of the reference method measurements is missing or invalid due to a specific, positively-identified physical cause (e.g., sampler malfunction, operator error, accidental damage to the filter, etc.; see paragraph (c)(2) of this section), then substitute zero for the missing measurement, *for the purposes of this outlier test only*.

(i) Calculate the quantities $2 \times R_{1,j}/(R_{1,j} + R_{2,j})$ and $2 \times R_{1,j}/(R_{1,j} + R_{3,j})$. If both quantities fall outside of the interval, (0.93, 1.07), then $R_{1,j}$ is an outlier.

(ii) Calculate the quantities $2 \times R_{2,j}/(R_{2,j} + R_{1,j})$ and $2 \times R_{2,j}/(R_{2,j} + R_{3,j})$. If both quantities fall outside of the interval, (0.93, 1.07), then $R_{2,j}$ is an outlier.

(iii) Calculate the quantities $2 \times R_{3,j}/(R_{3,j} + R_{1,j})$ and $2 \times R_{3,j}/(R_{3,j} + R_{2,j})$. If both quantities fall outside of the interval, (0.93, 1.07), then $R_{3,j}$ is an outlier.

(iv) If this test indicates that one of the reference method measurements in the measurement set is an outlier, the outlier measurement shall be eliminated from the measurement set, and the other two measurements considered valid. If the test indicates that more than one reference method measurement in the measurement set is an outlier, the entire measurement set (both reference and candidate method measurements) shall be excluded from further data analysis for the tests of this section.

(2) For each of the measurement sets for each test site, calculate the mean concentration for the reference method measurements, using equation 11 of this section:

*Equation 11*

$$\bar{R}_j = \frac{1}{n}\sum_{i=1}^{n} R_{i,j}$$

Where:

$\bar{R}_j$ = The mean concentration measured by the reference method for the measurement set;

$R_{i,j}$ = The measurement of reference method sampler i on test day j; and

n = The number of valid reference method measurements in the measurement set (normally 3).

(3) Any measurement set for which $\bar{R}_j$ does not fall in the acceptable concentration range specified in table C–4 of this subpart is not valid, and the entire measurement set (both reference and candidate method measurements) must be eliminated from further data analysis.

(4) For each of the valid measurement sets at each test site, calculate the mean concentration for the candidate method measurements, using equation 12 of this section. (The outlier test in paragraph (d)(1) of this section shall not be applied to the candidate method measurements.)

*Equation 12*

$$\bar{C}_j = \frac{1}{m}\sum_{i=1}^{m} C_{i,j}$$

Where:

$C_j$ = The mean concentration measured by the candidate method for the measurement set;

$C_{i,j}$ = The measurement of the candidate method sampler or analyzer i on test day j; and

m = The number of valid candidate method measurements in the measurement set (normally 3).

(e) *Test for reference method precision.* (1) For each of the measurement sets

for each site, calculate an estimate for the relative precision of the reference method measurements, $RP_j$, using equation 13 of this section:

*Equation 13*

$$RP_j = \frac{1}{\overline{R}_j}\sqrt{\frac{\sum_{i=1}^{n} R_{i,j}^2 - \frac{1}{n}\left(\sum_{i=1}^{n} R_{i,j}\right)^2}{n-1}} \times 100\%$$

(2) For each site, calculate an estimate of reference method relative precision for the site, RP, using the *root mean square* calculation of equation 14 of this section:

*Equation 14*

$$RP = \sqrt{\frac{1}{J}\sum_{j=1}^{J}\left(RP_j\right)^2}$$

Where, J is the total number of valid measurement sets for the site.

(3) Verify that the estimate for reference method relative precision for the site, RP, is not greater than the value specified for reference method precision in table C–4 of this subpart. A reference method relative precision greater than the value specified in table C–4 of this subpart indicates that quality control for the reference method is inadequate, and corrective measures must be implemented before proceeding with the test.

(f) *Test for candidate method precision.* (1) For each of the measurement sets, for each site, calculate an estimate for the relative precision of the candidate method measurements, $CP_j$, using equation 15 of this section:

*Equation 15*

$$CP_j = \frac{1}{\overline{C}_j}\sqrt{\frac{\sum_{i=1}^{m} C_{i,j}^2 - \frac{1}{m}\left(\sum_{i=1}^{m} C_{i,j}\right)^2}{m-1}} \times 100\%$$

(2) For each site, calculate an estimate of candidate method relative precision for the site, CP, using the *root mean square* calculation of equation 16 of this section:

*Equation 16*

$$CP = \sqrt{\frac{1}{J}\sum_{j=1}^{J}\left(CP_j\right)^2}$$

Where, J is the total number of valid measurement sets for the site.

(3) To pass the test for precision, the mean candidate method relative precision at each site must not be greater than the value for candidate method precision specified in table C–4 of this subpart.

(g) *Test for additive and multiplicative bias (comparative slope and intercept).* (1) For each test site, calculate the mean concentration measured by the reference method, $\bar{R}$, using equation 17 of this section:

*Equation 17*

$$\overline{R} = \frac{1}{J}\sum_{j=1}^{J}\overline{R}_j$$

(2) For each test site, calculate the mean concentration measured by the candidate method, $\bar{C}$, using equation 18 of this section:

*Equation 18*

$$\overline{C} = \frac{1}{J}\sum_{j=1}^{J}\overline{C}_j$$

(3) For each test site, calculate the linear regression slope and intercept of the mean candidate method measurements ($\bar{C}_j$) against the mean reference method measurements ($\bar{R}_j$), using equations 19 and 20 of this section, respectively:

*Equation 19*

$$\text{Slope} = \frac{\sum_{j=1}^{J}\left(\bar{R}_j - \bar{R}\right)\left(\bar{C}_j - \bar{C}\right)}{\sum_{j=1}^{J}\left(\bar{R}_j - \bar{R}\right)^2}$$

*Equation 20*

$$\text{Intercept} = \bar{C} - \text{slope} \times \bar{R}$$

(4) To pass this test, at each test site:

(i) The slope (calculated to at least 2 decimal places) must be in the interval specified for regression slope in table C–4 of this subpart; and

(ii) The intercept (calculated to at least 2 decimal places) must be in the interval specified for regression intercept in table C–4 of this subpart.

(iii) The slope and intercept limits are illustrated in figures C–2 and C–3 of this subpart.

(h) *Tests for comparison correlation*. (1) For each test site, calculate the (Pearson) correlation coefficient, r (not the coefficient of determination, $r^2$), using equation 21 of this section:

*Equation 21*

$$r = \frac{\sum_{j=1}^{J}\left(\bar{R}_j - \bar{R}\right)\left(\bar{C}_j - \bar{C}\right)}{\sqrt{\sum_{j=1}^{J}\left(\bar{R}_j - \bar{R}\right)^2 \sum_{j=1}^{J}\left(\bar{C}_j - \bar{C}\right)^2}}$$

(2) For each test site, calculate the concentration coefficient of variation, CCV, using equation 22 of this section:

*Equation 22*

$$\text{CCV} = \frac{1}{\bar{R}}\sqrt{\frac{\sum_{j=1}^{J}\left(\bar{R}_j - \bar{R}\right)^2}{J-1}}$$

(3) To pass the test, the correlation coefficient, r, for each test site must not be less than the values, for various values of CCV, specified for correlation in table C–4 of this subpart. These limits are illustrated in figure C–4 of this subpart.

[71 FR 61278, Oct. 17, 2006, as amended at 72 FR 32202, June 12, 2007]

TABLE C–1 TO SUBPART C OF PART 53—TEST CONCENTRATION RANGES, NUMBER OF MEASUREMENTS REQUIRED, AND MAXIMUM DISCREPANCY SPECIFICATIONS

| Pollutant | Concentration range, parts per million (ppm) | Simultaneous measurements required | | | | Maximum discrepancy specification, parts per million |
|---|---|---|---|---|---|---|
| | | 1-hour | | 24-hour | | |
| | | First set | Second set | First set | Second set | |
| Ozone | Low 0.06 to 0.10 | 5 | 6 | ..... | ..... | 0.02 |
| | Med. 0.15 to 0.25 | 5 | 6 | ..... | ..... | 0.03 |
| | High 0.35 to 0.46 | 4 | 6 | ..... | ..... | 0.04 |
| | Total | 14 | 18 | ..... | ..... | |
| Carbon monoxide | Low 7 to 11 | 5 | 6 | ..... | ..... | 1.5 |
| | Med. 20 to 30 | 5 | 6 | ..... | ..... | 2.0 |
| | High 25 to 45 | 4 | 6 | ..... | ..... | 3.0 |
| | Total | 14 | 18 | ..... | ..... | |
| Sulfur dioxide | Low 0.02 to 0.05 | 5 | 6 | 3 | 3 | 0.02 |
| | Med. 0.10 to 0.15 | 5 | 6 | 2 | 3 | 0.03 |
| | High 0.30 to 0.50 | 4 | 6 | 2 | 2 | 0.04 |
| | Total | 14 | 18 | 7 | 8 | |
| Nitrogen dioxide | Low 0.02 to 0.08 | ..... | ..... | 3 | 3 | 0.02 |
| | Med. 0.10 to 0.20 | ..... | ..... | 2 | 2 | 0.02 |
| | High 0.25 | ..... | ..... | 2 | 2 | 0.03 |

| Pollutant | Concentration range, parts per million (ppm) | Simultaneous measurements required | | | | Maximum discrepancy specification, parts per million |
|---|---|---|---|---|---|---|
| | | 1-hour | | 24-hour | | |
| | | First set | Second set | First set | Second set | |
| | Total | | | 7 | 8 | |

[75 FR 35601, June 22, 2010]

TABLE C–2 TO SUBPART C OF PART 53—SEQUENCE OF TEST MEASUREMENTS

| Measurement | Concentration range | |
|---|---|---|
| | First set | Second set |
| 1 | Low | Medium. |
| 2 | High | High. |
| 3 | Medium | Low. |
| 4 | High | High. |
| 5 | Low | Medium. |
| 6 | Medium | Low. |
| 7 | Low | Medium. |
| 8 | Medium | Low. |
| 9 | High | High. |
| 10 | Medium | Low. |
| 11 | High | Medium. |
| 12 | Low | High. |
| 13 | Medium | Medium. |
| 14 | Low | High. |
| 15 | | Low. |
| 16 | | Medium. |
| 17 | | Low. |
| 18 | | High. |

TABLE C–3 TO SUBPART C OF PART 53—TEST SPECIFICATIONS FOR PB IN TSP AND PB IN $PM_{10}$ METHODS

| | |
|---|---|
| Concentration range equivalent to percentage of NAAQS in μg/m³. | 30% to 250% |
| Minimum number of 24-hr measurements | 5 |
| Maximum reference method analytical bias, $D_q$. | ±5% |
| Maximum precision, $P_R$ or $P_C$ | ≤15% |
| Maximum difference (D) | ±20% |
| Estimated Method Detection Limit (MDL), μg/m³. | 5% of NAAQS level. |

[73 FR 67059, Nov. 12, 2008]

TABLE C–4 TO SUBPART C OF PART 53—TEST SPECIFICATIONS FOR $PM_{10}$, $PM_{2.5}$ AND $PM_{10-2.5}$ CANDIDATE EQUIVALENT METHODS

| Specification | $PM_{10}$ | $PM_{2.5}$ | | | $PM_{10-2.5}$ | |
|---|---|---|---|---|---|---|
| | | Class I | Class II | Class III | Class II | Class III |
| Acceptable concentration range ($R_j$), μg/m³. | 15–300 | 3–200 | 3–200 | 3–200 | 3–200 | 3–200 |
| Minimum number of test sites. | 2 | 1 | 2 | 4 | 2 | 4 |
| Minimum number of candidate method samplers or analyzers per site. | 3 | 3 | 3[1] | 3[1] | 3[1] | 3[1] |
| Number of reference method samplers per site. | 3 | 3 | 3[1] | 3[1] | 3[1] | 3[1] |
| Minimum number of acceptable sample sets per site for $PM_{10}$ methods: | | | | | | |
| $R_j$ <60 μg/m³ | 3 | | | | | |
| $R_j$ >60 μg/m³ | 3 | | | | | |
| Total | 10 | | | | | |
| Minimum number of acceptable sample sets per site for $PM_{2.5}$ and $PM_{10-2.5}$ candidate equivalent methods: | | | | | | |
| $R_j$ <30 μg/m³ for 24-hr or $R_j$ <20 μg/m³ for 48-hr samples. | | 3 | | | | |
| $R_j$ >30 μg/m³ for 24-hr or $R_j$ >20 μg/m³ for 48-hr samples. | 3 | | | | | |
| Each season | 10 | 23 | 23 | 23 | 23 | |
| Total, each site | 10 | 23 | 23 (46 for two-season sites) | 23 | 23 (46 for two-season sites) | |

| Specification | $PM_{10}$ | $PM_{2.5}$ | | | $PM_{10-2.5}$ | |
|---|---|---|---|---|---|---|
| | | Class I | Class II | Class III | Class II | Class III |
| Precision of replicate reference method measurements, $P_{Rj}$ or $RP_{Rj}$, respectively; RP for Class II or III $PM_{2.5}$ or $PM_{10-2.5}$, maximum. | 5 μg/m³ or 7%. | 2 μg/m³ or 5%. | 10% [2] | 10% [2] | 10% [2] | 10% [2] |
| Precision of $PM_{2.5}$ or $PM_{10-2.5}$ candidate method, CP, each site. | 10% [2] ... | 15% [2] ... | 15% [2] | 15% [2] | | |
| Slope of regression relationship. | 1 ±0.10 | 1 ±0.05 | 1 ±0.10 | 1 ±0.10 | 1 ±0.10 | 1 ±0.12 |
| Intercept of regression relationship, μg/m³. | 0 ±5 ..... | 0 ±1 ..... | Between: 13.55 − (15.05 × slope), but not less than −1.5; and 16.56 − (15.05 × slope), but not more than + 1.5 | Between: 15.05 − (17.32 × slope), but not less than −2.0; and 15.05 − (13.20 × slope), but not more than + 2.0 | Between: 62.05 − (70.5 × slope), but not less than −3.5; and 78.95 − (70.5 × slope), but not more than + 3.5 | Between: 70.50 − (82.93 × slope), but not less than −7.0; and 70.50 − (61.16 × slope), but not more than + 7.0 |
| Correlation of reference method and candidate method measurements. | ≥0.97 ... | ≥0.97 ... | ≥0.93—for CCV ≤0.4; ≥0.85 + 0.2 × CCV—for 0.4 ≤CCV ≤0.5; ≥0.95—for CCV ≥0.5 | | | |

[1] Some missing daily measurement values may be permitted; see test procedure.
[2] Calculated as the root mean square over all measurement sets.

[72 FR 32203, June 12, 2007]

TABLE C–5 TO SUBPART C OF PART 53—SUMMARY OF COMPARABILITY FIELD TESTING CAMPAIGN SITE AND SEASONAL REQUIREMENTS FOR CLASS II AND III FEMS FOR $PM_{10-2.5}$ AND $PM_{2.5}$

| Candidate method | Test site | A | B | C | D |
|---|---|---|---|---|---|
| $PM_{2.5}$ ..... | Test site location area. | Los Angeles basin or California Central Valley. | Western city such as Denver, Salt Lake City, or Albuquerque. | Midwestern city ..... | Northeastern or mid-Atlantic city. |
| | Test site characteristics. | Relatively high $PM_{2.5}$, nitrates, and semi-volatile organic pollutants. | Cold weather, higher elevation, winds, and dust. | Substantial temperature variation, high nitrates, wintertime conditions. | High sulfate and high relative humidity. |
| | *Class III* Field test campaigns (Total: 5). | Winter and summer. | Winter only ..... | Winter only ..... | Summer only. |
| | *Class II* Field test campaigns (Total: 2). | Site A or B, any season | | Site C or D, any season. | |
| $PM_{10-2.5}$ ..... | Test site location area. | Los Angeles basin or California Central Valley. | Western city such as Las Vegas or Phoenix. | Midwestern city ..... | Large city east of the Mississippi River. |
| | Test site characteristics. | Relatively high $PM_{2.5}$, nitrates, and semi-volatile organic pollutants. | High $PM_{10-2.5}$ to $PM_{2.5}$ ratio, windblown dust. | Substantial temperature variation, high nitrates, wintertime conditions. | High sulfate and high relative humidity. |
| | *Class III* Field test campaigns (Total: 5). | Winter and summer. | Winter only ..... | Winter only ..... | Summer only. |

| Candidate method | Test site | A | B | C | D |
|---|---|---|---|---|---|
| | *Class II* Field test campaigns (Total: 2). | Site A or B, any season | | Site C or D, any season. | |

FIGURE C–1 TO SUBPART C OF PART 53—SUGGESTED FORMAT FOR REPORTING TEST RESULTS FOR METHODS FOR $SO_2$, CO, $O_3$, $NO_2$

Candidate Method _____

Reference Method _____

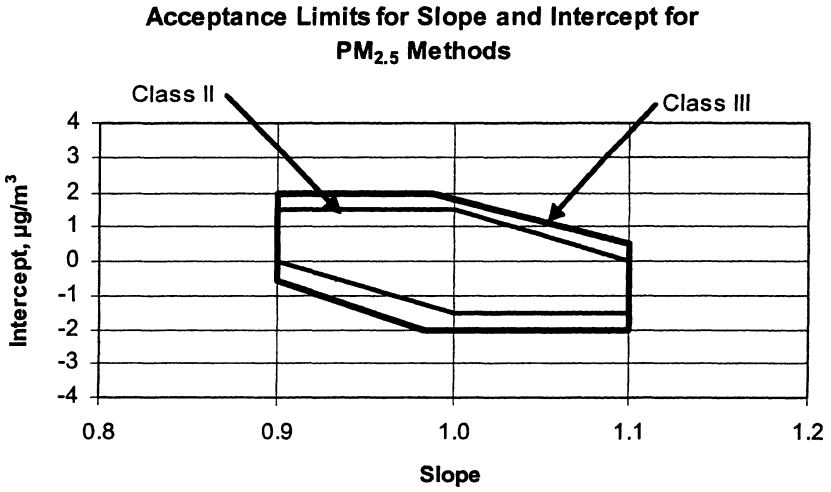
Applicant _____

☐ First Set ☐ Second Set ☐ Type ☐ 1 Hour ☐ 24 Hour

| Concentration range | | Date | Time | Concentration, ppm | | Difference | Table C–1 spec. | Pass or fail |
|---|---|---|---|---|---|---|---|---|
| | | | | Candidate | Reference | | | |
| Low | 1 | | | | | | | |
| _____ ppm | 2 | | | | | | | |
| to _____ ppm | 3 | | | | | | | |
| | 4 | | | | | | | |
| | 5 | | | | | | | |
| | 6 | | | | | | | |
| Medium | 1 | | | | | | | |
| _____ ppm | 2 | | | | | | | |
| to _____ ppm | 3 | | | | | | | |
| | 4 | | | | | | | |
| | 5 | | | | | | | |
| | 6 | | | | | | | |
| High | 1 | | | | | | | |
| _____ ppm | 2 | | | | | | | |
| to _____ ppm | 3 | | | | | | | |
| | 4 | | | | | | | |
| | 5 | | | | | | | |
| | 6 | | | | | | | |
| | 7 | | | | | | | |
| | 8 | | | | | | | |
| | | | | | | | Total Failures: | |

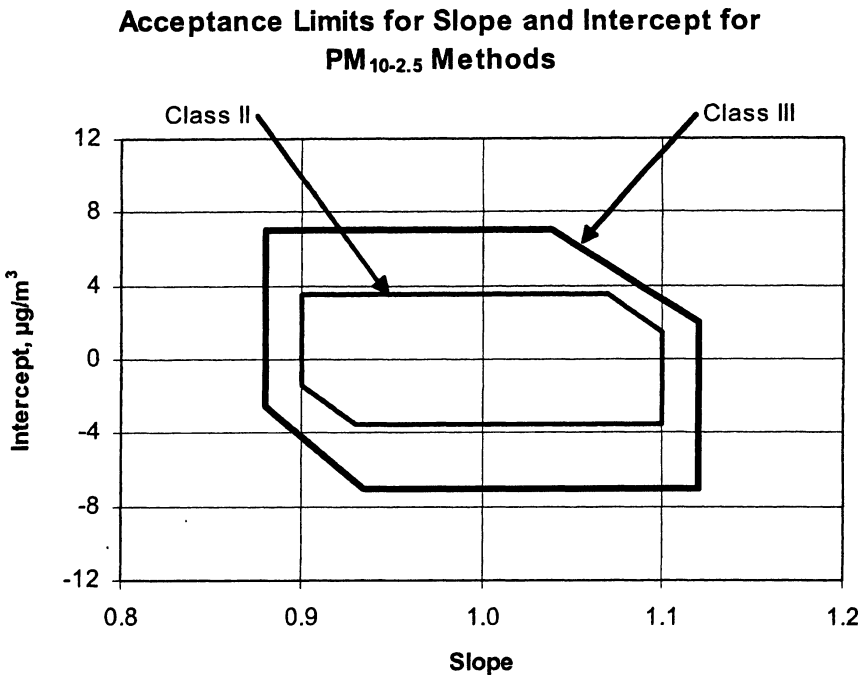
[72 FR 32204, June 12, 2007]

FIGURE C–2 TO SUBPART C OF PART 53—ILLUSTRATION OF THE SLOPE AND INTERCEPT LIMITS FOR CLASS II AND CLASS III $PM_{2.5}$ CANDIDATE EQUIVALENT METHODS



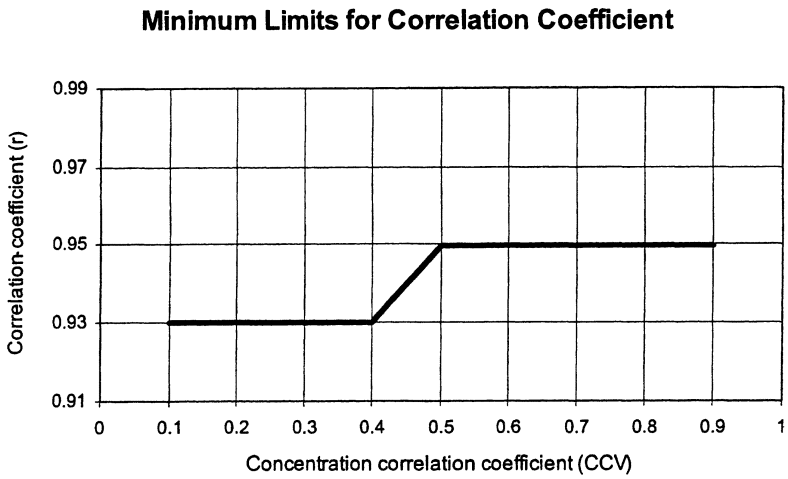

[72 FR 32204, June 12, 2007]

FIGURE C–3 TO SUBPART C OF PART 53—ILLUSTRATION OF THE SLOPE AND INTERCEPT LIMITS FOR CLASS II AND CLASS III $PM_{10-2.5}$ CANDIDATE EQUIVALENT METHODS




[72 FR 32204, June 12, 2007]

FIGURE C–4 TO SUBPART C OF PART 53—ILLUSTRATION OF THE MINIMUM LIMITS FOR CORRELATION COEFFICIENT FOR $PM_{2.5}$ AND $PM_{10-2.5}$ CLASS II AND III METHODS




[72 FR 32204, June 12, 2007]

APPENDIX A TO SUBPART C OF PART 53—REFERENCES

(1) 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*).

(2) 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/pmqainf.html*.

## Subpart D—Procedures for Testing Performance Characteristics of Methods for $PM_{10}$

SOURCE: 52 FR 24729, July 1, 1987, unless otherwise noted.

### §53.40 General provisions.

(a) The test procedures prescribed in this subpart shall be used to test the performance of candidate methods for $PM_{10}$ against the performance specifications given in table D–1. Except as provided in paragraph (b) of this section, a test sampler or samplers representative of the sampler described in the candidate method must exhibit performance better than, or equal to, the specified value for each performance parameter, to satisfy the requirements of this subpart.

(b) For a candidate method using a $PM_{10}$ sampler previously approved as part of a designated $PM_{10}$ method, only the test for precision need be conducted and passed to satisfy the requirements of this subpart. For a candidate method using a $PM_{10}$ sampler inlet previously approved as part of a designated $PM_{10}$ method, the tests for precision and flow rate stability must be conducted and passed to satisfy the requirements of this subpart; the tests for sampling effectiveness and 50 percent cutpoint need not be conducted if suitable rationale is provided to demonstrate that test results submitted for the previously approved method are applicable to the candidate method.

(c) The liquid particle sampling effectiveness and 50 percent cutpoint of a test sampler shall be determined in a wind tunnel using 10 particle sizes and three wind speeds as specified in table D–2. A minimum of 3 replicate measurements of sampling effectiveness shall be required for each of the 30 test

conditions for a minimum of 90 test measurements.

(d) For the liquid particle sampling effectiveness parameter, a smooth curve plot shall be constructed of sampling effectiveness (percent) versus aerodynamic particle diameter (μm) for each of the three wind speeds. These plots shall be used to calculate the expected mass concentration for the test sampler, using the procedure in § 53.43(a). The candidate method passes the liquid particle sampling effectiveness test if the expected mass concentration calculated for the test sampler at each wind speed differs by no more than ±10 percent from that predicted for the ''ideal'' sampler.*

(e) For the 50 percent cutpoint parameter, the test result for each wind speed shall be reported as the particle size at which the curve specified in § 53.40(d) crosses the 50 percent effectiveness line. The candidate method passes the 50 percent cutpoint test if the test result at each wind speed falls within 10±0.5 μm.

(f) The solid particle sampling effectiveness of a test sampler shall be determined in a wind tunnel using 25 μm particles at 2 wind speeds as specified in table D–2. A minimum of three replicate measurements of sampling effectiveness for the 25 μm solid particles shall be required at both wind speeds for a minimum of 6 test measurements.

(g) For the solid particle sampling effectiveness parameter, the test result for each wind speed shall be reported as the difference between the average of the replicate sampling effectiveness measurements obtained for the 25 μm solid particles and the average of the replicate measurements obtained for the 25 μm liquid particles. The candidate method passes the solid particle sampling effectiveness test if the test result for each wind speed is less than, or equal to, 5 percent.

(h) The precision and flow rate stability of three identical test samplers shall be determined at a suitable test site by simultaneously sampling the $PM_{10}$ concentration of the atmosphere for 10 periods of 24 hours.

(i) For the precision parameter, the test result for each of the 10 periods of 24 hours shall be calculated using the procedure in § 53.43(c). The candidate method passes the precision test if all of the test results meet the specifications in table D–1.

(j) For the flow rate stability parameter, the test results for each of the three test samplers and for each of the 10 periods of 24 hours shall be calculated using the procedure in § 53.43(d). The candidate method passes the flow rate stability test if all of the test results meet the specifications in table D–1.

(k) All 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 to EPA.

TABLE D–1—PERFORMANCE SPECIFICATIONS FOR $PM_{10}$ SAMPLERS

| Performance parameter | Units | Specification |
|---|---|---|
| 1. Sampling effectiveness: | | |
| A. Liquid particles | Percent | Such that the expected mass concentration is within ±10 percent of that predicted for the ideal sampler. |
| B. Solid particles | Percent | Sampling effectiveness is no more than 5 percent above that obtained for liquid particles of same size. |
| 2. 50 Percent cutpoint | μm | 10±μ.5 μm aerodynamic diameter. |
| 3. Precision | μg/m³ or percent | 5 μg/m³ or 7 percent for three collocated samplers. |
| 4. Flow rate stability | Percent | Average flow rate over 24 hours within ±5 percent of initial flow rate; all measured flow rates over 24 hours within ±10 percent of initial flow rate. |

*The sampling effectiveness curve for this ''ideal'' sampler is described by column 5 of table D–3 and is based on a model that approximates the penetration of particles into the human respiratory tract. Additional information on this model may be found in a document entitled, ''Particle Collection Criteria for 10 Micrometer Samplers,'' which is available from the Quality Assurance Division (MD–77), Environmental Monitoring Systems Laboratory, U.S. Environmental Protection Agency, Research Triangle Park, NC 27711.

