

SUBCHAPTER C—AIR PROGRAMS—(Continued)

PART 53—AMBIENT AIR MONITORING REFERENCE AND EQUIVALENT METHODS

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SOURCE: 40 FR 7049, Feb. 18, 1975, unless otherwise noted.

Subpart A—General Provisions

§ 53.1 Definitions.

(a) Terms used but not defined in this part shall have the meaning given them by the Act.

(b) *Act* means the Clean Air Act (42 U.S.C. 1857-1857J), as amended.

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

(d) *Administrator* means the Administrator of the Environmental Protection Agency or his authorized representative.

(e) *Reference method* means a method of sampling and analyzing the ambient air for an air pollutant that is specified as a reference method in an appendix to 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 cancelled in accordance with § 53.11 or § 53.16.

(f) *Equivalent method* means a method of sampling and analyzing the ambient air for an air pollutant that has been designated as an equivalent method in accordance with this part; it does not include a method for which an equivalent method designation has been cancelled in accordance with § 53.11 or § 53.16.

(g) *Candidate method* means a method of sampling and analyzing the ambient air for an air pollutant for which an application for a reference method determination or an 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.

(h) *Manual method* means a method for measuring concentrations of an ambient air pollutant in which sample

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collection, analysis, or measurement, or some combination thereof, is performed manually.

(i) *Automated method* or *analyzer* means a method for measuring concentrations of an ambient air pollutant in which sample collection, analysis, and measurement are performed automatically.

(j) *Test analyzer* means an analyzer subjected to testing as a candidate method in accordance with subparts B, C, and/or D of this part, as applicable.

(k) *Applicant* means a person who submits an application for a reference or equivalent method determination under § 53.4, or a person who assumes the rights and obligations of an applicant under § 53.7.

(l) *Ultimate purchaser* means the first person who purchases a reference method or an equivalent method for purposes other than resale.

(m) *PM₁₀ sampler* means a device, associated with a manual method for measuring PM₁₀, designed to collect PM₁₀ from an ambient air sample, but lacking the ability to automatically analyze or measure the collected sample to determine the mass concentration of PM₁₀ in the sampled air.

(n) *Test sampler* means a sampler subjected to testing as part of a candidate method in accordance with subpart C or D of this part.

[40 FR 7049, Feb. 18, 1975, as amended at 41 FR 11255, Mar. 17, 1976; 52 FR 24727, July 1, 1987]

§ 53.2 General requirements for a reference method determination.

(a) *Manual methods.* (1) For measuring SO₂ and lead, appendices A and G, respectively, of part 50 of this chapter specify unique manual reference methods for those pollutants. Except as provided in § 53.16, other manual methods for SO₂ and lead will not be considered for reference method determinations under this part.

(2) For measuring PM₁₀, a candidate method must be a manual method that meets the requirements specified in appendix J of part 50 of this chapter and must include a PM₁₀ sampler that meets the requirements specified in subpart D of this part.

(b) *Automated methods.* For measuring CO, O₃, and NO₂, a candidate auto-

rated method must utilize the measurement principle and calibration procedure specified in the appropriate appendix to part 50 of this chapter and must meet the requirements specified in subpart B of this part.

[52 FR 24727, July 1, 1987]

§ 53.3 General requirements for an equivalent method determination.

(a) *Manual methods.* Candidate manual methods must satisfy the requirements specified in subpart C of this part. In addition, samplers associated with manual methods for PM₁₀ must satisfy the requirements of subpart D of this part.

(b) *Automated methods.* Candidate automated methods for pollutants other than PM₁₀ must satisfy the requirements specified in subparts B and C of this part. Candidate automated methods for PM₁₀ must satisfy the requirements of subparts C and D of this part.

[52 FR 24727, July 1, 1987]

§ 53.4 Applications for reference or equivalent method determinations.

(a) Applications for reference or equivalent method determinations shall be submitted in triplicate to:

Director, Environmental Monitoring Systems Laboratory, Department E, U.S. Environmental Protection Agency, Research Triangle Park, North Carolina 27711.

(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 and by which it may be referred to unambiguously.

(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 listing of the significant components; schematic diagrams; and a detailed description of the apparatus and measurement procedures.

(3) A copy of a comprehensive operation or instruction manual providing a complete and detailed description of

the operational and calibration procedures prescribed for field use of the candidate method and all instruments utilized as part of that method. The manual shall include adequate warning of potential safety hazards that may result from normal use, or (if the method is automated) from normal use or malfunction, of the method and a description of necessary safety precautions (see § 53.9(b)). For samplers and automated methods, the manual shall include a clear description of installation and operation procedures and of necessary periodic maintenance, as well as comprehensive troubleshooting and corrective maintenance procedures and 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, and/or D of this part, as applicable.

(5) Test data, records, calculations, and test results as specified in subparts B, C, and/or D of this part, as applicable.

(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 PM₁₀, the application shall also contain the following:

(1) A detailed description of the quality control program 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 or sampler tested in accordance with this part.

(2) A description of the durability characteristics of such analyzers or samplers (see § 53.9(c)).

[40 FR 7049, Feb. 18, 1975, as amended at 52 FR 24727, July 1, 1987]

§ 53.5 Processing of applications.

After receiving an application for a reference or equivalent method determination, the Administrator will publish notice of the application in the FEDERAL REGISTER and, within 75 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 his 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 75-day period shall commence upon receipt of the additional information);

(d) 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 75-day period shall commence upon receipt of the additional test data); or

(e) 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 75-day period shall commence 1 calendar day after the additional tests have been completed).

§ 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 his 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 his judgment 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 his 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 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.

[40 FR 7049, Feb. 18, 1975, as amended at 41 FR 11256, Mar. 17, 1976]

§ 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 reference method or equivalent method (as applicable), and a notice of the designation shall be submitted for publication in the FEDERAL REGISTER not later than 15 days after the determination is made.

(b) A notice indicating that the method has been determined to be a reference method or an equivalent method shall be sent to the applicant. This notice shall constitute proof of the determination until a notice of designation is published in accordance with paragraph (a) of this section.

(c) The Administrator will maintain a current list of methods designated as reference or equivalent methods in accordance with this part and will send a copy of the list to any person or group upon request. A copy of the list will be available for inspection or copying at EPA Regional Offices.

§ 53.9 Conditions of designation.

Designation of a candidate method as a reference method or equivalent method shall be conditioned on 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 reference or equivalent method shall be accompanied by a copy of the manual referred to in § 53.4(b)(3) when delivered to any ultimate purchaser.

(b) Any method offered for sale as a reference or equivalent method shall generate no unreasonable hazard to operators or to the environment during normal use or (if the method is automated) during normal use or when malfunctioning.

(c) Any analyzer or PM₁₀ sampler offered for sale as a reference or equivalent method shall function within the limits of the performance specifications referred to in § 53.20(a) or § 53.40(a), as applicable, for at least one year after delivery and acceptance when maintained and operated in accordance with the manual referred to in § 53.4(b)(3).

(d) Any analyzer or PM₁₀ sampler offered for sale as a reference or equivalent method shall bear a prominent, permanently affixed label or sticker indicating that the analyzer or PM₁₀ sampler has been designated by EPA as a reference method or as an equivalent method (as applicable) in accordance with this part.

(e) If an analyzer is offered for sale as a reference or equivalent method but

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 reference or equivalent method.

(f) An applicant who offers analyzers or PM₁₀ samplers for sale as reference or equivalent methods shall maintain an accurate and current list of the names and mailing addresses of all ultimate purchasers of such analyzers or PM₁₀ samplers. For a period of seven years after publication of the reference or equivalent method designation applicable to such an analyzer or PM₁₀ sampler, the applicant shall notify all ultimate purchasers of the analyzer or PM₁₀ sampler within 30 days if the designation has been cancelled in accordance with § 53.11 or § 53.16 or if adjustment of the analyzer or PM₁₀ sampler is necessary under § 53.11(b).

(g) If an applicant modifies an analyzer or PM₁₀ sampler that has been designated as a reference or equivalent method, the applicant shall not sell the analyzer or PM₁₀ sampler as modified as a reference or equivalent method nor attach a label or sticker to the analyzer or PM₁₀ sampler as modified under paragraph (d) or (e) of this section until he has received notice under § 53.14(c) that the existing designation or a new designation will apply to the analyzer or PM₁₀ sampler as modified or has applied for and received notice under § 53.8(b) of a new reference or equivalent method determination for the analyzer or PM₁₀ sampler as modified.

[40 FR 7049, Feb. 18, 1975, as amended at 41 FR 11256, Mar. 17, 1976; 52 FR 24728, July 1, 1987]

§ 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 his 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 information available to him 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, he 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 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 cancelled, he will publish a notice of cancellation in the FEDERAL REGISTER and delete the designation 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 cancelled.

§ 53.12 Request for hearing on cancellation.

