

## **Appendix J to Part 50—Reference Method for the Determination of Particulate Matter as PM<sub>10</sub> in the Atmosphere**

### **1.0 Applicability.**

1.1 This method provides for the measurement of the mass concentration of particulate matter with an aerodynamic diameter less than or equal to a nominal 10 micrometers (PM<sub>10</sub>) in ambient air over a 24-hour period for purposes of determining attainment and maintenance of the primary and secondary national ambient air quality standards for particulate matter specified in § 50.6 of this chapter. The measurement process is nondestructive, and the PM<sub>10</sub> sample can be subjected to subsequent physical or chemical analyses. Quality assurance procedures and guidance are provided in part 58, appendices A and B, of this chapter and in References 1 and 2.

### **2.0 Principle.**

2.1 An air sampler draws ambient air at a constant flow rate into a specially shaped inlet where the suspended particulate matter is inertially separated into one or more size fractions within the PM<sub>10</sub> size range. Each size fraction in the PM<sub>10</sub> size range is then collected on a separate filter over the specified sampling period. The particle size discrimination characteristics (sampling effectiveness and 50 percent cutpoint) of the sampler inlet are prescribed as performance specifications in part 53 of this chapter.

2.2 Each filter is weighed (after moisture equilibration) before and after use to determine the net weight (mass) gain due to collected PM<sub>10</sub>. The total volume of air sampled, corrected to EPA reference conditions (25 C, 101.3 kPa), is determined from the measured flow rate and the sampling time. The mass concentration of PM<sub>10</sub> in the ambient air is computed as the total mass of collected particles in the PM<sub>10</sub> size range divided by the volume of air sampled, and is expressed in micrograms per standard cubic meter ( $\mu\text{g}/\text{std m}^3$ ). For PM<sub>10</sub> samples collected at temperatures and pressures significantly different from EPA reference conditions, these corrected concentrations sometimes differ substantially from actual concentrations (in micrograms per actual cubic meter), particularly at high elevations. Although not required, the actual PM<sub>10</sub> concentration can be calculated from the corrected concentration, using the average ambient temperature and barometric pressure during the sampling period.

2.3 A method based on this principle will be considered a reference method only if (a) the associated sampler meets the requirements specified in this appendix and the requirements in part 53 of this chapter, and (b) the method has been designated as a reference method in accordance with part 53 of this chapter.

### **3.0 Range.**

3.1 The lower limit of the mass concentration range is determined by the repeatability of filter tare weights, assuming the nominal air sample volume for the sampler. For samplers having an automatic filter-changing mechanism, there may be no upper limit. For samplers that do not have an automatic filter-changing mechanism, the upper limit is determined by the filter mass loading beyond which the sampler no longer maintains the operating flow rate within specified limits due to increased pressure drop across the loaded filter. This upper limit cannot be specified precisely because it is a complex function of the ambient particle size distribution and type, humidity, filter type, and perhaps other factors. Nevertheless, all samplers should be capable of measuring 24-hour PM<sub>10</sub> mass concentrations of at least 300  $\mu\text{g}/\text{std m}^3$  while maintaining the operating flow rate within the specified limits.

### **4.0 Precision.**

4.1 The precision of PM<sub>10</sub> samplers must be 5  $\mu\text{g}/\text{m}^3$  for PM<sub>10</sub> concentrations below 80  $\mu\text{g}/\text{m}^3$  and 7 percent for PM<sub>10</sub> concentrations above 80  $\mu\text{g}/\text{m}^3$ , as required by part 53 of this chapter, which prescribes a test procedure that

determines the variation in the PM<sub>10</sub> concentration measurements of identical samplers under typical sampling conditions. Continual assessment of precision via collocated samplers is required by part 58 of this chapter for PM<sub>10</sub> samplers used in certain monitoring networks.

