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(d) *Test procedure.* (1) Calculate and record the target time weighted concentration of Arizona road dust which is equivalent to exposing the sampler to an environment of 150 μg/m^3 over the time between cleaning specified by the candidate sampler's operations manual as:

EQUATION 40

$$\text{Target TWC} = 150\,\mu g/m^3 \times t$$

where:

t = the number of hours specified by the candidate method prior to periodic cleaning.

(2) Clean the candidate sampler. (i) Clean and dry the internal surfaces of the candidate sampler.

(ii) Prepare the internal surfaces in strict accordance with the operating manual referred to in section 7.4.18 of 40 CFR part 50, appendix L.

(3) Determine the preweight of the filter that shall be used in the isokinetic sampler. Record this value as InitWt.

(4) Install the candidate sampler's inlet and the isokinetic sampler within the test chamber or wind tunnel.

(5) Generate a dust cloud. (i) Generate a dust cloud composed of Arizona test dust.

(ii) Introduce the dust cloud into the chamber.

(iii) Allow sufficient time for the particle concentration to become steady within the chamber.

(6) Sample aerosol with a total filter and the candidate sampler. (i) Sample the aerosol for a time sufficient to produce an equivalent TWC equal to that of the target TWC ±15 percent.

(ii) Record the sampling time as t.

(7) Determine the time weighted concentration. (i) Determine the postweight of the isokinetic sampler's total filter.

(ii) Record this value as FinalWt.

(iii) Calculate and record the TWC as:

EQUATION 41

$$TWC = \frac{(FinalWt - InitWt) \times t}{Q}$$

where:

Q = the flow rate of the candidate method.

(iv) If the value of TWC deviates from the target TWC ±15 percent, then the loaded mass is unacceptable and the entire test procedure must be repeated.

(8) Determine the candidate sampler's effectiveness after loading. The candidate sampler's effectiveness as a function of particle aerodynamic diameter must then be evaluated by performing the test in § 53.62 (full wind tunnel test). A sampler which fits the category of inlet deviation in § 53.60(e)(1) may opt to perform the test in § 53.63 (inlet aspiration test) in lieu of the full wind tunnel test. A sampler which fits the category of fractionator deviation in § 53.60(e)(2) may opt to perform the test in § 53.64 (static fractionator test) in lieu of the full wind tunnel test.

(e) *Test results.* If the candidate sampler meets the acceptance criteria for the evaluation test performed in paragraph (d)(8) of this section, then the candidate sampler passes this test with the stipulation that the sampling train be cleaned as directed by and as frequently as that specified by the candidate sampler's operations manual.

§ 53.66 Test procedure: Volatility test.

(a) *Overview.* This test is designed to ensure that the candidate method's losses due to volatility when sampling semi-volatile ambient aerosol will be comparable to that of a federal reference method sampler. This is accomplished by challenging the candidate sampler with a polydisperse, semi-volatile liquid aerosol in three distinct phases. During phase A of this test, the aerosol is elevated to a steady-state, test-specified mass concentration and the sample filters are conditioned and preweighed. In phase B, the challenge aerosol is simultaneously sampled by the candidate method sampler and a reference method sampler onto the preweighed filters for a specified time period. In phase C (the blow-off phase), aerosol and aerosol-vapor free air is sampled by the samplers for an additional time period to partially volatilize the aerosol on the filters. The candidate sampler passes the volatility test if the acceptance criteria presented in table F–1 of this subpart are met or exceeded.

(b) *Technical definitions.* (1) Residual mass (RM) is defined as the weight of the filter after the blow-off phase subtracted from the initial weight of the filter.

(2) Corrected residual mass (CRM) is defined as the residual mass of the filter from the candidate sampler multiplied by the ratio of the reference method flow rate to the candidate method flow rate.

(c) *Facilities and equipment required—* (1) *Environmental chamber.* Because the nature of a volatile aerosol is greatly dependent upon environmental conditions, all phases of this test shall be conducted at a temperature of 22.0 ±0.5 °C and a relative humidity of 40 ±3 percent. For this reason, it is strongly advised that all weighing and experimental apparatus be housed in an environmental chamber capable of this level of control.

(2) *Aerosol generator.* The aerosol generator shall be a pressure nebulizer operated at 20 to 30 psig (140 to 207 kPa) to produce a polydisperse, semi-voltile aerosol with a mass median diameter larger than 1 μm and smaller than 2.5 μm. The nebulized liquid shall be A.C.S. reagent grade glycerol (C_3H_8O, FW = 92.09, CAS 56–81–5) of 99.5 percent minimum purity. For the purpose of this test the accepted mass median diameter is predicated on the stable aerosol inside the internal chamber and not on the aerosol emerging from the nebulizer nozzle. Aerosol monitoring and its stability are described in (c)(3) and (c)(4) of this section.