Within 60 days after publication of notice in accordance with § 53.11(b)(1), the applicant or any interested person may request a hearing on the Administrator's action. 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 and (b) data or other information in support of such objections. If, after reviewing the request and supporting data, the Administrator finds that the request raises a substantial issue of fact, he will grant a hearing in accordance with § 53.13 with respect to such issue.

§ 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 the Environmental Protection Agency, 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 his representative will represent the Environmental Protection Agency in any hearing under this section.

(5) Each party other than the Environmental Protection Agency may be represented by counsel or by any other duly authorized representative.

(b)(1) Upon his 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) At his discretion, 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 leave 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 the Environmental Protection Agency.

(d)(1) The presiding officer, upon the request of any party or at his discretion, may arrange for a prehearing conference at a time and place specified by him 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 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 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 therefor 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 which he would have in 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 therefor 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 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, to the Administrator prior to implementation of the modification (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) If the applicant believes the modification will or may affect the performance characteristics of the method, a brief statement of the probable effect; and

(4) Such further information, including test data, as may be necessary to explain and support any statement required by paragraphs (b)(2) and (b)(3) of this section.

(c) Within 30 calendar days after receiving a report under paragraph (a) of this section, the Administrator will take one or more of the following actions:

(1) Notify the applicant that the designation will continue to apply to the method if the modification is implemented.

(2) Send notice to the applicant that a new designation will apply to the method (as modified) if the modification is implemented, submit notice of the determination for publication in the FEDERAL REGISTER, and revise or supplement the list referred to in § 53.8(c) to reflect the determination.

(3) Send notice to the applicant that the designation will not apply to the method (as modified) if the modification is implemented and submit notice of the determination for publication in the FEDERAL REGISTER;

(4) Send notice to the applicant that additional information must be submitted before a determination can be made and specify the additional information that is needed (in such cases, the 30-day period shall commence upon receipt of the additional information);

(5) Send notice to the applicant that additional tests are necessary and specify what tests are necessary and how they shall be interpreted (in such cases, the 30-day period shall commence upon receipt of the additional test data); or

(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 one 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 he conducted that was a material factor in his determination. A representative of the applicant may be present during the performance of any such retest.

[40 FR 7049, Feb. 18, 1975, as amended at 40 FR 18168, Apr. 25, 1975; 41 FR 11256, Mar. 17, 1976]

§ 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 his 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; and (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 he desires such consideration. 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 75 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 in question and send notice of the determination to the applicant;

(2) Determine that it is inappropriate to propose a revision of the appendix 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 75-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 they shall be interpreted (in such cases, the 75-day period shall commence upon receipt of the additional test data); or

(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 75-day period shall commence one calendar day after the additional tests have been completed).

(e)(1) 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 will indicate that the Administrator proposes (i) to revise the appendix in question; (ii) where the appendix specifies a measurement principle and calibration procedure, to cancel reference method designations based on the appendix; and (iii) to cancel equivalent method designations based on the existing reference method(s). The notice 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) If, after consideration of comments received, the Administrator determines that the appendix in question should be revised, he will by publication in the FEDERAL REGISTER (i) promulgate the proposed revision, with such modifications as may be appropriate in view of comments received; (ii) where the appendix (prior to revision) specifies a measurement principle and calibration procedure, cancel reference method designations based on the appendix; (iii) cancel equivalent method designations based on the existing reference method(s); and (iv) 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. Cancelled 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.

(3) If the appendix 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.

(4) 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 cancelled 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.

(Secs. 110, 301(a), 313, 319, Clean Air Act (42 U.S.C. 7410, 7601(a), 7613, 7619))

[41 FR 11256, Mar. 17, 1976, as amended at 44 FR 27571, May 10, 1979]

Subpart B—Procedures for Testing Performance Characteristics of Automated Methods SO₂, CO, O₃, and NO₂

§ 53.20 General provisions.

(a) The test procedures given in this subpart shall be used to test the performance of candidate automated methods against the performance specifications given in table B-1. A test analyzer representative of the candidate automated method must exhibit performance better than, or equal to, the specified value for each such specification (except Range) to satisfy the requirements of this subpart. Except as provided in paragraph (b) of this section, the range of the candidate method must be the range specified in table B-1 to satisfy the requirements of this subpart.

(b) For a candidate method having more than one selectable range, one range must be that specified in table B-1 and a test analyzer representative of the method must pass the tests required by this subpart while operated in that range. The tests may be repeated for a broader range (i.e., one extending to higher concentrations) than that specified in table B-1 provided that the range does not extend to concentrations more than two times the upper range limit specified in table B-1. If the application is for a reference method determination, the tests may be repeated for a narrower range (one extending to lower concentrations) than that specified in table B-1.

If the tests are conducted or passed only for the specified range, any reference or equivalent method determination with respect to the method will be limited to that range. If the tests are passed for both the specified range and a broader range (or ranges), any such determination will include the broader range(s) as well as the specified range, provided that the tests required by subpart C of this part (if applicable) are met for the broader range(s). If the tests are passed for both the specified range and a narrower range, a reference method determination for the method will include the narrower range as well as the specified range. Appropriate test data shall be submitted for each range sought to be included in a reference or equivalent method determination under this paragraph (b).

(c) For each performance specification (except Range), the test procedure shall be initially repeated seven (7) times to yield 7 test results. Each result shall be compared with the corresponding specification in table B-1; a value higher than or outside that specified constitutes a failure. These 7 results for each parameter shall be interpreted as follows:

(1) Zero (0) failures: Candidate method passes the performance parameter.

(2) Three (3) or more failures: Candidate method fails the performance parameter.

(3) One (1) or two (2) failures: Repeat the test procedures for the parameter eight (8) additional times yielding a total of fifteen (15) test results. The combined total of 15 test results shall then be interpreted as follows:

(i) One (1) or two (2) failures: Candidate method passes the performance parameter.

(ii) Three (3) or more failures: Candidate method fails the performance parameter.

TABLE B-1—PERFORMANCE SPECIFICATIONS FOR AUTOMATED METHODS

Performance parameter	Units ¹	Sulfur dioxide	Photochemical oxidants	Carbon monoxide	Nitrogen dioxide	Definitions and test procedures
1. Range	Parts per million ...	0–0.5	0–0.5	0–50	0–0.5	Sec. 53.23(a).
2. Noisedo005	.005	.50	.005	Sec. 53.23(b).
3. Lower detectable limit	Parts per million01	.01	1.0	.01	Sec. 53.23(c).
4. Interference equivalentdo	Sec. 53.23(d).
Each interferant	Parts per million ...	±.02	±.02	±1.0	±0.02	

TABLE B-1—PERFORMANCE SPECIFICATIONS FOR AUTOMATED METHODS—Continued

Performance parameter	Units ¹	Sulfur di-oxide	Photo-chemical oxidants	Carbon monoxide	Nitrogen dioxide	Definitions and test procedures
Total interferantdo06	.06	1.5	.04	Sec. 52.23(e).
5. Zero drift, 12 and 24 hourdo	±.02	±.02	±1.0	±.02	
6. Span drift, 24 hourdododododo	Do.
20 percent of upper range limit	Percent	±20.0	±20.0	±10.0	±20.0	
80 percent of upper range limitdo	±5.0	±5.0	±2.5	±5.0	
7. Lag time	Minutes	20	20	10	20	Do.
8. Rise timedo	15	15	5	15	Do.
9. Fall timedo	15	15	5	15	Do.
10. Precisiondododododo	Do.
20 percent of upper range limit	Parts per million01	.01	.5	.02	
80 percent of upper range limitdo015	.01	.5	.03	

¹ To convert from parts per million to $\mu\text{g}/\text{m}^3$ at 25 °C and 760 mm Hg, multiply by $M/0.02447$, where M is the molecular weight of the gas.

(d) The tests for *zero drift*, *span drift*, *lag time*, *rise time*, *fall time*, and *precision* shall be combined into a single sequential procedure to be conducted at various line voltages and ambient temperatures specified in §53.23(e). The tests for *noise*, *lower detectable limit*, and *interference equivalents* shall be made at any temperature between 20 °C. and 30 °C. and at any normal line voltage between 105 and 125 volts, and shall be conducted such that not more than three (3) test results for each parameter are obtained per 24 hours.

(e) All response readings to be recorded shall first be converted to concentration units according to the calibration curve constructed in accordance with §53.21(b).

(f) All recorder chart tracings, records, test data and other documentation obtained from or pertinent to these tests shall be identified, dated, signed by the analyst performing the test, and submitted.

NOTE: Suggested formats for reporting the test results and calculations are provided in Figures B-2, B-3, B-4, B-5, and B-6 in appendix A. Symbols and abbreviations used in this subpart are listed in table B-5, appendix A.

[40 FR 7049, Feb. 18, 1975, as amended at 40 FR 18168, Apr. 25, 1975; 41 FR 52694, Dec. 1, 1976]

§ 53.21 Test conditions.

