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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 ¾ × 8-inch glass fiber strips containing known amounts of Pb at the following nominal levels: 100 micrograms per strip (μg/strip); 300 μg/strip; 750 μg/strip. The true amount of Pb, in total μg/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 (μg/m^3) for each analysis of each filter. Calculate the indicated total Pb amount for the audit samples in μg/strip for each analysis of each strip. 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 μg/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:

Equation 1

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

Where, i is the filter number.

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

the concentrations calculated from the three analyses using equation 2 of this section:

Equation 2

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

Where, i is audit sample number.

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

Equation 3

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

(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 (f) of this section, or the entire test procedure (starting with paragraph (d) of this section) must be repeated.

(i) *Acceptable filter pairs.* Disregard all filter pairs for which the Pb concentration, as determined in paragraph (g) of this section by the average of the three reference method determinations, falls outside the range of 0.5 to 4.0 μg/m³. All remaining filter pairs must be subjected to the tests for precision and comparability in paragraphs (j) and (k) of this section. At least five filter pairs must be within the 0.5 to 4.0 μg/m³ range for the tests to be valid.

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

Equation 4

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

or

Equation 5

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

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 (f) of this section, or the entire test procedure (starting with paragraph (d) of this section) 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.

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

Equation 6

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

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) exceeds ±20 percent, the candidate method passes the test for comparability.

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

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