### § 53.41 Test conditions.

(a) Set-up and start-up of all test samplers shall be in strict accordance with the operating instructions specified in the manual referred to in § 53.4(b)(3).

(b) If the internal surface or surfaces of the candidate method's sampler inlet on which the particles removed by the inlet are collected is a dry surface (i.e., not normally coated with oil or grease), those surfaces shall be cleaned prior to conducting wind tunnel tests with solid particles.

(c) Once the test sampler or samplers have been set up and the performance tests started, manual adjustment shall be permitted only between test points for the sampling effectiveness and 50 percent cutpoint tests or between test days for the precision and flow rate stability tests. The manual adjustments and any periodic maintenance shall be limited to only those procedures prescribed in the manual referred to in § 53.4(b)(3). The submitted records shall show clearly when any manual adjustment or periodic maintenance was made and shall describe the operations performed.

(d) If a test sampler malfunctions during any of the sampling effectiveness and 50 percent cutpoint tests, that test run shall be repeated. If a test sampler malfunctions during any of the precision and flow rate stability tests, that day's test shall be repeated. A detailed explanation of all malfunctions and the remedial actions taken shall be submitted to EPA with the application.

### § 53.42 Generation of test atmospheres for wind tunnel tests.

(a) A vibrating orifice aerosol generator shall be used to produce monodispersed liquid particles of oleic acid tagged with uranine dye and monodispersed solid particles of ammonium fluoroscein with equivalent aerodynamic diameters as specified in table D–2. The geometric standard deviation for each particle size and type generated shall not exceed 1.1 (for primary particles) and the proportion of multiplets (doublets and triplets) in a test particle atmosphere shall not exceed 10 percent. The particle delivery system shall consist of a blower system and a wind tunnel having a test section of sufficiently large cross-sectional area such that the test sampler, or portion thereof, as installed in the test section for testing, blocks no more than 15 percent of that area. To be acceptable, the blower system must be capable of achieving uniform wind speeds at the speeds specified in table D–2.

TABLE D–2—PARTICLE SIZES AND WIND SPEEDS FOR SAMPLING EFFECTIVENESS TESTS

| Particle size (μm) [a] | Wind speed (km/hr) | | |
|---|---|---|---|
| | 2 | 8 | 24 |
| 3±0.5 | *l* | *l* | *l* |
| 5±0.5 | *l* | *l* | *l* |
| 7±0.5 | *l* | *l* | *l* |
| 9±0.5 | *l* | *l* | *l* |
| 10±0.5 | *l* | *l* | *l* |
| 11±0.5 | *l* | *l* | *l* |
| 13±1.0 | *l* | *l* | *l* |
| 15±1.0 | *l* | *l* | *l* |
| 20±1.0 | *l* | *l* | *l* |
| 25±1.0 | *l* | *l*/s | *l*/s |

[a] Mass median aerodynamic diameter.
*l* = liquid particle.
s = solid particle.
Number of liquid particle test points (minimum of 3 replicates for each combination of particle size and wind speed): 90.
Number of solid particle test points (minimum of 3 replicates for each combination of particle size and wind speed): 6.
Total number of test points: 96.

(b) The size of the test particles delivered to the test section of the wind tunnel shall be established using the operating parameters of the vibrating orifice aerosol generator and shall be verified during the tests by microscopic examination of samples of the particles collected on glass slides or other suitable substrates. When sizing liquid particles on glass slides, the slides should be pretreated with an oleophobic surfactant and an appropriate flattening factor shall be used in the calculation of aerodynamic diameter. The particle size, as established by the operating parameters of the vibrating orifice aerosol generator, shall be within the tolerance specified in table D–2. The precision of the particle size verification technique shall be 0.5 μm or better, and particle size determined by the verification technique shall not differ by more than 0.5 μm or 10 percent, whichever is higher, from that established by the operating parameters of the vibrating orifice aerosol generator.

(c) The population of multiplets in a test particle atmosphere shall be determined during the tests and shall not exceed 10 percent. Solid particles shall be checked for dryness and evidence of breakage or agglomeration during the microscopic examination. If the solid particles in a test atmosphere are wet or show evidence of significant breakage or agglomeration (µ5 percent), the solid particle test atmosphere is unacceptable for purposes of these tests.

(d) The concentration of particles in the wind tunnel is not critical. However, the cross-sectional uniformity of the particle concentration in the sampling zone of the test section shall be established during the tests using isokinetic samplers. An array of not less than five evenly spaced isokinetic samplers shall be used to determine the particle concentration uniformity in the sampling zone. If the particle concentration measured by any single isokinetic sampler in the sampling zone differs by more than 10 percent from the mean concentration, the particle delivery system is unacceptable in terms of uniformity of particle concentration. The sampling zone shall be a rectangular area having a horizontal dimension not less than 1.2 times the width of the test sampler at its inlet opening and a vertical dimension not less than 25 centimeters. The sampling zone is an area in the test section of the wind tunnel that is horizontally and vertically symmetrical with respect to the test sampler inlet opening.

(e) The wind speed in the wind tunnel shall be determined during the tests using an appropriate technique capable of a precision of 5 percent or better (e.g., hot-wire anemometry). The mean wind speed in the test section of the wind tunnel during the tests shall be within 10 percent of the value specified in table D–2. The wind speed measured at any test point in the test section shall not differ by more than 10 percent from the mean wind speed in the test section. The turbulence intensity (longitudinal component and macroscale) in the test section shall be determined during the tests using an appropriate technique (e.g., hot-wire anemometry).

(f) The accuracy of all flow measurements used to calculate the test atmosphere concentrations and the test results shall be documented to be within ±2 percent, referenced to a primary standard. Any flow measurement corrections shall be clearly shown. All flow measurements shall be given in actual volumetric units.

(g) Schematic drawings of the particle delivery system (wind tunnel and blower system) and other information showing complete procedural details of the test atmosphere generation, verification, and delivery techniques shall be submitted to EPA. All pertinent calculations shall be clearly presented.

### § 53.43 Test procedures.

(a) *Sampling effectiveness*—(1) *Technical definition.* The ratio (expressed as a percentage) of the mass concentration of particles of a given size reaching the sampler filter or filters to the mass concentration of particles of the same size approaching the sampler.

(2) *Test procedure.* (i) Establish a wind speed specified in table D–2 and measure the wind speed and turbulence intensity (longitudinal component and macroscale) at a minimum of 12 test points in a cross-sectional area of the test section of the wind tunnel. The mean wind speed in the test section must be within ±10 percent of the value specified in table D–2 and the variation at any test point in the test section may not exceed 10 percent of the mean.

(ii) Generate particles of a size and type specified in table D–2 using a vibrating orifice aerosol generator. Check for the presence of satellites and adjust the generator as necessary. Calculate the aerodynamic particle size using the operating parameters of the vibrating orifice aerosol generator and record. The calculated aerodynamic diameter must be within the tolerance specified in table D–2.

(iii) Collect a sample of the particles on a glass slide or other suitable substrate at the particle injection point. If a glass slide is used, it should be pretreated with an appropriate oleophobic surfactant when collecting liquid particles. Use a microscopic technique to size a minimum of 25 primary particles in three viewing fields (do not include multiplets). Determine

the geometric mean aerodynamic diameter and geometric standard deviation using the bulk density of the particle type (and an appropriate flattening factor for liquid particles if collected on a glass slide). The measured geometric mean aerodynamic diameter must be within 0.5 μm or 10 percent of the aerodynamic diameter calculated from the operating parameters of the vibrating orifice aerosol generator. The geometric standard deviation must not exceed 1.1.

(iv) Determine the population of multiplets (doublets and triplets) in the collected sample by counting a minimum of 100 particles in three viewing fields. The multiplet population of the particle test atmosphere must not exceed 10 percent.

(v) Introduce the particles into the wind tunnel and allow the particle concentration to stabilize.

(vi) Install an array of five or more evenly spaced isokinetic samplers in the sampling zone (see §53.42(d)) of the wind tunnel. Collect particles on appropriate filters (e.g., glass fiber) over a time period such that the relative error of the measured particle concentration is less than 5 percent. Relative error is defined as (p × 100%)/(X), where p is the precision of the fluorometer on the appropriate range, X is the measured concentration, and the units of p and X are the same.

(vii) Determine the quantity of material collected with each isokinetic sampler in the array using a calibrated fluorometer. Calculate and record the mass concentration for each isokinetic sampler as:

$$C_{iso(ij)} = \frac{\text{mass of material collected with isokinetic sampler}}{\text{sample flow rate} \times \text{sampling time}}$$

where

i = replicate number and j = isokinetic sampler number.

(viii) Calculate and record the mean mass concentration as:

$$C_{iso(i)} = \frac{\sum_{j=1}^{n} C_{iso(ij)}}{n}$$

where

n = total number of isokinetic samplers.

(ix) Calculate and record the coefficient of variation of the mass concentration measurements as:

$$CV_{iso(i)} = \sqrt{\frac{\sum_{j=1}^{n} C^2 iso(ij) - \sum_{j=1}^{n} C_{iso(ij)}^{2/n}}{n-1}} \Big/ \overline{C}_{iso(i)}$$

If the value of $CV_{iso(i)}$ exceeds 0.10, the particle concentration uniformity is unacceptable and steps (vi) through (ix) must be repeated. If adjustment of the vibrating orifice aerosol generator or changes in the particle delivery system are necessary to achieve uniformity, steps (ii) through (ix) must be repeated. Remove the array of isokinetic samplers from the wind tunnel. NOTE: A single isokinetic sampler, operated at the same nominal flow rate as the test sampler, may be used in place of the array of isokinetic samplers for the determination of particle mass concentration used in the calculation of sampling effectiveness of the test sampler in step (xiii). In this case, the

array of isokinetic samplers must be used to demonstrate particle concentration uniformity prior to the replicate measurements of sampling effectiveness.

(x) If a single isokinetic sampler is used, install the sampler in the wind tunnel with the sampler nozzle centered in the sampling zone (see § 53.42(d)). Collect particles on an appropriate filter (e.g., glass fiber) for a time period such that the relative error of the measured concentration (as defined in step (vi)) is less than 5 percent. Determine the quantity of material collected with the isokinetic sampler using a calibrated fluorometer. Calculate and record the mass concentration as $C_{iso(i)}$ as in step vii. Remove the isokinetic sampler from the wind tunnel.

(xi) Install the test sampler (or portion thereof) in the wind tunnel with the sampler inlet opening centered in the sampling zone (see § 53.42(d)). To meet the maximum blockage limit of § 53.42(a) or for convenience, part of the test sampler may be positioned external to the wind tunnel provided that neither the geometry of the sampler nor the length of any connecting tube or pipe is altered. Collect particles on an appropriate filter or filters (e.g., glass fiber) for a time period such that the relative error of the measured concentration (as defined in step (vi)) is less than 5 percent.

(xii) Determine the quantity of material collected with the test sampler using a calibrated fluorometer. Calculate and record the mass concentration as:

$$C_{sam(i)} = \frac{\text{mass of material collected with test sampler}}{\text{sample flow rate} \times \text{sampling time}}$$

where i = replicate number.

(xiii) Calculate and record the sampling effectiveness of the test sampler as:

$$E_{(i)} = \frac{C_{sam(i)}}{C_{iso(i)}} \times 100\%$$

where i = replicate number.

NOTE: If a single isokinetic sampler is used for the determination of particle mass concentration, replace $C_{iso(i)}$ with $C_{iso(i)}$.

(xiv) Remove the test sampler from the wind tunnel. Repeat steps (vi) through (xiii), as appropriate, to obtain a minimum of three replicate measurements of sampling effectiveness.

(xv) Calculate and record the average sampling effectiveness of the test sampler as:

$$\overline{E} = \frac{\sum_{i=1}^{n} E_{(i)}}{n}$$

where n = number of replicates.

(xvi) Calculate and record the coefficient of variation for the replicate sampling effectiveness measurements of the test sampler as:

$$CV_E = \sqrt{\frac{\sum_{i=1}^{n} E^2{}_{(i)} - \left(\sum_{i=1}^{n} E_{(i)}\right)^{2/n}}{n-1}} \Big/ \overline{E}$$

If the value of $CV_E$ exceeds 0.10, the test run (steps (ii) through (xvi)) must be repeated.

(xvii) Repeat steps i through xvi for each wind speed, particle size, and particle type specified in table D–2.

(xviii) For each of the three wind speeds (nominally 2, 8, and 24 km/hr), correct the liquid particle sampling effectiveness data for the presence of multiplets (doublets and triplets) in the test particle atmospheres.

(xix) For each wind speed, plot the corrected liquid particle sampling effectiveness of the test sampler ($E_{corr}$) as a function of particle size ($d_p$) on semilogarithmic graph paper where $d_p$ is the particle size established by the operating parameters of the vibrating orifice aerosol generator. Construct a smooth curve through the data.

(xx) For each wind speed, calculate the expected mass concentration for the test sampler under the assumed particle size distribution and compare it to the mass concentration predicted for the ideal sampler, as follows:

(A) Extrapolate the upper and lower ends of the corrected liquid particle sampling effectiveness curve to 100 percent and 0 percent, respectively, using smooth curves. Assume that $E_{corr}$ = 100 percent at a particle size of 1.0 µm and $E_{corr}$ = 0 percent at a particle size of 50 µm.

(B) Determine the value of $E_{corr}$ at each of the particle sizes specified in the first column of table D–3. Record each $E_{corr}$ value as a decimal between 0 and 1 in the second column of table D–3.

(C) Multiply the values of $E_{corr}$ in column 2 by the interval mass concentration values in column 3 and enter the products in column 4 of table D–3.

(D) Sum the values in column 4 and enter the total as the expected mass concentration for the test sampler at the bottom of column 4 of table D–3.

(E) Calculate and record the percent difference in expected mass concentration between the test sampler and the ideal sampler as:

$$\Delta C = \frac{C_{sam(exp)} - C_{ideal(exp)}}{C_{ideal(exp)}} \times 100\%$$

where:

$C_{sam(exp)}$ = expected mass concentration for the test sampler, µg/m³

$C_{ideal(exp)}$ = expected mass concentration for the ideal sampler, µg/m³ (calculated for the ideal sampler and given at the bottom of column 7 of table D–3.)

(F) The candidate method passes the liquid particle sampling effectiveness test if the Δ C value for each wind speed meets the specification in table D–1.

(xxi) For each of the two wind speeds (nominally 8 and 24 km/hr), calculate the difference between the average sampling effectiveness value for the 25 µm solid particles and the average sampling effectiveness value for the 25 µm liquid particles (uncorrected for multiplets).

(xxii) The candidate method passes the solid particle sampling effectiveness test if each such difference meets the specification in table D–1.

TABLE D–3—EXPECTED MASS CONCENTRATION FOR $PM_{10}$ SAMPLERS

| Particle size (um) | Test sampler | | | Ideal Sampler | | |
|---|---|---|---|---|---|---|
| | Sampling effectiveness | Interval mass concentration (µg/m³) | Expected mass concentration (µg/m³) | Sampling effectiveness | Interval mass concentration (µg/m³) | Expected mass concentration (µg/m³) |
| (1) | (2) | (3) | (4) | (5) | (6) | (7) |
| <1.0 | 1.000 | 62.813 | 62.813 | 1.000 | 62.813 | 62.813 |
| 1.5 | | 9.554 | | 0.949 | 9.554 | 9.067 |
| 02.0 | | 2.164 | | 0.942 | 2.164 | 2.038 |
| 02.5 | | 1.785 | | 0.933 | 1.785 | 1.665 |
| 03.0 | | 2.084 | | 0.922 | 2.084 | 1.921 |
| 03.5 | | 2.618 | | 0.909 | 2.618 | 2.380 |
| 04.0 | | 3.211 | | 0.893 | 3.211 | 2.867 |
| 04.5 | | 3.784 | | 0.876 | 3.784 | 3.315 |
| 05.0 | | 4.300 | | 0.857 | 4.300 | 3.685 |
| 05.5 | | 4.742 | | 0.835 | 4.742 | 3.960 |
| 06.0 | | 5.105 | | 0.812 | 5.105 | 4.145 |
| 06.5 | | 5.389 | | 0.786 | 5.389 | 4.236 |
| 07.0 | | 5.601 | | 0.759 | 5.601 | 4.251 |
| 07.5 | | 5.746 | | 0.729 | 5.746 | 4.189 |
| 08.0 | | 5.834 | | 0.697 | 5.834 | 4.066 |
| 08.5 | | 5.871 | | 0.664 | 5.871 | 3.898 |
| 09.0 | | 5.864 | | 0.628 | 5.864 | 3.683 |
| 09.5 | | 5.822 | | 0.590 | 5.822 | 3.435 |
| 10.0 | | 5.750 | | 0.551 | 5.750 | 3.168 |
| 10.5 | | 5.653 | | 0.509 | 5.653 | 2.877 |
| 11.0 | | 8.257 | | 0.465 | 8.257 | 3.840 |
| 12.0 | | 10.521 | | 0.371 | 10.521 | 3.903 |
| 13.0 | | 9.902 | | 0.269 | 9.902 | 2.664 |
| 14.0 | | 9.250 | | 0.159 | 9.250 | 1.471 |
| 15.0 | | 8.593 | | 0.041 | 8.593 | 0.352 |
| 16.0 | | 7.948 | | 0.000 | 7.948 | 0.000 |

TABLE D–3—EXPECTED MASS CONCENTRATION FOR $PM_{10}$ SAMPLERS—Continued

| Particle size (um) | Test sampler | | | Ideal Sampler | | |
|---|---|---|---|---|---|---|
| | Sampling effectiveness | Interval mass concentration (μg/m³) | Expected mass concentration (μg/m³) | Sampling effectiveness | Interval mass concentration (μg/m³) | Expected mass concentration (μg/m³) |
| (1) | (2) | (3) | (4) | (5) | (6) | (7) |
| 17.0 | | 7.329 | | 0.000 | 7.329 | 0.000 |
| 18.0 | | 9.904 | | 0.000 | 9.904 | 0.000 |
| 20.0 | | 11.366 | | 0.000 | 11.366 | 0.000 |
| 22.0 | | 9.540 | | 0.000 | 9.540 | 0.000 |
| 24.0 | | 7.997 | | 0.000 | 7.997 | 0.000 |
| 26.0 | | 6.704 | | 0.000 | 6.704 | 0.000 |
| 28.0 | | 5.627 | | 0.000 | 5.627 | 0.000 |
| 30.0 | | 7.785 | | 0.000 | 7.785 | 0.000 |
| 35.0 | | 7.800 | | 0.000 | 7.800 | 0.000 |
| 40.0 | | 5.192 | | 0.000 | 5.192 | 0.000 |
| 45.0 | | 4.959 | | 0.000 | 4.959 | 0.000 |
| | | $C_{sam(exp)}$ = D | | | $C_{ideal(exp)}$ = | 143.889 |

(b) *50 Percent cutpoint*—(1) *Technical definition*. The particle size for which the sampling effectiveness of the sampler is 50 percent.

(2) *Test procedure*. (i) From the corrected liquid particle sampling effectiveness curves for each of the three wind speeds, determine the particle size at which the curve crosses the 50 percent effectiveness line and record as $D_{50}$ on the corresponding sampling effectiveness plot.

(ii) The candidate method passes the 50 percent cutpoint test if the $D_{50}$ value at each wind speed meets the specification in table D–1.

(c) *Precision*—(1) *Technical definition*. The variation in the measured particle concentration among identical samplers under typical sampling conditions.

(2) *Test procedure*. (i) Set up three identical test samplers at the test site in strict accordance with the instructions in the manual referred to in § 53.4(b)(3). Locate the test sampler inlet openings at the same height and between 2 and 4 meters apart. The samplers shall be oriented in a manner that will minimize spatial and wind directional effects on sample collection. Perform a flow calibration for each test sampler in accordance with the instructions given in the instruction manual and/or appendix J to part 50 of this chapter. Set the operating flow rate to the value prescribed in the sampler instruction manual.

NOTE: For candidate equivalent methods, this test may be used to satisfy part of the requirements of subpart C of this chapter. In that case, three reference method samplers are also used at the test site, measurements with the candidate and reference methods are compared as specified in § 53.34, and the test site must meet the requirements of § 53.30(b).

(ii) Measure the $PM_{10}$ concentration of the atmosphere using the three test samplers for 10 periods (test days) of 24 hours each. On each of the 10 test days, measure the initial and final flow rates of each test sampler. On three of the test days, measure the flow rate of each test sampler after 6, 12, and 18 hours of operation. All measurements of flow rate and mass collected must be made in accordance with the procedures prescribed in the sampler instruction manual and/or appendix J to part 50 of this chapter. All measurements of flow rate must be in actual volumetric units. Record the $PM_{10}$ concentration for each sampler and each test day as $C_{(i)(j)}$ where i is the sampler number and j is the test day.

(iii) For each test day, calculate and record the average of the three measured $PM_{10}$ concentrations as $C_{(j)}$ where j is the test day. If $C_{(j)}$<30 μg/m³ for any test day, data from that test day are unacceptable and the tests for that day must be repeated.

(iv) Calculate and record the precision for each of the 10 test days as:

$$P_j = \sqrt{\frac{\sum_{i=1}^{3} C^2_{(i)(j)} - \left(\sum_{i=1}^{3} C_{(i)(j)}\right)^{2/3}}{2}}$$

if $\overline{C}j$ is below 80 $\mu g / m^3$, or

$$RP_j = 100\% \times \sqrt{\frac{\sum_{i=1}^{3} C^2_{(i)(j)} - \left(\sum_{i=1}^{3} C_{(i)(j)}\right)^{2/3}}{2}} \quad \overline{C}_{(j)}$$

if $\overline{C}j$ is above 80 $\mu g / m^3$.

(v) The candidate method passes the precision test if all 10 $P_j$ or $RP_j$ values meet the specifications in table D–1.

(d) *Flow rate stability*—(1) *Technical definition*. Freedom from variation in the operating flow rate of the sampler under typical sampling conditions.

(2) *Test procedure*. (i) For each of the three test samplers and each of the 10 test days of the precision test, record each measured flow rate as $F_{(i)(j)(t)}$, where i is the sampler number, j is the test day, and t is the time of flow rate measurement (t = 0, 6, 12, 18, or 24 hours).

(ii) For each sampler and for each test day, calculate and record the average flow rate as:

$$\overline{F}_{(i)(j)} = \frac{\sum_{t=0}^{24} F_{(i)(j)(t)}}{n}$$

where n = number of flow rate measurements during the 24-hour test day.

(iii) For each sampler and for each test day, calculate and record the percent difference between the average flow rate and the initial flow rate as:

$$\Delta F_{(i)(j)} = \frac{\overline{F}_{(i)(j)} - F_{(i)(j)(0)}}{F_{(i)(j)(0)}} \times 100\%$$

where $F_{(i)(j)(0)}$ is the initial flow rate (t = 0).

(iv) For each sampler and for each of the 3 test days on which flow measurements were obtained at 6-hour intervals throughout the 24-hour sampling period, calculate and record the percent differences between each measured flow rate and the initial flow rate as:

$$\Delta F_{(i)(j)(t)} = \frac{F_{(i)(j)} - F_{(i)(j)(0)}}{F_{(i)(j)(0)}} \times 100\%$$

where t = 6, 12, 18, or 24 hours.

(v) The candidate method passes the flow rate stability test if all of the $\Delta F_{(i)(j)}$ and $\Delta F_{(i)(j)(t)}$ values meet the specifications in table D–1.

## Subpart E—Procedures for Testing Physical (Design) and Performance Characteristics of Reference Methods and Class I and Class II Equivalent Methods for $PM_{2.5}$ or $PM_{10-2.5}$

SOURCE: 62 FR 38799, July 18, 1997, unless otherwise noted.

### § 53.50 General provisions.

(a) A candidate method for $PM_{2.5}$ or $PM_{10-2.5}$ described in an application for a FRM or FEM determination submitted under § 53.4 shall be determined by the EPA to be a FRM or a Class I, II, or III FEM on the basis of the definitions for such methods given in § 53.1. This subpart sets forth the specific tests that must be carried out and the test results, evidence, documentation,

and other materials that must be provided to EPA to demonstrate that a $PM_{2.5}$ or $PM_{10-2.5}$ sampler associated with a candidate reference method or Class I or Class II equivalent method meets all design and performance specifications set forth in appendix L or O, respectively, of part 50 of this chapter as well as additional requirements specified in this subpart E. Some or all of these tests may also be applicable to a candidate Class III equivalent method or analyzer, as may be determined under § 53.3(b)(3).

(b) *$PM_{2.5}$ methods*—(1) *Reference method.* A sampler associated with a candidate reference method for $PM_{2.5}$ shall be subject to the provisions, specifications, and test procedures prescribed in §§ 53.51 through 53.58.

(2) *Class I method.* A sampler associated with a candidate Class I equivalent method for $PM_{2.5}$ shall be subject to the provisions, specifications, and test procedures prescribed in all sections of this subpart.

(3) *Class II method.* A sampler associated with a candidate Class II equivalent method for $PM_{2.5}$ shall be subject to the provisions, specifications, and test procedures prescribed in all applicable sections of this subpart, as specified in subpart F of this part or as specified in § 53.3(a)(3).

(c) *$PM_{10-2.5}$ methods*—(1) *Reference method.* A sampler associated with a reference method for $PM_{10-2.5}$, as specified in appendix O to part 50 of this chapter, shall be subject to the requirements in this paragraph (c)(1).

(i) The $PM_{2.5}$ sampler of the $PM_{10-2.5}$ sampler pair shall be verified to be either currently designated under this part 53 as a FRM for $PM_{2.5}$, or shown to meet all requirements for designation as a FRM for $PM_{2.5}$, in accordance with this part 53.

(ii) The $PM_{10C}$ sampler of the $PM_{10-2.5}$ sampler pair shall be verified to be of like manufacturer, design, configuration, and fabrication to the $PM_{2.5}$ sampler of the $PM_{10-2.5}$ sampler pair, except for replacement of the particle size separator specified in section 7.3.4 of appendix L to part 50 of this chapter with the downtube extension as specified in Figure O–1 of appendix O to part 50 of this chapter.

(iii) For samplers that meet the provisions of paragraphs (c)(1)(i) and (ii) of this section, the candidate $PM_{10-2.5}$ reference method may be determined to be a FRM without further testing.

(2) *Class I method.* A sampler associated with a Class I candidate equivalent method for $PM_{10-2.5}$ shall meet the requirements in this paragraph (c)(2).

(i) The $PM_{2.5}$ sampler of the $PM_{10-2.5}$ sampler pair shall be verified to be either currently designated under this part 53 as a FRM or Class I FEM for $PM_{2.5}$, or shown to meet all requirements for designation as a FRM or Class I FEM for $PM_{2.5}$, in accordance with this part 53.

(ii) The $PM_{10c}$ sampler of the $PM_{10-2.5}$ sampler pair shall be verified to be of similar design to the $PM_{10-2.5}$ sampler and to meet all requirements for designation as a FRM or Class I FRM for $PM_{2.5}$, in accordance with this part 53, except for replacement of the particle size separator specified in section 7.3.4 of appendix L to part 50 of this chapter with the downtube extension as specified in Figure O–1 of appendix O to part 50 of this chapter.

(iii) For samplers that meet the provisions of paragraphs (c)(2)(i) and (ii) of this section, the candidate $PM_{10-2.5}$ method may be determined to be a Class I FEM without further testing.