(a) *Set-up and start-up* of the test analyzer shall be in strict accordance with the operating instructions specified in the manual referred to in §53.4(b)(3). Allow adequate warm-up or stabiliza-

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

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

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

shall consist of at least seven (7) approximately equally spaced, identifiable points, including 0 and 90±5 percent of full scale.

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

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

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

§ 53.22 Generation of test atmospheres.

(a) Table B-2 specifies preferred methods for generating test atmospheres and suggested methods of verifying the concentrations. Only one means of establishing the concentration of a test atmosphere is normally required. If the method of generation can produce reproducible concentrations, verification is optional. If the method of generation is not reproducible, then establishment of the concentration by some verification method is required. However, when a method of generation other than that given in table B-2 is used, the test concentration shall be verified.

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

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

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

TABLE B-2—TEST ATMOSPHERES

Test gas	Generation	Verification
Ammonia	Permeation device. Similar to system described in references 1 and 2.	Indophenol method, reference 3.
Carbon dioxide	Cylinder of zero air or nitrogen containing CO ₂ as required to obtain the concentration specified in table B-3.	Use NBS-certified standards whenever possible. If NBS standards are not available, obtain 2 standards from independent sources which agree within 2 percent; or obtain one standard and submit it to an independent laboratory for analysis which must agree within 2 percent of the supplier's nominal analysis.
Carbon monoxide.	Cylinder of zero air or nitrogen containing CO as required to obtain the concentration specified in table B-3.	Do.
Ethane	Cylinder of zero air or nitrogen containing ethane as required to obtain the concentration specified in table B-3.	Do.
Ethylene	Cylinder of prepurified nitrogen containing ethylene as required to obtain the concentration specified in table B-3.	Do.
Hydrogen chloride.	Cylinder ¹ of prepurified nitrogen containing approximately 100 p/m of gaseous HCl. Dilute with zero air to concentration specified in table B-3.	Collect samples in bubbler containing distilled water and analyze by the mercuric thiocyanate method, ASTM (D512), p. 29, reference 4.
Hydrogen sulfide.	Permeation device system described in references 1 and 2.	Tentative method of analysis for H ₂ S content of the atmosphere, p. 426, reference 5.

TABLE B-2—TEST ATMOSPHERES—Continued

Test gas	Generation	Verification
Methane	Cylinder of zero air containing methane as required to obtain the concentration specified in table B-3.	Use NBS-certified standards whenever possible. If NBS standards are not available, obtain 2 standards from independent sources which agree within 2 percent; or obtain one standard and submit it to an independent laboratory for an analysis which must agree within 2 percent of the supplier's nominal analysis.
Nitric oxide	Cylinder ¹ of prepurified nitrogen containing approximately 100 p/m NO. Dilute with zero air to required concentration.	Gas-phase titration as described in reference 6, section 7.1.
Nitrogen dioxide	1. Gas phase titration as described in reference 6 2. Permeation device, similar to system described in references 1 and 2.	1. Use an NO ₂ analyzer calibrated with a gravimetrically calibrated permeation device. 2. Use an NO ₂ analyzer calibrated by gas-phase titration as described in reference 6.
Ozone	Calibrated ozone generator as described in reference 7, appendix D.	Use an ozone analyzer calibrated by gas-phase titration as described in reference 6.
Sulfur dioxide	Permeation device Similar to system described in reference method for SO ₂ , reference 7, appendix A.	P-rosaniline method. Reference 7, appendix A.
Water	Pass zero air through distilled water at a fixed known temperature between 20° and 30 °C. such that the air stream becomes saturated. Dilute with zero air to concentration specified in table B-3.	Measure relative humidity by means of a dew-point indicator, calibrated electrolytic or piezo electric hygrometer, or wet/dry bulb thermometer.
Xylene	Cylinder of prepurified nitrogen containing 100 p/m xylene. Dilute with zero air to concentration specified in table B-3.	Use NBS-certified standards whenever possible. If NBS standards are not available, obtain 2 standards from independent sources which agree within 2 percent; or obtain one standard and submit it to an independent laboratory for an analysis which must agree within 2 percent of the supplier's nominal analysis.
Zero air	1. Ambient air purified by appropriate scrubbers or other devices such that it is free of contaminants likely to cause a detectable response on the analyzer. 2. Cylinder of compressed zero air certified by the supplier or an independent laboratory to be free of contaminants likely to cause a detectable response on the analyzer.	

¹ Use stainless steel pressure regulator dedicated to the pollutant measured.
Reference 1. O'Keeffe, A. E., and Ortman, G. C. "Primary Standards for Trace Gas Analysis," *Anal. Chem.* 38, 760 (1966).
Reference 2. Scaringelli, F. P., A. E., Rosenberg, E., and Bell, J. P., "Primary Standards for Trace Gas Analysis." *Anal. Chem.* 42, 871 (1970).
Reference 3. "Tentative Method of Analysis for Ammonia in the Atmosphere (Indophenol Method)", *Health Lab Sciences*, vol. 10, No. 2, 115-118, April 1973.
Reference 4. *1973 Annual Book of ASTM Standards*, American Society for Testing and Materials, 1916 Race St., Philadelphia, PA.
Reference 5. *Methods for Air Sampling and Analysis*, Intersociety Committee, 1972, American Public Health Association, 1015.
Reference 6. *Federal Register*, vol. 38, No. 110, Tentative Method for the Continuous Measurement of Nitrogen Dioxide (Chemiluminescent) addenda C. (June 8, 1973).
Reference 7. *Federal Register*, vol. 36, No. 228, National Primary and Secondary Ambient Air Quality Standards, Nov. 25, 1971.

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

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

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

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

[40 FR 7049, Feb. 18, 1975, as amended at 40 FR 18168, Apr. 25, 1975]

§ 53.23 Test procedures.

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

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

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

NOTE: A single calibration curve will normally suffice.

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

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

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

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

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

(iv) Convert each DM reading to concentration units (p/m) by reference to the test analyzer's calibration curve as determined in § 53.21(b). Label the converted DM readings $r_1, r_2, r_3 \dots r_1 \dots r_{25}$.

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

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

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

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

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

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

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

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

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

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

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

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

(d) *Interference equivalent*—(1) *Technical definition.* Positive or negative response caused by a substance other than the one being measured.

(2) *Test procedure.* The test analyzer shall be tested for all substances likely to cause a detectable response. The test analyzer shall be challenged, in turn, with each interfering agent specified in table B-3. In the event that there are substances likely to cause a significant interference which have not been specified in table B-3, these substances shall be tested at a concentration substantially higher than that

normally found in the ambient air. The interference may be either positive or negative, depending on whether the test analyzer's response is increased or decreased by the presence of the interferent. Interference equivalents shall be determined by mixing each interferent, one at a time, with the pollutant at the concentrations specified in table B-3, and comparing the test analyzer's response to the response caused by the pollutant alone. Known gas-phase reactions that might occur between an interferent and the pollut-

ant are designated by footnote 3 in table B-3. In these cases, the interference equivalent shall be determined in the absence of the pollutant.

(i) Allow sufficient time for warm-up and stabilization of the test analyzer.

(ii) For a candidate method using a prefilter or scrubber based upon a chemical reaction to derive part of its specificity, and which requires periodic service or maintenance, the test analyzer shall be "conditioned" prior to each interference test as follows:

TABLE B-3—INTERFERANT TEST CONCENTRATION,¹ PARTS PER MILLION

Pollutant	Analyzer type ²	Hydrochloric acid	Ammonia	Hydrogen sulfide	Sulfur dioxide	Nitrogen dioxide	Nitric oxide	Carbon dioxide	Ethylene	Ozone	M-xylene	Water vapor	Carbon monoxide	Methane	Ethane
SO ₂	Flame photometric (FPD)	0.1	10.14	750	3 20,000	50
SO ₂	Gas chromatography (FPD)1	4.14	750	3 20,000	50
SO ₂	Spectrophotometric-wet chemical (pararosaniline reaction)	0.2	30.1	.1	4.14	0.5	750	0.5
SO ₂	Electrochemical2	3.1	.1	4.14	.5	0.5	0.2	.5	3 20,000
SO ₂	Conductivity2	3.1	4.14	.5	7505	0.2
SO ₂	Spectrophotometric-gas phase	4.14	.5
O ₃	Chemiluminescent	3.1	750	4.08	3 20,000
O ₃	Electrochemical	3.15	.5	4.08	3 20,000
O ₃	Spectrophotometric-wet chemical (potassium iodide reaction)	3.15	.5	3.5	4.08
O ₃	Spectrophotometric-gas phase5	.5	3.5	4.08
CO	Infrared	750	20,000	4 10
CO	Gas chromatography with flame ionization detector	20,000	4 10	0.5
CO	Electrochemical	20,000	4 10
CO	Catalytic combustion-thermal detection	750	.2	20,000	4 10	5.0
CO	IR fluorescence	750	20,000	4 105
CO	Mercury replacement UV photometric	4 105
NO ₂	Chemiluminescent	3.15	4.1	.5	20,000
NO ₂	Spectrophotometric-wet chemical (azo-dye reaction)5	4.1	.5	7505
NO ₂	Electrochemical	0.2	3.15	4.1	.5	7505	20,000	50
NO ₂	Spectrophotometric-gas phase	3.15	4.1	.55	20,000	50

¹ Concentrations of interferant listed must be prepared and controlled to ± 10 percent of the state value.