## **5.0 Accuracy.**

5.1 Because the size of the particles making up ambient particulate matter varies over a wide range and the concentration of particles varies with particle size, it is difficult to define the absolute accuracy of PM<sub>10</sub> samplers. Part 53 of this chapter provides a specification for the sampling effectiveness of PM<sub>10</sub> samplers. This specification requires that the expected mass concentration calculated for a candidate PM<sub>10</sub> sampler, when sampling a specified particle size distribution, be within  $\pm 10$  percent of that calculated for an ideal sampler whose sampling effectiveness is explicitly specified. Also, the particle size for 50 percent sampling effectiveness is required to be  $10 \pm 0.5$  micrometers. Other specifications related to accuracy apply to flow measurement and calibration, filter media, analytical (weighing) procedures, and artifact. The flow rate accuracy of PM<sub>10</sub> samplers used in certain monitoring networks is required by part 58 of this chapter to be assessed periodically via flow rate audits.

## **6.0 Potential Sources of Error.**

6.1 Volatile Particles. Volatile particles collected on filters are often lost during shipment and/or storage of the filters prior to the post-sampling weighing<sup>3</sup>. Although shipment or storage of loaded filters is sometimes unavoidable, filters should be reweighed as soon as practical to minimize these losses.

6.2 Artifacts. Positive errors in PM<sub>10</sub> concentration measurements may result from retention of gaseous species on filters.<sup>4 5</sup> Such errors include the retention of sulfur dioxide and nitric acid. Retention of sulfur dioxide on filters, followed by oxidation to sulfate, is referred to as artifact sulfate formation, a phenomenon which increases with increasing filter alkalinity.<sup>6</sup> Little or no artifact sulfate formation should occur using filters that meet the alkalinity specification in section 7.2.4. Artifact nitrate formation, resulting primarily from retention of nitric acid, occurs to varying degrees on many filter types, including glass fiber, cellulose ester, and many quartz fiber filters.<sup>5 7,8,9,10</sup> Loss of true atmospheric particulate nitrate during or following sampling may also occur due to dissociation or chemical reaction. This phenomenon has been observed on Teflon<sup>®</sup> filters<sup>8</sup> and inferred for quartz fiber filters.<sup>11 12</sup> The magnitude of nitrate artifact errors in PM<sub>10</sub> mass concentration measurements will vary with location and ambient temperature; however, for most sampling locations, these errors are expected to be small.

6.3 Humidity. The effects of ambient humidity on the sample are unavoidable. The filter equilibration procedure in section 9.0 is designed to minimize the effects of moisture on the filter medium.

6.4 Filter Handling. Careful handling of filters between presampling and postsampling weighings is necessary to avoid errors due to damaged filters or loss of collected particles from the filters. Use of a filter cartridge or cassette may reduce the magnitude of these errors. Filters must also meet the integrity specification in section 7.2.3.

6.5 Flow Rate Variation. Variations in the sampler's operating flow rate may alter the particle size discrimination characteristics of the sampler inlet. The magnitude of this error will depend on the sensitivity of the inlet to variations in flow rate and on the particle distribution in the atmosphere during the sampling period. The use of a flow control device (section 7.1.3) is required to minimize this error.

6.6 Air Volume Determination. Errors in the air volume determination may result from errors in the flow rate and/or sampling time measurements. The flow control device serves to minimize errors in the flow rate determination, and an elapsed time meter (section 7.1.5) is required to minimize the error in the sampling time measurement.

## **7.0 Apparatus.**

7.1 PM<sub>10</sub> Sampler.

7.1.1 The sampler shall be designed to:

- a. Draw the air sample into the sampler inlet and through the particle collection filter at a uniform face velocity.
- b. Hold and seal the filter in a horizontal position so that sample air is drawn downward through the filter.
- c. Allow the filter to be installed and removed conveniently.
- d. Protect the filter and sampler from precipitation and prevent insects and other debris from being sampled.
- e. Minimize air leaks that would cause error in the measurement of the air volume passing through the filter.
- f. Discharge exhaust air at a sufficient distance from the sampler inlet to minimize the sampling of exhaust air.
- g. Minimize the collection of dust from the supporting surface.

7.1.2 The sampler shall have a sample air inlet system that, when operated within a specified flow rate range, provides particle size discrimination characteristics meeting all of the applicable performance specifications prescribed in part 53 of this chapter. The sampler inlet shall show no significant wind direction dependence. The latter requirement can generally be satisfied by an inlet shape that is circularly symmetrical about a vertical axis.