(3) *Class II method.* A sampler associated with a Class II candidate equivalent method for $PM_{10-2.5}$ shall be subject to the applicable requirements of this subpart E, as described in § 53.3(a)(5).

(d) The provisions of § 53.51 pertain to test results and documentation required to demonstrate compliance of a candidate method sampler with the design specifications set forth in 40 CFR part 50, appendix L or O, as applicable. The test procedures prescribed in §§ 53.52 through 53.59 pertain to performance tests required to demonstrate compliance of a candidate method sampler with the performance specifications set forth in 40 CFR part 50, appendix L or O, as applicable, as well as additional requirements specified in this subpart E. These latter test procedures shall be used to test the performance of candidate samplers against the performance specifications and requirements specified in each procedure and

summarized in table E–1 of this subpart.

(e) Test procedures prescribed in § 53.59 do not apply to candidate reference method samplers. These procedures apply primarily to candidate Class I or Class II equivalent method samplers for $PM_{2.5}$ or $PM_{10-2.5}$ that have a sample air flow path configuration upstream of the sample filter that is modified from that specified for the FRM sampler, as set forth in 40 CFR part 50, appendix L, Figures L–1 to L–29 or 40 CFR part 50 appendix O, Figure O–1, if applicable, such as might be necessary to provide for sequential sample capability. The additional tests determine the adequacy of aerosol transport through any altered components or supplemental devices that are used in a candidate sampler upstream of the filter. In addition to the other test procedures in this subpart, these test procedures shall be used to further test the performance of such an equivalent method sampler against the performance specifications given in the procedure and summarized in table E–1 of this subpart.

(f) A 10-day operational field test of measurement precision is required under § 53.58 for both FRM and Class I FEM samplers for $PM_{2.5}$. This test requires collocated operation of three candidate method samplers at a field test site. For candidate FEM samplers, this test may be combined and carried out concurrently with the test for comparability to the FRM specified under § 53.34, which requires collocated operation of three FRM samplers and three candidate FEM samplers.

(g) All tests and collection of test data shall be performed in accordance with the requirements of reference 1, section 4.10.5 (ISO 9001) and reference 2, part B, (section 6) and Part C, (section 7) in appendix A of this subpart. All test data and other documentation obtained specifically from or pertinent to these tests shall be identified, dated, signed by the analyst performing the test, and submitted to EPA in accordance with subpart A of this part.

[71 FR 61289, Oct. 17, 2006]

**§ 53.51 Demonstration of compliance with design specifications and manufacturing and test requirements.**

(a) *Overview*. (1) Paragraphs (a) through (f) of this section specify certain documentation that must be submitted and tests that are required to demonstrate that samplers associated with a designated FRM or FEM for $PM_{2.5}$ or $PM_{10-2.5}$ are properly manufactured to meet all applicable design and performance specifications and have been properly tested according to all applicable test requirements for such designation. Documentation is required to show that instruments and components of a $PM_{2.5}$ or $PM_{10-2.5}$ sampler are manufactured in an ISO 9001-registered facility under a quality system that meets ISO–9001 requirements for manufacturing quality control and testing.

(2) In addition, specific tests are required by paragraph (d) of this section to verify that critical features of FRM samplers—the particle size separator and the surface finish of surfaces specified to be anodized—meet the specifications of 40 CFR part 50, appendix L or appendix O, as applicable. A checklist is required to provide certification by an ISO-certified auditor that all performance and other required tests have been properly and appropriately conducted, based on a reasonable and appropriate sample of the actual operations or their documented records. Following designation of the method, another checklist is required initially to provide an ISO-certified auditor's certification that the sampler manufacturing process is being implemented under an adequate and appropriate quality system.

(3) For the purposes of this section, the definitions of ISO 9001-registered facility and ISO-certified auditor are found in § 53.1. An exception to the reliance by EPA on ISO-certified auditors is the requirement for the submission of the operation or instruction manual associated with the candidate method to EPA as part of the application. This manual is required under § 53.4(b)(3). The EPA has determined that acceptable technical judgment for review of this manual may not be assured by ISO-certified auditors, and approval of this manual will therefore be performed by EPA.

(b) *ISO registration of manufacturing facility*. The applicant must submit documentation verifying that the samplers identified and sold as part of a designated $PM_{2.5}$ or $PM_{10-2.5}$ FRM or FEM will be manufactured in an ISO 9001-registered facility and that the manufacturing facility is maintained in compliance with all applicable ISO 9001 requirements (reference 1 in appendix A of this subpart). The documentation shall indicate the date of the original ISO 9001 registration for the facility and shall include a copy of the most recent certification of continued ISO 9001 facility registration. If the manufacturer does not wish to initiate or complete ISO 9001 registration for the manufacturing facility, documentation must be included in the application to EPA describing an alternative method to demonstrate that the facility meets the same general requirements as required for registration to ISO–9001. In this case, the applicant must provide documentation in the application to demonstrate, by required ISO-certified auditor's inspections, that a quality system is in place which is adequate to document and monitor that the sampler system components and final assembled samplers all conform to the design, performance and other requirements specified in this part and in 40 CFR part 50, appendix L.

(c) *Sampler manufacturing quality control*. The manufacturer must ensure that all components used in the manufacture of $PM_{2.5}$ or $PM_{10-2.5}$ samplers to be sold as part of a FRM or FEM and that are specified by design in 40 CFR part 50, appendix L or O (as applicable), are fabricated or manufactured exactly as specified. If the manufacturer's quality records show that its quality control (QC) and quality assurance (QA) system of standard process control inspections (of a set number and frequency of testing that is less than 100 percent) complies with the applicable QA provisions of section 4 of reference 4 in appendix A of this subpart and prevents nonconformances, 100 percent testing shall not be required until that conclusion is disproved by customer return or other independent manufacturer or customer test records. If problems are uncovered, inspection to verify conformance to the drawings, specifications, and tolerances shall be performed. Refer also to paragraph (e) of this section—final assembly and inspection requirements.

(d) *Specific tests and supporting documentation required to verify conformance to critical component specifications*—(1) *Verification of $PM_{2.5}$ (WINS) impactor jet diameter*. For samplers utilizing the WINS impactor particle size separator specified in paragraphs 7.3.4.1, 7.3.4.2, and 7.3.4.3 of appendix L to part 50 of this chapter, the diameter of the jet of each impactor manufactured for a $PM_{2.5}$ or $PM_{10-2.5}$ sampler under the impactor design specifications set forth in 40 CFR part 50, appendix L, shall be verified against the tolerance specified on the drawing, using standard, NIST-traceable ZZ go/no go plug gages. This test shall be a final check of the jet diameter following all fabrication operations, and a record shall be kept of this final check. The manufacturer shall submit evidence that this procedure is incorporated into the manufacturing procedure, that the test is or will be routinely implemented, and that an appropriate procedure is in place for the disposition of units that fail this tolerance test.

(2) *VSCC separator*. For samplers utilizing the BGI VSCC™ Very Sharp Cut Cyclone particle size separator specified in paragraph 7.3.4.4 of appendix L to part 50 of this chapter, the VSCC manufacturer shall identify the critical dimensions and manufacturing tolerances for the device, develop appropriate test procedures to verify that the critical dimensions and tolerances are maintained during the manufacturing process, and carry out those procedures on each VSCC manufactured to verify conformance of the manufactured products. The manufacturer shall also maintain records of these tests and their results and submit evidence that this procedure is incorporated into the manufacturing procedure, that the test is or will be routinely implemented, and that an appropriate procedure is in place for the disposition of units that fail this tolerance test.

(3) *Verification of surface finish*. The anodization process used to treat surfaces specified to be anodized shall be verified by testing treated specimen

surfaces for weight and corrosion resistance to ensure that the coating obtained conforms to the coating specification. The specimen surfaces shall be finished in accordance with military standard specification 8625F, Type II, Class I (reference 4 in appendix A of this subpart) in the same way the sampler surfaces are finished, and tested, prior to sealing, as specified in section 4.5.2 of reference 4 in appendix A of this subpart.

(e) *Final assembly and inspection requirements.* Each sampler shall be tested after manufacture and before delivery to the final user. Each manufacturer shall document its post-manufacturing test procedures. As a minimum, each test shall consist of the following: Tests of the overall integrity of the sampler, including leak tests; calibration or verification of the calibration of the flow measurement device, barometric pressure sensor, and temperature sensors; and operation of the sampler with a filter in place over a period of at least 48 hours. The results of each test shall be suitably documented and shall be subject to review by an ISO-certified auditor.

(f) *Manufacturer's audit checklists.* Manufacturers shall require an ISO-certified auditor to sign and date a statement indicating that the auditor is aware of the appropriate manufacturing specifications contained in 40 CFR part 50, appendix L or O (as applicable), and the test or verification requirements in this subpart. Manufacturers shall also require an ISO-certified auditor to complete the checklists, shown in figures E–1 and E–2 of this subpart, which describe the manufacturer's ability to meet the requirements of the standard for both designation testing and product manufacture.

(1) *Designation testing checklist.* The completed statement and checklist as shown in figure E–1 of this subpart shall be submitted with the application for FRM or FEM determination.

(2) *Product manufacturing checklist.* Manufacturers shall require an ISO-certified auditor to complete a Product Manufacturing Checklist (figure E–2 of this subpart), which evaluates the manufacturer on its ability to meet the requirements of the standard in maintaining quality control in the production of FRM or FEM devices. The completed checklist shall be submitted with the application for FRM or FEM determination.

[71 FR 61290, Oct. 17, 2006]

### § 53.52 Leak check test.

(a) *Overview.* In section 7.4.6 of 40 CFR part 50, appendix L, the sampler is required to include the facility, including components, instruments, operator controls, a written procedure, and other capabilities as necessary, to allow the operator to carry out a leak test of the sampler at a field monitoring site without additional equipment. This test procedure is intended to test the adequacy and effectiveness of the sampler's leak check facility. Because of the variety of potential sampler configurations and leak check procedures possible, some adaptation of this procedure may be necessary to accommodate the specific sampler under test. The test conditions and performance specifications associated with this test are summarized in table E–1 of this subpart. The candidate test sampler must meet all test parameters and test specifications to successfully pass this test.

(b) *Technical definitions.* (1) External leakage includes the total flow rate of external ambient air which enters the sampler other than through the sampler inlet and which passes through any one or more of the impactor, filter, or flow rate measurement components.

(2) Internal leakage is the total sample air flow rate that passes through the filter holder assembly without passing through the sample filter.

(c) *Required test equipment.* (1) Flow rate measurement device, range 70 mL/min to 130 mL/min, 2 percent certified accuracy, NIST-traceable.

(2) Flow rate measurement adaptor (40 CFR part 50, appendix L, figure L-30) or equivalent adaptor to facilitate measurement of sampler flow rate at the top of the downtube.

(3) Impermeable membrane or disk, 47 mm nominal diameter.

(4) Means, such as a micro-valve, of providing a simulated leak flow rate through the sampler of approximately 80 mL/min under the conditions specified for the leak check in the sampler's leak check procedure.

(5) Teflon sample filter, as specified in section 6 of 40 CFR part 50, appendix L.

(d) *Calibration of test measurement instruments.* Submit documentation showing evidence of appropriately recent calibration, certification of calibration accuracy, and NIST-traceability (if required) of all measurement instruments used in the tests. The accuracy of flow rate meters shall be verified at the highest and lowest pressures and temperatures used in the tests and shall be checked at zero and one or more non-zero flow rates within 7 days of use for this test.

(e) *Test setup.* (1) The test sampler shall be set up for testing as described in the sampler's operation or instruction manual referred to in § 53.4(b)(3). The sampler shall be installed upright and set up in its normal configuration for collecting PM samples, except that the sample air inlet shall be removed and the flow rate measurement adaptor shall be installed on the sampler's downtube.

(2) The flow rate control device shall be set up to provide a constant, controlled flow rate of 80 mL/min into the sampler downtube under the conditions specified for the leak check in the sampler's leak check procedure.

(3) The flow rate measurement device shall be set up to measure the controlled flow rate of 80 mL/min into the sampler downtube under the conditions specified for the leak check in the sampler's leak check procedure.

(f) *Procedure.* (1) Install the impermeable membrane in a filter cassette and install the cassette into the sampler. Carry out the internal leak check procedure as described in the sampler's operation/instruction manual and verify that the leak check acceptance criterion specified in table E–1 of this subpart is met.

(2) Replace the impermeable membrane with a Teflon filter and install the cassette in the sampler. Remove the inlet from the sampler and install the flow measurement adaptor on the sampler's downtube. Close the valve of the adaptor to seal the flow system. Conduct the external leak check procedure as described in the sampler's operation/instruction manual and verify that the leak check acceptance criteria specified in table E–1 of this subpart are met.

(3) Arrange the flow control device, flow rate measurement device, and other apparatus as necessary to provide a simulated leak flow rate of 80 mL/min into the test sampler through the downtube during the specified external leak check procedure. Carry out the external leak check procedure as described in the sampler's operation/instruction manual but with the simulated leak of 80 mL/min.

(g) *Test results.* The requirements for successful passage of this test are:

(1) That the leak check procedure indicates no significant external or internal leaks in the test sampler when no simulated leaks are introduced.

(2) That the leak check procedure properly identifies the occurrence of the simulated external leak of 80 mL/min.

[62 FR 38799, July 18, 1997, as amended at 71 FR 61291, Oct. 17, 2006]

### § 53.53 Test for flow rate accuracy, regulation, measurement accuracy, and cut-off.

(a) *Overview.* This test procedure is designed to evaluate a candidate sampler's flow rate accuracy with respect to the design flow rate, flow rate regulation, flow rate measurement accuracy, coefficient of variability measurement accuracy, and the flow rate cut-off function. The tests for the first four parameters shall be conducted over a 6-hour time period during which reference flow measurements are made at intervals not to exceed 5 minutes. The flow rate cut-off test, conducted separately, is intended to verify that the sampler carries out the required automatic sample flow rate cut-off function properly in the event of a low-flow condition. The test conditions and performance specifications associated with this test are summarized in table E–1 of this subpart. The candidate test sampler must meet all test parameters and test specifications to successfully pass this test.

(b) *Technical definitions.* (1) Sample flow rate means the quantitative volumetric flow rate of the air stream caused by the sampler to enter the sampler inlet and pass through the

sample filter, measured in actual volume units at the temperature and pressure of the air as it enters the inlet.

(2) The flow rate cut-off function requires the sampler to automatically stop sample flow and terminate the current sample collection if the sample flow rate deviates by more than the variation limits specified in table E–1 of this subpart (±10 percent from the nominal sample flow rate) for more than 60 seconds during a sample collection period. The sampler is also required to properly notify the operator with a flag warning indication of the out-of-specification flow rate condition and if the flow rate cut-off results in an elapsed sample collection time of less than 23 hours.

(c) *Required test equipment.* (1) Flow rate meter, suitable for measuring and recording the actual volumetric sample flow rate at the sampler downtube, with a minimum range of 10 to 25 L/min, 2 percent certified, NIST-traceable accuracy. Optional capability for continuous (analog) recording capability or digital recording at intervals not to exceed 30 seconds is recommended. While a flow meter which provides a direct indication of volumetric flow rate is preferred for this test, an alternative certified flow measurement device may be used as long as appropriate volumetric flow rate corrections are made based on measurements of actual ambient temperature and pressure conditions.

(2) Ambient air temperature sensor, with a resolution of 0.1 °C and certified to be accurate to within 0.5 °C (if needed). If the certified flow meter does not provide direct volumetric flow rate readings, ambient air temperature measurements must be made using continuous (analog) recording capability or digital recording at intervals not to exceed 5 minutes.

(3) Barometer, range 600 mm Hg to 800 mm Hg, certified accurate to 2 mm Hg (if needed). If the certified flow meter does not provide direct volumetric flow rate readings, ambient pressure measurements must be made using continuous (analog) recording capability or digital recording at intervals not to exceed 5 minutes.

(4) Flow measurement adaptor (40 CFR part 50, appendix L, figure L-30) or equivalent adaptor to facilitate measurement of sample flow rate at the sampler downtube.

(5) Valve or other means to restrict or reduce the sample flow rate to a value at least 10 percent below the design flow rate (16.67 L/min). If appropriate, the valve of the flow measurement adaptor may be used for this purpose.

(6) Means for creating an additional pressure drop of 55 mm Hg in the sampler to simulate a heavily loaded filter, such as an orifice or flow restrictive plate installed in the filter holder or a valve or other flow restrictor temporarily installed in the flow path near the filter.

(7) Teflon sample filter, as specified in section 6 of 40 CFR part 50, appendix L (if required).

(d) *Calibration of test measurement instruments.* Submit documentation showing evidence of appropriately recent calibration, certification of calibration accuracy, and NIST-traceability (if required) of all measurement instruments used in the tests. The accuracy of flow-rate meters shall be verified at the highest and lowest pressures and temperatures used in the tests and shall be checked at zero and at least one flow rate within ±3 percent of 16.7 L/min within 7 days prior to use for this test. Where an instrument's measurements are to be recorded with an analog recording device, the accuracy of the entire instrument-recorder system shall be calibrated or verified.

(e) *Test setup.* (1) Setup of the sampler shall be as required in this paragraph (e) and otherwise as described in the sampler's operation or instruction manual referred to in §53.4(b)(3). The sampler shall be installed upright and set up in its normal configuration for collecting PM samples. A sample filter and (or) the device for creating an additional 55 mm Hg pressure drop shall be installed for the duration of these tests. The sampler's ambient temperature, ambient pressure, and flow rate measurement systems shall all be calibrated per the sampler's operation or instruction manual within 7 days prior to this test.

(2) The inlet of the candidate sampler shall be removed and the flow measurement adaptor installed on the sampler's downtube. A leak check as described in the sampler's operation or instruction manual shall be conducted and must be properly passed before other tests are carried out.

(3) The inlet of the flow measurement adaptor shall be connected to the outlet of the flow rate meter.

(4) For the flow rate cut-off test, the valve or means for reducing sampler flow rate shall be installed between the flow measurement adaptor and the downtube or in another location within the sampler such that the sampler flow rate can be manually restricted during the test.

(f) *Procedure*. (1) Set up the sampler as specified in paragraph (e) of this section and otherwise prepare the sampler for normal sample collection operation as directed in the sampler's operation or instruction manual. Set the sampler to automatically start a 6-hour sample collection period at a convenient time.

(2) During the 6-hour operational flow rate portion of the test, measure and record the sample flow rate with the flow rate meter at intervals not to exceed 5 minutes. If ambient temperature and pressure corrections are necessary to calculate volumetric flow rate, ambient temperature and pressure shall be measured at the same frequency as that of the certified flow rate measurements. Note and record the actual start and stop times for the 6-hour flow rate test period.

(3) Following completion of the 6-hour flow rate test period, install the flow rate reduction device and change the sampler flow rate recording frequency to intervals of not more than 30 seconds. Reset the sampler to start a new sample collection period. Manually restrict the sampler flow rate such that the sampler flow rate is decreased slowly over several minutes to a flow rate slightly less than the flow rate cut-off value (15.0 L/min). Maintain this flow rate for at least 2.0 minutes or until the sampler stops the sample flow automatically. Manually terminate the sample period, if the sampler has not terminated it automatically.

(g) *Test results*. At the completion of the test, validate the test conditions and determine the test results as follows:

(1) *Mean sample flow rate*. (i) From the certified measurements ($Q_{ref}$) of the test sampler flow rate obtained by use of the flow rate meter, tabulate each flow rate measurement in units of L/min. If ambient temperature and pressure corrections are necessary to calculate volumetric flow rate, each measured flow rate shall be corrected using its corresponding temperature and pressure measurement values. Calculate the mean flow rate for the sample period ($Q_{ref,ave}$) as follows:

EQUATION 1

$$Q_{ref,ave} = \frac{\sum_{i=1}^{n} Q_{ref,i}}{n}$$

where:

n equals the number of discrete certified flow rate measurements over the 6-hour test period.

(ii)(A) Calculate the percent difference between this mean flow rate value and the design value of 16.67 L/min, as follows:

EQUATION 2

$$\%\ \text{Difference} = \frac{Q_{ref,ave} - 16.67}{16.67} \text{ x } 100\%$$

(B) To successfully pass the mean flow rate test, the percent difference calculated in Equation 2 of this paragraph (g)(1)(ii) must be within ±5 percent.

(2) *Sample flow rate regulation*. (i) From the certified measurements of the test sampler flow rate, calculate the sample coefficient of variation (CV) of the discrete measurements as follows:

EQUATION 3

$$\%CV_{ref} = \frac{1}{Q_{ref,ave}} \text{ x } \sqrt{\frac{\sum_{i=1}^{n} Q_{ref,i}^2 - \frac{1}{n}\left(\sum_{i-1}^{n} Q_{ref,i}\right)^2}{n-1}} \text{ x } 100\%$$

(ii) To successfully pass the flow rate regulation test, the calculated coefficient of variation for the certified flow rates must not exceed 2 percent.

(3) *Flow rate measurement accuracy*. (i) Using the mean volumetric flow rate reported by the candidate test sampler at the completion of the 6-hour test period ($Q_{ind,ave}$), determine the accuracy of the reported mean flow rate as:

EQUATION 4

$$\%\ \text{Difference} = \frac{|Q_{ind,ave} - Q_{ref,ave}|}{Q_{ref,ave}} \times 100\%$$

(ii) To successfully pass the flow rate measurement accuracy test, the percent difference calculated in Equation 4 of this paragraph (g)(3) shall not exceed 2 percent.

(4) *Flow rate coefficient of variation measurement accuracy*. (i) Using the flow rate coefficient of variation indicated by the candidate test sampler at the completion of the 6-hour test ($\%CV_{ind}$), determine the accuracy of this reported coefficient of variation as:

EQUATION 5

$$\text{Difference}\ (\%) = |\%CV_{ind} - \%CV_{ref}|$$

(ii) To successfully pass the flow rate CV measurement accuracy test, the absolute difference in values calculated in Equation 5 of this paragraph (g)(4) must not exceed 0.3 (CV%).

(5) *Flow rate cut-off*. (i) Inspect the measurements of the sample flow rate during the flow rate cut-off test and determine the time at which the sample flow rate decreased to a value less than the cut-off value specified in table E–1 of this subpart. To pass this test, the sampler must have automatically stopped the sample flow at least 30 seconds but not more than 90 seconds after the time at which the sampler flow rate was determined to have decreased to a value less than the cut-off value.

(ii) At the completion of the flow rate cut-off test, download the archived data from the test sampler and verify that the sampler's required Flow-out-of-spec and Incorrect sample period flag indicators are properly set.

[62 FR 38799, July 18, 1997, as amended at 71 FR 61291, Oct. 17, 2006]

### § 53.54 Test for proper sampler operation following power interruptions.

(a) *Overview*. (1) This test procedure is designed to test certain performance parameters of the candidate sampler during a test period in which power interruptions of various duration occur. The performance parameters tested are:

(i) Proper flow rate performance of the sampler.

(ii) Accuracy of the sampler's average flow rate, CV, and sample volume measurements.

(iii) Accuracy of the sampler's reported elapsed sampling time.

(iv) Accuracy of the reported time and duration of power interruptions.

(2) This test shall be conducted during operation of the test sampler over a continuous 6-hour test period during which the sampler's flow rate shall be measured and recorded at intervals not to exceed 5 minutes. The performance parameters tested under this procedure, the corresponding minimum performance specifications, and the applicable test conditions are summarized in table E–1 of this subpart. Each performance parameter tested, as described or determined in the test procedure, must meet or exceed the associated performance specification to successfully pass this test.

(b) *Required test equipment*. (1) Flow rate meter, suitable for measuring and recording the actual volumetric sample flow rate at the sampler downtube, with a minimum range of 10 to 25 L/min, 2 percent certified, NIST-traceable accuracy. Optional capability for continuous (analog) recording capability or digital recording at intervals not to exceed 5 minutes is recommended. While a flow meter which provides a direct indication of volumetric flow rate is preferred for this test, an alternative certified flow measurement device may be used as long as appropriate volumetric flow rate corrections are made based on measurements of actual ambient temperature and pressure conditions.

(2) Ambient air temperature sensor (if needed for volumetric corrections to flow rate measurements), with a resolution of 0.1 °C, certified accurate to within 0.5 °C, and continuous (analog) recording capability or digital recording at intervals not to exceed 5 minutes.

(3) Barometer (if needed for volumetric corrections to flow rate measurements), range 600 mm Hg to 800 mm Hg, certified accurate to 2 mm Hg, with continuous (analog) recording capability or digital recording at intervals not to exceed 5 minutes.

(4) Flow measurement adaptor (40 CFR part 50, appendix L, figure L-30) or equivalent adaptor to facilitate measurement of sample flow rate at the sampler downtube.

(5) Means for creating an additional pressure drop of 55 mm Hg in the sampler to simulate a heavily loaded filter, such as an orifice or flow restrictive plate installed in the filter holder or a valve or other flow restrictor temporarily installed in the flow path near the filter.

(6) Teflon sample filter, as specified in section 6 of 40 CFR part 50, appendix L (if required).

(7) Time measurement system, accurate to within 10 seconds per day.

(c) *Calibration of test measurement instruments.* Submit documentation showing evidence of appropriately recent calibration, certification of calibration accuracy, and NIST-traceability (if required) of all measurement instruments used in the tests. The accuracy of flow rate meters shall be verified at the highest and lowest pressures and temperatures used in the tests and shall be checked at zero and at least one flow rate within ±3 percent of 16.7 L/min within 7 days prior to use for this test. Where an instrument's measurements are to be recorded with an analog recording device, the accuracy of the entire instrument-recorder system shall be calibrated or verified.

(d) *Test setup.* (1) Setup of the sampler shall be performed as required in this paragraph (d) and otherwise as described in the sampler's operation or instruction manual referred to in § 53.4(b)(3). The sampler shall be installed upright and set up in its normal configuration for collecting PM samples. A sample filter and (or) the device for creating an additional 55 mm Hg pressure drop shall be installed for the duration of these tests. The sampler's ambient temperature, ambient pressure, and flow measurement systems shall all be calibrated per the sampler's operating manual within 7 days prior to this test.

(2) The inlet of the candidate sampler shall be removed and the flow measurement adaptor installed on the sample downtube. A leak check as described in the sampler's operation or instruction manual shall be conducted and must be properly passed before other tests are carried out.

(3) The inlet of the flow measurement adaptor shall be connected to the outlet of the flow rate meter.

(e) *Procedure.* (1) Set up the sampler as specified in paragraph (d) of this section and otherwise prepare the sampler for normal sample collection operation as directed in the sampler's operation or instruction manual. Set the sampler to automatically start a 6-hour sample collection period at a convenient time.

(2) During the entire 6-hour operational flow rate portion of the test, measure and record the sample flow rate with the flow rate meter at intervals not to exceed 5 minutes. If ambient temperature and pressure corrections are necessary to calculate volumetric flow rate, ambient temperature and pressure shall be measured at the same frequency as that of the certified flow rate measurements. Note and record the actual start and stop times for the 6-hour flow rate test period.

(3) During the 6-hour test period, interrupt the AC line electrical power to the sampler 5 times, with durations of 20 seconds, 40 seconds, 2 minutes, 7 minutes, and 20 minutes (respectively), with not less than 10 minutes of normal electrical power supplied between each power interruption. Record the hour and minute and duration of each power interruption.

(4) At the end of the test, terminate the sample period (if not automatically terminated by the sampler) and download all archived instrument data from the test sampler.

(f) *Test results.* At the completion of the sampling period, validate the test

conditions and determine the test results as follows:

(1) *Mean sample flow rate.* (i) From the certified measurements ($Q_{ref}$) of the test sampler flow rate, tabulate each flow rate measurement in units of L/min. If ambient temperature and pressure corrections are necessary to calculate volumetric flow rate, each measured flow rate shall be corrected using its corresponding temperature and pressure measurement values. Calculate the mean flow rate for the sample period ($Q_{ref,ave}$) as follows:

EQUATION 6

$$Q_{ref,ave} = \frac{\sum_{i=1}^{n} Q_{ref,i}}{n}$$

where:

n equals the number of discrete certified flow rate measurements over the 6-hour test period, excluding flow rate values obtained during periods of power interruption.