² Analyzer types not listed will be considered by the administrator as special cases.

³ Do not mix with pollutant.

⁴ Concentration of pollutant used for test. These pollutant concentrations must be prepared to ± 10 percent of the stated value.

(A) Service or perform the indicated maintenance on the scrubber or prefilter as directed in the manual referred to in § 53.4(b)(3).

(B) Before testing for each interferent, allow the test analyzer to sample through the scrubber a test atmosphere containing the interferent at a concentration equal to the value specified in table B-3. Sampling shall be at the normal flow rate and shall be continued for 6 continuous hours prior to testing.

(iii) Generate three test atmosphere streams as follows:

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

(B) Test atmosphere *I*: Interference concentration.

(C) Test atmosphere *Z*: Zero air.

(iv) Adjust the individual flow rates and the pollutant or interferent generators for the three test atmospheres as follows:

(A) The flow rates of test atmospheres *I* and *Z* shall be identical.

(B) The concentration of pollutant in test atmosphere *P* shall be adjusted such that when *P* is mixed (diluted) with either test atmosphere *I* or *Z*, the resulting concentration of pollutant shall be as specified in table B-3.

(C) The concentration of interferent in test atmosphere *I* shall be adjusted such that when *I* is mixed (diluted) with test atmosphere *P*, the resulting concentration of interferent shall be equal to the value specified in table B-3.

(D) To minimize concentration errors due to flow rate differences between *I* and *Z*, it is recommended that, when possible, the flow rate of *P* be from 10 to 20 times larger than the flow rates of *I* and *Z*.

(v) Mix test atmospheres *P* and *Z* by passing the total flow of both atmospheres through a mixing flask.

(vi) Sample and measure the mixture of test atmospheres *P* and *Z* with the test analyzer. Allow for a stable reading, and record the reading, in concentration units, as *R* (see Figure B-3).

(vii) Mix test atmospheres *P* and *I* by passing the total flow of both atmospheres through a mixing flask.

(viii) Sample and measure this mixture. Record the stable reading, in concentration units, as *R_I*.

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

$$IE = R_I - R$$

IE must be equal to or less than the specification given in table B-1 for each interferent to pass the test.

(x) Follow steps (iii) through (ix) of this section, in turn, to determine the interference equivalent for each interferent.

(xi) For those interferents which cannot be mixed with the pollutant, as indicated by footnote (3) in table B-3, adjust the concentration of test atmosphere *I* to the specified value without being mixed or diluted by the pollutant test atmosphere. Determine *IE* as follows:

(A) Sample and measure test atmosphere *Z* (zero air). Allow for a stable reading and record the reading, in concentration units, as *R*.

(B) Sample and measure the interferent test atmosphere *I*. If the test analyzer is not capable of negative readings, adjust the analyzer (not the recorder) to give an offset zero. Record the stable reading in concentration units as *R_I*, extrapolating the calibration curve, if necessary, to represent negative readings.

(C) Calculate $IE = R_I - R$. *IE* must be equal to or less than the specification in table B-1 to pass the test.

(xii) Sum the absolute value of all the individual interference equivalents. This sum must be equal to or less than the total interferent specification given in table B-1 to pass the test.

(e) *Zero drift, span drift, lag time, rise time, fall time, and precision*—(1) *Technical definitions*—(i) *Zero drift*: The change in response to zero pollutant concentration, over 12- and 24-hour periods of continuous unadjusted operation.

(ii) *Span drift*: The percent change in response to an up-scale pollutant concentration over a 24-hour period of continuous unadjusted operation.

(iii) *Lag time*: The time interval between a step change in input concentration and the first observable corresponding change in response.

(iv) *Rise time*: The time interval between initial response and 95 percent of

final response after a step increase in input concentration.

(v) *Fall time*: The time interval between initial response and 95 percent of final response after a step decrease in input concentration.

(vi) *Precision*: Variation about the mean of repeated measurements of the same pollutant concentration, expressed as one standard deviation about the mean.

(2) Tests for these performance parameters shall be accomplished over a period of seven (7) or more days. During this time, the line voltage supplied to the test analyzer and the ambient temperature surrounding the analyzer shall be varied from day to day. One test result for each performance parameter shall be obtained each test day, for seven (7) or fifteen (15) test days as necessary. The tests are performed sequentially in a single procedure.

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

(4) Table B-4 specifies the line voltage and room temperature to be used for each test day. The line voltage and temperature shall be changed to the specified values at the start of each test day (i.e., at the start of the 12-hour zero test). Initial adjustments (day zero) shall be made at a line voltage of 115 volts (rms) and a room temperature of 25 °C.

(5) The tests shall be conducted in blocks consisting of 3 test days each until 7 or 15 test results have been obtained. (The final block may contain

fewer than three test days.) If a test is interrupted by an occurrence other than a malfunction of the test analyzer, only the block during which the interruption occurred shall be repeated.

(6) During each block, manual adjustments to the electronics, gas, or reagent flows or periodic maintenance shall not be permitted. Automatic adjustments which the test analyzer performs by itself are permitted at any time.

(7) At least 4 hours prior to the start of the first test day of each block, the test analyzer may be adjusted and/or serviced according to the periodic maintenance procedures specified in the manual referred to in §53.4(b)(3). If a new block is to immediately follow a previous block, such adjustments or servicing may be done immediately after completion of the day's tests for the last day of the previous block and at the voltage and temperature specified for that day, but only on test days 3, 6, 9, and 12.

NOTE: If necessary, the beginning of the test days succeeding such maintenance or adjustment may be delayed as necessary to complete the service or adjustment operation.

(8) All response readings to be recorded shall first be converted to concentration units according to the calibration curve. Whenever a test atmosphere is to be measured but a stable reading is not required, the test atmosphere shall be measured long enough to cause a change in response of at least 10% of full scale. Identify all readings and other pertinent data on the strip chart. (See Figure B-1 illustrating the pattern of the required readings.)

TABLE B-4—LINE VOLTAGE AND ROOM TEMPERATURE TEST CONDITIONS

Test day	Line voltage, ¹ rms	Room temperature, ² °C	Comments
0	115	25	Initial set-up and adjustments.
1	125	20	
2	105	20	
3	125	30	Adjustments and/or periodic maintenance permitted at end of tests.
4	105	30	
5	125	20	
6	105	20	Adjustments and/or periodic maintenance permitted at end of tests.
7	125	30	Examine test results to ascertain if further testing is required.
8	105	30	
9	125	20	Adjustments and/or periodic maintenance permitted at end of tests.
10	105	20	
11	125	30	
12	105	30	Adjustments and/or periodic maintenance permitted at end of tests.

TABLE B-4—LINE VOLTAGE AND ROOM TEMPERATURE TEST CONDITIONS—Continued

Test day	Line voltage, ¹ rms	Room temperature, ² °C	Comments
13	125	20	
14	105	20	
15	125	30	

¹ Voltage specified shall be controlled to ± 1 volt.
² Temperature specified shall be controlled to ± 1 °C.