(3) *Aerosol monitoring equipment.* The evaporation and condensation dynamics of a volatile aerosol is greatly dependent upon the vapor pressure of the volatile component in the carrier gas. The size of an aerosol becomes fixed only when an equilibrium is established between the aerosol and the surrounding vapor; therefore, aerosol size measurement shall be used as a surrogate measure of this equilibrium. A suitable instrument with a range of 0.3 to 10 μm, an accuracy of 0.5 μm, and a resolution of 0.2 μm (e.g., an optical particle sizer, or a time-of-flight instrument) shall be used for this purpose. The parameter monitored for stability shall be the mass median instrument measured diameter (i.e. optical diameter if an optical particle counter is used). A stable aerosol shall be defined as an aerosol with a mass median diameter that has changed less than 0.25 μm over a 4 hour time period.

(4) *Internal chamber.* The time required to achieve a stable aerosol depends upon the time during which the aerosol is resident with the surrounding air. This is a function of the internal volume of the aerosol transport system and may be facilitated by recirculating the challenge aerosol. A chamber with a volume of 0.5 m^3 and a recirculating loop (airflow of approximately 500 cfm) is recommended for this purpose. In addition, a baffle is recommended to dissipate the jet of air that the recirculating loop can create. Furthermore, a HEPA filtered hole in the wall of the chamber is suggested to allow makeup air to enter the chamber or excess air to exit the chamber to maintain a system flow balance. The concentration inside the chamber shall be maintained at 1 mg/m^3 ±20 percent to obtain consistent and significant filter loading.

(5) *Aerosol sampling manifold.* A manifold shall be used to extract the aerosol from the area in which it is equilibrated and transport it to the candidate method sampler, the reference method sampler, and the aerosol monitor. The losses in each leg of the manifold shall be equivalent such that the three devices will be exposed to an identical aerosol.

(6) *Chamber air temperature recorders.* Minimum range 15-25 °C, certified accuracy to within 0.2 °C, resolution of 0.1 °C. Measurement shall be made at the intake to the sampling manifold and adjacent to the weighing location.

(7) *Chamber air relative humidity recorders.* Minimum range 30 - 50 percent, certified accuracy to within 1 percent, resolution of 0.5 percent. Measurement shall be made at the intake to the sampling manifold and adjacent to the weighing location.

(8) *Clean air generation system.* A source of aerosol and aerosol-vapor free air is required for phase C of this test. This clean air shall be produced by filtering air through an absolute (HEPA) filter.

(9) *Balance*. Minimum range 0 - 200 mg, certified accuracy to within 10 μg, resolution of 1 μg.

(d) *Additional filter handling conditions*—(1) *Filter handling*. Careful handling of the filter during sampling, conditioning, and weighing is necessary to avoid errors due to damaged filters or loss of collected particles from the filters. All filters must be weighed immediately after phase A dynamic conditioning and phase C.

(2) *Dynamic conditioning of filters*. Total dynamic conditioning is required prior to the initial weight determined in phase A. Dynamic conditioning refers to pulling clean air from the clean air generation system through the filters. Total dynamic conditioning can be established by sequential filter weighing every 30 minutes following repetitive dynamic conditioning. The filters are considered sufficiently conditioned if the sequential weights are repeatable to ±3 μg.

(3) *Static charge*. The following procedure is suggested for minimizing charge effects. Place six or more Polonium static control devices (PSCD) inside the microbalance weighing chamber, (MWC). Two of them must be placed horizontally on the floor of the MWC and the remainder placed vertically on the back wall of the MWC. Taping two PSCD's together or using double-sided tape will help to keep them from falling. Place the filter that is to be weighed on the horizontal PSCDs facing aerosol coated surface up. Close the MWC and wait 1 minute. Open the MWC and place the filter on the balance dish. Wait 1 minute. If the charges have been neutralized the weight will stabilize within 30-60 seconds. Repeat the procedure of neutralizing charges and weighing as prescribed above several times (typically 2-4 times) until consecutive weights will differ by no more than 3 micrograms. Record the last measured weight and use this value for all subsequent calculations.

(e) *Test procedure*—(1) *Phase A - Preliminary steps*. (i) Generate a polydisperse glycerol test aerosol.

(ii) Introduce the aerosol into the transport system.

(iii) Monitor the aerosol size and concentration until stability and level have been achieved.

(iv) Condition the candidate method sampler and reference method sampler filters until total dynamic conditioning is achieved as specified in paragraph (d)(2) of this section.

(v) Record the dynamically conditioned weight as $InitWt_c$ and $InitWt_r$ where c is the candidate method sampler and r is the reference method sampler.

(2) *Phase B - Aerosol loading*. (i) Install the dynamically conditioned filters into the appropriate samplers.

(ii) Attach the samplers to the manifold.