(ii)(A) Calculate the percent difference between this mean flow rate value and the design value of 16.67 L/min, as follows:

EQUATION 7

$$\%\ \text{Difference} = \frac{Q_{ref,ave} - 16.67}{16.67} \text{ x } 100\%$$

(B) To successfully pass this test, the percent difference calculated in Equation 7 of this paragraph (f)(1)(ii) must be within ±5 percent.

(2) *Sample flow rate regulation.* (i) From the certified measurements of the test sampler flow rate, calculate the sample coefficient of variation of the discrete measurements as follows:

EQUATION 8

$$\%CV_{ref} = \frac{1}{Q_{ref,ave}} \text{ x } \sqrt{\frac{\sum_{i=1}^{n} Q_{ref,i}^2 - \frac{1}{n}\left(\sum_{i-1}^{n} Q_{ref,i}\right)^2}{n-1}} \text{ x } 100\%$$

(ii) To successfully pass this test, the calculated coefficient of variation for the certified flow rates must not exceed 2 percent.

(3) *Flow rate measurement accuracy.* (i) Using the mean volumetric flow rate reported by the candidate test sampler at the completion of the 6-hour test ($Q_{ind,ave}$), determine the accuracy of the reported mean flow rate as:

EQUATION 9

$$\%\ \text{Difference} = \frac{|Q_{ind,ave} - Q_{ref,ave}|}{Q_{ref,ave}} \text{ x } 100\%$$

(ii) To successfully pass this test, the percent difference calculated in Equation 9 of this paragraph (f)(3) shall not exceed 2 percent.

(4) *Flow rate CV measurement accuracy.* (i) Using the flow rate coefficient of variation indicated by the candidate test sampler at the completion of the 6-hour test ($\%CV_{ind}$), determine the accuracy of the reported coefficient of variation as:

EQUATION 10

$$\text{Difference}\ (\%) = |\%CV_{ind} - \%CV_{ref}|$$

(ii) To successfully pass this test, the absolute difference in values calculated in Equation 10 of this paragraph (f)(4) must not exceed 0.3 (CV%).

(5) Verify that the sampler properly provided a record and visual display of the correct year, month, day-of-month, hour, and minute with an accuracy of ±2 minutes, of the start of each power interruption of duration greater than 60 seconds.

(6) Calculate the actual elapsed sample time, excluding the periods of electrical power interruption. Verify that the elapsed sample time reported by the sampler is accurate to within ±20 seconds for the 6-hour test run.

(7) Calculate the sample volume as $Q_{ref.ave}$ multiplied by the sample time, excluding periods of power interruption. Verify that the sample volume reported by the sampler is within 2 percent of the calculated sample volume to successfully pass this test.

(8) Inspect the downloaded instrument data from the test sampler and verify that all data are consistent with normal operation of the sampler.

[62 FR 38799, July 18, 1997; 63 FR 7714, Feb. 17, 1998, as amended at 71 FR 61291, Oct. 17, 2006]

**§ 53.55 Test for effect of variations in power line voltage and ambient temperature.**

(a) *Overview.* (1) This test procedure is a combined procedure to test various performance parameters under variations in power line voltage and ambient temperature. Tests shall be conducted in a temperature-controlled environment over four 6-hour time periods during which reference temperature and flow rate measurements shall be made at intervals not to exceed 5 minutes. Specific parameters to be evaluated at line voltages of 105 and 125 volts and temperatures of −20 °C and + 40 °C are as follows:

(i) Sample flow rate.

(ii) Flow rate regulation.

(iii) Flow rate measurement accuracy.

(iv) Coefficient of variability measurement accuracy.

(v) Ambient air temperature measurement accuracy.

(vi) Proper operation of the sampler when exposed to power line voltage and ambient temperature extremes.

(2) The performance parameters tested under this procedure, the corresponding minimum performance specifications, and the applicable test conditions are summarized in table E–1 of this subpart. Each performance parameter tested, as described or determined in the test procedure, must meet or exceed the associated performance specification given. The candidate sampler must meet all specifications for the associated $PM_{2.5}$ or $PM_{10-2.5}$ method (as applicable) to pass this test procedure.

(b) *Technical definition.* Sample flow rate means the quantitative volumetric flow rate of the air stream caused by the sampler to enter the sampler inlet and pass through the sample filter, measured in actual volume units at the temperature and pressure of the air as it enters the inlet.

(c) *Required test equipment.* (1) Environmental chamber or other temperature-controlled environment or environments, capable of obtaining and maintaining temperatures at −20 °C and = 40 °C as required for the test with an accuracy of ±2 °C. The test environment(s) must be capable of maintaining these temperatures within the specified limits continuously with the additional heat load of the operating test sampler in the environment. Henceforth, where the test procedures specify a test or environmental ''chamber,'' an alternative temperature-controlled environmental area or areas may be substituted, provided the required test temperatures and all other test requirements are met.

(2) Variable voltage AC power transformer, range 100 Vac to 130 Vac, with sufficient current capacity to operate the test sampler continuously under the test conditions.

(3) Flow rate meter, suitable for measuring and recording the actual volumetric sample flow rate at the sampler downtube, with a minimum range of 10 to 25 actual L/min, 2 percent certified, NIST-traceable accuracy. Optional capability for continuous (analog) recording capability or digital recording at intervals not to exceed 5 minutes is recommended. While a flow meter which provides a direct indication of volumetric flow rate is preferred for this test, an alternative certified flow measurement device may be used as long as appropriate volumetric flow rate corrections are made based on measurements of actual ambient temperature and pressure conditions.

(4) Ambient air temperature recorder, range −30 °C to = 50 °C, with a resolution of 0.1 °C and certified accurate to within 0.5 °C. Ambient air temperature measurements must be made using continuous (analog) recording capability or digital recording at intervals not to exceed 5 minutes.

(5) Barometer, range 600 mm Hg to 800 mm Hg, certified accurate to 2 mm Hg. If the certified flow rate meter does not provide direct volumetric flow rate readings, ambient pressure measurements must be made using continuous (analog) recording capability or digital recording at intervals not to exceed 5 minutes.

(6) Flow measurement adaptor (40 CFR part 50, appendix L, figure L-30) or equivalent adaptor to facilitate measurement of sampler flow rate at the sampler downtube.

(7) Means for creating an additional pressure drop of 55 mm Hg in the sampler to simulate a heavily loaded filter,

such as an orifice or flow restrictive plate installed in the filter holder or a valve or other flow restrictor temporarily installed in the flow path near the filter.

(8) AC RMS voltmeter, accurate to 1.0 volt.

(9) Teflon sample filter, as specified in section 6 of 40 CFR part 50, appendix L (if required).

(d) *Calibration of test measurement instruments.* Submit documentation showing evidence of appropriately recent calibration, certification of calibration accuracy, and NIST-traceability (if required) of all measurement instruments used in the tests. The accuracy of flow rate meters shall be verified at the highest and lowest pressures and temperatures used in the tests and shall be checked at zero and at least one flow rate within ±3 percent of 16.7 L/min within 7 days prior to use for this test. Where an instrument's measurements are to be recorded with an analog recording device, the accuracy of the entire instrument-recorder system shall be calibrated or verified.

(e) *Test setup.* (1) Setup of the sampler shall be performed as required in this paragraph (e) and otherwise as described in the sampler's operation or instruction manual referred to in § 53.4(b)(3). The sampler shall be installed upright and set up in the temperature-controlled chamber in its normal configuration for collecting PM samples. A sample filter and (or) the device for creating an additional 55 mm Hg pressure drop shall be installed for the duration of these tests. The sampler's ambient temperature, ambient pressure, and flow measurement systems shall all be calibrated per the sampler's operating manual within 7 days prior to this test.

(2) The inlet of the candidate sampler shall be removed and the flow measurement adaptor installed on the sampler's downtube. A leak check as described in the sampler's operation or instruction manual shall be conducted and must be properly passed before other tests are carried out.

(3) The inlet of the flow measurement adaptor shall be connected to the outlet of the flow rate meter.

(4) The ambient air temperature recorder shall be installed in the test chamber such that it will accurately measure the temperature of the air in the vicinity of the candidate sampler without being unduly affected by the chamber's air temperature control system.

(f) *Procedure.* (1) Set up the sampler as specified in paragraph (e) of this section and otherwise prepare the sampler for normal sample collection operation as directed in the sampler's operation or instruction manual.

(2) The test shall consist of four test runs, one at each of the following conditions of chamber temperature and electrical power line voltage (respectively):

(i) −20 °C ±2 °C and 105 ±1 Vac.

(ii) −20 °C ±2 °C and 125 ±1 Vac.

(iii) = 40 °C ±2 °C and 105 ±1 Vac.

(iv) = 40 °C ±2 °C and 125 ±1 Vac.

(3) For each of the four test runs, set the selected chamber temperature and power line voltage for the test run. Upon achieving each temperature setpoint in the chamber, the candidate sampler and flow meter shall be thermally equilibrated for a period of at least 2 hours prior to the test run. Following the thermal conditioning time, set the sampler to automatically start a 6-hour sample collection period at a convenient time.

(4) During each 6-hour test period:

(i) Measure and record the sample flow rate with the flow rate meter at intervals not to exceed 5 minutes. If ambient temperature and pressure corrections are necessary to calculate volumetric flow rate, ambient temperature and pressure shall be measured at the same frequency as that of the certified flow rate measurements. Note and record the actual start and stop times for the 6-hour flow rate test period.

(ii) Determine and record the ambient (chamber) temperature indicated by the sampler and the corresponding ambient (chamber) temperature measured by the ambient temperature recorder specified in paragraph (c)(4) of this section at intervals not to exceed 5 minutes.

(iii) Measure the power line voltage to the sampler at intervals not greater than 1 hour.

(5) At the end of each test run, terminate the sample period (if not automatically terminated by the sampler) and download all archived instrument data from the test sampler.

(g) *Test results*. For each of the four test runs, examine the chamber temperature measurements and the power line voltage measurements. Verify that the temperature and line voltage met the requirements specified in paragraph (f) of this section at all times during the test run. If not, the test run is not valid and must be repeated. Determine the test results as follows:

(1) *Mean sample flow rate*. (i) From the certified measurements ($Q_{ref}$) of the test sampler flow rate, tabulate each flow rate measurement in units of L/min. If ambient temperature and pressure corrections are necessary to calculate volumetric flow rate, each measured flow rate shall be corrected using its corresponding temperature and pressure measurement values. Calculate the mean flow rate for each sample period ($Q_{ref,ave}$) as follows:

EQUATION 11

$$Q_{ref,ave} = \frac{\sum_{i=1}^{n} Q_{ref,i}}{n}$$

where:

n equals the number of discrete certified flow rate measurements over each 6-hour test period.

(ii)(A) Calculate the percent difference between this mean flow rate value and the design value of 16.67 L/min, as follows:

EQUATION 12

$$\%\ \text{Difference} = \frac{Q_{ref,ave} - 16.67}{16.67} \text{ x } 100\%$$

(B) To successfully pass this test, the percent difference calculated in Equation 12 of this paragraph (g)(1)(ii) must be within ±5 percent for each test run.

(2) *Sample flow rate regulation*. (i) From the certified measurements of the test sampler flow rate, calculate the sample coefficient of variation of the discrete measurements as follows:

EQUATION 13

$$\%CV_{ref} = \frac{1}{Q_{ref,ave}} \text{ x} \sqrt{\frac{\sum_{i=1}^{n} Q_{ref,i}^2 - \frac{1}{n}\left(\sum_{i-1}^{n} Q_{ref,i}\right)^2}{n-1}} \text{ x } 100\%$$

(ii) To successfully pass this test, the calculated coefficient of variation for the certified flow rates must not exceed 2 percent.

(3) *Flow rate measurement accuracy*. (i) Using the mean volumetric flow rate reported by the candidate test sampler at the completion of each 6-hour test ($Q_{ind,ave}$), determine the accuracy of the reported mean flow rate as:

EQUATION 14

$$\%\ \text{Difference} = \frac{|Q_{ind,ave} - Q_{ref,ave}|}{Q_{ref,ave}} \text{ x } 100\%$$

(ii) To successfully pass this test, the percent difference calculated in Equation 14 of this paragraph (g)(3) shall not exceed 2 percent for each test run.

(4) *Flow rate coefficient of variation measurement accuracy*. (i) Using the flow rate coefficient of variation indicated by the candidate test sampler ($\%CV_{ind}$), determine the accuracy of the reported coefficient of variation as:

EQUATION 15

$$\text{Difference}\ \% = |\%CV_{ind} - \%CV_{ref}|$$

(ii) To successfully pass this test, the absolute difference calculated in Equation 15 of this paragraph (g)(4) must not exceed 0.3 (CV%) for each test run.

(5) *Ambient temperature measurement accuracy*. (i) Calculate the absolute value of the difference between the mean ambient air temperature indicated by the test sampler and the mean ambient (chamber) air temperature measured with the ambient air temperature recorder as:

*Equation 16*

$$T_{diff} = \left|T_{ind,ave} - T_{ref,ave}\right|$$

Where:

$T_{ind,ave}$ = The mean ambient air temperature indicated by the test sampler, °C; and

$T_{ref,ave}$ = The mean ambient air temperature measured by the reference temperature instrument, °C.

(ii) The calculated temperature difference must be less than 2 °C for each test run.

(6) *Sampler functionality*. To pass the sampler functionality test, the following two conditions must both be met for each test run:

(i) The sampler must not shutdown during any portion of the 6-hour test.

(ii) An inspection of the downloaded data from the test sampler verifies that all the data are consistent with normal operation of the sampler.

[62 FR 38799, July 18, 1997, as amended at 71 FR 61291, Oct. 17, 2006]

### § 53.56 Test for effect of variations in ambient pressure.

(a) *Overview*. (1) This test procedure is designed to test various sampler performance parameters under variations in ambient (barometric) pressure. Tests shall be conducted in a pressure-controlled environment over two 6-hour time periods during which reference pressure and flow rate measurements shall be made at intervals not to exceed 5 minutes. Specific parameters to be evaluated at operating pressures of 600 and 800 mm Hg are as follows:

(i) Sample flow rate.

(ii) Flow rate regulation.

(iii) Flow rate measurement accuracy.

(iv) Coefficient of variability measurement accuracy.

(v) Ambient pressure measurement accuracy.

(vi) Proper operation of the sampler when exposed to ambient pressure extremes.

(2) The performance parameters tested under this procedure, the corresponding minimum performance specifications, and the applicable test conditions are summarized in table E–1 of this subpart. Each performance parameter tested, as described or determined in the test procedure, must meet or exceed the associated performance specification given. The candidate sampler must meet all specifications for the associated $PM_{2.5}$ or $PM_{10-2.5}$ method (as applicable) to pass this test procedure.

(b) *Technical definition*. Sample flow rate means the quantitative volumetric flow rate of the air stream caused by the sampler to enter the sampler inlet and pass through the sample filter, measured in actual volume units at the temperature and pressure of the air as it enters the inlet.

(c) *Required test equipment*. (1) Hypobaric chamber or other pressure-controlled environment or environments, capable of obtaining and maintaining pressures at 600 mm Hg and 800 mm Hg required for the test with an accuracy of 5 mm Hg. Henceforth, where the test procedures specify a test or environmental chamber, an alternative pressure-controlled environmental area or areas may be substituted, provided the test pressure requirements are met. Means for simulating ambient pressure using a closed-loop sample air system may also be approved for this test; such a proposed method for simulating the test pressure conditions may be described and submitted to EPA at the address given in § 53.4(a) prior to conducting the test for a specific individual determination of acceptability.

(2) Flow rate meter, suitable for measuring and recording the actual volumetric sampler flow rate at the sampler downtube, with a minimum range of 10 to 25 L/min, 2 percent certified, NIST-traceable accuracy. Optional capability for continuous (analog) recording capability or digital recording at intervals not to exceed 5 minutes is recommended. While a flow meter which provides a direct indication of volumetric flow rate is preferred for this test, an alternative certified flow measurement device may be used as long as appropriate volumetric flow rate corrections are made based on measurements of actual ambient temperature and pressure conditions.

(3) Ambient air temperature recorder (if needed for volumetric corrections to flow rate measurements) with a range −30 °C to = 50 °C, certified accurate to within 0.5 °C. If the certified flow meter does not provide direct volumetric flow rate readings, ambient temperature measurements must be made using continuous (analog) recording capability or digital recording at intervals not to exceed 5 minutes.

(4) Barometer, range 600 mm Hg to 800 mm Hg, certified accurate to 2 mm Hg. Ambient air pressure measurements must be made using continuous (analog) recording capability or digital recording at intervals not to exceed 5 minutes.

(5) Flow measurement adaptor (40 CFR part 50, appendix L, figure L–30) or equivalent adaptor to facilitate measurement of sampler flow rate at the sampler downtube.

(6) Means for creating an additional pressure drop of 55 mm Hg in the sampler to simulate a heavily loaded filter, such as an orifice or flow restrictive plate installed in the filter holder or a valve or other flow restrictor temporarily installed in the flow path near the filter.

(7) Teflon sample filter, as specified in section 6 of 40 CFR part 50, appendix L (if required).

(d) *Calibration of test measurement instruments.* Submit documentation showing evidence of appropriately recent calibration, certification of calibration accuracy, and NIST-traceability (if required) of all measurement instruments used in the tests. The accuracy of flow rate meters shall be verified at the highest and lowest pressures and temperatures used in the tests and shall be checked at zero and at least one flow rate within ±3 percent of 16.7 L/min within 7 days prior to use for this test. Where an instrument's measurements are to be recorded with an analog recording device, the accuracy of the entire instrument-recorder system shall be calibrated or verified.

(e) *Test setup.* (1) Setup of the sampler shall be performed as required in this paragraph (e) and otherwise as described in the sampler's operation or instruction manual referred to in § 53.4(b)(3). The sampler shall be installed upright and set up in the pressure-controlled chamber in its normal configuration for collecting PM samples. A sample filter and (or) the device for creating an additional 55 mm Hg pressure drop shall be installed for the duration of these tests. The sampler's ambient temperature, ambient pressure, and flow measurement systems shall all be calibrated per the sampler's operating manual within 7 days prior to this test.

(2) The inlet of the candidate sampler shall be removed and the flow measurement adaptor installed on the sampler's downtube. A leak check as described in the sampler's operation or instruction manual shall be conducted and must be properly passed before other tests are carried out.

(3) The inlet of the flow measurement adaptor shall be connected to the outlet of the flow rate meter.

(4) The barometer shall be installed in the test chamber such that it will accurately measure the air pressure to which the candidate sampler is subjected.

(f) *Procedure.* (1) Set up the sampler as specified in paragraph (e) of this section and otherwise prepare the sampler for normal sample collection operation as directed in the sampler's operation or instruction manual.

(2) The test shall consist of two test runs, one at each of the following conditions of chamber pressure:

(i) 600 mm Hg.

(ii) 800 mm Hg.

(3) For each of the two test runs, set the selected chamber pressure for the test run. Upon achieving each pressure setpoint in the chamber, the candidate sampler shall be pressure-equilibrated for a period of at least 30 minutes prior to the test run. Following the conditioning time, set the sampler to automatically start a 6-hour sample collection period at a convenient time.

(4) During each 6-hour test period:

(i) Measure and record the sample flow rate with the flow rate meter at intervals not to exceed 5 minutes. If ambient temperature and pressure corrections are necessary to calculate volumetric flow rate, ambient temperature and pressure shall be measured at the same frequency as that of the certified flow rate measurements. Note and record the actual start and stop times for the 6-hour flow rate test period.

(ii) Determine and record the ambient (chamber) pressure indicated by the sampler and the corresponding ambient (chamber) pressure measured by the barometer specified in paragraph (c)(4) of this section at intervals not to exceed 5 minutes.

(5) At the end of each test period, terminate the sample period (if not automatically terminated by the sampler) and download all archived instrument data for the test run from the test sampler.

(g) *Test results*. For each of the two test runs, examine the chamber pressure measurements. Verify that the pressure met the requirements specified in paragraph (f) of this section at all times during the test. If not, the test run is not valid and must be repeated. Determine the test results as follows:

(1) *Mean sample flow rate*. (i) From the certified measurements ($Q_{ref}$) of the test sampler flow rate, tabulate each flow rate measurement in units of L/min. If ambient temperature and pressure corrections are necessary to calculate volumetric flow rate, each measured flow rate shall be corrected using its corresponding temperature and pressure measurement values. Calculate the mean flow rate for the sample period ($Q_{ref,ave}$) as follows:

EQUATION 17

$$Q_{ref,ave} = \frac{\sum_{i=1}^{n} Q_{ref,i}}{n}$$

where:

n equals the number of discrete certified flow measurements over the 6-hour test period.

(ii)(A) Calculate the percent difference between this mean flow rate value and the design value of 16.67 L/min, as follows:

EQUATION 18

$$\%\ \text{Difference} = \frac{Q_{ref,ave} - 16.67}{16.67} \text{ x } 100\%$$

(B) To successfully pass this test, the percent difference calculated in Equation 18 of this paragraph (g)(1) must be within ±5 percent for each test run.

(2) *Sample flow rate regulation*. (i) From the certified measurements of the test sampler flow rate, calculate the sample coefficient of variation of the discrete measurements as follows:

EQUATION 19

$$\%CV_{ref} = \frac{1}{Q_{ref,ave}} \text{ x} \sqrt{\frac{\sum_{i=1}^{n} Q_{ref,i}^2 - \frac{1}{n}\left(\sum_{i-1}^{n} Q_{ref,i}\right)^2}{n-1}} \text{ x } 100\%$$

(ii) To successfully pass this test, the calculated coefficient of variation for the certified flow rates must not exceed 2 percent.

(3) *Flow rate measurement accuracy*. (i) Using the mean volumetric flow rate reported by the candidate test sampler at the completion of each 6-hour test ($Q_{ind,ave}$), determine the accuracy of the reported mean flow rate as:

EQUATION 20

$$\%\ \text{Difference} = \frac{|Q_{ind,ave} - Q_{ref,ave}|}{Q_{ref,ave}} \text{ x } 100\%$$

(ii) To successfully pass this test, the percent difference calculated in Equation 20 of this paragraph (g)(3) shall not exceed 2 percent for each test run.

(4) *Flow rate CV measurement accuracy*. (i) Using the flow rate coefficient of variation indicated by the candidate test sampler at the completion of the 6-hour test ($\%CV_{ind}$), determine the accuracy of the reported coefficient of variation as:

EQUATION 21

$$\text{Difference}\ (\%) = |\%CV_{ind} - \%CV_{ref}|$$

(ii) To successfully pass this test, the absolute difference in values calculated in Equation 21 of this paragraph (g)(4) must not exceed 0.3 (CV%) for each test run.

(5) *Ambient pressure measurement accuracy*. (i) Calculate the absolute difference between the mean ambient air pressure indicated by the test sampler and the ambient (chamber) air pressure measured with the reference barometer as:

EQUATION 22

$$P_{diff} = |P_{ind,ave} - P_{ref,ave}|$$

where:

$P_{ind,ave}$ = mean ambient pressure indicated by the test sampler, mm Hg; and

$P_{ref,ave}$ = mean barometric pressure measured by the reference barometer, mm Hg.

(ii) The calculated pressure difference must be less than 10 mm Hg for each test run to pass the test.

(6) *Sampler functionality*. To pass the sampler functionality test, the following two conditions must both be met for each test run:

(i) The sampler must not shut down during any part of the 6-hour tests; and

(ii) An inspection of the downloaded data from the test sampler verifies that all the data are consistent with normal operation of the sampler.

[62 FR 38799, July 18, 1997; 63 FR 7714, Feb. 17, 1998, as amended at 71 FR 61292, Oct. 17, 2006]

### § 53.57 Test for filter temperature control during sampling and post-sampling periods.

(a) *Overview*. This test is intended to measure the candidate sampler's ability to prevent excessive overheating of the PM sample collection filter (or filters) under conditions of elevated solar insolation. The test evaluates radiative effects on filter temperature during a 4-hour period of active sampling as well as during a subsequent 4-hour non-sampling time period prior to filter retrieval. Tests shall be conducted in an environmental chamber which provides the proper radiant wavelengths and energies to adequately simulate the sun's radiant effects under clear conditions at sea level. For additional guidance on conducting solar radiative tests under controlled conditions, consult military standard specification 810–E (reference 6 in appendix A of this subpart). The performance parameters tested under this procedure, the corresponding minimum performance specifications, and the applicable test conditions are summarized in table E–1 of this subpart. Each performance parameter tested, as described or determined in the test procedure, must meet or exceed the associated performance specification to successfully pass this test.

(b) *Technical definition*. Filter temperature control during sampling is the ability of a sampler to maintain the temperature of the particulate matter sample filter within the specified deviation (5 °C) from ambient temperature during any active sampling period. Post-sampling temperature control is the ability of a sampler to maintain the temperature of the particulate matter sample filter within the specified deviation from ambient temperature during the period from the end of active sample collection by the sampler until the filter is retrieved from the sampler for laboratory analysis.

(c) *Required test equipment*. (1) Environmental chamber providing the means, such as a bank of solar-spectrum lamps, for generating or simulating thermal radiation in approximate spectral content and intensity equivalent to solar insulation of 1000 ±50 W/m$^2$ inside the environmental chamber. To properly simulate the sun's radiative effects on the sampler, the solar bank must provide the spectral energy distribution and permitted tolerances specified in table E–2 of this subpart. The solar radiation source area shall be such that the width of the candidate sampler shall not exceed one-half the dimensions of the solar bank. The solar bank shall be located a minimum of 76 cm (30 inches) from any surface of the candidate sampler. To meet requirements of the solar radiation tests, the chamber's internal volume shall be a minimum of 10 times that of the volume of the candidate sampler. Air velocity in the region of the sampler must be maintained continuously during the radiative tests at 2.0 ±0.5 m/sec.

(2) Ambient air temperature recorder, range −30 °C to = 50 °C, with a resolution of 0.1 °C and certified accurate to within 0.5 °C. Ambient air temperature measurements must be made using continuous (analog) recording capability or digital recording at intervals not to exceed 5 minutes.

(3) Flow measurement adaptor (40 CFR part 50, appendix L, figure L-30) or equivalent adaptor to facilitate measurement of sampler flow rate at the sampler downtube.

(4) Miniature temperature sensor(s), capable of being installed in the sampler without introducing air leakage and capable of measuring the sample air temperature within 1 cm of the center of the filter, downstream of the filter; with a resolution of 0.1 °C, certified

accurate to within 0.5 °C, NIST-traceable, with continuous (analog) recording capability or digital recording at intervals of not more than 5 minutes.

(5) Solar radiometer, to measure the intensity of the simulated solar radiation in the test environment, range of 0 to approximately 1500 W/m$^2$. Optional capability for continuous (analog) recording or digital recording at intervals not to exceed 5 minutes is recommended.

(6) Sample filter or filters, as specified in section 6 of 40 CFR part 50, appendix L.

(d) *Calibration of test measurement instruments.* Submit documentation showing evidence of appropriately recent calibration, certification of calibration accuracy, and NIST-traceability (if required) of all measurement instruments used in the tests. The accuracy of flow rate meters shall be verified at the highest and lowest pressures and temperatures used in the tests and shall be checked at zero and at least one flow rate within ±3 percent of 16.7 L/min within 7 days prior to use for this test. Where an instrument's measurements are to be recorded with an analog recording device, the accuracy of the entire instrument-recorder system shall be calibrated or verified.