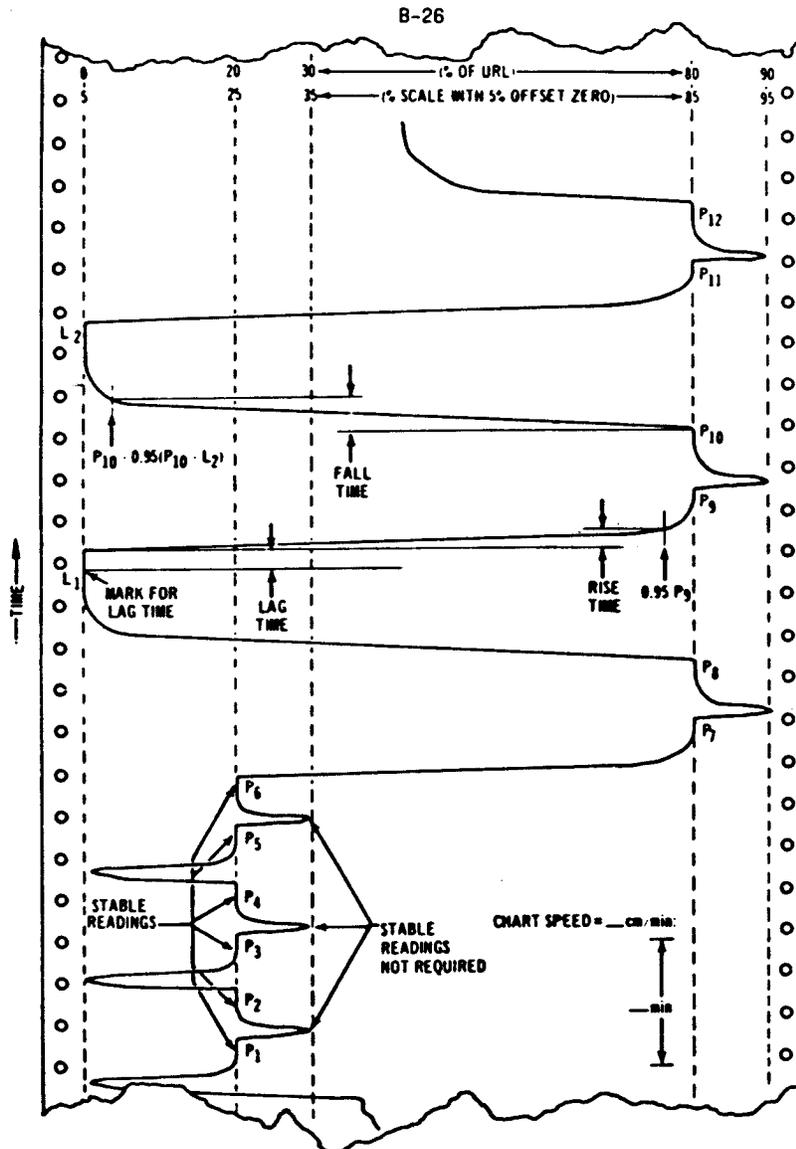


Figure B-1. Example showing the nature of the tracing obtained during the test for drift, lag time, rise time, fall time, and precision. The time scale has been greatly compressed.

(9) Test procedure. (i) Arrange to generate pollutant test atmospheres as follows:

Test atmosphere	Pollutant concentration (percent)
A ₀	Zero air.
A ₂₀	20±5 of the upper range limit.
A ₃₀	30±5 of the upper range limit.

Test atmosphere	Pollutant concentration (percent)
A ₈₀	80±5 of the upper range limit.
A ₉₀	90±5 of the upper range limit.

Test atmospheres A₀, A₂₀, and A₈₀ shall be consistent during the tests and from day to day.

(ii) For steps (xxv) through (xxxi) of this section, a chart speed of at least 10 centimeters per hour shall be used. The actual chart speed, chart speed changes, and time checks shall be clearly marked on the chart.

(iii) Allow sufficient time for test analyzer to warm up and stabilize at a line voltage of 115 volts and a room temperature of 25 °C. Recalibrate, if necessary, and adjust the zero baseline to 5 percent of chart. No further adjustments shall be made to the analyzer until the end of the tests on the third day.

(iv) Measure test atmosphere A₀ until a stable reading is obtained, and record this reading (in ppm) as Z_n, where n=0 (see Figure B-4 in appendix A).

(v) Measure test atmosphere A₂₀. Allow for a stable reading and record it as M_n, where n=0.

(vi) Measure test atmosphere A₈₀. Allow for a stable reading and record it as S_n, where n=0.

(vii) The above readings for Z₀, M₀, and S₀ should be taken at least four (4) hours prior to the beginning of test day 1.

(viii) At the beginning of each test day, adjust the line voltage and room temperature to the values given in table B-4.

(ix) Measure test atmosphere A₀ continuously for at least twelve (12) continuous hours during each test day.

(x) After the 12-hour zero drift test (step ix), sample test atmosphere A₀. A stable reading is not required.

(xi) Measure test atmosphere A₂₀ and record the stable reading (in ppm) as P₁. (See Figure B-4 in appendix A.)

(xii) Sample test atmosphere A₃₀; a stable reading is not required.

(xiii) Measure test atmosphere A₂₀ and record the stable reading as P₂.

(xiv) Sample test atmosphere A₀; a stable reading is not required.

(xv) Measure test atmosphere A₂₀ and record the stable reading as P₃.

(xvi) Sample test atmosphere A₃₀; a stable reading is not required.

(xvii) Measure test atmosphere A₂₀ and record the stable reading as P₄.

(xviii) Sample test atmosphere A₀; a stable reading is not required.

(xix) Measure test atmosphere A₂₀ and record the stable reading as P₅.

(xx) Sample test atmosphere A₃₀; a stable reading is not required.

(xxi) Measure test atmosphere A₂₀ and record the stable reading as P₆.

(xxii) Measure test atmosphere A₃₀ and record the stable reading as P₇.

(xxiii) Sample test atmosphere A₉₀; a stable reading is not required.

(xxiv) Measure test atmosphere A₈₀ and record the stable reading as P₈. Increase chart speed to at least 10 centimeters per hour.

(xxv) Measure test atmosphere A₀. Record the stable reading as L₁.

(xxvi) Quickly switch the test analyzer to measure test atmosphere A₈₀ and mark the recorder chart to show the exact time when the switch occurred.

(xxvii) Measure test atmosphere A₉₀ and record the stable reading as P₈₀.

(xxviii) Sample test atmosphere A₉₀; a stable reading is not required.

(xxix) Measure test atmosphere A₈₀ and record the stable reading as P₁₀.

(xxx) Measure test atmosphere A₀ and record the stable reading as L₂.

(xxxi) Measure test atmosphere A₈₀ and record the stable reading as P₁₁.

(xxxii) Sample test atmosphere A₉₀; a stable reading is not required.

(xxxiii) Measure test atmosphere A₈₀ and record the stable reading as P₁₂.

(xxxiv) Repeat steps (viii) through (xxxiii) of this section, each test day.

(xxxv) If zero and span adjustments are made after the readings are taken on test days 3, 6, 9, or 12, complete all adjustments; then measure test atmospheres A₀, A₈₀, and A₂₀. Allow for a stable reading on each, and record the readings as Z_n, S_n, and M_n respectively, where n=the test day number.

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

(i) *Zero drift.* (A) 12-hour. Examine the strip chart pertaining to the 12-

hour continuous zero air test. Determine the minimum (Cmin.) and maximum (Cmax.) readings (in p/m) during this period of 12 consecutive hours, extrapolating the calibration curve to negative concentration units if necessary. Determine the 12-hour zero drift (12ZD) as $12ZD = C_{max.} - C_{min.}$. (See Figure B-5 in appendix A.)

(B) Calculate the 24-hour zero drift (24ZD) for the *n*-th test day as $24ZD_n = Z_n - Z_{n-1}$, or $24ZD_n = Z_n - Z_{n-1}$ if zero adjustment was made on the previous day, where $Z_n = \frac{1}{2} (L_1 + L_2)$ for L_1 and L_2 taken on the *n*-th test day.

(C) Compare 12 ZD and 24 ZD to the zero drift specification in table B-1. Both 12 ZD and 24 ZD must be equal to or less than the specified value to pass the test for zero drift.

(ii) *Span drift.* (A) Span drift at 20 percent of URL (MSD)

$$MSD_n = \frac{M_n - M_{n-1}}{M_{n-1}} \times 100\%$$

$$MSD_n = \frac{M_n - M'_{n-1}}{M'_{n-1}} \times 100\%$$

If span adjustment was made on the previous day, where

$$M_n = \frac{1}{6} \sum_{i=1}^6 P_i$$

n indicates the *n*-th test day, and *i* indicates the *i*-th reading on the *n* th day.

(B) Span drift at 80 percent of URL (USD):

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

or

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

If span adjustment was made on the previous day, where

$$S_n = \frac{1}{6} \sum_{i=7}^{12} P_i$$

n indicates the *n*-th test day, and *i* indicates the *i*-th reading on the *n*-th test day.

(C) Both USD and MSD must be equal to or less than the respective specifications given in table B-1 to pass the test for span draft.

(iii) *Lag time.* Determine, from the strip chart, the elapsed time in minutes between the mark made in step (xxvi) and the first observable (two times the noise level) response. This time must be equal to or less than the time specified in table B-1 to pass the test for lag time.

(iv) *Rise time.* Calculate 95 percent of reading P_9 and determine from the recorder chart, the elapsed time between the first observable (two times noise level) response and a response equal to 95 percent of the P_9 reading. This time must be equal to or less than the rise time specified in table B-1 to pass the test for rise time.

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

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

(A)

$$P_{30} = \sqrt{\frac{1}{5} \left[\sum_{i=1}^6 P_i^2 - \frac{1}{6} \left(\sum_{i=1}^6 P_i \right)^2 \right]}$$

(B)

$$P_{30} = \sqrt{\frac{1}{5} \left[\sum_{i=7}^{12} P_i^2 - \frac{1}{6} \left(\sum_{i=7}^{12} P_i \right)^2 \right]}$$

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(C) Both P_{20} and P_{80} must be equal to or less than the specification given in table B-1 to pass the test for precision.

