

§ 53.31

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

(b) *Selection of test sites.* (1) Each test site shall be in an area which can be shown to have at least moderate concentrations of various pollutants. Each site shall be clearly identified and shall be justified as an appropriate test site with suitable supporting evidence such as a description of the surrounding area, characterization of the sources and pollutants typical in the area, maps, population density data, vehicular traffic data, emission inventories, pollutant measurements from previous years, concurrent pollutant measurements, meteorological data, and other information useful in supporting the suitability of the site for the comparison test or tests.

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

(c) *Test atmosphere.* Ambient air sampled at an appropriate test site or sites shall be used for these tests. Simultaneous concentration measurements shall be made in each of the concentra-

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tion ranges specified in tables C-1, C-3, or C-4 of this subpart, as appropriate.

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

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

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

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

§ 53.31 [Reserved]

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

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

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

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

(b) *Test measurements.* All test measurements are to be made at the same

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

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

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

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

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

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

(2) For a candidate method having more than one selectable range, one

range must be that specified in table B-1 of subpart B of this part, and a test analyzer representative of the method must pass the tests required by this subpart while operated on that range. The tests may be repeated for a broader range (i.e., one extending to higher concentrations) than the one specified in table B-1 of subpart B of this part, provided that the range does not extend to concentrations more than two times the upper range limit specified in table B-1 of subpart B of this part and that the test analyzer has passed the tests required by subpart B of this part (if applicable) for the broader range. If the tests required by this subpart are conducted or passed only for the range specified in table B-1 of subpart B of this part, any equivalent method determination with respect to the method will be limited to that range. If the tests are passed for both the specified range and a broader range (or ranges), any such determination will include the broader range(s) as well as the specified range. Appropriate test data shall be submitted for each range sought to be included in such a determination.

(f) *Operation of automated methods.* (1) Once the test analyzer has been set up and calibrated and tests started, manual adjustment or normal periodic maintenance, as specified in the manual referred to in §53.4(b)(3), is permitted only every 3 days. Automatic adjustments which the test analyzer performs by itself are permitted at any time. The submitted records shall show clearly when manual adjustments were made and describe the operations performed.

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

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

(4) Ambient air shall be sampled from a common intake and distribution

manifold designed to deliver homogeneous air samples to both methods. Precautions shall be taken in the design and construction of this manifold to minimize the removal of particulate matter and trace gases, and to insure that identical samples reach the two methods. If necessary, the concentration of pollutant in the sampled ambient air may be augmented with artificially generated pollutant. However, at all times the air sample measured by the candidate and reference methods under test shall consist of not less than 80 percent ambient air by volume. Schematic drawings, physical illustrations, descriptions, and complete details of the manifold system and the augmentation system (if used) shall be submitted.

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

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

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

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

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

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

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

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

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

urements shall be interpreted as follows:

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

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

(iv) For SO₂, the 1-hour and 24-hour measurements shall be interpreted separately, and the candidate method must pass the tests for both 1- and 24-hour measurements to pass the test for comparability.

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

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

(6) For O₃ and CO, no more than six 1-hour measurements shall be made per day. For SO₂, no more than four 1-hour measurements or one 24-hour measurement shall be made per day. One-hour measurements may be made concurrently with 24-hour measurements if appropriate.

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

made with the method, and the correction procedure shall become a part of the method.

§ 53.33 Test procedure for methods for Pb.

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

(1) Measurements made by a candidate method, and

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

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

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

(d) *Sample collection.* Collect simultaneous 24-hour samples (filters) of Pb at the test site or sites with both the reference and candidate methods until at least 10 filter pairs have been obtained. A candidate method which employs a sampler and sample collection procedure that are identical to the sampler and sample collection procedure specified in the reference method, but uses a different analytical procedure, may be tested by analyzing common samples. The common samples shall be collected according to the sample collection procedure specified by the reference method and each shall be divided for respective analysis in accordance with the analytical procedures of the candidate method and the reference method.

(e) *Audit samples.* Three audit samples must be obtained from the address given in § 53.4(a). The audit samples are $\frac{3}{4} \times 8$ -inch glass fiber strips containing

known amounts of Pb at the following nominal levels: 100 micrograms per strip ($\mu\text{g}/\text{strip}$); 300 $\mu\text{g}/\text{strip}$; 750 $\mu\text{g}/\text{strip}$. The true amount of Pb, in total $\mu\text{g}/\text{strip}$, will be provided with each audit sample.

(f) *Filter analysis.* (1) For both the reference method samples and the audit samples, analyze each filter extract three times in accordance with the reference method analytical procedure. The analysis of replicates should not be performed sequentially, i.e., a single sample should not be analyzed three times in sequence. Calculate the indicated Pb concentrations for the reference method samples in micrograms per cubic meter ($\mu\text{g}/\text{m}^3$) for each analysis of each filter. Calculate the indicated total Pb 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 indicate the filter number, and A, B, C indicate the first, second, and third analysis of each filter, respectively.

(2) For the candidate method samples, analyze each sample filter or filter extract three times and calculate, in accordance with the candidate method, the indicated Pb concentration in $\mu\text{g}/\text{m}^3$ 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 Pb concentrations without a separable procedure, $C_{1A}=C_{1B}=C_{1C}$, $C_{2A}=C_{2B}=C_{2C}$, etc.

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

$$\text{Equation 1}$$

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

Where, i is the filter number.

(h) *Accuracy.* (1)(i) For the audit samples, calculate the average Pb concentration for each strip by averaging