(e) *Test setup.* (1) Setup of the sampler shall be performed as required in this paragraph (e) and otherwise as described in the sampler's operation or instruction manual referred to in §53.4(b)(3). The sampler shall be installed upright and set up in the solar radiation environmental chamber in its normal configuration for collecting PM samples (with the inlet installed). The sampler's ambient and filter temperature measurement systems shall be calibrated per the sampler's operating manual within 7 days prior to this test. A sample filter shall be installed for the duration of this test. For sequential samplers, a sample filter shall also be installed in each available sequential channel or station intended for collection of a sequential sample (or at least five additional filters for magazine-type sequential samplers) as directed by the sampler's operation or instruction manual.

(2) The miniature temperature sensor shall be temporarily installed in the test sampler such that it accurately measures the air temperature 1 cm from the center of the filter on the downstream side of the filter. The sensor shall be installed such that no external or internal air leakage is created by the sensor installation. The sensor's dimensions and installation shall be selected to minimize temperature measurement uncertainties due to thermal conduction along the sensor mounting structure or sensor conductors. For sequential samplers, similar temperature sensors shall also be temporarily installed in the test sampler to monitor the temperature 1 cm from the center of each filter stored in the sampler for sequential sample operation.

(3) The solar radiant energy source shall be installed in the test chamber such that the entire test sampler is irradiated in a manner similar to the way it would be irradiated by solar radiation if it were located outdoors in an open area on a sunny day, with the radiation arriving at an angle of between 30° and 45° from vertical. The intensity of the radiation received by all sampler surfaces that receive direct radiation shall average 1000 ±50 W/m$^2$, measured in a plane perpendicular to the incident radiation. The incident radiation shall be oriented with respect to the sampler such that the area of the sampler's ambient temperature sensor (or temperature shield) receives full, direct radiation as it would or could during normal outdoor installation. Also, the temperature sensor must not be shielded or shaded from the radiation by a sampler part in a way that would not occur at other normal insulation angles or directions.

(4) The solar radiometer shall be installed in a location where it measures thermal radiation that is generally representative of the average thermal radiation intensity that the upper portion of the sampler and sampler inlet receive. The solar radiometer shall be oriented so that it measures the radiation in a plane perpendicular to its angle of incidence.

(5) The ambient air temperature recorder shall be installed in the test chamber such that it will accurately measure the temperature of the air in

the chamber without being unduly affected by the chamber's air temperature control system or by the radiant energy from the solar radiation source that may be present inside the test chamber.

(f) *Procedure*. (1) Set up the sampler as specified in paragraph (e) of this section and otherwise prepare the sampler for normal sample collection operation as directed in the sampler's operation or instruction manual.

(2) Remove the inlet of the candidate test sampler and install the flow measurement adaptor on the sampler's downtube. Conduct a leak check as described in the sampler's operation or instruction manual. The leak test must be properly passed before other tests are carried out.

(3) Remove the flow measurement adaptor from the downtube and re-install the sampling inlet.

(4) Activate the solar radiation source and verify that the resulting energy distribution prescribed in table E–2 of this subpart is achieved.

(5) Program the test sampler to conduct a single sampling run of 4 continuous hours. During the 4-hour sampling run, measure and record the radiant flux, ambient temperature, and filter temperature (all filter temperatures for sequential samplers) at intervals not to exceed 5 minutes.

(6) At the completion of the 4-hour sampling phase, terminate the sample period, if not terminated automatically by the sampler. Continue to measure and record the radiant flux, ambient temperature, and filter temperature or temperatures for 4 additional hours at intervals not to exceed 5 minutes. At the completion of the 4-hour post-sampling period, discontinue the measurements and turn off the solar source.

(7) Download all archived sampler data from the test run.

(g) *Test results*. Chamber radiant flux control. Examine the continuous record of the chamber radiant flux and verify that the flux met the requirements specified in table E–2 of this subpart at all times during the test. If not, the entire test is not valid and must be repeated.

(1) *Filter temperature measurement accuracy*. (i) For each 4-hour test period, calculate the absolute value of the difference between the mean filter temperature indicated by the sampler (active filter) and the mean filter temperature measured by the reference temperature sensor installed within 1 cm downstream of the (active) filter as:

EQUATION 23

$$T_{diff,filter} = |T_{ind,filter} - T_{ref,filter}|$$

where:

$T_{ind,filter}$ = mean filter temperature indicated by the test sampler, °C; and

$T_{ref,filter}$ = mean filter temperature measured by the reference temperature sensor, °C.

(ii) To successfully pass the indicated filter temperature accuracy test, the calculated difference between the measured means ($T_{diff,filter}$) must not exceed 2 °C for each 4-hour test period.

(2) *Ambient temperature measurement accuracy*. (i) For each 4-hour test period, calculate the absolute value of the difference between the mean ambient air temperature indicated by the test sampler and the mean ambient air temperature measured by the reference ambient air temperature recorder as:

EQUATION 24

$$T_{diff,ambient} = |T_{ind,ambient} - T_{ref,ambient}|$$

where:

$T_{ind,ambient}$ = mean ambient air temperature indicated by the test sampler, °C; and

$T_{ref,ambient}$ = mean ambient air temperature measured by the reference ambient air temperature recorder, °C.

(ii) To successfully pass the indicated ambient temperature accuracy test, the calculated difference between the measured means ($T_{diff,ambient}$) must not exceed 2 °C for each 4-hour test period.

(3) *Filter temperature control accuracy*. (i) For each temperature measurement interval over each 4-hour test period, calculate the difference between the filter temperature indicated by the reference temperature sensor and the ambient temperature indicated by the test sampler as:

EQUATION 25

$$T_{diff} = T_{ref,filter} - T_{ind,ambient}$$

(ii) Tabulate and inspect the calculated differences as a function of time. To successfully pass the indicated filter temperature control test, the calculated difference between the measured values must not exceed 5 °C for any consecutive intervals covering more than a 30–minute time period.

(iii) For sequential samplers, repeat the test calculations for each of the stored sequential sample filters. All stored filters must also meet the 5 °C temperature control test.

[62 FR 38799, July 18, 1997; 63 FR 7714, Feb. 17, 1998, as amended at 71 FR 61292, Oct. 17, 2006]

### § 53.58 Operational field precision and blank test.

(a) *Overview*. This test is intended to determine the operational precision of the candidate sampler during a minimum of 10 days of field operation, using three collocated test samplers. Measurements of PM are made at a test site with all of the samplers and then compared to determine replicate precision. Candidate sequential samplers are also subject to a test for possible deposition of particulate matter on inactive filters during a period of storage in the sampler. This procedure is applicable to both reference and equivalent methods. In the case of equivalent methods, this test may be combined and conducted concurrently with the comparability test for equivalent methods (described in subpart C of this part), using three reference method samplers collocated with three candidate equivalent method samplers and meeting the applicable site and other requirements of subpart C of this part.

(b) *Technical definition*. (1) Field precision is defined as the standard deviation or relative standard deviation of a set of PM measurements obtained concurrently with three or more collocated samplers in actual ambient air field operation.

(2) Storage deposition is defined as the mass of material inadvertently deposited on a sample filter that is stored in a sequential sampler either prior to or subsequent to the active sample collection period.

(c) *Test site*. Any outdoor test site having $PM_{2.5}$ (or $PM_{10-2.5}$, as applicable) concentrations that are reasonably uniform over the test area and that meet the minimum level requirement of paragraph (g)(2) of this section is acceptable for this test.

(d) *Required facilities and equipment*. (1) An appropriate test site and suitable electrical power to accommodate three test samplers are required.

(2) Teflon sample filters, as specified in section 6 of 40 CFR part 50, appendix L, conditioned and preweighed as required by section 8 of 40 CFR part 50, appendix L, as needed for the test samples.

(e) *Test setup*. (1) Three identical test samplers shall be installed at the test site in their normal configuration for collecting PM samples in accordance with the instructions in the associated manual referred to in § 53.4(b)(3) and also in accordance with applicable supplemental guidance provided in reference 3 in appendix A of this subpart. The test samplers' inlet openings shall be located at the same height above ground and between 2 (1 for samplers with flow rates less than 200 L/min.) and 4 meters apart horizontally. The samplers shall be arranged or oriented in a manner that will minimize the spatial and wind directional effects on sample collection of one sampler on any other sampler.

(2) Each test sampler shall be successfully leak checked, calibrated, and set up for normal operation in accordance with the instruction manual and with any applicable supplemental guidance provided in reference 3 in appendix A of this subpart.

(f) *Test procedure*. (1) Install a conditioned, preweighed filter in each test sampler and otherwise prepare each sampler for normal sample collection. Set identical sample collection start and stop times for each sampler. For sequential samplers, install a conditioned, preweighed specified filter in each available channel or station intended for automatic sequential sample filter collection (or at least five additional filters for magazine-type sequential samplers), as directed by the sampler's operation or instruction manual. Since the inactive sequential channels are used for the storage deposition part of the test, they may not be used to collect the active PM test samples.

(2) Collect either a nominal 24-hour or 48-hour atmospheric PM sample simultaneously with each of the three test samplers.

(3) Following sample collection, retrieve the collected sample from each sampler. For sequential samplers, retrieve the additional stored (blank, unsampled) filters after at least 5 days (120 hours) storage in the sampler if the active samples are 24-hour samples, or after at least 10 days (240 hours) if the active samples are 48-hour samples.

(4) Determine the measured PM mass concentration for each sample in accordance with the applicable procedures prescribed for the candidate method in appendix L or appendix O, as applicable, of part 50 of this chapter, and in accordance with the associated manual referred to in § 53.4(b)(3) and supplemental guidance in reference 2 in appendix A of this subpart. For sequential samplers, also similarly determine the storage deposition as the net weight gain of each blank, unsampled filter after the 5-day (or 10-day) period of storage in the sampler.

(5) Repeat this procedure to obtain a total of 10 sets of any combination of (nominal) 24-hour or 48-hour PM measurements over 10 test periods. For sequential samplers, repeat the 5-day (or 10-day) storage test of additional blank filters once for a total of two sets of blank filters.

(g) *Calculations*. (1) Record the PM concentration for each test sampler for each test period as $C_{i,j}$, where i is the sampler number (i = 1,2,3) and j is the test period (j = 1,2, * * * 10).

(2)(i) For each test period, calculate and record the average of the three measured PM concentrations as $C_{ave,j}$ where j is the test period using equation 26 of this section:

*Equation 26*

$$C_{ave,j} = \frac{1}{3} \times \sum_{i=1}^{3} C_{i,j}$$

(ii) If $C_{ave,j}$ <3 μg/m³ for any test period, data from that test period are unacceptable, and an additional sample collection set must be obtained to replace the unacceptable data.

(3)(i) Calculate and record the precision for each of the 10 test periods, as the standard deviation, using equation 27 of this section:

*Equation 27*

$$P_j = \sqrt{\frac{\sum_{i=1}^{3} C_{i,j}^2 - \frac{1}{3}\left(\sum_{i=1}^{3} C_{i,j}\right)^2}{2}}$$

(ii) For each of the 10 test periods, also calculate and record the precision as the relative standard deviation, in percent, using equation 28 of this section:

*Equation 28*

$$RP_j = 100\% \times \frac{P_j}{C_{ave,j}}$$

(h) *Test results*. (1) The candidate method passes the precision test if *either* $P_j$ or $RP_j$ is less than or equal to the corresponding specification in table E–1 of this subpart for all 10 test periods.

(2) The candidate sequential sampler passes the blank filter storage deposition test if the average net storage deposition weight gain of each set of blank filters (total of the net weight gain of each blank filter divided by the number of filters in the set) from each test sampler (six sets in all) is less than 50 μg.

[71 FR 61292, Oct. 17, 2006,as amended at 72 FR 32208, June 12, 2007]

### § 53.59 Aerosol transport test for Class I equivalent method samplers.

(a) *Overview*. This test is intended to verify adequate aerosol transport through any modified or air flow splitting components that may be used in a Class I candidate equivalent method sampler such as may be necessary to achieve sequential sampling capability. This test is applicable to all Class I candidate samplers in which the aerosol flow path (the flow path through which sample air passes upstream of sample collection filter) differs significantly from that specified for reference method samplers as specified in 40 CFR

part 50, appendix L or appendix O, as applicable. The test requirements and performance specifications for this test are summarized in table E–1 of this subpart.

(b) *Technical definitions.* (1) Aerosol transport is the percentage of a laboratory challenge aerosol which penetrates to the active sample filter of the candidate equivalent method sampler.

(2) The active sample filter is the exclusive filter through which sample air is flowing during performance of this test.

(3) A no-flow filter is a sample filter through which no sample air is intended to flow during performance of this test.

(4) A channel is any of two or more flow paths that the aerosol may take, only one of which may be active at a time.

(5) An added component is any physical part of the sampler which is different in some way from that specified for a reference method sampler in 40 CFR part 50, appendix L or appendix O, as applicable, such as a device or means to allow or cause the aerosol to be routed to one of several channels.

(c) *Required facilities and test equipment.* (1) Aerosol generation system, as specified in §53.62(c)(2).

(2) Aerosol delivery system, as specified in §53.64(c)(2).

(3) Particle size verification equipment, as specified in §53.62(c)(3).

(4) Fluorometer, as specified in §53.62(c)(7).

(5) Candidate test sampler, with the inlet and impactor or impactors removed, and with all internal surfaces of added components electroless nickel coated as specified in §53.64(d)(2).

(6) Filters that are appropriate for use with fluorometric methods (e.g., glass fiber).

(d) *Calibration of test measurement instruments.* Submit documentation showing evidence of appropriately recent calibration, certification of calibration accuracy, and NIST-traceability (if required) of all measurement instruments used in the tests. The accuracy of flow rate meters shall be verified at the highest and lowest pressures and temperatures used in the tests and shall be checked at zero and at least one flow rate within ±3 percent of 16.7 L/min within 7 days prior to use for this test. Where an instrument's measurements are to be recorded with an analog recording device, the accuracy of the entire instrument-recorder system shall be calibrated or verified.

(e) *Test setup.* (1) The candidate test sampler shall have its inlet and impactor or impactors removed. The lower end of the down tube shall be reconnected to the filter holder, using an extension of the downtube, if necessary. If the candidate sampler has a separate impactor for each channel, then for this test, the filter holder assemblies must be connected to the physical location on the sampler where the impactors would normally connect.

(2) The test particle delivery system shall be connected to the sampler downtube so that the test aerosol is introduced at the top of the downtube.

(f) *Test procedure.* (1) All surfaces of the added or modified component or components which come in contact with the aerosol flow shall be thoroughly washed with 0.01 N NaOH and then dried.

(2) Generate aerosol. (i) Generate aerosol composed of oleic acid with a uranine fluorometric tag of 3 ±0.25 μm aerodynamic diameter using a vibrating orifice aerosol generator according to conventions specified in §53.61(g).

(ii) Check for the presence of satellites and adjust the generator to minimize their production.

(iii) Calculate the aerodynamic particle size using the operating parameters of the vibrating orifice aerosol generator. The calculated aerodynamic diameter must be 3 ±0.25 μm aerodynamic diameter.

(3) Verify the particle size according to procedures specified in §53.62(d)(4)(i).

(4) Collect particles on filters for a time period such that the relative error of the resulting measured fluorometric concentration for the active filter is less than 5 percent.

(5) Determine the quantity of material collected on the active filter using a calibrated fluorometer. Record the mass of fluorometric material for the active filter as $M_{active\ (i)}$ where i = the active channel number.

(6) Determine the quantity of material collected on each no-flow filter using a calibrated fluorometer. Record

the mass of fluorometric material on each no-flow filter as $M_{no\text{-}flow}$.

(7) Using 0.01 N NaOH, wash the surfaces of the added component or components which contact the aerosol flow. Determine the quantity of material collected using a calibrated fluorometer. Record the mass of fluorometric material collected in the wash as $M_{wash}$.

(8) Calculate the aerosol transport as:

EQUATION 29

$$T_{(i)} = \frac{M_{active}}{M_{active+}M_{wash+}\sum M_{no\text{-}flow}} \text{ x } 100\%$$

where:

i = the active channel number.

(9) Repeat paragraphs (f)(1) through (8) of this section for each channel, making each channel in turn the exclusive active channel.

(g) *Test results.* The candidate Class I sampler passes the aerosol transport test if $T_{(i)}$ is at least 97 percent for each channel.

[62 FR 38799, July 18, 1997, as amended at 71 FR 61293, Oct. 17, 2006]

TABLE E–1 TO SUBPART E OF PART 53—SUMMARY OF TEST REQUIREMENTS FOR REFERENCE AND CLASS I EQUIVALENT METHODS FOR $PM_{2.5}$ AND $PM_{10-2.5}$

| Subpart E procedure | Performance test | Performance specification | Test conditions | Part 50, appendix L reference |
|---|---|---|---|---|
| § 53.52 Sample leak check test. | Sampler leak check facility. | External leakage: 80 mL/min, max.<br>Internal leakage: 80 mL/min, max. | Controlled leak flow rate of 80 mL/min. | Sec. 7.4.6. |
| § 53.53 Base flow rate test. | Sample flow rate .....<br>1. Mean .....<br>2. Regulation .....<br>3. Meas accuracy .....<br>4. CV accuracy .....<br>5. Cut-off ..... | 1. 16.67 ±5%, L/min .....<br>2. 2%, max .....<br>3. 2%, max .....<br>4. 0.3% max .....<br>5. Flow rate cut-off if flow rate deviates more than 10% from design flow rate for >60 ±30 seconds. | (a) 6-hour normal operational test plus flow rate cut-off test.<br>(b) Normal conditions .....<br>(c) Additional 55 mm Hg pressure drop to simulate loaded filter.<br>(d) Variable flow restriction used for cut-off test. | Sec. 7.4.1.<br>Sec. 7.4.2.<br>Sec. 7.4.3.<br>Sec. 7.4.4.<br>Sec. 7.4.5. |
| § 53.54 Power interruption test. | Sample flow rate .....<br>1. Mean .....<br>2. Regulation .....<br>3. Meas. accuracy .....<br>4. CV accuracy .....<br>5. Occurrence time of power interruptions.<br>6. Elapsed sample time<br>7. Sample volume ..... | 1. 16.67 ±5%, L/min .....<br>2. 2%, max .....<br>3. 2%, max .....<br>4. 0.3% max .....<br>5. ±2 min if >60 seconds.<br>6. ±20 seconds .....<br>7. ±2%, max ..... | (a) 6-hour normal operational test.<br>(b) Nominal conditions .....<br>(c) Additional 55 mm Hg pressure drop to simulate loaded filter.<br>(d) 6 power interruptions of various durations. | Sec. 7.4.1.<br>Sec. 7.4.2.<br>Sec. 7.4.3.<br>Sec. 7.4.5.<br>Sec. 7.4.12.<br>Sec. 7.4.13.<br>Sec. 7.4.15.4.<br>Sec. 7.4.15.5. |
| § 53.55 Temperature and line voltage test. | Sample flow rate .....<br>1. Mean .....<br>2. Regulation .....<br>3. Meas. accuracy .....<br>4. CV accuracy .....<br>5. Temperature meas. accuracy.<br>6. Proper operation. | 1. 16.67 ±5%, L/min .....<br>2. 2%, max .....<br>3. 2%, max .....<br>4. 0.3% max .....<br>5. 2 °C ..... | (a) 6-hour normal operational test.<br>(b) Normal conditions .....<br>(c) Additional 55 mm Hg pressure drop to simulate loaded filter.<br>(d) Ambient temperature at −20 and + 40 °C.<br>(e) Line voltage: 105 Vac to 125 Vac. | Sec. 7.4.1.<br>Sec. 7.4.2.<br>Sec. 7.4.3.<br>Sec. 7.4.5.<br>Sec. 7.4.8.<br>Sec. 7.4.15.1. |
| § 53.56 Barometric pressure effect test. | Sample flow rate .....<br>1. Mean .....<br>2. Regulation .....<br>3. Meas. accuracy .....<br>4. CV accuracy .....<br>5. Pressure meas. accuracy.<br>6. Proper operation. | 1. 16.67 ±5%, L/p;min .....<br>2. 2%, max .....<br>3. 2%, max .....<br>4. 0.3% max .....<br>5. 10 mm Hg ..... | (a) 6-hour normal operational test.<br>(b) Normal conditions .....<br>(c) Additional 55 mm Hg pressure drop to simulate loaded filter.<br>(d) Barometric pressure at 600 and 800 mm Hg. | Sec. 7.4.1.<br>Sec. 7.4.2.<br>Sec. 7.4.3.<br>Sec. 7.4.5.<br>Sec. 7.4.9. |

| Subpart E procedure | Performance test | Performance specification | Test conditions | Part 50, appendix L reference |
|---|---|---|---|---|
| § 53.57 Filter temperature control test. | 1. Filter temp. meas. accuracy.<br>2. Ambient temp. meas. accuracy.<br>3. Filter temp. control accuracy, sampling and non-sampling. | 1. 2 °C .....<br>2. 2 °C .....<br>3. Not more than 5 °C above ambient temp. for more than 30 min. | (a) 4-hour simulated solar radiation, sampling.<br>(b) 4-hour simulated solar radiation, non-sampling.<br>(c) Solar flux of 1000 ±50 W/m $^2$. | Sec. 7.4.8.<br>Sec. 7.4.10.<br>Sec. 7.4.11. |
| § 53.58 Field precision test. | 1. Measurement precision.<br>2. Storage deposition test for sequential samplers. | 1. $P_j$ <2 μg/m $^3$ or $RP_j$ <5%.<br>2. 50 μg max. average weight gain/blank filter. | (a) 3 collocated samplers at 1 site for at least 10 days.<br>(b) $PM_{2.5}$ conc. >3 μg/m $^3$ ...<br>(c) 24- or 48-hour samples<br>(d) 5- or 10-day storage period for inactive stored filters. | Sec. 5.1.<br>Sec. 7.3.5.<br>Sec. 8.<br>Sec. 9.<br>Sec. 10. |
| The Following Requirement Is Applicable to Class I Candidate Equivalent Methods Only | | | | |
| § 53.59 Aerosol transport test. | Aerosol transport ..... | 97%, min. for all channels.. | Determine aerosol transport through any new or modified components with respect to the reference method sampler before the filter for each channel. | |

[72 FR 32208, June 12, 2007]

TABLE E–2 TO SUBPART E OF PART 53—SPECTRAL ENERGY DISTRIBUTION AND PERMITTED TOLERANCE FOR CONDUCTING RADIATIVE TESTS

| Characteristic | Spectral Region | | | |
|---|---|---|---|---|
| | Ultraviolet | | Visible | Infrared |
| Bandwidth (μm) | 0.28 to 0.32 | 0.32 to 0.40 | 0.40 to 0.78 | 0.78 to 3.00 |
| Irradiance (W/m$^2$) | 5 | 56 | 450 to 550 | 439 |
| Allowed Tolerance | ±35% | ±25% | ±10% | ±10% |

[62 FR 38799, July 18, 1997; 63 FR 7714, Feb. 17, 1998]

FIGURE E–1 TO SUBPART E OF PART 53—DESIGNATION TESTING CHECKLIST

**DESIGNATION TESTING CHECKLIST**

| Auditee | Auditor signature | Date |
|---|---|---|

| Compliance Status: Y = Yes N = No NA = Not applicable/Not available | | | | Verification Comments (Includes documentation of who, what, where, when, why) (Doc. #, Rev. #, Rev. Date) |
|---|---|---|---|---|
| Verification | | | Verified by Direct Observation of Process or of Documented Evidence: Performance, Design or Application Spec. Corresponding to Sections of 40 CFR Part 53 or 40 CFR Part 50, Appendix L | |
| Y | N | NA | | |
| | | | **Performance Specification Tests**<br>Sample flow rate coefficient of variation (§ 53.53) (L–7.4.3) | |
| | | | Filter temperature control (sampling) (§ 53.57) (L–7.4.10) | |
| | | | Elapsed sample time accuracy (§ 53.54) (L–7.4.13) | |
| | | | Filter temperature control (post sampling) (§ 53.57) (L–7.4.10) | |
| | | | **Application Specification Tests** | |
| | | | Field Precision (§ 53.58) (L–5.1) | |

| Compliance Status: Y = Yes N = No NA = Not applicable/Not available | | | | Verification Comments (Includes documentation of who, what, where, when, why) (Doc. #, Rev. #, Rev. Date) |
|---|---|---|---|---|
| Verification | | | Verified by Direct Observation of Process or of Documented Evidence: Performance, Design or Application Spec. Corresponding to Sections of 40 CFR Part 53 or 40 CFR Part 50, Appendix L | |
| Y | N | NA | | |
| | | | Meets all Appendix L requirements (part 53, subpart A, § 53.2(a)(3)) (part 53, subpart E, § 53.51(a),(d)) | |
| | | | Filter Weighing (L–8) | |
| | | | Field Sampling Procedure (§ 53.30, .31, .34) | |
| | | | **Design Specification Tests** | |
| | | | Filter (L–6) | |
| | | | Range of Operational Conditions (L–7.4.7) | |
| The Following Requirements Apply Only to Class I Candidate Equivalent Methods | | | | |
| | | | Aerosol Transport (§ 53.59) | |

## APPENDIX A TO SUBPART E 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) 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/pmqainf.html*.

(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.

(5) Quality Assurance Handbook for Air Pollution Measurement Systems, Volume IV: Meteorological Measurements. Revised March, 1995. EPA–600/R–94–038d. Available from National Technical Information Service, Springfield, VA 22161, (800–553–6847, *http://www.ntis.gov*). NTIS number PB95–199782INZ.

(6) Military standard specification (mil. spec.) 810-E 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.

[62 FR 38799, July 18, 1997, as amended at 71 FR 61295, Oct. 17, 2006]

# Subpart F—Procedures for Testing Performance Characteristics of Class II Equivalent Methods for $PM_{2.5}$

SOURCE: 62 FR 38814, July 18, 1997, unless otherwise noted.

### § 53.60 General provisions.

(a) This subpart sets forth the specific requirements that a $PM_{2.5}$ sampler associated with a candidate Class II equivalent method must meet to be designated as an equivalent method for $PM_{2.5}$. This subpart also sets forth the explicit test procedures that must be carried out and the test results, evidence, documentation, and other materials that must be provided to EPA to demonstrate that a sampler meets all specified requirements for designation as an equivalent method.

(b) A candidate method described in an application for a FRM or FEM determination submitted under § 53.4 shall be determined by the EPA to be a Class II candidate equivalent method on the basis of the definition of a Class II FEM in § 53.1.

(c) Any sampler associated with a Class II candidate equivalent method (Class II sampler) must meet all applicable requirements for FRM samplers

or Class I FEM samplers specified in subpart E of this part, as appropriate. Except as provided in §53.3(a)(3), a Class II $PM_{2.5}$ sampler must meet the additional requirements as specified in paragraph (d) of this section.

(d) Except as provided in paragraphs (d)(1), (2), and (3) of this section, all Class II samplers are subject to the additional tests and performance requirements specified in §53.62 (full wind tunnel test), §53.65 (loading test), and §53.66 (volatility test). Alternative tests and performance requirements, as described in paragraphs (d)(1), (2), and (3) of this section, are optionally available for certain Class II samplers which meet the requirements for reference method or Class I equivalent method samplers given in 40 CFR part 50, appendix L, and in subpart E of this part, except for specific deviations of the inlet, fractionator, or filter.

(1) *Inlet deviation*. A sampler which has been determined to be a Class II sampler solely because the design or construction of its inlet deviates from the design or construction of the inlet specified in 40 CFR part 50, appendix L, for reference method samplers shall not be subject to the requirements of §53.62 (full wind tunnel test), provided that it meets all requirements of §53.63 (wind tunnel inlet aspiration test), §53.65 (loading test), and §53.66 (volatility test).