[40 FR 7049, Feb. 18, 1975, as amended at 41 FR 52694, Dec. 1, 1976]

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

TABLE B-5—SYMBOLS AND ABBREVIATIONS

B_L	Analyzer reading at specified <i>LDL</i> concentration.
B_x	Analyzer reading at 0 concentration for <i>LDL</i> test.
<i>DM</i>	Digital meter.
C_{max}	Maximum analyzer reading during 12 <i>ZD</i> test.
C_{min}	Minimum analyzer reading during 12 <i>ZD</i> test.
i	Subscript indicating the i -th quantity in a series.
<i>IE</i>	Interference equivalent.
L_1	First analyzer zero reading for 24 <i>ZD</i> test.
L_2	Second analyzer zero reading for 24 <i>ZD</i> test.
M_n	Average of $P_1 \dots P_6$ for the n -th test day.
M_n	Adjusted span reading at 20 percent of <i>URL</i> on the n -th test day.
<i>MSD</i>	Span drift at 20 percent of <i>URL</i> .

TABLE B-5—SYMBOLS AND ABBREVIATIONS—Continued

n	Subscript indicating the test day number.
P	Analyzer reading for precision test.
P_i	The i -th analyzer reading for precision test.
P_{20}	Precision at 20 percent of <i>URL</i> .
P_{80}	Precision at 80 percent of <i>URL</i> .
R	Analyzer reading of pollutant alone for <i>IE</i> test.
R_I	Analyzer reading with interferent added for <i>IE</i> test.
r_i	The i -th <i>DM</i> reading for noise test.
S	Standard deviation of noise readings.
S_0	Noise value (S) measured at 0 concentration.
S_{80}	Noise value (S) measured at 80 percent of <i>URL</i> .
S_n	Average of $P_7 \dots P_{12}$ for the n -th test day.
S_n	Adjusted span reading at 80 percent of <i>URL</i> on the n -th test day.
<i>URL</i>	Upper range limit.
<i>USD</i>	Span drift at 80 percent of o
Z	Average of L_1 and L_2 .
Z_n	Average of L_1 and L_2 on the n -th test day.
Z_n	Adjusted zero reading on the n -th test day.
<i>ZD</i>	Zero drift.
12 <i>ZD</i>	12-hour zero drift.
24 <i>ZD</i>	24-hour zero drift.

Applicant _____ Date _____

Test No. _____

Analyzer _____ Range _____

READING NUMBER (i)	TIME	0% of URL		80% of URL	
		DM READING	f_i , ppm	DM READING	f_i , ppm
1					
2					
3					
4					
5					
6					
7					
8					
9					
10					
11					
12					
13					
14					
15					
16					
17					
18					
19					
20					
21					
22					
23					
24					
25					
$\sum_{i=1}^{25} f_i$					
$\sum_{i=1}^{25} f_i^2$					
S			$S_0 =$		$S_{80} =$

Figure B-2. Form for noise data.

Applicant _____ Range _____
 Analyzer _____

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_2															
	B_L															
	$LDL = B_L - B_2$															
INTERFERENCE EQUIVALENT	R_1															
	R_{11}															
	$IE_1 = R_{11} \cdot R_1$															
	R_2															
	R_{12}															
	$IE_2 = R_{12} \cdot R_2$															
	R_3															
	R_{13}															
	$IE_3 = R_{13} \cdot R_3$															
	R_4															
	R_{14}															
	$IE_4 = R_{14} \cdot R_4$															
	R_5															
	R_{15}															
	$IE_5 = R_{15} \cdot R_5$															
TOTAL	$IE_T = \sum_{i=1}^5 IE_i$															

Figure B-3. Form for data and calculations for lower detectable limit and interference equivalent.

Applicant _____
 Analyzer _____ Range _____

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

Figure B-4. Form recording data for drift and precision.

Applicant		Range														
Analyzer		n - th TEST DAY														
TEST PARAMETER	CALCULATION	1	2	3	4	5	6	7	8	9	10	11	12	13	14	15
Zero drift	12 hour $12ZD = C_{max} - C_{min}$															
	$Z = \frac{1}{2}(L_1 + L_2)$															
24 hour	$24ZD_n = Z_n - Z_{n-1}$															
	$24ZD_n = Z_n - Z'_{n-1}$															
20% URL	$M_n = \frac{1}{6} \sum_{i=1}^6 P_i$															
	$MSD_n = \frac{M_n - M_{n-1}}{M_{n-1}} \times 100\%$															
Span drift	$MSD_n = \frac{M_n - M'_{n-1}}{M'_{n-1}} \times 100\%$															
	$S_n = \frac{1}{6} \sum_{i=1}^6 P_i$															
80% URL	$USD_n = \frac{S_n - S_{n-1}}{S_{n-1}} \times 100\%$															
	$USD_n = \frac{S_n - S'_{n-1}}{S'_{n-1}} \times 100\%$															
Precision	$P_{20} = \sqrt{\frac{1}{5} \left[\sum_{i=1}^6 P_i^2 - \frac{1}{6} \left(\sum_{i=1}^6 P_i \right)^2 \right]}$															
	$P_{80} = \sqrt{\frac{1}{5} \left[\sum_{i=1}^{12} P_i^2 - \frac{1}{6} \left(\sum_{i=1}^{12} P_i \right)^2 \right]}$															

Figure B-5. Form for calculating zero drift, span drift and precision.

Applicant _____		Analysis _____															
Analyzer _____		Range _____															
PERFORMANCE PARAMETER	Table B-1 spec.	TEST															No. of test failures
NOISE, ppm	0% URL (5g)	1	2	3	4	5	6	7	8	9	10	11	12	13	14	15	
	80% URL (5g)																
LDL (must be 2 × noise)																	
INTER-FERENCE EQUIV. ALENT, ppm	IE ₁																
	IE ₂																
	IE ₃																
	IE ₄																
	IE ₅																
TOTAL (IE _T)																	
ZERO DRIFT, ppm	12 hour (1ZZD)																
	24 hour (2AZD)																
SPAN DRIFT, %	20% URL (MSD)																
	80% URL (USD)																
LAG TIME, min																	
RISE TIME, min																	
FALL TIME, min																	
PRECISION, ppm	20% URL (P ₂₀)																
	80% URL (P ₈₀)																

^aCompare each test LDL reading with the corresponding noise measurements. LDL reading must exceed the 0% URL noise value by a factor of 2 to pass the test for LDL.

Figure B-6. Form for summary of test results.

[40 FR 7049, Feb. 18, 1975, as amended at 40 FR 18169, Apr. 25, 1975]

Subpart C—Procedures for Determining Comparability Between Candidate Methods and Reference Methods

AUTHORITY: Secs. 110, 301(a), Clean Air Act, as amended (42 U.S.C. 7410, 7601).

§ 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.

(1) Comparability is shown for SO₂, CO, O₃, and NO₂ methods when the differences between:

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

(ii) Measurements made simultaneously by a reference method, are less than or equal to the values specified in the last column of table C-1.

(2) Comparability is shown for lead methods when the differences between:

(i) Measurements made by a candidate method, and (ii) measurements made by the reference method on simultaneously collected lead samples (or the same sample, if applicable), are less than or equal to the value specified in table C-3.

(3) Comparability is shown for PM₁₀ methods when the relationship between: (i) Measurements made by a candidate method, and (ii) measurements made by a reference method on simultaneously collected PM₁₀ samples (or the same sample, if applicable) at each of two test sites, is such that the linear regression parameters (slope, intercept, and correlation coefficient) describing the relationship meet the values specified in table C-4.

(b) *Selection of test sites*—(1) *All methods*. Each test site shall be in a predominately urban area which can be shown to have at least moderate concentrations of various pollutants. The site shall be clearly identified and shall be justified as an appropriate test site with suitable supporting evidence such as maps, population density data, vehicular traffic data, emission inventories, pollutant measurements from previous years, concurrent pollutant measurements, and wind or weather data. If desired, a request for approval of the test site or sites may be submitted prior to conducting the tests. The Administrator may in his discretion select a different site (or sites) for any additional tests he decides to conduct.

(2) *Methods for SO₂, CO, O₃, and NO₂*. 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 speci-

fied ranges. (See paragraph (d)(2) of this section.)

(3) *Methods for lead*. 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 lead concentrations in the specified range. Test sites for lead measurements must be located near a heavily traveled roadway.

(4) *Methods for PM₁₀*. Test measurements must be made at, 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 PM₁₀ concentrations in the specified range.

(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 table C-1, C-3, or C-4, as appropriate.

(d) *Sample collection*—(1) *All methods*. All test concentration measurements or samples shall be taken in such a way that both the candidate method and the reference method receive air samples that are homogenous or as nearly identical as practical.