7.1.3 The sampler shall have a flow control device capable of maintaining the sampler's operating flow rate within the flow rate limits specified for the sampler inlet over normal variations in line voltage and filter pressure drop.

7.1.4 The sampler shall provide a means to measure the total flow rate during the sampling period. A continuous flow recorder is recommended but not required. The flow measurement device shall be accurate to  $\pm 2$  percent.

7.1.5 A timing/control device capable of starting and stopping the sampler shall be used to obtain a sample collection period of  $24 \pm 1$  hr ( $1,440 \pm 60$  min). An elapsed time meter, accurate to within  $\pm 15$  minutes, shall be used to measure sampling time. This meter is optional for samplers with continuous flow recorders if the sampling time measurement obtained by means of the recorder meets the  $\pm 15$  minute accuracy specification.

7.1.6 The sampler shall have an associated operation or instruction manual as required by part 53 of this chapter which includes detailed instructions on the calibration, operation, and maintenance of the sampler.

## 7.2 Filters.

7.2.1 Filter Medium. No commercially available filter medium is ideal in all respects for all samplers. The user's goals in sampling determine the relative importance of various filter characteristics (e.g., cost, ease of handling, physical and chemical characteristics, etc.) and, consequently, determine the choice among acceptable filters. Furthermore, certain types of filters may not be suitable for use with some samplers, particularly under heavy loading conditions (high mass concentrations), because of high or rapid increase in the filter flow resistance that would exceed the capability of the sampler's flow control device. However, samplers equipped with automatic filter-changing mechanisms may allow use of these types of filters. The specifications given below are minimum requirements to ensure acceptability of the filter medium for measurement of  $PM_{10}$  mass concentrations. Other filter evaluation criteria should be considered to meet individual sampling and analysis objectives.

7.2.2 Collection Efficiency.  $\geq 99$  percent, as measured by the DOP test (ASTM-2986) with  $0.3 \mu m$  particles at the sampler's operating face velocity.

7.2.3 Integrity.  $\pm 5 \mu g/m^3$  (assuming sampler's nominal 24-hour air sample volume). Integrity is measured as the  $PM_{10}$  concentration equivalent corresponding to the average difference between the initial and the final weights of a random sample of test filters that are weighed and handled under actual or simulated sampling conditions, but have no air

sample passed through them ( *i.e.* , filter blanks). As a minimum, the test procedure must include initial equilibration and weighing, installation on an inoperative sampler, removal from the sampler, and final equilibration and weighing.

7.2.4 Alkalinity. <25 microequivalents/gram of filter, as measured by the procedure given in Reference 13 following at least two months storage in a clean environment (free from contamination by acidic gases) at room temperature and humidity.

7.3 Flow Rate Transfer Standard. The flow rate transfer standard must be suitable for the sampler's operating flow rate and must be calibrated against a primary flow or volume standard that is traceable to the National Bureau of Standards (NBS). The flow rate transfer standard must be capable of measuring the sampler's operating flow rate with an accuracy of  $\pm 2$  percent.

7.4 Filter Conditioning Environment.

7.4.1 Temperature range: 15 to 30 C.

7.4.2 Temperature control:  $\pm 3$  C.

7.4.3 Humidity range: 20% to 45% RH.

7.4.4 Humidity control:  $\pm 5$ % RH.

7.5 Analytical Balance. The analytical balance must be suitable for weighing the type and size of filters required by the sampler. The range and sensitivity required will depend on the filter tare weights and mass loadings. Typically, an analytical balance with a sensitivity of 0.1 mg is required for high volume samplers (flow rates  $>0.5$  m<sup>3</sup> /min). Lower volume samplers (flow rates  $<0.5$  m<sup>3</sup> /min) will require a more sensitive balance.

## **8.0 Calibration.**

8.1 General Requirements.

8.1.1 Calibration of the sampler's flow measurement device is required to establish traceability of subsequent flow measurements to a primary standard. A flow rate transfer standard calibrated against a primary flow or volume standard shall be used to calibrate or verify the accuracy of the sampler's flow measurement device.