(2) *Fractionator deviation*. A sampler which has been determined to be a Class II sampler solely because the design or construction of its particle size fractionator deviates from the design or construction of the particle size fractionator specified in 40 CFR part 50, appendix L for reference method samplers shall not be subject to the requirements of §53.62 (full wind tunnel test), provided that it meets all requirements of §53.64 (static fractionator test), §53.65 (loading test), and §53.66 (volatility test).

(3) *Filter size deviation*. A sampler which has been determined to be a Class II sampler solely because its effective filtration area deviates from that of the reference method filter specified in 40 CFR part 50, appendix L, for reference method samplers shall not be subject to the requirements of §53.62 (full wind tunnel test) nor §53.65 (loading test), provided it meets all requirements of §53.66 (volatility test).

(e) *The test specifications and acceptance criteria for each test are summarized in table F–1 of this subpart*. The candidate sampler must demonstrate performance that meets the acceptance criteria for each applicable test to be designated as an equivalent method.

(f) *Overview of various test procedures for Class II samplers*—(1) *Full wind tunnel test*. This test procedure is designed to ensure that the candidate sampler's effectiveness (aspiration of an ambient aerosol and penetration of the sub 2.5-micron fraction to its sample filter) will be comparable to that of a reference method sampler. The candidate sampler is challenged at wind speeds of 2 and 24 km/hr with monodisperse aerosols of the size specified in table F–2 of this subpart. The experimental test results are then integrated with three idealized ambient distributions (typical, fine, and coarse) to yield the expected mass concentration measurement for each. The acceptance criteria are based on the results of this numerical analysis and the particle diameter for which the sampler effectiveness is 50 percent.

(2) *Wind tunnel inlet aspiration test*. The wind tunnel inlet aspiration test directly compares the inlet of the candidate sampler to the inlet of a reference method sampler with the single-sized, liquid, monodisperse challenge aerosol specified in table F–2 of this subpart at wind speeds of 2 km/hr and 24 km/hr. The acceptance criteria, presented in table F–1 of this subpart, is based on the relative aspiration between the candidate inlet and the reference method inlet.

(3) *Static fractionator test*. The static fractionator test determines the effectiveness of the candidate sampler's 2.5-micron fractionator under static conditions for aerosols of the size specified in table F–2 of this subpart. The numerical analysis procedures and acceptance criteria are identical to those in the full wind tunnel test.

(4) *Loading test*. The loading test is conducted to ensure that the performance of a candidate sampler is not significantly affected by the amount of particulate deposited on its interior surfaces between periodic cleanings.

The candidate sampler is artificially loaded by sampling a test environment containing aerosolized, standard test dust. The duration of the loading phase is dependent on both the time between cleaning as specified by the candidate method and the aerosol mass concentration in the test environment. After loading, the candidate's performance must then be evaluated by § 53.62 (full wind tunnel evaluation), § 53.63 (wind tunnel inlet aspiration test), or § 53.64 (static fractionator test). If the results of the appropriate test meet the criteria presented in table F–1 of this subpart, then the candidate sampler passes the loading test under the condition that it be cleaned at least as often as the cleaning frequency proposed by the candidate method and that has been demonstrated to be acceptable by this test.

(5) *Volatility test.* The volatility test challenges the candidate sampler with a polydisperse, semi-volatile liquid aerosol. This aerosol is simultaneously sampled by the candidate method sampler and a reference method sampler for a specified time period. Clean air is then passed through the samplers during a blow-off time period. Residual mass is then calculated as the weight of the filter after the blow-off phase is subtracted from the initial weight of the filter. Acceptance criteria are based on a comparison of the residual mass measured by the candidate sampler (corrected for flow rate variations from that of the reference method) to the residual mass measured by the reference method sampler for several specified clean air sampling time periods.

(g) *Test data.* All 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 to EPA as part of the equivalent method application. Schematic drawings of each particle delivery system and other information showing complete procedural details of the test atmosphere generation, verification, and delivery techniques for each test performed shall be submitted to EPA. All pertinent calculations shall be clearly presented. In addition, manufacturers are required to submit as part of the application, a Designation Testing Checklist (Figure F–1 of this subpart) which has been completed and signed by an ISO-certified auditor.

[62 FR 38814, July 18, 1997, as amended at 71 FR 61295, Oct. 17, 2006]

### § 53.61 Test conditions.

(a) *Sampler surface preparation.* Internal surfaces of the candidate sampler shall be cleaned and dried prior to performing any Class II sampler test in this subpart. The internal collection surfaces of the sampler shall then be prepared in strict accordance with the operating instructions specified in the sampler's operating manual referred to in section 7.4.18 of 40 CFR part 50, appendix L.

(b) *Sampler setup.* Set up and start up of all test samplers shall be in strict accordance with the operating instructions specified in the manual referred to in section 7.4.18 of 40 CFR part 50, appendix L, unless otherwise specified within this subpart.

(c) *Sampler adjustments.* Once the test sampler or samplers have been set up and the performance tests started, manual adjustment shall be permitted only between test points for all applicable tests. Manual adjustments and any periodic maintenance shall be limited to only those procedures prescribed in the manual referred to in section 7.4.18 of 40 CFR part 50, appendix L. The submitted records shall clearly indicate when any manual adjustment or periodic maintenance was made and shall describe the operations performed.

(d) *Sampler malfunctions.* If a test sampler malfunctions during any of the applicable tests, that test run shall be repeated. A detailed explanation of all malfunctions and the remedial actions taken shall be submitted as part of the equivalent method application.

(e) *Particle concentration measurements.* All measurements of particle concentration must be made such that the relative error in measurement is less than 5.0 percent. Relative error is defined as $(s \times 100 \text{ percent})/(X)$, where s is the sample standard deviation of the particle concentration detector, X is the measured concentration, and the units of s and X are identical.

(f) *Operation of test measurement equipment.* All test measurement equipment shall be set up, calibrated, and maintained by qualified personnel according to the manufacturer's instructions. All appropriate calibration information and manuals for this equipment shall be kept on file.

(g) *Vibrating orifice aerosol generator conventions.* This section prescribes conventions regarding the use of the vibrating orifice aerosol generator (VOAG) for the size-selective performance tests outlined in §§53.62, 53.63, 53.64, and 53.65.

(1) *Particle aerodynamic diameter.* The VOAG produces near-monodisperse droplets through the controlled break-up of a liquid jet. When the liquid solution consists of a non-volatile solute dissolved in a volatile solvent, the droplets dry to form particles of near-monodisperse size.

(i) The physical diameter of a generated spherical particle can be calculated from the operating parameters of the VOAG as:

EQUATION 1

$$D_p = \left(\frac{6\ Q\ C_{vol}}{\pi\ f}\right)^{1/3}$$

where:

$D_p$ = particle physical diameter, μm;
Q = liquid volumetric flow rate, $\mu m^3$/sec;
$C_{vol}$ = volume concentration (particle volume produced per drop volume), dimensionless; and
f = frequency of applied vibrational signal, 1/sec.

(ii) A given particle's aerodynamic behavior is a function of its physical particle size, particle shape, and density. Aerodynamic diameter is defined as the diameter of a unit density ($\rho_o$ = 1g/$cm^3$) sphere having the same settling velocity as the particle under consideration. For converting a spherical particle of known density to aerodynamic diameter, the governing relationship is:

EQUATION 2

$$D_{ae} = \frac{\sqrt{\rho_p}\sqrt{C_{D_p}}D_p}{\sqrt{\rho_o}\sqrt{C_{D_{ae}}}}$$

where:

$D_{ae}$ = particle aerodynamic diameter, μm;
$\rho_p$ = particle density, g/$cm^3$;
$\rho_o$ = aerodynamic particle density = 1 g/$cm^3$;
$C_{Dp}$ = Cunningham's slip correction factor for physical particle diameter, dimensionless; and
$C_{Dae}$ = Cunningham's slip correction factor for aerodynamic particle diameter, dimensionless.

(iii) At room temperature and standard pressure, the Cunningham's slip correction factor is solely a function of particle diameter:

EQUATION 3

$$C_{Dae} = 1 + \frac{0.1659}{D_{ae}} + \frac{0.053}{D_{ae}}\ \exp\left(-8.33\ D_{ae}\right)$$

or

EQUATION 4

$$C_{D_p} = 1 + \frac{0.1659}{D_p} + \frac{0.053}{D_p}\ \exp\left(-8.33\ D_p\right)$$

(iv) Since the slip correction factor is itself a function of particle diameter, the aerodynamic diameter in equation 2 of paragraph (g)(1)(ii) of this section cannot be solved directly but must be determined by iteration.

(2) *Solid particle generation.* (i) Solid particle tests performed in this subpart shall be conducted using particles composed of ammonium fluorescein. For use in the VOAG, liquid solutions of known volumetric concentration can be prepared by diluting fluorescein powder ($C_20H_{12}O_5$, FW = 332.31, CAS 2321-07-5) with aqueous ammonia. Guidelines for preparation of fluorescein solutions of the desired volume concentration ($C_{vol}$) are presented by Vanderpool and Rubow (1988) (Reference 2 in appendix A of this subpart). For purposes of converting particle physical diameter to aerodynamic diameter, an ammonium fluorescein density of 1.35 g/$cm^3$ shall be used.

(ii) Mass deposits of ammonium fluorescein shall be extracted and analyzed using solutions of 0.01 N ammonium hydroxide.

(3) *Liquid particle generation.* (i) Tests prescribed in § 53.63 for inlet aspiration require the use of liquid particle tests composed of oleic acid tagged with uranine to enable subsequent fluorometric quantitation of collected aerosol mass deposits. Oleic acid ($C_{18}H_{34}O_2$, FW = 282.47, CAS 112-80-1) has a density of 0.8935 g/cm$^3$. Because the viscosity of oleic acid is relatively high, significant errors can occur when dispensing oleic acid using volumetric pipettes. For this reason, it is recommended that oleic acid solutions be prepared by quantifying dispensed oleic acid gravimetrically. The volume of oleic acid dispensed can then be calculated simply by dividing the dispensed mass by the oleic acid density.

(ii) Oleic acid solutions tagged with uranine shall be prepared as follows. A known mass of oleic acid shall first be diluted using absolute ethanol. The desired mass of the uranine tag should then be diluted in a separate container using absolute ethanol. Uranine ($C_{20}H_{10}O_5Na_2$, FW = 376.3, CAS 518-47-8) is the disodium salt of fluorescein and has a density of 1.53 g/cm$^3$. In preparing uranine tagged oleic acid particles, the uranine content shall not exceed 20 percent on a mass basis. Once both oleic acid and uranine solutions are properly prepared, they can then be combined and diluted to final volume using absolute ethanol.

(iii) Calculation of the physical diameter of the particles produced by the VOAG requires knowledge of the liquid solution's volume concentration ($C_{vol}$). Because uranine is essentially insoluble in oleic acid, the total particle volume is the sum of the oleic acid volume and the uranine volume. The volume concentration of the liquid solution shall be calculated as:

EQUATION 5

$$C_{vol} = \frac{V_u + V_{oleic}}{V_{sol}} = \frac{\left(M_u/\rho_u\right) + \left(M_{oleic}/\rho_{oleic}\right)}{V_{sol}}$$

where:

$V_u$ = uranine volume, ml;
$V_{oleic}$ = oleic acid volume, ml;
$V_{sol}$ = total solution volume, ml;
$M_u$ = uranine mass, g;
$\rho_u$ = uranine density, g/cm$^3$;
$M_{oleic}$ = oleic acid mass, g; and
$\rho_{oleic}$ = oleic acid density, g/cm.$^3$

(iv) For purposes of converting the particles' physical diameter to aerodynamic diameter, the density of the generated particles shall be calculated as:

EQUATION 6

$$\rho_p = \frac{M_u + M_{oleic}}{\left(M_u/\rho_u\right) + \left(M_{oleic}/\rho_{oleic}\right)}$$

(v) Mass deposits of oleic acid shall be extracted and analyzed using solutions of 0.01 N sodium hydroxide.

[62 FR 38814, July 18, 1997; 63 FR 7714, Feb. 17, 1998]

**§ 53.62 Test procedure: Full wind tunnel test.**

(a) *Overview.* The full wind tunnel test evaluates the effectiveness of the candidate sampler at 2 km/hr and 24 km/hr for aerosols of the size specified in table F–2 of this subpart (under the heading, ''Full Wind Tunnel Test''). For each wind speed, a smooth curve is fit to the effectiveness data and corrected for the presence of multiplets in the wind tunnel calibration aerosol. The cutpoint diameter ($D_{p50}$) at each wind speed is then determined from the corrected effectiveness curves. The two resultant penetration curves are then each numerically integrated with three idealized ambient particle size distributions to provide six estimates of measured mass concentration. Critical parameters for these idealized distributions are presented in table F–3 of this subpart.

(b) *Technical definitions.* Effectiveness is the ratio (expressed as a percentage) of the mass concentration of particles of a specific size reaching the sampler filter or filters to the mass concentration of particles of the same size approaching the sampler.

(c) *Facilities and equipment required*—(1) *Wind tunnel.* The particle delivery system shall consist of a blower system and a wind tunnel having a test section of sufficiently large cross-sectional

area such that the test sampler, or portion thereof, as installed in the test section for testing, blocks no more than 15 percent of the test section area. The wind tunnel blower system must be capable of maintaining uniform wind speeds at the 2 km/hr and 24 km/hr in the test section.

(2) *Aerosol generation system.* A vibrating orifice aerosol generator shall be used to produce monodisperse solid particles of ammonium fluorescein with equivalent aerodynamic diameters as specified in table F–2 of this subpart. The geometric standard deviation for each particle size generated shall not exceed 1.1 (for primary particles) and the proportion of multiplets (doublets and triplets) in all test particle atmosphere shall not exceed 10 percent of the particle population. The aerodynamic particle diameter, as established by the operating parameters of the vibrating orifice aerosol generator, shall be within the tolerance specified in table F–2 of this subpart.

(3) *Particle size verification equipment.* The size of the test particles shall be verified during this test by use of a suitable instrument (e.g., scanning electron microscope, optical particle sizer, time-of-flight apparatus). The instrument must be capable of measuring solid and liquid test particles with a size resolution of 0.1 μm or less. The accuracy of the particle size verification technique shall be 0.15 μm or better.

(4) *Wind speed measurement.* The wind speed in the wind tunnel shall be determined during the tests using an appropriate technique capable of a precision of 2 percent and an accuracy of 5 percent or better (e.g., hot-wire anemometry). For the wind speeds specified in table F–2 of this subpart, the wind speed shall be measured at a minimum of 12 test points in a cross-sectional area of the test section of the wind tunnel. The mean wind speed in the test section must be within ±10 percent of the value specified in table F–2 of this subpart, and the variation at any test point in the test section may not exceed 10 percent of the measured mean.

(5) *Aerosol rake.* The cross-sectional uniformity of the particle concentration in the sampling zone of the test section shall be established during the tests using an array of isokinetic samplers, referred to as a rake. Not less than five evenly spaced isokinetic samplers shall be used to determine the particle concentration spatial uniformity in the sampling zone. The sampling zone shall be a rectangular area having a horizontal dimension not less than 1.2 times the width of the test sampler at its inlet opening and a vertical dimension not less than 25 centimeters.

(6) *Total aerosol isokinetic sampler.* After cross-sectional uniformity has been confirmed, a single isokinetic sampler may be used in place of the array of isokinetic samplers for the determination of particle mass concentration used in the calculation of sampling effectiveness of the test sampler in paragraph (d)(5) of this section. In this case, the array of isokinetic samplers must be used to demonstrate particle concentration uniformity prior to the replicate measurements of sampling effectiveness.

(7) *Fluorometer.* A fluorometer used for quantifying extracted aerosol mass deposits shall be set up, maintained, and calibrated according to the manufacturer's instructions. A series of calibration standards shall be prepared to encompass the minimum and maximum concentrations measured during size-selective tests. Prior to each calibration and measurement, the fluorometer shall be zeroed using an aliquot of the same solvent used for extracting aerosol mass deposits.

(8) *Sampler flow rate measurements.* All flow rate measurements used to calculate the test atmosphere concentrations and the test results must be accurate to within ±2 percent, referenced to a NIST-traceable primary standard. Any necessary flow rate measurement corrections shall be clearly documented. All flow rate measurements shall be performed and reported in actual volumetric units.

(d) *Test procedures*—(1) *Establish and verify wind speed.* (i) Establish a wind speed specified in table F–2 of this subpart.

(ii) Measure the wind speed at a minimum of 12 test points in a cross-sectional area of the test section of the wind tunnel using a device as described in paragraph (c)(4) of this section.

(iii) Verify that the mean wind speed in the test section of the wind tunnel during the tests is within 10 percent of the value specified in table F–2 of this subpart. The wind speed measured at any test point in the test section shall not differ by more than 10 percent from the mean wind speed in the test section.

(2) *Generate aerosol.* (i) Generate particles of a size specified in table F–2 of this subpart using a vibrating orifice aerosol generator.

(ii) Check for the presence of satellites and adjust the generator as necessary.

(iii) Calculate the physical particle size using the operating parameters of the vibrating orifice aerosol generator and record.

(iv) Determine the particle's aerodynamic diameter from the calculated physical diameter and the known density of the generated particle. The calculated aerodynamic diameter must be within the tolerance specified in table F–2 of this subpart.

(3) *Introduce particles into the wind tunnel.* Introduce the generated particles into the wind tunnel and allow the particle concentration to stabilize.

(4) *Verify the quality of the test aerosol.* (i) Extract a representative sample of the aerosol from the sampling test zone and measure the size distribution of the collected particles using an appropriate sizing technique. If the measurement technique does not provide a direct measure of aerodynamic diameter, the geometric mean aerodynamic diameter of the challenge aerosol must be calculated using the known density of the particle and the measured mean physical diameter. The determined geometric mean aerodynamic diameter of the test aerosol must be within 0.15 μm of the aerodynamic diameter calculated from the operating parameters of the vibrating orifice aerosol generator. The geometric standard deviation of the primary particles must not exceed 1.1.

(ii) Determine the population of multiplets in the collected sample. The multiplet population of the particle test atmosphere must not exceed 10 percent of the total particle population.

(5) *Aerosol uniformity and concentration measurement.* (i) Install an array of five or more evenly spaced isokinetic samplers in the sampling zone (paragraph (c)(5) of this section). Collect particles on appropriate filters over a time period such that the relative error of the measured particle concentration is less than 5.0 percent.

(ii) Determine the quantity of material collected with each isokinetic sampler in the array using a calibrated fluorometer. Calculate and record the mass concentration for each isokinetic sampler as:

EQUATION 7

$$C_{iso(ij)} = \frac{M_{iso(ij)}}{Q_{(ij)} \times t_{(ij)}}$$

where:

i = replicate number;
j = isokinetic sampler number;
$M_{iso}$ = mass of material collected with the isokinetic sampler;
Q = isokinetic sampler volumetric flow rate; and
t = sampling time.

(iii) Calculate and record the mean mass concentration as:

EQUATION 8

$$\overline{C}_{iso(i)} = \frac{\sum_{j=1}^{n} C_{iso(ij)}}{n}$$

where:

i = replicate number;
j = isokinetic sampler number; and
n = total number of isokinetic samplers.

(iv) Precision calculation. (A) Calculate the coefficient of variation of the mass concentration measurements as:

EQUATION 9

$$CV_{iso(i)} = \frac{\sqrt{\sum_{j=1}^{n} C_{iso(ij)}^2 - \frac{1}{n}\left(\sum_{j=1}^{n} C_{iso(ij)}\right)^2}}{n-1} / \overline{C}_{iso(i)} \times 100\%$$

where:

i = replicate number;
j = isokinetic sampler number; and
n = total number of isokinetic samplers.

(B) If the value of $CV_{iso(i)}$ for any replicate exceeds 10 percent, the particle concentration uniformity is unacceptable and step 5 must be repeated. If adjustment of the vibrating orifice aerosol generator or changes in the particle delivery system are necessary to achieve uniformity, steps 1 through 5 must be repeated. When an acceptable aerosol spatial uniformity is achieved, remove the array of isokinetic samplers from the wind tunnel.

(6) *Alternative measure of wind tunnel total concentration.* If a single isokinetic sampler is used to determine the mean aerosol concentration in the wind tunnel, install the sampler in the wind tunnel with the sampler nozzle centered in the sampling zone (paragraph (c)(6) of this section).

(i) Collect particles on an appropriate filter over a time period such that the relative error of the measured concentration is less than 5.0 percent.

(ii) Determine the quantity of material collected with the isokinetic sampler using a calibrated fluorometer.

(iii) Calculate and record the mass concentration as $C_{iso(i)}$ as in paragraph (d)(5)(ii) of this section.

(iv) Remove the isokinetic sampler from the wind tunnel.

(7) *Measure the aerosol with the candidate sampler.* (i) Install the test sampler (or portion thereof) in the wind tunnel with the sampler inlet opening centered in the sampling zone. To meet the maximum blockage limit of paragraph (c)(1) of this section or for convenience, part of the test sampler may be positioned external to the wind tunnel provided that neither the geometry of the sampler nor the length of any connecting tube or pipe is altered. Collect particles for a time period such that the relative error of the measured concentration is less than 5.0 percent.

(ii) Remove the test sampler from the wind tunnel.

(iii) Determine the quantity of material collected with the test sampler using a calibrated fluorometer. Calculate and record the mass concentration for each replicate as:

EQUATION 10

$$C_{cand(i)} = \frac{M_{cand(i)}}{Q_{(i)} \times t_{(i)}}$$

where:

i = replicate number;

$M_{cand}$ = mass of material collected with the candidate sampler;

Q = candidate sampler volumetric flow rate; and

t = sampling time.

(iv)(A) Calculate and record the sampling effectiveness of the candidate sampler as:

EQUATION 11

$$E_{(i)} = \frac{C_{cand(i)}}{\overline{C}_{iso(i)}} \times 100\%$$

where:

i = replicate number.

(B) If a single isokinetic sampler is used for the determination of particle mass concentration, replace $C_{iso(i)}$ with $C_{iso}$.

(8) *Replicate measurements and calculation of mean sampling effectiveness.* (i) Repeat steps in paragraphs (d)(5) through (d)(7) of this section, as appropriate, to obtain a minimum of three valid replicate measurements of sampling effectiveness.

(ii) Calculate and record the average sampling effectiveness of the test sampler for the particle size as:

EQUATION 12

$$\overline{E} = \frac{\sum_{i=1}^{n} E_{(i)}}{n}$$

where:

i = replicate number; and

n = number of replicates.

(iii) Sampling effectiveness precision. (A) Calculate and record the coefficient of variation for the replicate sampling effectiveness measurements of the test sampler as:

EQUATION 13

$$CV_E = \frac{\sqrt{\sum_{i=1}^{n} E_{(i)}^2 - \frac{1}{n}\left(\sum_{i=1}^{n} E_{(i)}\right)^2}}{n-1} \frac{1}{\overline{E}} \times 100\%$$

where:

i = replicate number, and
n = number of replicates.

(B) If the value of $CV_E$ exceeds 10 percent, the test run (steps in paragraphs (d)(2) through (d)(8) of this section) must be repeated until an acceptable value is obtained.

(9) Repeat steps in paragraphs (d)(2) through (d)(8) of this section until the sampling effectiveness has been measured for all particle sizes specified in table F–2 of this subpart.

(10) Repeat steps in paragraphs (d)(1) through (d)(9) of this section until tests have been successfully conducted for both wind speeds of 2 km/hr and 24 km/hr.

(e) *Calculations*—(1) *Graphical treatment of effectiveness data.* For each wind speed given in table F–2 of this subpart, plot the particle average sampling effectiveness of the candidate sampler as a function of aerodynamic particle diameter ($D_{ae}$) on semi-logarithmic graph paper where the aerodynamic particle diameter is the particle size established by the parameters of the VOAG in conjunction with the known particle density. Construct a best-fit, smooth curve through the data by extrapolating the sampling effectiveness curve through 100 percent at an aerodynamic particle size of 0.5 μm and 0 percent at an aerodynamic particle size of 10 μm. Correction for the presence of multiplets shall be performed using the techniques presented by Marple, et al (1987). This multiplet-corrected effectiveness curve shall be used for all remaining calculations in this paragraph (e).

(2) *Cutpoint determination.* For each wind speed determine the sampler $Dp_{50}$ cutpoint defined as the aerodynamic particle size corresponding to 50 percent effectiveness from the multiplet corrected smooth curve.

(3) *Expected mass concentration calculation.* For each wind speed, calculate the estimated mass concentration measurement for the test sampler under each particle size distribution (Tables F–4, F–5, and F–6 of this subpart) and compare it to the mass concentration predicted for the reference sampler as follows:

(i) Determine the value of corrected effectiveness using the best-fit, multiplet-corrected curve at each of the particle sizes specified in the first column of table F–4 of this subpart. Record each corrected effectiveness value as a decimal between 0 and 1 in column 2 of table F–4 of this subpart.

(ii) Calculate the interval estimated mass concentration measurement by multiplying the values of corrected effectiveness in column 2 by the interval mass concentration values in column 3 and enter the products in column 4 of table F–4 of this subpart.

(iii) Calculate the estimated mass concentration measurement by summing the values in column 4 and entering the total as the estimated mass concentration measurement for the test sampler at the bottom of column 4 of table F–4 of this subpart.

(iv) Calculate the estimated mass concentration ratio between the candidate method and the reference method as:

EQUATION 14

$$R_c = \frac{C_{cand(est)}}{C_{ref(est)}} \times 100\%$$

where:

$C_{cand(est)}$ = estimated mass concentration measurement for the test sampler, μg/m³; and

$C_{ref(est)}$ = estimated mass concentration measurement for the reference sampler, μg/m³ (calculated for the reference sampler and specified at the bottom of column 7 of table F–4 of this subpart).

(v) Repeat steps in paragraphs (e) (1) through (e)(3) of this section for tables F–5 and F–6 of this subpart.

(f) *Evaluation of test results.* The candidate method passes the wind tunnel effectiveness test if the $R_c$ value for each wind speed meets the specification in table F–1 of this subpart for each of the three particle size distributions.

**§53.63 Test procedure: Wind tunnel inlet aspiration test.**

(a) *Overview*. This test applies to a candidate sampler which differs from the reference method sampler only with respect to the design of the inlet. The purpose of this test is to ensure that the aspiration of a Class II candidate sampler is such that it representatively extracts an ambient aerosol at elevated wind speeds. This wind tunnel test uses a single-sized, liquid aerosol in conjunction with wind speeds of 2 km/hr and 24 km/hr. The test atmosphere concentration is alternately measured with the candidate sampler and a reference method device, both of which are operated without the 2.5-micron fractionation device installed. The test conditions are summarized in table F–2 of this subpart (under the heading of ''wind tunnel inlet aspiration test''). The candidate sampler must meet or exceed the acceptance criteria given in table F–1 of this subpart.

(b) *Technical definition*. Relative aspiration is the ratio (expressed as a percentage) of the aerosol mass concentration measured by the candidate sampler to that measured by a reference method sampler.

(c) *Facilities and equipment required*. The facilities and equipment are identical to those required for the full wind tunnel test (§53.62(c)).

(d) *Setup*. The candidate and reference method samplers shall be operated with the $PM_{2.5}$ fractionation device removed from the flow path throughout this entire test procedure. Modifications to accommodate this requirement shall be limited to removal of the fractionator and insertion of the filter holder directly into the downtube of the inlet.

(e) *Test procedure*—(1) *Establish the wind tunnel test atmosphere*. Follow the procedures in §53.62(d)(1) through (d)(4) to establish a test atmosphere for one of the two wind speeds specified in table F–2 of this subpart.

(2) *Measure the aerosol concentration with the reference sampler*. (i) Install the reference sampler (or portion thereof) in the wind tunnel with the sampler inlet opening centered in the sampling zone. To meet the maximum blockage limit of §53.62(c)(1) or for convenience, part of the test sampler may be positioned external to the wind tunnel provided that neither the geometry of the sampler nor the length of any connecting tube or pipe is altered. Collect particles for a time period such that the relative error of the measured concentration is less than 5.0 percent.