(iii) Operate the candidate and the reference samplers such that they simultaneously sample the test aerosol for 2 hours for a candidate sampler operating at 16.7 L/min or higher, or proportionately longer for a candidate sampler operating at a lower flow rate.

(3) *Phase C - Blow-off*. (i) Alter the intake of the samplers to sample air from the clean air generation system.

(ii) Sample clean air for one of the required blow-off time durations (1, 2, 3, and 4 hours).

(iii) Remove the filters from the samplers.

(iv) Weigh the filters immediately and record this weight, $FinalWt_c$ and $FinalWt_r$, where c is the candidate method sampler and r is the reference method sampler.

(v) Calculate the residual mass for the reference method sampler:

EQUATION 41A

$$RM_{(ij)} = \left(FinalWt_r - InitWt_r\right)$$

where:

i = repetition number; and
j = blow-off time period.

(vi) Calculate the corrected residual mass for the candidate method sampler as:

EQUATION 41B

$$CRM_{(ij)} = \left(FinalWt_r - InitWt_r\right) \times \frac{Q_r}{Q_c}$$

where:

i = repetition number;
j = blow-off time period;
Q_c = candidate method sampler flow rate, and
Q_r = reference method sampler flow rate.

(4) Repeat steps in paragraph (e)(1) through (e)(3) of this section until three repetitions have been completed for each of the required blow-off time durations (1, 2, 3, and 4 hours).

(f) *Calculations and analysis.* (1) Perform a linear regression with the candidate method CRM as the dependent variable and the reference method RM as the independent variable.

(2) Determine the following regression parameters: slope, intercept, and correlation coefficient (r).

(g) *Test results.* The candidate method passes the volatility test if the regression parameters meet the acceptance criteria specified in table F–1 of this subpart.

[62 FR 38814, July 18, 1997, as amended at 71 FR 61295, Oct. 17, 2006]

TABLE F–1 TO SUBPART F OF PART 53—PERFORMANCE SPECIFICATIONS FOR $PM_{2.5}$ CLASS II EQUIVALENT SAMPLERS

Performance test	Specifications	Acceptance criteria
§ 53.62 Full Wind Tunnel Evaluation	Solid VOAG produced aerosol at 2 km/hr and 24 km/hr.	Dp_{50} = 2.5 μm ± 0.2 μm Numerical Analysis Results: 95% ≤ R_c ≤ 105%.
§ 53.63 Wind Tunnel Inlet Aspiration Test	Liquid VOAG produced aerosol at 2 km/hr and 24 km/hr.	Relative Aspiration: 95% ≤ A ≤ 105%.
§ 53.64 Static Fractionator Test	Evaluation of the fractionator under static conditions.	Dp_{50} = 2.5 μm ± 0.2 μm Numerical Analysis Results: 95% ≤ R_c ≤ 105%.
§ 53.65 Loading Test	Loading of the clean candidate under laboratory conditions.	Acceptance criteria as specified in the post-loading evaluation test (§ 53.62, § 53.63, or § 53.64).
§ 53.66 Volatility Test	Polydisperse liquid aerosol produced by air nebulization of A.C.S. reagent grade glycerol, 99.5% minimum purity.	Regression Parameters Slope = 1 ± 0.1, Intercept = 0 ± 0.15 mg, r ≥ 0.97.

[72 FR 32209, June 12, 2007]

TABLE F–2 TO SUBPART F OF PART 53—PARTICLE SIZES AND WIND SPEEDS FOR FULL WIND TUNNEL TEST, WIND TUNNEL INLET ASPIRATION TEST, AND STATIC CHAMBER TEST

Primary Partical Mean Size[a] (μm)	Full Wind Tunnel Test		Inlet Aspiration Test		Static Fractionator Test	Volatility Test
	2 km/hr	24 km/hr	2 km/hr	24 km/hr		
1.5±0.25	S	S			S	
2.0±0.25	S	S			S	
2.2±0.25	S	S			S	
2.5±0.25	S	S			S	
2.8±0.25	S	S			S	
3.0±0.25			L	L		
3.5±0.25	S	S			S	
4.0±0.5	S	S			S	
Polydisperse Glycerol Aerosol				L		

[a] Aerodynamic diameter.
S=Solid particles.
L=Liquid particles.

TABLE F–3 TO SUBPART F OF PART 53—CRITICAL PARAMETERS OF IDEALIZED AMBIENT PARTICLE SIZE DISTRIBUTIONS

Idealized Distribution	Fine Particle Mode			Coarse Particle Mode			$PM_{2.5}$/PM_{10} Ratio	FRM Sampler Expected Mass Conc. (μg/m³)
	MMD (μm)	Geo. Std. Dev.	Conc. (μg/m³)	MMD (μm)	Geo. Std. Dev.	Conc. (μg/m³)		
Coarse	0.50	2	12.0	10	2	88.0	0.27	13.814