8.1.2 Particle size discrimination by inertial separation requires that specific air velocities be maintained in the sampler's air inlet system. Therefore, the flow rate through the sampler's inlet must be maintained throughout the sampling period within the design flow rate range specified by the manufacturer. Design flow rates are specified as actual volumetric flow rates, measured at existing conditions of temperature and pressure ( $Q_a$  ). In contrast, mass concentrations of PM<sub>10</sub> are computed using flow rates corrected to EPA reference conditions of temperature and pressure ( $Q_{std}$  ).

8.2 Flow Rate Calibration Procedure.

8.2.1 PM<sub>10</sub> samplers employ various types of flow control and flow measurement devices. The specific procedure used for flow rate calibration or verification will vary depending on the type of flow controller and flow indicator employed. Calibration in terms of actual volumetric flow rates ( $Q_a$  ) is generally recommended, but other measures of flow rate (e.g.,  $Q_{std}$  ) may be used provided the requirements of section 8.1 are met. The general procedure given here is based on actual volumetric flow units ( $Q_a$  ) and serves to illustrate the steps involved in the calibration of a PM<sub>10</sub> sampler. Consult the sampler manufacturer's instruction manual and Reference 2 for specific guidance on calibration. Reference 14 provides additional information on the use of the commonly used measures of flow rate and their interrelationships.

8.2.2 Calibrate the flow rate transfer standard against a primary flow or volume standard traceable to NBS. Establish a calibration relationship (e.g., an equation or family of curves) such that traceability to the primary standard is accurate to

within 2 percent over the expected range of ambient conditions ( *i.e.* , temperatures and pressures) under which the transfer standard will be used. Recalibrate the transfer standard periodically.

8.2.3 Following the sampler manufacturer's instruction manual, remove the sampler inlet and connect the flow rate transfer standard to the sampler such that the transfer standard accurately measures the sampler's flow rate. Make sure there are no leaks between the transfer standard and the sampler.

8.2.4 Choose a minimum of three flow rates (actual  $\text{m}^3/\text{min}$ ), spaced over the acceptable flow rate range specified for the inlet (see 7.1.2) that can be obtained by suitable adjustment of the sampler flow rate. In accordance with the sampler manufacturer's instruction manual, obtain or verify the calibration relationship between the flow rate (actual  $\text{m}^3/\text{min}$ ) as indicated by the transfer standard and the sampler's flow indicator response. Record the ambient temperature and barometric pressure. Temperature and pressure corrections to subsequent flow indicator readings may be required for certain types of flow measurement devices. When such corrections are necessary, correction on an individual or daily basis is preferable. However, seasonal average temperature and average barometric pressure for the sampling site may be incorporated into the sampler calibration to avoid daily corrections. Consult the sampler manufacturer's instruction manual and Reference 2 for additional guidance.

8.2.5 Following calibration, verify that the sampler is operating at its design flow rate (actual  $\text{m}^3/\text{min}$ ) with a clean filter in place.

8.2.6 Replace the sampler inlet.

## **9.0 Procedure.**

9.1 The sampler shall be operated in accordance with the specific guidance provided in the sampler manufacturer's instruction manual and in Reference 2. The general procedure given here assumes that the sampler's flow rate calibration is based on flow rates at ambient conditions ( $Q_a$ ) and serves to illustrate the steps involved in the operation of a  $\text{PM}_{10}$  sampler.

9.2 Inspect each filter for pinholes, particles, and other imperfections. Establish a filter information record and assign an identification number to each filter.

9.3 Equilibrate each filter in the conditioning environment (see 7.4) for at least 24 hours.

9.4 Following equilibration, weigh each filter and record the presampling weight with the filter identification number.

9.5 Install a preweighed filter in the sampler following the instructions provided in the sampler manufacturer's instruction manual.

9.6 Turn on the sampler and allow it to establish run-temperature conditions. Record the flow indicator reading and, if needed, the ambient temperature and barometric pressure. Determine the sampler flow rate (actual  $\text{m}^3/\text{min}$ ) in accordance with the instructions provided in the sampler manufacturer's instruction manual. NOTE.—No onsite temperature or pressure measurements are necessary if the sampler's flow indicator does not require temperature or pressure corrections or if seasonal average temperature and average barometric pressure for the sampling site are incorporated into the sampler calibration (see step 8.2.4). If individual or daily temperature and pressure corrections are required, ambient temperature and barometric pressure can be obtained by on-site measurements or from a nearby weather station. Barometric pressure readings obtained from airports must be station pressure, not corrected to sea level, and may need to be corrected for differences in elevation between the sampling site and the airport.