(ii) Determine the quantity of material collected with the reference method sampler using a calibrated fluorometer. Calculate and record the mass concentration as:

EQUATION 15

$$C_{ref(i)} = \frac{M_{ref(i)}}{Q_{(i)} \times t_{(i)}}$$

where:

i = replicate number;

$M_{ref}$ = mass of material collected with the reference method sampler;

Q = reference method sampler volumetric flow rate; and

t = sampling time.

(iii) Remove the reference method sampler from the tunnel.

(3) *Measure the aerosol concentration with the candidate sampler*. (i) Install the candidate sampler (or portion thereof) in the wind tunnel with the sampler inlet centered in the sampling zone. To meet the maximum blockage limit of §53.62(c)(1) or for convenience, part of the test sampler may be positioned external to the wind tunnel provided that neither the geometry of the sampler nor the length of any connecting tube or pipe is altered. Collect particles for a time period such that the relative error of the measured concentration is less than 5.0 percent.

(ii) Determine the quantity of material collected with the candidate sampler using a calibrated fluorometer. Calculate and record the mass concentration as:

EQUATION 16

$$C_{cand(i)} = \frac{M_{cand(i)}}{Q_{(i)} \times t_{(i)}}$$

where:

i = replicate number;

$M_{cand}$ = mass of material collected with the candidate sampler;

Q = candidate sampler volumetric flow rate; and
t = sampling time.

(iii) Remove the candidate sampler from the wind tunnel.

(4) Repeat steps in paragraphs (d) (2) and (d)(3) of this section. Alternately measure the tunnel concentration with the reference sampler and the candidate sampler until four reference sampler and three candidate sampler measurements of the wind tunnel concentration are obtained.

(5) *Calculations.* (i) Calculate and record aspiration ratio for each candidate sampler run as:

EQUATION 17

$$A_{(i)} = \frac{C_{cand(i)}}{\left(C_{ref(i)} + C_{ref(i+1)}\right) \times \frac{1}{2}}$$

where:

i = replicate number.

(ii) Calculate and record the mean aspiration ratio as:

EQUATION 18

$$\overline{A} = \frac{\sum_{i=1}^{n} A_{(i)}}{n}$$

where:

i = replicate number; and
n = total number of measurements of aspiration ratio.

(iii) Precision of the aspiration ratio. (A) Calculate and record the precision of the aspiration ratio measurements as the coefficient of variation as:

EQUATION 19

$$CV_A = \frac{\sqrt{\sum_{i=1}^{n} A_{(i)}^2 - \frac{1}{n}\left(\sum_{i=1}^{n} A_{(i)}\right)^2}}{n-1} / \overline{A}_{(i)} \times 100\%$$

where:

i = replicate number; and
n = total number of measurements of aspiration ratio.

(B) If the value of $CV_A$ exceeds 10 percent, the entire test procedure must be repeated.

(f) *Evaluation of test results.* The candidate method passes the inlet aspiration test if all values of A meet the acceptance criteria specified in table F–1 of this subpart.

**§ 53.64 Test procedure: Static fractionator test.**

(a) *Overview.* This test applies only to those candidate methods in which the sole deviation from the reference method is in the design of the 2.5-micron fractionation device. The purpose of this test is to ensure that the fractionation characteristics of the candidate fractionator are acceptably similar to that of the reference method sampler. It is recognized that various methodologies exist for quantifying fractionator effectiveness. The following commonly-employed techniques are provided for purposes of guidance. Other methodologies for determining sampler effectiveness may be used contingent upon prior approval by the Agency.

(1) *Wash-off method.* Effectiveness is determined by measuring the aerosol mass deposited on the candidate sampler's after filter versus the aerosol mass deposited in the fractionator. The material deposited in the fractionator is recovered by washing its internal surfaces. For these wash-off tests, a fluorometer must be used to quantitate the aerosol concentration. Note that if this technique is chosen, the candidate must be reloaded with coarse aerosol prior to each test point when reevaluating the curve as specified in the loading test.

(2) *Static chamber method.* Effectiveness is determined by measuring the aerosol mass concentration sampled by the candidate sampler's after filter versus that which exists in a static chamber. A calibrated fluorometer shall be used to quantify the collected aerosol deposits. The aerosol concentration is calculated as the measured aerosol mass divided by the sampled air volume.

(3) *Divided flow method.* Effectiveness is determined by comparing the aerosol concentration upstream of the candidate sampler's fractionator versus

that concentration which exists downstream of the candidate fractionator. These tests may utilize either fluorometry or a real-time aerosol measuring device to determine the aerosol concentration.

(b) *Technical definition*. Effectiveness under static conditions is the ratio (expressed as a percentage) of the mass concentration of particles of a given size reaching the sampler filter to the mass concentration of particles of the same size existing in the test atmosphere.

(c) *Facilities and equipment required*—(1) *Aerosol generation*. Methods for generating aerosols shall be identical to those prescribed in § 53.62(c)(2).

(2) *Particle delivery system*. Acceptable apparatus for delivering the generated aerosols to the candidate fractionator is dependent on the effectiveness measurement methodology and shall be defined as follows:

(i) *Wash-off test apparatus*. The aerosol may be delivered to the candidate fractionator through direct piping (with or without an in-line mixing chamber). Validation particle size and quality shall be conducted at a point directly upstream of the fractionator.

(ii) *Static chamber test apparatus*. The aerosol shall be introduced into a chamber and sufficiently mixed such that the aerosol concentration within the chamber is spatially uniform. The chamber must be of sufficient size to house at least four total filter samplers in addition to the inlet of the candidate method size fractionator. Validation of particle size and quality shall be conducted on representative aerosol samples extracted from the chamber.

(iii) *Divided flow test apparatus*. The apparatus shall allow the aerosol concentration to be measured upstream and downstream of the fractionator. The aerosol shall be delivered to a manifold with two symmetrical branching legs. One of the legs, referred to as the bypass leg, shall allow the challenge aerosol to pass unfractionated to the detector. The other leg shall accommodate the fractionation device.

(3) *Particle concentration measurement*—(i) *Fluorometry*. Refer to § 53.62(c)(7).

(ii) *Number concentration measurement*. A number counting particle sizer may be used in conjunction with the divided flow test apparatus in lieu of fluorometric measurement. This device must have a minimum range of 1 to 10 μm, a resolution of 0.1 μm, and an accuracy of 0.15 μm such that primary particles may be distinguished from multiplets for all test aerosols. The measurement of number concentration shall be accomplished by integrating the primary particle peak.

(d) *Setup*—(1) *Remove the inlet and downtube from the candidate fractionator*. All tests procedures shall be conducted with the inlet and downtube removed from the candidate sampler.

(2) *Surface treatment of the fractionator*. Rinsing aluminum surfaces with alkaline solutions has been found to adversely affect subsequent fluorometric quantitation of aerosol mass deposits. If wash-off tests are to be used for quantifying aerosol penetration, internal surfaces of the fractionator must first be plated with electroless nickel. Specifications for this plating are specified in Society of Automotive Engineers Aerospace Material Specification (SAE AMS) 2404C, Electroless Nickel Plating (Reference 3 in appendix A of subpart F).

(e) *Test procedure: Wash-off method*—(1) *Clean the candidate sampler*. Note: The procedures in this step may be omitted if this test is being used to evaluate the fractionator after being loaded as specified in § 53.65.

(i) Clean and dry the internal surfaces of the candidate sampler.

(ii) Prepare the internal fractionator surfaces in strict accordance with the operating instructions specified in the sampler's operating manual referred to in section 7.4.18 of 40 CFR part 50, appendix L.

(2) *Generate aerosol*. Follow the procedures for aerosol generation prescribed in § 53.62(d)(2).

(3) *Verify the quality of the test aerosol*. Follow the procedures for verification of test aerosol size and quality prescribed in § 53.62(d)(4).

(4) *Determine effectiveness for the particle size being produced*. (i) Collect particles downstream of the fractionator

on an appropriate filter over a time period such that the relative error of the fluorometric measurement is less than 5.0 percent.

(ii) Determine the quantity of material collected on the after filter of the candidate method using a calibrated fluorometer. Calculate and record the aerosol mass concentration for the sampler filter as:

EQUATION 20

$$C_{cand(i)} = \frac{M_{cand(i)}}{Q_{(i)} \times t_{(i)}}$$

where:

i = replicate number;
$M_{cand}$ = mass of material collected with the candidate sampler;
Q = candidate sampler volumetric flowrate; and
t = sampling time.

(iii) Wash all interior surfaces upstream of the filter and determine the quantity of material collected using a calibrated fluorometer. Calculate and record the fluorometric mass concentration of the sampler wash as:

EQUATION 21

$$C_{wash(i)} = \frac{M_{wash(i)}}{Q_{(i)} \times t_{(i)}}$$

where:

i = replicate number;
$M_{wash}$ = mass of material washed from the interior surfaces of the fractionator;
Q = candidate sampler volumetric flowrate; and
t = sampling time.

(iv) Calculate and record the sampling effectiveness of the test sampler for this particle size as:

EQUATION 22

$$E_{(i)} = \frac{C_{wash(i)}}{C_{cand(i)} + C_{wash(i)}} \times 100\%$$

where:

i = replicate number.

(v) Repeat steps in paragraphs (e)(4) of this section, as appropriate, to obtain a minimum of three replicate measurements of sampling effectiveness. Note: The procedures for loading the candidate in § 53.65 must be repeated between repetitions if this test is being used to evaluate the fractionator after being loaded as specified in § 53.65.

(vi) Calculate and record the average sampling effectiveness of the test sampler as:

EQUATION 23

$$\overline{E} = \frac{\sum_{i=1}^{n} E_{(i)}}{n}$$

where:

i = replicate number; and
n = number of replicates.

(vii)(A) Calculate and record the coefficient of variation for the replicate sampling effectiveness measurements of the test sampler as:

EQUATION 24

$$CV_E = \frac{\sqrt{\sum_{i=1}^{n} E_{(i)}^2 - \frac{1}{n}\left(\sum_{i=1}^{n} E_{(i)}\right)^2}}{n-1} \times \frac{1}{\overline{E}} \times 100\%$$

where:

i = replicate number; and
n = total number of measurements.

(B) If the value of $CV_E$ exceeds 10 percent, then steps in paragraphs (e) (2) through (e)(4) of this section must be repeated.

(5) Repeat steps in paragraphs (e) (1) through (e)(4) of this section for each particle size specified in table F–2 of this subpart.

(f) *Test procedure: Static chamber method*—(1) *Generate aerosol*. Follow the procedures for aerosol generation prescribed in § 53.62(d)(2).

(2) *Verify the quality of the test aerosol*. Follow the procedures for verification of test aerosol size and quality prescribed in § 53.62(d)(4).

(3) *Introduce particles into chamber*. Introduce the particles into the static chamber and allow the particle concentration to stabilize.

(4) *Install and operate the candidate sampler's fractionator and its after-filter*

*and at least four total filters.* (i) Install the fractionator and an array of four or more equally spaced total filter samplers such that the total filters surround and are in the same plane as the inlet of the fractionator.

(ii) Simultaneously collect particles onto appropriate filters with the total filter samplers and the fractionator for a time period such that the relative error of the measured concentration is less than 5.0 percent.

(5) *Calculate the aerosol spatial uniformity in the chamber.* (i) Determine the quantity of material collected with each total filter sampler in the array using a calibrated fluorometer. Calculate and record the mass concentration for each total filter sampler as:

EQUATION 25

$$C_{total(ij)} = \frac{M_{total(ij)}}{Q_{(ij)} \times t_{(ij)}}$$

where:

i = replicate number;
j = total filter sampler number;
$M_{total}$ = mass of material collected with the total filter sampler;
Q = total filter sampler volumetric flowrate; and
t = sample time.

(ii) Calculate and record the mean mass concentration as:

EQUATION 26

$$\overline{C}_{total(i)} = \frac{\sum_{j=1}^{n} C_{total(ij)}}{n}$$

where:

n = total number of samplers;
i = replicate number; and
j = filter sampler number.

(iii) (A) Calculate and record the coefficient of variation of the total mass concentration as:

EQUATION 27

$$CV_{total} = \frac{\sqrt{\sum_{j=1}^{n} C_{total(ij)}^{2} - \frac{1}{n}\left(\sum_{j=1}^{n} C_{total(ij)}\right)^{2}}}{n-1} \times \frac{1}{\overline{C}_{total(i)}} \times 100\%$$

where:

i = replicate number;
j = total filter sampler number; and
n = number of total filter samplers.

(B) If the value of $CV_{total}$ exceeds 10 percent, then the particle concentration uniformity is unacceptable, alterations to the static chamber test apparatus must be made, and steps in paragraphs (f)(1) through (f)(5) of this section must be repeated.

(6) *Determine the effectiveness of the candidate sampler.* (i) Determine the quantity of material collected on the candidate sampler's after filter using a calibrated fluorometer. Calculate and record the mass concentration for the candidate sampler as:

EQUATION 28

$$C_{cand(i)} = \frac{M_{cand(i)}}{Q_{(i)} \times t_{(i)}}$$

where:

i = replicate number;
$M_{cand}$ = mass of material collected with the candidate sampler;
Q = candidate sampler volumetric flowrate; and
t = sample time.

(ii) Calculate and record the sampling effectiveness of the candidate sampler as:

EQUATION 29

$$E_{(i)} = \frac{C_{cand(i)}}{\overline{C}_{total(i)}} \times 100\%$$

where:

i = replicate number.

(iii) Repeat step in paragraph (f)(4) through (f)(6) of this section, as appropriate, to obtain a minimum of three replicate measurements of sampling effectiveness.

(iv) Calculate and record the average sampling effectiveness of the test sampler as:

EQUATION 30

$$\overline{E} = \frac{\sum_{i=1}^{n} E_{(i)}}{n}$$

where:

i= replicate number.

(v)(A) Calculate and record the coefficient of variation for the replicate sampling effectiveness measurements of the test sampler as:

EQUATION 31

$$CV_E = \frac{\sqrt{\sum_{i=1}^{n} E_{(i)}^2 - \frac{1}{n}\left(\sum_{i=1}^{n} E_{(i)}\right)^2}}{n-1} \times \frac{1}{\overline{E}} \times 100\%$$

where:

i = replicate number; and

n = number of measurements of effectiveness.

(B) If the value of $CV_E$ exceeds 10 percent, then the test run (steps in paragraphs (f)(2) through (f)(6) of this section) is unacceptable and must be repeated.

(7) Repeat steps in paragraphs (f)(1) through (f)(6) of this section for each particle size specified in table F–2 of this subpart.

(g) *Test procedure: Divided flow method*—(1) *Generate calibration aerosol.* Follow the procedures for aerosol generation prescribed in § 53.62(d)(2).

(2) *Verify the quality of the calibration aerosol.* Follow the procedures for verification of calibration aerosol size and quality prescribed in § 53.62(d)(4).

(3) *Introduce aerosol.* Introduce the calibration aerosol into the static chamber and allow the particle concentration to stabilize.

(4) *Validate that transport is equal for the divided flow option.* (i) With fluorometry as a detector:

(A) Install a total filter on each leg of the divided flow apparatus.

(B) Collect particles simultaneously through both legs at 16.7 L/min onto an appropriate filter for a time period such that the relative error of the measured concentration is less than 5.0 percent.

(C) Determine the quantity of material collected on each filter using a calibrated fluorometer. Calculate and record the mass concentration measured in each leg as:

EQUATION 32

$$C_{(i)} = \frac{M_{(i)}}{Q_{(i)} \times t_{(i)}}$$

where:

i = replicate number,

M = mass of material collected with the total filter; and

Q = candidate sampler volumetric flowrate.

(D) Repeat steps in paragraphs (g)(4)(i)(A) through (g)(4)(i)(C) of this section until a minimum of three replicate measurements are performed.

(ii) With an aerosol number counting device as a detector:

(A) Remove all flow obstructions from the flow paths of the two legs.

(B) Quantify the aerosol concentration of the primary particles in each leg of the apparatus.

(C) Repeat steps in paragraphs (g)(4)(ii)(A) through (g)(4)(ii)(B) of this section until a minimum of three replicate measurements are performed.

(iii) (A) Calculate the mean concentration and coefficient of variation as:

EQUATION 33

$$\overline{C} = \frac{\sum_{i=1}^{n} C_{(i)}}{n}$$

EQUATION 34

$$CV = \frac{\sqrt{\sum_{i=1}^{n} C_{(i)}^2 - \frac{1}{n}\left(\sum_{i=1}^{n} C_{(i)}\right)^2}}{n-1} \times \frac{1}{\overline{C}} \times 100\%$$

where:

i = replicate number; and
n = number of replicates.

(B) If the measured mean concentrations through the two legs do not agree within 5 percent, then adjustments may be made in the setup, and this step must be repeated.

(5) *Determine effectiveness.* Determine the sampling effectiveness of the test sampler with the inlet removed by one of the following procedures:

(i) With fluorometry as a detector:

(A) Prepare the divided flow apparatus for particle collection. Install a total filter into the bypass leg of the divided flow apparatus. Install the particle size fractionator with a total filter placed immediately downstream of it into the other leg.

(B) Collect particles simultaneously through both legs at 16.7 L/min onto appropriate filters for a time period such that the relative error of the measured concentration is less than 5.0 percent.

(C) Determine the quantity of material collected on each filter using a calibrated fluorometer. Calculate and record the mass concentration measured by the total filter and that measured after penetrating through the candidate fractionator as follows:

EQUATION 35

$$C_{total_{(i)}} = \frac{M_{total(i)}}{Q_{(i)} \times t_{(i)}}$$

EQUATION 36

$$C_{cand_{(i)}} = \frac{M_{cand(i)}}{Q_{(i)} \times t_{(i)}}$$

where:

i = replicate number.

(ii) With a number counting device as a detector:

(A) Install the particle size fractionator into one of the legs of the divided flow apparatus.

(B) Quantify and record the aerosol number concentration of the primary particles passing through the fractionator as $C_{cand(i)}$.

(C) Divert the flow from the leg containing the candidate fractionator to the bypass leg. Allow sufficient time for the aerosol concentration to stabilize.

(D) Quantify and record the aerosol number concentration of the primary particles passing through the bypass leg as $C_{total(i)}$.

(iii) Calculate and record sampling effectiveness of the candidate sampler as:

EQUATION 37

$$E_{(i)} = \frac{C_{cand(i)}}{\overline{C}_{total(i)}} \times 100\%$$

where:

i = replicate number.

(6) Repeat step in paragraph (g)(5) of this section, as appropriate, to obtain a minimum of three replicate measurements of sampling effectiveness.

(7) *Calculate the mean and coefficient of variation for replicate measurements of effectiveness.* (i) Calculate and record the mean sampling effectiveness of the candidate sampler as:

EQUATION 38

$$\overline{E} = \frac{\sum_{i=1}^{n} E_{(i)}}{n}$$

where:

i = replicate number.

(ii)(A) Calculate and record the coefficient of variation for the replicate sampling effectiveness measurements of the candidate sampler as:

EQUATION 39

$$CV_E = \frac{\sqrt{\sum_{i=1}^{n} E_{(i)}^2 - \frac{1}{n}\left(\sum_{i=1}^{n} E_{(i)}\right)^2}}{n-1} \times \frac{1}{\overline{E}} \times 100\%$$

where:

i = replicate number; and
n = number of replicates.

(B) If the coefficient of variation is not less than 10 percent, then the test run must be repeated (steps in paragraphs (g)(1) through (g)(7) of this section).

(8) Repeat steps in paragraphs (g)(1) through (g)(7) of this section for each particle size specified in table F–2 of this subpart.

(h) *Calculations*—(1) *Treatment of multiplets*. For all measurements made by fluorometric analysis, data shall be corrected for the presence of multiplets as described in § 53.62(f)(1). Data collected using a real-time device (as described in paragraph (c)(3)(ii)) of this section will not require multiplet correction.

(2) *Cutpoint determination*. For each wind speed determine the sampler $Dp_{50}$ cutpoint defined as the aerodynamic particle size corresponding to 50 percent effectiveness from the multiplet corrected smooth curve.

(3) *Graphical analysis and numerical integration with ambient distributions*. Follow the steps outlined in § 53.62 (e)(3) through (e)(4) to calculate the estimated concentration measurement ratio between the candidate sampler and a reference method sampler.

(i) *Test evaluation*. The candidate method passes the static fractionator test if the values of Rc and $Dp_{50}$ for each distribution meets the specifications in table F–1 of this subpart.

[62 FR 38814, July 18, 1997; 63 FR 7714, Feb. 17, 1998]

### § 53.65 Test procedure: Loading test.

(a) *Overview*. (1) The loading tests are designed to quantify any appreciable changes in a candidate method sampler's performance as a function of coarse aerosol collection. The candidate sampler is exposed to a mass of coarse aerosol equivalent to sampling a mass concentration of 150 μg/m³ over the time period that the manufacturer has specified between periodic cleaning. After loading, the candidate sampler is then evaluated by performing the test in § 53.62 (full wind tunnel test), § 53.63 (wind tunnel inlet aspiration test), or § 53.64 (static fractionator test). If the acceptance criteria are met for this evaluation test, then the candidate sampler is approved for multiday sampling with the periodic maintenance schedule as specified by the candidate method. For example, if the candidate sampler passes the reevaluation tests following loading with an aerosol mass equivalent to sampling a 150 μg/m³ aerosol continuously for 7 days, then the sampler is approved for 7 day field operation before cleaning is required.

(2) [Reserved]

(b) *Technical definition*. Effectiveness after loading is the ratio (expressed as a percentage) of the mass concentration of particles of a given size reaching the sampler filter to the mass concentration of particles of the same size approaching the sampler.

(c) *Facilities and equipment required*—(1) *Particle delivery system*. The particle delivery system shall consist of a static chamber or a low velocity wind tunnel having a sufficiently large cross-sectional area such that the test sampler, or portion thereof, may be installed in the test section. At a minimum, the system must have a sufficiently large cross section to house the candidate sampler inlet as well as a collocated isokinetic nozzle for measuring total aerosol concentration. The mean velocity in the test section of the static chamber or wind tunnel shall not exceed 2 km/hr.

(2) *Aerosol generation equipment*. For purposes of these tests, the test aerosol shall be produced from commercially available, bulk Arizona road dust. To provide direct interlaboratory comparability of sampler loading characteristics, the bulk dust is specified as 0-10 μm ATD available from Powder Technology Incorporated (Burnsville, MN). A fluidized bed aerosol generator,

Wright dust feeder, or sonic nozzle shall be used to efficiently deagglomerate the bulk test dust and transform it into an aerosol cloud. Other dust generators may be used contingent upon prior approval by the Agency.

(3) *Isokinetic sampler*. Mean aerosol concentration within the static chamber or wind tunnel shall be established using a single isokinetic sampler containing a preweighed high-efficiency total filter.

(4) *Analytic balance*. An analytical balance shall be used to determine the weight of the total filter in the isokinetic sampler. The precision and accuracy of this device shall be such that the relative measurement error is less than 5.0 percent for the difference between the initial and final weight of the total filter. The identical analytic balance shall be used to perform both initial and final weighing of the total filter.

(d) *Test procedure*. (1) Calculate and record the target time weighted concentration of Arizona road dust which is equivalent to exposing the sampler to an environment of 150 μg/m³ over the time between cleaning specified by the candidate sampler's operations manual as:

EQUATION 40

$$\text{Target TWC} = 150\,\mu g/m^3 \times t$$

where:

t = the number of hours specified by the candidate method prior to periodic cleaning.

(2) Clean the candidate sampler. (i) Clean and dry the internal surfaces of the candidate sampler.

(ii) Prepare the internal surfaces in strict accordance with the operating manual referred to in section 7.4.18 of 40 CFR part 50, appendix L.

(3) Determine the preweight of the filter that shall be used in the isokinetic sampler. Record this value as InitWt.

(4) Install the candidate sampler's inlet and the isokinetic sampler within the test chamber or wind tunnel.

(5) Generate a dust cloud. (i) Generate a dust cloud composed of Arizona test dust.

(ii) Introduce the dust cloud into the chamber.

(iii) Allow sufficient time for the particle concentration to become steady within the chamber.

(6) Sample aerosol with a total filter and the candidate sampler. (i) Sample the aerosol for a time sufficient to produce an equivalent TWC equal to that of the target TWC ±15 percent.

(ii) Record the sampling time as t.

(7) Determine the time weighted concentration. (i) Determine the postweight of the isokinetic sampler's total filter.

(ii) Record this value as FinalWt.

(iii) Calculate and record the TWC as:

EQUATION 41

$$TWC = \frac{(FinalWt - InitWt) \times t}{Q}$$

where:

Q = the flow rate of the candidate method.

(iv) If the value of TWC deviates from the target TWC ±15 percent, then the loaded mass is unacceptable and the entire test procedure must be repeated.

(8) Determine the candidate sampler's effectiveness after loading. The candidate sampler's effectiveness as a function of particle aerodynamic diameter must then be evaluated by performing the test in §53.62 (full wind tunnel test). A sampler which fits the category of inlet deviation in §53.60(e)(1) may opt to perform the test in §53.63 (inlet aspiration test) in lieu of the full wind tunnel test. A sampler which fits the category of fractionator deviation in §53.60(e)(2) may opt to perform the test in §53.64 (static fractionator test) in lieu of the full wind tunnel test.

(e) *Test results*. If the candidate sampler meets the acceptance criteria for the evaluation test performed in paragraph (d)(8) of this section, then the candidate sampler passes this test with the stipulation that the sampling train be cleaned as directed by and as frequently as that specified by the candidate sampler's operations manual.

**§53.66 Test procedure: Volatility test.**

(a) *Overview*. This test is designed to ensure that the candidate method's losses due to volatility when sampling

semi-volatile ambient aerosol will be comparable to that of a federal reference method sampler. This is accomplished by challenging the candidate sampler with a polydisperse, semi-volatile liquid aerosol in three distinct phases. During phase A of this test, the aerosol is elevated to a steady-state, test-specified mass concentration and the sample filters are conditioned and preweighed. In phase B, the challenge aerosol is simultaneously sampled by the candidate method sampler and a reference method sampler onto the preweighed filters for a specified time period. In phase C (the blow-off phase), aerosol and aerosol-vapor free air is sampled by the samplers for an additional time period to partially volatilize the aerosol on the filters. The candidate sampler passes the volatility test if the acceptance criteria presented in table F–1 of this subpart are met or exceeded.

(b) *Technical definitions.* (1) Residual mass (RM) is defined as the weight of the filter after the blow-off phase subtracted from the initial weight of the filter.

(2) Corrected residual mass (CRM) is defined as the residual mass of the filter from the candidate sampler multiplied by the ratio of the reference method flow rate to the candidate method flow rate.

(c) *Facilities and equipment required*—(1) *Environmental chamber.* Because the nature of a volatile aerosol is greatly dependent upon environmental conditions, all phases of this test shall be conducted at a temperature of 22.0 ±0.5 °C and a relative humidity of 40 ±3 percent. For this reason, it is strongly advised that all weighing and experimental apparatus be housed in an environmental chamber capable of this level of control.

(2) *Aerosol generator.* The aerosol generator shall be a pressure nebulizer operated at 20 to 30 psig (140 to 207 kPa) to produce a polydisperse, semi-voltile aerosol with a mass median diameter larger than 1 μm and smaller than 2.5 μm. The nebulized liquid shall be A.C.S. reagent grade glycerol ($C_3H_8O$, FW = 92.09, CAS 56–81–5) of 99.5 percent minimum purity. For the purpose of this test the accepted mass median diameter is predicated on the stable aerosol inside the internal chamber and not on the aerosol emerging from the nebulizer nozzle. Aerosol monitoring and its stability are described in (c)(3) and (c)(4) of this section.