(2) *Methods for SO₂, CO, O₃, and NO₂*. 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 particulates and trace gases, and to insure that identical samples reach to 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.

(3) *Methods for lead and PM₁₀*. The ambient air intake points of the candidate and reference method samplers for lead or PM₁₀ shall be located 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.

(4) *Methods employing a common sampling procedure*. Candidate methods which employ a sampler and sample collection procedure that are identical to the sampler and sample collection procedure specified in the reference method 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.

(e) *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.

[44 FR 37916, June 29, 1979 and 52 FR 24728, July 1, 1987]

§ 53.31 Test conditions.

(a) *All methods*. All test measurements made or test samples collected by means of a sample manifold as specified in § 53.30(d)(2) 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 paragraph (c) of this section prior to initiation of the tests.

(b) *Samplers and automated methods*. Set-up and start-up of the test analyzer, test sampler(s), and reference method (if applicable) shall be in strict accordance with the applicable operation manual(s). If the test analyzer does not have an integral strip chart recorder, connect the analyzer output to a suitable strip chart recorder of the servo, null-balance type. 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.

NOTE: 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 evaluation of data submitted.

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

(c) *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 manual method (or portion thereof) shall be calibrated if such calibration is a part of the method.

(d) *Range*. (1) Except as provided in paragraph (d)(2) of this section, each method shall be operated in the range specified for the reference method in the appropriate appendix to part 50 (for manual reference methods), or specified in table B-1 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 and a test analyzer representative of the method must pass the tests required by this subpart while operated in that range. The tests may be repeated for a broader range (i.e., one extending to higher concentrations) than the one specified in table B-1, provided that the range does not extend to concentrations more than two times the upper range limit specified in table B-1 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, 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.

(e) *Operation of automated methods.* (1) Once the test analyzer has been set up and calibrated and 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. At 3-day intervals only adjustments and periodic maintenance as specified in the manual referred to in § 53.4(b)(3) are permitted. 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.

[40 FR 7049, Feb. 18, 1975, as amended at 40 FR 18169, Apr. 25, 1975; 44 FR 37917, June 29, 1979; 52 FR 24728, July 1, 1987]

§ 53.32 Test procedures for methods for SO₂, CO, O₃, and NO₂.

(a) Conduct the first set of simultaneous measurements with the candidate and reference methods:

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

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

(3) The measurements shall be made in the sequence specified in table C-2, except for the 1-hour SO₂ measurements, which are all in the high range.

(b) 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

constitutes a failure. (See Figure C-1 in appendix A for a suggested format for reporting the test results).

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

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

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

(3) One (1) or two (2) failures: Conduct a second set of simultaneous measurements as specified in table C-1. The results of the combined total of first-set and second-set measurements shall be interpreted as follows:

(i) One (1) or two (2) failures: The candidate method passes the test for comparability.

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

(4) For sulfur dioxide, 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.

(d) 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.

(e) 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.

(f) For oxidant and carbon monoxide, no more than six (6) 1-hour measurements shall be made per day. For sulfur dioxide, no more than four (4) 1-hour measurements or one (1) 24-hour measurement shall be made per day. One-hour measurements may be made concurrently with 24-hour measurements if appropriate.

(g) For applicable methods, control or calibration checks may be per-

formed 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.

TABLE C-1—TEST CONCENTRATION RANGES, NUMBER OF MEASUREMENTS REQUIRED, AND MAXIMUM DISCREPANCY SPECIFICATION

Pollutant	Concentration range, parts per million	Simultaneous measurements required				Maximum discrepancy specification, parts per million
		1-hr		24-hr		
		First set	Second set	First set	Second set	
Oxidants	Low 0.06 to 0.10	5	6	0.02
	Med 0.15 to 0.25	5	603
	High 0.35 to 0.45	4	604
Total	14	18
Carbon monoxide	Low 7 to 11	5	6	1.5
	Med 20 to 30	5	6	2.0
	High 35 to 45	4	6	3.0
Total	14	18
Sulfur dioxide	Low 0.02 to 0.05	3	3	0.02
	Med 0.10 to 0.15	2	3	.03
	High 0.30 to 0.50	7	8	2	2	.04
Total	7	8	7	8
Nitrogen dioxide	Low 0.02 to 0.08	3	3	0.02
	Med 0.10 to 0.20	2	3	.02
	High 0.25 to 0.35	2	2	.03
Total	7	8

TABLE C-2—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.

[40 FR 7049, Feb. 18, 1975, as amended at 41 FR 52693, Dec. 12, 1976; 44 FR 37917, June 29, 1979; 52 FR 24728, July 1, 1987]

§ 53.33 Test procedure for methods for lead.

(a) *Sample collection.* Collect simultaneous 24-hour samples (filters) of lead at the test site or sites with both the reference and candidate methods until at least 10 filter pairs have been obtained. If the conditions of § 53.30(d)(4) apply, collect at least 10 common samples (filters) in accordance with § 53.30(d)(4) and divide each to form the filter pairs.

(b) *Audit samples.* Three audit samples must be obtained from the Director, Quality Assurance Division (MD-77), Environmental Monitoring Systems Laboratory, U.S. Environmental

Protection Agency, Research Triangle Park, NC 27711. The audit samples are $\frac{3}{4} \times 8$ -inch glass fiber strips containing known amounts of lead at the following nominal levels: 100 $\mu\text{g}/\text{strip}$; 300 $\mu\text{g}/\text{strip}$; 750 $\mu\text{g}/\text{strip}$. The true amount of lead in total $\mu\text{g}/\text{strip}$ will be provided with each audit sample.

(c) *Filter analysis.* (1) For both the reference method and the audit samples, analyze each filter extract 3 times in accordance with the reference method analytical procedure. The analysis of replicates should not be performed sequentially (i.e., and single sample should not be analyzed three times in sequence). Calculate the indicated lead concentrations for the reference method samples in $\mu\text{g}/\text{m}^3$ for each analysis of each filter. Calculate the indicated total lead amount for the audit samples in $\mu\text{g}/\text{strip}$ for each analysis of each strip. Label these test results as R_{1A} , R_{1B} , R_{1C} , R_{2A} , R_{2B} , . . . , Q_{1A} , Q_{1B} , Q_{1C} , . . . , where R denotes results from the reference method samples; Q denotes results from the audit samples; 1, 2, 3 indicates filter number and A, B, C indicates 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 lead concentration in $\mu\text{g}/\text{m}^3$ for each analysis of each filter. Label these test results as C_{1A} , C_{1B} , C_{2C} , . . . , where C denotes results from the candidate method. (For candidate methods which provide a direct measurement of lead concentrations without a separable procedure, $C_{1A}=C_{1B}=C_{1C}$, $C_{2A}=C_{2B}=C_{2C}$, etc.)

(d) For the reference method, calculate the average lead concentration for each filter by averaging the concentrations calculated from the three analyses:

$$R_{i\text{ave}} = \frac{R_{iA} + R_{iB} + R_{iC}}{3},$$

where i is the filter number.

(e) Disregard all filter pairs for which the lead concentration as determined in the previous paragraph (d) by the average of the three reference method determinations, falls outside the range of

0.5 to 4.0 $\mu\text{g}/\text{m}^3$. All remaining filter pairs must be subjected to both of the following tests for precision and comparability. At least five filter pairs must be within the 0.5 to 4.0 $\mu\text{g}/\text{m}^3$ range for the tests to be valid.

(f) *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, as follows:

$$P_{Ri} = \frac{R_{i\text{max}} - R_{i\text{min}}}{R_{i\text{ave}}} \times 100\%,$$

or

$$P_{Ci} = \frac{C_{i\text{max}} - C_{i\text{min}}}{C_{i\text{ave}}} \times 100\%,$$

where i indicates the filter number.

(2) 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 (c) of this section, or the entire test procedure (starting with paragraph (a)) must be repeated.

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

(4) 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.

(g) *Test for accuracy.* (1) For the audit samples calculate the average lead concentration for each strip by averaging the concentrations calculated from the three analyses:

$$Q_{i\text{ave}} = \frac{Q_{iA} + Q_{iB} + Q_{iC}}{3}$$

where i is audit sample number.

Calculate the percent difference (D_q) between the indicated lead concentration for each audit sample and the true lead concentration (T_q) as follows:

$$D_{qi} = \frac{Q_{i\text{ave}} - T_{qi}}{T_{qi}} \times 100$$

(2) If any difference value (D_{qi}) exceeds ± 5 percent the accuracy 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 (c) of this section or the entire test procedure (starting with paragraph (a)) must be repeated.

(h) *Test for comparability.* (1) For each filter 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, as:

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

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) exceed ± 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.

(i) The candidate method must pass both the precision test and the comparability test to qualify for designation as an equivalent method.

