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) of this section) 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:

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

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

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

(a) *Collocated measurements.* Set up three reference method samplers collocated with three candidate method samplers or analyzers at each of the number of test sites specified in table C–4 of this subpart. At each site, obtain as many sets of simultaneous PM_{10} or $PM_{2.5}$ measurements as necessary (see paragraph (c)(3) of this section), each set consisting of three reference method and three candidate method measurements, all obtained simultaneously. For $PM_{2.5}$ candidate Class II equivalent methods, at least two collocated PM_{10} reference method samplers are also required to obtain $PM_{2.5}/PM_{10}$ ratios for each sample set. Candidate PM_{10} method measurements shall be 24-hour integrated measurements; $PM_{2.5}$ measurements may be either 24- or 48-hour integrated measurements. All collocated measurements in a sample set must cover the same 24- or 48-hour time period. For samplers, retrieve the samples promptly after sample collection and analyze each sample according to the reference method or candidate method, as appropriate, and determine the PM_{10} or $PM_{2.5}$ concentration in $\mu g/m^3$. If the conditions of § 53.30(d)(4) apply, collect sample sets only with the three reference method samplers. Guidance for quality assurance procedures for $PM_{2.5}$ methods is found in section 2.12 of the Quality Assurance Handbook (reference 6 of appendix A to subpart A of this part).

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

(c) *Test for comparability and precision.* (1) For each of the measurement sets, calculate the average PM_{10} or $PM_{2.5}$ concentration obtained with the reference method samplers:

Equation 7

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

where:

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

(2)(i) For each of the measurement sets, calculate the precision of the reference method PM_{10} or $PM_{2.5}$ measurements as:

Equation 8

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

If the corresponding $\bar{R}_j$ is below:

80 µg/m³ for PM_{10} methods.
40 µg/m³ for 24-hour $PM_{2.5}$ at single test sites for Class I candidate methods.
40 µg/m³ for 24-hour $PM_{2.5}$ at sites having $PM_{2.5}/PM_{10}$ ratios >0.75.
30 µg/m³ for 48-hour $PM_{2.5}$ at single test sites for Class I candidate methods.
30 µg/m³ for 48-hour $PM_{2.5}$ at sites having $PM_{2.5}/PM_{10}$ ratios >0.75.
30 µg/m³ for 24-hour $PM_{2.5}$ at sites having $PM_{2.5}/PM_{10}$ ratios <0.40.
20 µg/m³ for 48-hour $PM_{2.5}$ at sites having $PM_{2.5}/PM_{10}$ ratios >0.75.

(ii) Otherwise, calculate the precision of the reference method PM_{10} or $PM_{2.5}$ measurements as:

Equation 9

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

(3) If $\bar{R}_j$ falls outside the acceptable concentration range specified in table C–4 of this subpart for any set, or if P_j RP_j as applicable, exceeds the value specified in table C–4 of this subpart for any set, that set of measurements shall be discarded. For each site, table C–4 of this subpart specifies the minimum number of sample sets required for various conditions, and §53.30(b)(5) specifies the $PM_{2.5}/PM_{10}$ ratio requirements applicable to Class II candidate equivalent methods. Additional measurement sets shall be collected and analyzed, as necessary, to provide a minimum of 10 acceptable measurement sets for each test site. If more than 10 measurement sets are collected that meet the above criteria, all such measurement sets shall be used to demonstrate comparability.

(4) For each of the acceptable measurement sets, calculate the average PM_{10} or $PM_{2.5}$ concentration obtained with the candidate method samplers:

Equation 10

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

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} or $PM_{2.5}$ measurements obtained with the candidate method (R_j) against the corresponding average PM_{10} or $PM_{2.5}$ 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 under paragraph (c)(5) of this section meet the values specified in table C–4 of this subpart for all test sites, the candidate method passes the test for comparability.