(3) *Aerosol monitoring equipment.* The evaporation and condensation dynamics of a volatile aerosol is greatly dependent upon the vapor pressure of the volatile component in the carrier gas. The size of an aerosol becomes fixed only when an equilibrium is established between the aerosol and the surrounding vapor; therefore, aerosol size measurement shall be used as a surrogate measure of this equilibrium. A suitable instrument with a range of 0.3 to 10 μm, an accuracy of 0.5 μm, and a resolution of 0.2 μm (e.g., an optical particle sizer, or a time-of-flight instrument) shall be used for this purpose. The parameter monitored for stability shall be the mass median instrument measured diameter (i.e. optical diameter if an optical particle counter is used). A stable aerosol shall be defined as an aerosol with a mass median diameter that has changed less than 0.25 μm over a 4 hour time period.

(4) *Internal chamber.* The time required to achieve a stable aerosol depends upon the time during which the aerosol is resident with the surrounding air. This is a function of the internal volume of the aerosol transport system and may be facilitated by recirculating the challenge aerosol. A chamber with a volume of 0.5 $m^3$ and a recirculating loop (airflow of approximately 500 cfm) is recommended for this purpose. In addition, a baffle is recommended to dissipate the jet of air that the recirculating loop can create. Furthermore, a HEPA filtered hole in the wall of the chamber is suggested to allow makeup air to enter the chamber or excess air to exit the chamber to maintain a system flow balance. The concentration inside the chamber shall be maintained at 1 $mg/m^3$ ±20 percent to obtain consistent and significant filter loading.

(5) *Aerosol sampling manifold.* A manifold shall be used to extract the aerosol from the area in which it is equilibrated and transport it to the candidate method sampler, the reference

method sampler, and the aerosol monitor. The losses in each leg of the manifold shall be equivalent such that the three devices will be exposed to an identical aerosol.

(6) *Chamber air temperature recorders.* Minimum range 15-25 °C, certified accuracy to within 0.2 °C, resolution of 0.1 °C. Measurement shall be made at the intake to the sampling manifold and adjacent to the weighing location.

(7) *Chamber air relative humidity recorders.* Minimum range 30 - 50 percent, certified accuracy to within 1 percent, resolution of 0.5 percent. Measurement shall be made at the intake to the sampling manifold and adjacent to the weighing location.

(8) *Clean air generation system.* A source of aerosol and aerosol-vapor free air is required for phase C of this test. This clean air shall be produced by filtering air through an absolute (HEPA) filter.

(9) *Balance.* Minimum range 0 - 200 mg, certified accuracy to within 10 μg, resolution of 1 μg.

(d) *Additional filter handling conditions*—(1) *Filter handling.* Careful handling of the filter during sampling, conditioning, and weighing is necessary to avoid errors due to damaged filters or loss of collected particles from the filters. All filters must be weighed immediately after phase A dynamic conditioning and phase C.

(2) *Dynamic conditioning of filters.* Total dynamic conditioning is required prior to the initial weight determined in phase A. Dynamic conditioning refers to pulling clean air from the clean air generation system through the filters. Total dynamic conditioning can be established by sequential filter weighing every 30 minutes following repetitive dynamic conditioning. The filters are considered sufficiently conditioned if the sequential weights are repeatable to ±3 μg.

(3) *Static charge.* The following procedure is suggested for minimizing charge effects. Place six or more Polonium static control devices (PSCD) inside the microbalance weighing chamber, (MWC). Two of them must be placed horizontally on the floor of the MWC and the remainder placed vertically on the back wall of the MWC. Taping two PSCD's together or using double-sided tape will help to keep them from falling. Place the filter that is to be weighed on the horizontal PSCDs facing aerosol coated surface up. Close the MWC and wait 1 minute. Open the MWC and place the filter on the balance dish. Wait 1 minute. If the charges have been neutralized the weight will stabilize within 30-60 seconds. Repeat the procedure of neutralizing charges and weighing as prescribed above several times (typically 2-4 times) until consecutive weights will differ by no more than 3 micrograms. Record the last measured weight and use this value for all subsequent calculations.

(e) *Test procedure*—(1) *Phase A - Preliminary steps.* (i) Generate a polydisperse glycerol test aerosol.

(ii) Introduce the aerosol into the transport system.

(iii) Monitor the aerosol size and concentration until stability and level have been achieved.

(iv) Condition the candidate method sampler and reference method sampler filters until total dynamic conditioning is achieved as specified in paragraph (d)(2) of this section.

(v) Record the dynamically conditioned weight as $InitWt_c$ and $InitWt_r$ where c is the candidate method sampler and r is the reference method sampler.

(2) *Phase B - Aerosol loading.* (i) Install the dynamically conditioned filters into the appropriate samplers.

(ii) Attach the samplers to the manifold.

(iii) Operate the candidate and the reference samplers such that they simultaneously sample the test aerosol for 2 hours for a candidate sampler operating at 16.7 L/min or higher, or proportionately longer for a candidate sampler operating at a lower flow rate.

(3) *Phase C - Blow-off.* (i) Alter the intake of the samplers to sample air from the clean air generation system.

(ii) Sample clean air for one of the required blow-off time durations (1, 2, 3, and 4 hours).

(iii) Remove the filters from the samplers.

(iv) Weigh the filters immediately and record this weight, $FinalWt_c$ and $FinalWt_r$, where c is the candidate

method sampler and r is the reference method sampler.

(v) Calculate the residual mass for the reference method sampler:

EQUATION 41A

$$RM_{(ij)} = \left(FinalWt_r - InitWt_r\right)$$

where:

i = repetition number; and
j = blow-off time period.

(vi) Calculate the corrected residual mass for the candidate method sampler as:

EQUATION 41B

$$CRM_{(ij)} = \left(FinalWt_r - InitWt_r\right) \times \frac{Q_r}{Q_c}$$

where:

i = repetition number;
j = blow-off time period;
$Q_c$ = candidate method sampler flow rate, and
$Q_r$ = reference method sampler flow rate.

(4) Repeat steps in paragraph (e)(1) through (e)(3) of this section until three repetitions have been completed for each of the required blow-off time durations (1, 2, 3, and 4 hours).

(f) *Calculations and analysis.* (1) Perform a linear regression with the candidate method CRM as the dependent variable and the reference method RM as the independent variable.

(2) Determine the following regression parameters: slope, intercept, and correlation coefficient (r).

(g) *Test results.* The candidate method passes the volatility test if the regression parameters meet the acceptance criteria specified in table F–1 of this subpart.

[62 FR 38814, July 18, 1997, as amended at 71 FR 61295, Oct. 17, 2006]

TABLE F–1 TO SUBPART F OF PART 53—PERFORMANCE SPECIFICATIONS FOR $PM_{2.5}$ CLASS II EQUIVALENT SAMPLERS

| Performance test | Specifications | Acceptance criteria |
|---|---|---|
| § 53.62 Full Wind Tunnel Evaluation | Solid VOAG produced aerosol at 2 km/hr and 24 km/hr. | $Dp_{50}$ = 2.5 μm ±0.2 μm Numerical Analysis Results: 95% ≤$R_c$ ≤105%. |
| § 53.63 Wind Tunnel Inlet Aspiration Test | Liquid VOAG produced aerosol at 2 km/hr and 24 km/hr. | Relative Aspiration: 95% ≤A ≤105%. |
| § 53.64 Static Fractionator Test | Evaluation of the fractionator under static conditions. | $Dp_{50}$ = 2.5 μm ±0.2 μm Numerical Analysis Results: 95% ≤$R_c$ ≤105%. |
| § 53.65 Loading Test | Loading of the clean candidate under laboratory conditions. | Acceptance criteria as specified in the post-loading evaluation test (§ 53.62, § 53.63, or § 53.64). |
| § 53.66 Volatility Test | Polydisperse liquid aerosol produced by air nebulization of A.C.S. reagent grade glycerol, 99.5% minimum purity. | Regression Parameters Slope = 1 ±0.1, Intercept = 0 ±0.15 mg, r ≥0.97. |

[72 FR 32209, June 12, 2007]

TABLE F–2 TO SUBPART F OF PART 53—PARTICLE SIZES AND WIND SPEEDS FOR FULL WIND TUNNEL TEST, WIND TUNNEL INLET ASPIRATION TEST, AND STATIC CHAMBER TEST

| Primary Partical Mean Size[a] (μm) | Full Wind Tunnel Test | | Inlet Aspiration Test | | Static Fractionator Test | Volatility Test |
|---|---|---|---|---|---|---|
| | 2 km/hr | 24 km/hr | 2 km/hr | 24 km/hr | | |
| 1.5±0.25 | S | S | | | S | |
| 2.0±0.25 | S | S | | | S | |
| 2.2±0.25 | S | S | | | S | |
| 2.5±0.25 | S | S | | | S | |
| 2.8±0.25 | S | S | | | S | |
| 3.0±0.25 | | | L | L | | |
| 3.5±0.25 | S | S | | | S | |
| 4.0±0.5 | S | S | | | S | |

| Primary Partical Mean Size[a] (μm) | Full Wind Tunnel Test | | Inlet Aspiration Test | | Static Fractionator Test | Volatility Test |
|---|---|---|---|---|---|---|
| | 2 km/hr | 24 km/hr | 2 km/hr | 24 km/hr | | |
| Polydisperse Glycerol Aerosol | | | | L | | |

[a] Aerodynamic diameter.
S = Solid particles.
L = Liquid particles.

TABLE F–3 TO SUBPART F OF PART 53—CRITICAL PARAMETERS OF IDEALIZED AMBIENT PARTICLE SIZE DISTRIBUTIONS

| Idealized Distribution | Fine Particle Mode | | | Coarse Particle Mode | | | $PM_{2.5}/PM_{10}$ Ratio | FRM Sampler Expected Mass Conc. (μg/m³) |
|---|---|---|---|---|---|---|---|---|
| | MMD (μm) | Geo. Std. Dev. | Conc. (μg/m³) | MMD (μm) | Geo. Std. Dev. | Conc. (μg/m³) | | |
| Coarse | 0.50 | 2 | 12.0 | 10 | 2 | 88.0 | 0.27 | 13.814 |
| "Typical" | 0.50 | 2 | 33.3 | 10 | 2 | 66.7 | 0.55 | 34.284 |
| Fine | 0.85 | 2 | 85.0 | 15 | 2 | 15.0 | 0.94 | 78.539 |

TABLE F–4 TO SUBPART F OF PART 53—ESTIMATED MASS CONCENTRATION MEASUREMENT OF $PM_{2.5}$ FOR IDEALIZED COARSE AEROSOL SIZE DISTRIBUTION

| Particle Aerodynamic Diameter (μm) | Test Sampler | | | Ideal Sampler | | |
|---|---|---|---|---|---|---|
| | Fractional Sampling Effectiveness | Interval Mass Concentration (μg/m³) | Estimated Mass Concentration Measurement (μg/m³) | Fractional Sampling Effectiveness | Interval Mass Concentration (μg/m³) | Estimated Mass Concentration Measurement (μg/m³) |
| (1) | (2) | (3) | (4) | (5) | (6) | (7) |
| <0.500 | 1.000 | 6.001 | | 1.000 | 6.001 | 6.001 |
| 0.625 | | 2.129 | | 0.999 | 2.129 | 2.127 |
| 0.750 | | 0.982 | | 0.998 | 0.982 | 0.980 |
| 0.875 | | 0.730 | | 0.997 | 0.730 | 0.728 |
| 1.000 | | 0.551 | | 0.995 | 0.551 | 0.548 |
| 1.125 | | 0.428 | | 0.991 | 0.428 | 0.424 |
| 1.250 | | 0.346 | | 0.987 | 0.346 | 0.342 |
| 1.375 | | 0.294 | | 0.980 | 0.294 | 0.288 |
| 1.500 | | 0.264 | | 0.969 | 0.264 | 0.256 |
| 1.675 | | 0.251 | | 0.954 | 0.251 | 0.239 |
| 1.750 | | 0.250 | | 0.932 | 0.250 | 0.233 |
| 1.875 | | 0.258 | | 0.899 | 0.258 | 0.232 |
| 2.000 | | 0.272 | | 0.854 | 0.272 | 0.232 |
| 2.125 | | 0.292 | | 0.791 | 0.292 | 0.231 |
| 2.250 | | 0.314 | | 0.707 | 0.314 | 0.222 |
| 2.375 | | 0.339 | | 0.602 | 0.339 | 0.204 |
| 2.500 | | 0.366 | | 0.480 | 0.366 | 0.176 |
| 2.625 | | 0.394 | | 0.351 | 0.394 | 0.138 |
| 2.750 | | 0.422 | | 0.230 | 0.422 | 0.097 |
| 2.875 | | 0.449 | | 0.133 | 0.449 | 0.060 |
| 3.000 | | 0.477 | | 0.067 | 0.477 | 0.032 |
| 3.125 | | 0.504 | | 0.030 | 0.504 | 0.015 |
| 3.250 | | 0.530 | | 0.012 | 0.530 | 0.006 |
| 3.375 | | 0.555 | | 0.004 | 0.555 | 0.002 |
| 3.500 | | 0.579 | | 0.001 | 0.579 | 0.001 |
| 3.625 | | 0.602 | | 0.000000 | 0.602 | 0.000000 |
| 3.750 | | 0.624 | | 0.000000 | 0.624 | 0.000000 |
| 3.875 | | 0.644 | | 0.000000 | 0.644 | 0.000000 |
| 4.000 | | 0.663 | | 0.000000 | 0.663 | 0.000000 |
| 4.125 | | 0.681 | | 0.000000 | 0.681 | 0.000000 |
| 4.250 | | 0.697 | | 0.000000 | 0.697 | 0.000000 |
| 4.375 | | 0.712 | | 0.000000 | 0.712 | 0.000000 |
| 4.500 | | 0.726 | | 0.000000 | 0.726 | 0.000000 |
| 4.625 | | 0.738 | | 0.000000 | 0.738 | 0.000000 |
| 4.750 | | 0.750 | | 0.000000 | 0.750 | 0.000000 |
| 4.875 | | 0.760 | | 0.000000 | 0.760 | 0.000000 |
| 5.000 | | 0.769 | | 0.000000 | 0.769 | 0.000000 |
| 5.125 | | 0.777 | | 0.000000 | 0.777 | 0.000000 |
| 5.250 | | 0.783 | | 0.000000 | 0.783 | 0.000000 |
| 5.375 | | 0.789 | | 0.000000 | 0.789 | 0.000000 |

| Particle Aerodynamic Diameter (μm) | Test Sampler | | | Ideal Sampler | | |
|---|---|---|---|---|---|---|
| | Fractional Sampling Effectiveness | Interval Mass Concentration (μg/m³) | Estimated Mass Concentration Measurement (μg/m³) | Fractional Sampling Effectiveness | Interval Mass Concentration (μg/m³) | Estimated Mass Concentration Measurement (μg/m³) |
| 5.500 | | 0.794 | | 0.000000 | 0.794 | 0.000000 |
| 5.625 | | 0.798 | | 0.000000 | 0.798 | 0.000000 |
| 5.75 | | 0.801 | | 0.000000 | 0.801 | 0.000000 |
| | | $C_{sam(exp)}$= | | | $C_{ideal(exp)}$= | 13.814 |

TABLE F–5 TO SUBPART F OF PART 53—ESTIMATED MASS CONCENTRATION MEASUREMENT OF $PM_{2.5}$ FOR IDEALIZED ''TYPICAL'' COARSE AEROSOL SIZE DISTRIBUTION

| Particle Aerodynamic Diameter (μm) | Test Sampler | | | Ideal Sampler | | |
|---|---|---|---|---|---|---|
| | Fractional Sampling Effectiveness | Interval Mass Concentration (μg/m³) | Estimated Mass Concentration Measurement (μg/m³) | Fractional Sampling Effectiveness | Interval Mass Concentration (μg/m³) | Estimated Mass Concentration Measurement (μg/m³) |
| (1) | (2) | (3) | (4) | (5) | (6) | (7) |
| <0.500 | 1.000 | 16.651 | | 1.000 | 16.651 | 16.651 |
| 0.625 | | 5.899 | | 0.999 | 5.899 | 5.893 |
| 0.750 | | 2.708 | | 0.998 | 2.708 | 2.703 |
| 0.875 | | 1.996 | | 0.997 | 1.996 | 1.990 |
| 1.000 | | 1.478 | | 0.995 | 1.478 | 1.471 |
| 1.125 | | 1.108 | | 0.991 | 1.108 | 1.098 |
| 1.250 | | 0.846 | | 0.987 | 0.846 | 0.835 |
| 1.375 | | 0.661 | | 0.980 | 0.661 | 0.648 |
| 1.500 | | 0.532 | | 0.969 | 0.532 | 0.516 |
| 1.675 | | 0.444 | | 0.954 | 0.444 | 0.424 |
| 1.750 | | 0.384 | | 0.932 | 0.384 | 0.358 |
| 1.875 | | 0.347 | | 0.899 | 0.347 | 0.312 |
| 2.000 | | 0.325 | | 0.854 | 0.325 | 0.277 |
| 2.125 | | 0.314 | | 0.791 | 0.314 | 0.248 |
| 2.250 | | 0.312 | | 0.707 | 0.312 | 0.221 |
| 2.375 | | 0.316 | | 0.602 | 0.316 | 0.190 |
| 2.500 | | 0.325 | | 0.480 | 0.325 | 0.156 |
| 2.625 | | 0.336 | | 0.351 | 0.336 | 0.118 |
| 2.750 | | 0.350 | | 0.230 | 0.350 | 0.081 |
| 2.875 | | 0.366 | | 0.133 | 0.366 | 0.049 |
| 3.000 | | 0.382 | | 0.067 | 0.382 | 0.026 |
| 3.125 | | 0.399 | | 0.030 | 0.399 | 0.012 |
| 3.250 | | 0.416 | | 0.012 | 0.416 | 0.005 |
| 3.375 | | 0.432 | | 0.004 | 0.432 | 0.002 |
| 3.500 | | 0.449 | | 0.001 | 0.449 | 0.000000 |
| 3.625 | | 0.464 | | 0.000000 | 0.464 | 0.000000 |
| 3.750 | | 0.480 | | 0.000000 | 0.480 | 0.000000 |
| 3.875 | | 0.494 | | 0.000000 | 0.494 | 0.000000 |
| 4.000 | | 0.507 | | 0.000000 | 0.507 | 0.000000 |
| 4.125 | | 0.520 | | 0.000000 | 0.520 | 0.000000 |
| 4.250 | | | | 0.000000 | 0.532 | 0.000000 |
| 4.375 | | | | 0.000000 | 0.543 | 0.000000 |
| 4.500 | | | | 0.000000 | 0.553 | 0.000000 |
| 4.625 | | | | 0.000000 | 0.562 | 0.000000 |
| 4.750 | | | | 0.000000 | 0.570 | 0.000000 |
| 4.875 | | | | 0.000000 | 0.577 | 0.000000 |
| 5.000 | | | | 0.000000 | 0.584 | 0.000000 |
| 5.125 | | | | 0.000000 | 0.590 | 0.000000 |
| 5.250 | | | | 0.000000 | 0.595 | 0.000000 |
| 5.375 | | | | 0.000000 | 0.599 | 0.000000 |
| 5.500 | | | | 0.000000 | 0.603 | 0.000000 |
| 5.625 | | | | 0.000000 | 0.605 | 0.000000 |
| 5.75 | | | | 0.000000 | 0.608 | 0.000000 |
| | | $C_{sam(exp)}$= | | | $C_{ideal(exp)}$= | 34.284 |

TABLE F–6 TO SUBPART F OF PART 53—ESTIMATED MASS CONCENTRATION MEASUREMENT OF $PM_{2.5}$ FOR IDEALIZED FINE AEROSOL SIZE DISTRIBUTION

| Particle Aerodynamic Diameter (μm) | Test Sampler | | | Ideal Sampler | | |
|---|---|---|---|---|---|---|
| | Fractional Sampling Effectiveness | Interval Mass Concentration (μg/m³) | Estimated Mass Concentration Measurement (μg/m³) | Fractional Sampling Effectiveness | Interval Mass Concentration (μg/m³) | Estimated Mass Concentration Measurement (μg/m³) |
| (1) | (2) | (3) | (4) | (5) | (6) | (7) |
| <0.500 | 1.000 | 18.868 | | 1.000 | 18.868 | 18.868 |
| 0.625 | | 13.412 | | 0.999 | 13.412 | 13.399 |
| 0.750 | | 8.014 | | 0.998 | 8.014 | 7.998 |
| 0.875 | | 6.984 | | 0.997 | 6.984 | 6.963 |
| 1.000 | | 5.954 | | 0.995 | 5.954 | 5.924 |
| 1.125 | | 5.015 | | 0.991 | 5.015 | 4.970 |
| 1.250 | | 4.197 | | 0.987 | 4.197 | 4.142 |
| 1.375 | | 3.503 | | 0.980 | 3.503 | 3.433 |
| 1.500 | | 2.921 | | 0.969 | 2.921 | 2.830 |
| 1.675 | | 2.438 | | 0.954 | 2.438 | 2.326 |
| 1.750 | | 2.039 | | 0.932 | 2.039 | 1.900 |
| 1.875 | | 1.709 | | 0.899 | 1.709 | 1.536 |
| 2.000 | | 1.437 | | 0.854 | 1.437 | 1.227 |
| 2.125 | | 1.212 | | 0.791 | 1.212 | 0.959 |
| 2.250 | | 1.026 | | 0.707 | 1.026 | 0.725 |
| 2.375 | | 0.873 | | 0.602 | 0.873 | 0.526 |
| 2.500 | | 0.745 | | 0.480 | 0.745 | 0.358 |
| 2.625 | | 0.638 | | 0.351 | 0.638 | 0.224 |
| 2.750 | | 0.550 | | 0.230 | 0.550 | 0.127 |
| 2.875 | | 0.476 | | 0.133 | 0.476 | 0.063 |
| 3.000 | | 0.414 | | 0.067 | 0.414 | 0.028 |
| 3.125 | | 0.362 | | 0.030 | 0.362 | 0.011 |
| 3.250 | | 0.319 | | 0.012 | 0.319 | 0.004 |
| 3.375 | | 0.282 | | 0.004 | 0.282 | 0.001 |
| 3.500 | | 0.252 | | 0.001 | 0.252 | 0.000000 |
| 3.625 | | 0.226 | | 0.000000 | 0.226 | 0.000000 |
| 3.750 | | 0.204 | | 0.000000 | 0.204 | 0.000000 |
| 3.875 | | 0.185 | | 0.000000 | 0.185 | 0.000000 |
| 4.000 | | 0.170 | | 0.000000 | 0.170 | 0.000000 |
| 4.125 | | 0.157 | | 0.000000 | 0.157 | 0.000000 |
| 4.250 | | 0.146 | | 0.000000 | 0.146 | 0.000000 |
| 4.375 | | 0.136 | | 0.000000 | 0.136 | 0.000000 |
| 4.500 | | 0.129 | | 0.000000 | 0.129 | 0.000000 |
| 4.625 | | 0.122 | | 0.000000 | 0.122 | 0.000000 |
| 4.750 | | 0.117 | | 0.000000 | 0.117 | 0.000000 |
| 4.875 | | 0.112 | | 0.000000 | 0.112 | 0.000000 |
| 5.000 | | 0.108 | | 0.000000 | 0.108 | 0.000000 |
| 5.125 | | 0.105 | | 0.000000 | 0.105 | 0.000000 |
| 5.250 | | 0.102 | | 0.000000 | 0.102 | 0.000000 |
| 5.375 | | 0.100 | | 0.000000 | 0.100 | 0.000000 |
| 5.500 | | 0.098 | | 0.000000 | 0.098 | 0.000000 |
| 5.625 | | 0.097 | | 0.000000 | 0.097 | 0.000000 |
| 5.75 | | 0.096 | | 0.000000 | 0.096 | 0.000000 |
| | | $C_{sam(exp)}$= | | | $C_{ideal(exp)}$= | 78.539 |

FIGURE F–1 TO SUBPART F OF PART 53—DESIGNATION TESTING CHECKLIST

**DESIGNATION TESTING CHECKLIST FOR CLASS II**

Auditee _____ Auditor signature _____ Date _____

| Compliance Status: Y = Yes N = No NA = Not applicable/Not available | | | | |
|---|---|---|---|---|
| Verification | | | Verified by Direct Observation of Process or of Documented Evidence: Performance, Design or Application Spec. Corresponding to Sections of 40 CFR Part 53, Subparts E and F | Verification Comments (Includes documentation of who, what, where, when, why) (Doc. #, Rev. #, Rev. Date) |
| Y | N | NA | | |
| | | | **Subpart E: Performance Specification Tests** | |

| Compliance Status: Y = Yes N = No NA = Not applicable/Not available | | | | |
|---|---|---|---|---|
| Verification | | | Verified by Direct Observation of Process or of Documented Evidence: Performance, Design or Application Spec. Corresponding to Sections of 40 CFR Part 53, Subparts E and F | Verification Comments (Includes documentation of who, what, where, when, why) (Doc. #, Rev. #, Rev. Date) |
| Y | N | NA | | |
| | | | Evaluation completed according to Subpart E § 53.50 to § 53.56 | |
| | | | **Subpart E: Class I Sequential Tests** | |
| | | | Class II samplers that are also Class I (sequentialized) have passed the tests in § 53.57 | |
| | | | **Subpart F: Performance Spec/Test** | |
| | | | Evaluation of Physical Characteristics of Clean Sampler - One of these tests must be performed:<br>§ 53.62 - Full Wind Tunnel<br>§ 53.63 - Inlet Aspiration<br>§ 53.64 - Static Fractionator | |
| | | | Evaluation of Physical Characteristics of Loaded Sampler<br>§ 53.65 Loading Test<br>One of the following tests must be performed for evaluation after loading: § 53.62, § 53.63, § 53.64 | |
| | | | Evaluation of the Volatile Characteristics of the Class II Sampler § 53.66 | |

APPENDIX A TO SUBPART F OF PART 53—REFERENCES

(1) Marple, V.A., K.L. Rubow, W. Turner, and J.D. Spangler, Low Flow Rate Sharp Cut Impactors for Indoor Air Sampling: Design and Calibration., JAPCA, 37: 1303-1307 (1987).

(2) Vanderpool, R.W. and K.L. Rubow, Generation of Large, Solid Calibration Aerosols, J. of Aer. Sci. and Tech., 9:65-69 (1988).

(3) Society of Automotive Engineers Aerospace Material Specification (SAE AMS) 2404C, Electroless Nickel Planting, SAE, 400 Commonwealth Drive, Warrendale PA-15096, Revised 7-1-84, pp. 1-6.

# PART 54—PRIOR NOTICE OF CITIZEN SUITS

Sec.
54.1 Purpose.
54.2 Service of notice.
54.3 Contents of notice.

AUTHORITY: Sec. 304 of the Clean Air Act, as amended (sec. 12, Pub. L. 91–604, 84 Stat. 1706).

SOURCE: 36 FR 23386, Dec. 9, 1971, unless otherwise noted.

## § 54.1 Purpose.

Section 304 of the Clean Air Act, as amended, authorizes the commencement of civil actions to enforce the Act or to enforce certain requirements promulgated pursuant to the Act. The purpose of this part is to prescribe procedures governing the giving of notices required by subsection 304(b) of the Act (sec. 12, Pub. L. 91–604; 84 Stat. 1706) as a prerequisite to the commencement of such actions.

## § 54.2 Service of notice.

(a) Notice to Administrator: Service of notice given to the Administrator under this part shall be accomplished by certified mail addressed to the Administrator, Environmental Protection Agency, Washington, DC 20460. Where notice relates to violation of an emission standard or limitation or to violation of an order issued with respect to an emission standard or limitation, a copy of such notice shall be mailed to the Regional Administrator of the Environmental Protection Agency for the Region in which such violation is alleged to have occurred.