TABLE C-3—TEST SPECIFICATIONS FOR LEAD METHODS

Concentration range, $\mu\text{g}/\text{m}^3$	0.5-4.0
Minimum number of 24-hr measurements	5
Maximum analytical precision, percent	15
Maximum analytical accuracy, percent	± 5
Maximum difference, percent of reference method ..	± 20

[44 FR 37917, June 29, 1979, as amended at 52 FR 24728, July 1, 1987]

§ 53.34 Test procedure for methods for PM_{10} .

(a) *Sample collection.* Using three reference method samplers collocated with three candidate method samplers, collect a minimum of 15 sets of simultaneous 24-hour PM_{10} samples at each of two test sites (i.e., a minimum of 30 sets of samples, each consisting of three reference method and three candidate method samples collected simultaneously, 180 samples total). If the conditions of § 53.30(d)(4) apply, collect sample sets only with the three reference method samplers.

(b) *Sample analysis.* Analyze each sample (or the same sample if § 53.30(d)(4) applies) according to the reference method or candidate method, as appropriate, and determine the PM_{10} concentration in $\mu\text{g}/\text{m}^3$.

(c) *Test for comparability.* (1) For each of the sample sets, calculate the average PM_{10} concentration obtained with the reference method samplers:

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

where R denotes results from the reference method, i is the sampler number, and j is the set.

(2) For each of the sample sets, calculate the precision of the reference method PM_{10}

MEASUREMENTS:

$$P_j = \sqrt{\frac{\sum_{i=1}^3 R^2_{ij} - \left(\sum_{i=1}^3 R_{ij}\right)^2}{2}}$$

if \bar{R}_j is above $80 \mu\text{g}/\text{m}^3$, or

$$RP_j = 100\% \times \sqrt{\frac{\sum_{i=1}^3 R^2_{ij} - \left(\sum_{i=1}^3 R_{ij}\right)^2}{2}} / \bar{R}_j$$

if \bar{R}_j is above $80 \mu\text{g}/\text{m}^3$.

(3) If R_j falls outside the acceptable concentration range specified in table C-4 for any set, or if P_j or RP_j , as applicable, exceeds the value specified in table C-4 for any set, that set of samples shall be discarded. For each site, at least three of the sample sets shall have R_j values below $80 \mu\text{g}/\text{m}^3$ and at least three of the sample sets shall have R_j values above $80 \mu\text{g}/\text{m}^3$. Additional sample sets shall be collected and analyzed, as necessary, to provide a minimum of 10 acceptable sample sets for each site. If more than 10 sample sets meet the above criteria, all such sample sets shall be used to demonstrate comparability.

(4) For each of the acceptable sample sets, calculate the average PM_{10} concentration obtained with the candidate method samplers:

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

where C denotes results from the candidate method, i is the sampler number, and j is the set.

(5) For each site, plot the average PM_{10} measurements obtained with the candidate method (C_j) against the corresponding average PM_{10} measurements obtained with the reference method (R_j). For each site, calculate and record the linear regression slope and intercept, and the correlation coefficient.

(6) If the linear regression parameters calculated above meet the values specified in table C-4 for each test site, the candidate method passes the test for comparability.

TABLE C-4—TEST SPECIFICATIONS FOR PM_{10} METHODS

Acceptable concentration range, $\mu\text{g}/\text{m}^3$	30 to 500.
Minimum number of test sites	2.
Number of candidate method samplers per site	3.
Number of reference method samplers per site	3.
Minimum number of 24-hour samples per sampler per site.	15.
Minimum number of acceptable sample sets per site.	10.
Precision of replicate reference method measurements.	$5 \mu\text{g}/\text{m}^3$ or 7 percent.
Slope of regression relationship	1 ± 0.1 .
Intercept of regression relationship, $\mu\text{g}/\text{m}^3$	0 ± 5 .
Correlation of reference method and candidate method measurements.	≥ 0.97 .

[52 FR 24729, July 1, 1987]

APPENDIX A TO SUBPART C—OPTIONAL FORM FOR REPORTING TEST RESULTS

Candidate Method _____
 Reference Method _____
 Applicant _____ Pollutant _____
 First Set Second Set Type 1 hour 24 hour

CONCENTRATION RANGE	DATE	TIME	CONCENTRATION, ppm		DIFFER- ENCE	TABLE C-1 SPEC.	PASS OR FAIL
			CANDIDATE	REFERENCE			
LOW _____ ppm to _____ ppm	1						
	2						
	3						
	4						
	5						
	6						
MEDIUM _____ ppm to _____ ppm	1						
	2						
	3						
	4						
	5						
	6						
HIGH _____ ppm to _____ ppm	1						
	2						
	3						
	4						
	5						
	6						
	7						
	8						
TOTAL FAILURES:							

Figure C 1. Form for Subpart C test results.

[40 FR 7049, Feb. 18, 1975, as amended at 40 FR 18169, Apr. 25, 1975]

Subpart D—Procedures for Testing Performance Characteristics of Methods for PM₁₀

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 PM₁₀ 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 PM₁₀ sampler previously approved as part of a designated PM₁₀ method, only the test for precision need be conducted and passed to satisfy the requirements of this subpart. For a candidate method using a PM₁₀ sampler inlet previously approved as part of a designated PM₁₀ 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.*

*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 Cri-

(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 PM₁₀ 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.

teria 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.

(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 PM₁₀ 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. Sampling effectiveness is no more than 5 percent above that obtained for liquid particles of same size.
B. Solid particles	Percent	
2. 50 Percent cutpoint	µm	10±1.5 µm aerodynamic diameter.
3. Precision	µg/m ³ or percent.	5 µg/m ³ 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.

§ 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 fluorescein 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	/	/	/
5±0.5	/	/	/
7±0.5	/	/	/
9±0.5	/	/	/
10±0.5	/	/	/
11±0.5	/	/	/
13±1.0	/	/	/
15±1.0	/	/	/
20±1.0	/	/	/

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

Particle size (µm) ^a	Wind speed (km/hr)		
	2	8	24
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 multipliets 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 $(p \times 100\%) / (X)$, 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:

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

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:

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

where n=number of replicates.

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

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

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 semi-logarithmic 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:

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

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 PM₁₀ SAMPLERS

Particle size (μm)	Test sampler			Ideal Sampler		
	Sampling effectiveness	Interval mass concentration (μg/m ³)	Expected mass concentration (μg/m ³)	Sampling effectiveness	Interval mass concentration (μg/m ³)	Expected mass concentration (μg/m ³)
(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

TABLE D-3—EXPECTED MASS CONCENTRATION FOR PM₁₀ SAMPLERS—Continued

Particle size (um)	Test sampler			Ideal Sampler		
	Sampling effectiveness	Interval mass concentration (µg/m ³)	Expected mass concentration (µg/m ³)	Sampling effectiveness	Interval mass concentration (µg/m ³)	Expected mass concentration (µg/m ³)
(1)	(2)	(3)	(4)	(5)	(6)	(7)
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
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 _{san(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 D₅₀ on the corresponding sampling effectiveness plot.

(ii) The candidate method passes the 50 percent cutpoint test if the D₅₀ 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 PM₁₀ 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 in-

struction manual and/or appendix J to part 50 of this chapter. All measurements of flow rate must be in actual volumetric units. Record the PM₁₀ 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 PM₁₀ 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:

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

if \bar{C}_j is below 80 μg/m³, or

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

if \bar{C}_j is above 80 μg/m³.

(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:

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

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:

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

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

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(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.

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.

(b) Notice to State: Service of notice given to a State under this part regarding violation of an emission standard or limitation, or an order issued with respect to an emission standard or limitation shall be accomplished by certified mail addressed to an authorized representative of the State agency charged with responsibility for air pollution control in the State. A copy of such notice shall be mailed to the Governor of the State.

(c) Notice to alleged violator: Service of notice given to an alleged violator under this part shall be accomplished by certified mail addressed to, or by personal service upon, the owner or managing agent of the building, plant, installation, or facility alleged to be in violation of an emission standard or limitation, or an order issued with respect to an emission standard or limitation. Where the alleged violator is a corporation, a copy of such notice shall be sent by certified mail to the registered agent, if any, of such corporation in the State in which such violation is alleged to have occurred.

(d) Notice served in accordance with the provisions of this part shall be deemed given on the postmark date, if served by mail, or on the date of receipt, if personally served.

§ 54.3 Contents of notice.

(a) *Failure to act.* Notice regarding a failure of the Administrator to perform an act or duty which is not discretionary shall identify the provisions of the Act which requires such act or creates such duty, shall describe with reasonable specificity the action taken or not taken by the Administrator which is claimed to constitute a failure to perform such act or duty, and shall state the full name and address of the person giving the notice.

(b) *Violation of standard, limitation or order.* Notices to the Administrator, States, and alleged violators regarding violation of an emission standard or limitation or an order issued with respect to an emission standard or limitation, shall include sufficient information to permit the recipient to identify the specific standard, limitation,