[62 FR 38792, July 19, 1997; 63 FR 7714, Feb. 17, 1998]

TABLE C–1 TO SUBPART C OF PART 53—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	
Ozone	Low 0.06 to 0.10	5	6			0.02
	Med 0.15 to 0.25	5	6			.03
	High 0.35 to 0.45	4	6			.04
	Total	14	18			

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	
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	.03
	High 0.25 to 0.35			2	2	.03
	Total			7	8	

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

Measurement	Concentration Range	
	First Set	Second Set
1	Low	Medium
2	High	High
3	Medium	Low
4	High	High
5	Low	Medium
6	Medium	Low
7	Low	Medium
8	Medium	Low
9	High	High
10	Medium	Low
11	High	Medium
12	Low	High
13	Medium	Medium
14	Low	High
15		Low
16		Medium
17		Low
18		High

TABLE C–3 TO SUBPART C OF PART 53—TEST SPECIFICATIONS FOR LEAD METHODS

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

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

Specification	PM_{10}	$PM_{2.5}$	
		Class I	Class II
Acceptable concentration range ($\bar{R}_j$), $\mu g/m^3$	30–300	10–200	10–200
Minimum number of test sites	2	1	2
Number of candidate method samplers per site	3	3	3
Number of reference method samplers per site	3	3	3
Minimum number of acceptable sample sets per site for PM_{10}:			
$\bar{R}_j < 80\ \mu g/m^3$	3		
$\bar{R}_j > 80\ \mu g/m^3$	3		
Total	10		
Minimum number of acceptable sample sets per site for $PM_{2.5}$:			

Specification	PM_{10}	$PM_{2.5}$	
		Class I	Class II
Single test site for Class I candidate equivalent methods:			
$\bar{R}_j$ < 40 μg/m³ for 24-hr or $\bar{R}_j$ < 30 μg/m³ for 48-hr samples		3	
$\bar{R}_j$ > 40 μg/m³ for 24-hr or $\bar{R}_j$ > 30 μg/m³ for 48-hr samples		3	
Sites at which the $PM_{2.5}/PM_{10}$ ratio must be > 0.75:			
$\bar{R}_j$ < 40 μg/m³ for 24-hr or $\bar{R}_j$ < 30 μg/m³ for 48-hr samples			3
$\bar{R}_j$ > 40 μg/m³ for 24-hr or $\bar{R}_j$ > 30 μg/m³ for 48-hr samples			3
Sites at which the $PM_{2.5}/PM_{10}$ ratio must be < 0.40:			
$\bar{R}_j$ < 30 μg/m³ for 24-hr or $\bar{R}_j$ < 20 μg/m³ for 48-hr samples			3
$\bar{R}_j$ > 30 μg/m³ for 24-hr or $\bar{R}_j$ > 20 μg/m³ for 48-hr samples			3
Total, each site		10	10
Precision of replicate reference method measurements, P_j or RP_j respectively, maximum	5 μg/m³ or 7%	2 μg/m³ or 5%	2 μg/m³ or 5%
Slope of regression relationship	1±0.1	1±0.05	1±0.05
Intercept of regression relationship, μg/m³	0±5	0±1	0±1
Correlation of reference method and candidate method measurements	≥0.97	≥0.97	≥0.97

[62 FR 38792, July 18, 1997; 63 FR 7714, Feb. 17, 1998]

FIGURE C–1 TO SUBPART C OF PART 53—SUGGESTED FORMAT FOR REPORTING TEST RESULTS

Candidate Method_____

Reference Method_____

Applicant_____

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

Concentration Range		Date	Time	Concentration, ppm		Difference	Table C–1 Spec.	Pass or Fail
				Candidate	Reference			
Low _____ ppm to _____ ppm1	1							
	2							
	3							
	4							
	5							
	6							
Medium _____ ppm to _____ ppm1	1							
	2							
	3							
	4							
	5							
	6							
High _____ ppm to _____ ppm1	1							
	2							
	3							
	4							

Candidate Method_____

Reference Method_____

Applicant_____

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

Concentration Range		Date	Time	Concentration, ppm		Difference	Table C–1 Spec.	Pass or Fail
				Candidate	Reference			
	5							
	6							
	7							
	8							
							Total Failures:	

APPENDIX A TO SUBPART C OF PART 53—REFERENCES

(1) American National Standard—Specifications and Guidelines for Quality Systems for Environmental Data Collection and Environmental Technology Programs, ANSI/ASQC E4-1994. Available from American Society for Quality Control, 611 East Wisconsin Avenue, Milwaukee, WI 53202.

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

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

§ 53.40 General provisions.

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

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

(c) The liquid particle sampling effectiveness and 50 percent cutpoint of a test sampler shall be determined in a wind tunnel using 10 particle sizes and three wind speeds as specified in table D–2. A minimum of 3 replicate measurements of sampling effectiveness shall be required for each of the 30 test conditions for a minimum of 90 test measurements.

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

*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

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