9.7 If the flow rate is outside the acceptable range specified by the manufacturer, check for leaks, and if necessary, adjust the flow rate to the specified setpoint. Stop the sampler.

9.8 Set the timer to start and stop the sampler at appropriate times. Set the elapsed time meter to zero or record the initial meter reading.

9.9 Record the sample information (site location or identification number, sample date, filter identification number, and sampler model and serial number).

9.10 Sample for  $24 \pm 1$  hours.

9.11 Determine and record the average flow rate ( $\bar{Q}_a$ ) in actual  $m^3 / \text{min}$  for the sampling period in accordance with the instructions provided in the sampler manufacturer's instruction manual. Record the elapsed time meter final reading and, if needed, the average ambient temperature and barometric pressure for the sampling period (see note following step 9.6).

9.12 Carefully remove the filter from the sampler, following the sampler manufacturer's instruction manual. Touch only the outer edges of the filter.

9.13 Place the filter in a protective holder or container (e.g., petri dish, glassine envelope, or manila folder).

9.14 Record any factors such as meteorological conditions, construction activity, fires or dust storms, etc., that might be pertinent to the measurement on the filter information record.

9.15 Transport the exposed sample filter to the filter conditioning environment as soon as possible for equilibration and subsequent weighing.

9.16 Equilibrate the exposed filter in the conditioning environment for at least 24 hours under the same temperature and humidity conditions used for presampling filter equilibration (see 9.3).

9.17 Immediately after equilibration, reweigh the filter and record the postsampling weight with the filter identification number.

## 10.0 Sampler Maintenance.

10.1 The  $PM_{10}$  sampler shall be maintained in strict accordance with the maintenance procedures specified in the sampler manufacturer's instruction manual.

## 11.0 Calculations.

11.1 Calculate the average flow rate over the sampling period corrected to EPA reference conditions as  $\bar{Q}_{std}$ . When the sampler's flow indicator is calibrated in actual volumetric units ( $Q_a$ ),  $\bar{Q}_{std}$  is calculated as:

$$\bar{Q}_{std} = \bar{Q}_a \times (P_{av} / T_{av}) (T_{std} / P_{std})$$

where

$\bar{Q}_{std}$  = average flow rate at EPA reference conditions,  $m^3 / \text{min}$ ;

$\bar{Q}_a$  = average flow rate at ambient conditions,  $m^3 / \text{min}$ ;

$P_{av}$  = average barometric pressure during the sampling period or average barometric pressure for the sampling site, kPa (or mm Hg);

$T_{av}$  = average ambient temperature during the sampling period or seasonal average ambient temperature for the sampling site, K;

$T_{std}$  = standard temperature, defined as 298 K;

$P_{std}$  = standard pressure, defined as 101.3 kPa (or 760 mm Hg).

11.2 Calculate the total volume of air sampled as:

$$V_{std} = \bar{Q}_{std} \times t$$

where

$V_{std}$  = total air sampled in standard volume units, std  $m^3$  ;

$t$  = sampling time, min.

11.3 Calculate the  $PM_{10}$  concentration as:

$$PM_{10} = (W_f - W_i) \times 10^6 / V_{std}$$

where

$PM_{10}$  = mass concentration of  $PM_{10}$  ,  $\mu g/std\ m^3$  ;

$W_f$  ,  $W_i$  = final and initial weights of filter collecting  $PM_{10}$  particles, g;

$10^6$  = conversion of g to  $\mu g$ .

NOTE: If more than one size fraction in the  $PM_{10}$  size range is collected by the sampler, the sum of the net weight gain by each collection filter [ $\Sigma(W_f - W_i)$ ] is used to calculate the  $PM_{10}$  mass concentration.

## 12.0 References.

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[52 FR 24664, July 1, 1987; 52 FR 29467, Aug. 7, 1987]