Desiccate for 24 hours, and weigh to a constant weight. Report the results to the nearest 0.1 mg.

NOTE: In order to facilitate the evaporation of TCE liquid samples, these samples may be dried in a controlled temperature oven at temperatures up to 38 °C (100 °F) until the liquid is evaporated.

12.0 Data Analysis and Calculations

Carry out calculations, retaining at least one extra significant figure beyond that of the acquired data. Round off figures after the final calculation. Other forms of the equations may be used as long as they give equivalent results.

12.1 Nomenclature. Same as Method 5, section 12.1, with the following additions:

C_t = TCE blank residue concentration, mg/g.
m_t = Mass of residue of TCE blank after evaporation, mg.
V_{pc} = Volume of water collected in precollector, ml.
V_t = Volume of TCE blank, ml.
V_{tw} = Volume of TCE used in wash, ml.
W_t = Weight of residue in TCE wash, mg.
ρ_t = Density of TCE (see label on bottle), g/ml.

12.2 Dry Gas Meter Temperature, Orifice Pressure Drop, and Dry Gas Volume. Same as Method 5, sections 12.2 and 12.3, except use data obtained in performing this test.

12.3 Volume of Water Vapor.

$$V_{w(std)} = K_2\left(V_{lc} + V_{pc}\right) \qquad \text{Eq. 5A-1}$$

Where:

K_2 = 0.001333 m^3/ml for metric units.
= 0.04706 ft^3/ml for English units.

12.4 Moisture Content.

$$B_{ws} = \frac{V_{w(std)}}{V_{m(std)} + V_{w(std)}} \qquad \text{Eq. 5A-2}$$

NOTE: In saturated or water droplet-laden gas streams, two calculations of the moisture content of the stack gas shall be made, one from the impinger and precollector analysis (Equations 5A–1 and 5A–2) and a second from the assumption of saturated conditions. The lower of the two values of moisture content shall be considered correct. The procedure for determining the moisture content based upon assumption of saturated conditions is given in section 4.0 of Method 4. For the purpose of this method, the average stack gas temperature from Figure 5–3 of Method 5 may be used to make this determination, provided that the accuracy of the in-stack temperature sensor is within 1 °C (2 °F).

12.5 TCE Blank Concentration.

$$C_t = \frac{m_t}{V_t \rho_t} \qquad \text{Eq. 5A-3}$$

NOTE: In no case shall a blank value of greater than 0.001 percent of the weight of TCE used be subtracted from the sample weight.

12.6 TCE Wash Blank.

$$W_t = C_t V_{tw} \rho_t \qquad \text{Eq. 5A-4}$$

12.7 Total PM Weight. Determine the total PM catch from the sum of the weights obtained from Containers 1 and 2, less the TCE blank.

12.8 PM Concentration.

$$c_s = K_3 \frac{m_n}{V_{m(std)}} \qquad \text{Eq. 5A-5}$$

Where:

K_3 = 0.001 g/mg for metric units
= 0.0154 gr/mg for English units

12.9 Isokinetic Variation. Same as in Method 5, section 12.11.

13.0 Method Performance [Reserved]

14.0 Pollution Prevention [Reserved]

15.0 Waste Management [Reserved]

16.0 References

Same as Method 5, section 17.0.

17.0 Tables, Diagrams, Flowcharts, and Validation Data

Plant _____
Date _____
Run No. _____
Filter No. _____
Amount liquid lost during transport _____
Acetone blank volume, ml _____
Acetone blank concentration, mg/mg (Equation 5–4) _____
Acetone wash blank, mg (Equation 5–5) _____

Container number	Weight of particulate collected, mg		
	Final weight	Tare weight	Weight gain
1.			
2.			
Total: Less acetone blank.			

Container number	Weight of particulate collected, mg		
	Final weight	Tare weight	Weight gain
Weight of particulate matter.			

	Volume of liquid water collected	
	Impinger volume, ml	Silica gel weight, g
Final		
Initial		
Liquid collected		
Total volume collected		g* ml

* Convert weight of water to volume by dividing total weight increase by density of water (1 g/ml).

$$\frac{\text{Increase, g}}{(1\text{g/ml})} = \text{Volume water, ml}$$

METHOD 5B—DETERMINATION OF NONSULFURIC ACID PARTICULATE MATTER EMISSIONS FROM STATIONARY SOURCES

NOTE: This method does not include all of the specifications (*e.g.*, equipment and supplies) and procedures (*e.g.*, sampling and analytical) essential to its performance. Some material is incorporated by reference from other methods in this part. Therefore, to obtain reliable results, persons using this method should have a thorough knowledge of at least the following additional test methods: Method 1, Method 2, Method 3, Method 5.

1.0 Scope and Application

1.1 Analyte. Nonsulfuric acid particulate matter. No CAS number assigned.

1.2 Applicability. This method is determining applicable for the determination of nonsulfuric acid particulate matter from stationary sources, only where specified by an applicable subpart of the regulations or where approved by the Administrator for a particular application.

1.3 Data Quality Objectives. Adherence to the requirements of this method will enhance the quality of the data obtained from air pollutant sampling methods.

2.0 Summary of Method

Particulate matter is withdrawn isokinetically from the source and collected on a glass fiber filter maintained at a temperature of 160 ±14 °C (320 ±25 °F). The collected sample is then heated in an oven at 160 °C (320 °F) for 6 hours to volatilize any condensed sulfuric acid that may have been collected, and the nonsulfuric acid particulate mass is determined gravimetrically.

3.0 Definitions [Reserved]

4.0 Interferences [Reserved]

5.0 Safety

5.1 Disclaimer. This method may involve hazardous materials, operations, and equipment. This test method may not address all of the safety problems associated with its use. It is the responsibility of the user of this test method to establish appropriate safety and health practices and to determine the applicability of regulatory limitations prior to performing this test method.

6.0 Equipment and Supplies

Same as Method 5, section 6.0, with the following addition and exceptions:

6.1 Sample Collection. The probe liner heating system and filter heating system must be capable of maintaining a sample gas temperature of 160 ±14 °C (320 ±25 °F).

6.2 Sample Preparation. An oven is required for drying the sample.

7.0 Reagents and Standards

Same as Method 5, section 7.0.

8.0 Sample Collection, Preservation, Storage, and Transport.

Same as Method 5, with the exception of the following:

8.1 Initial Filter Tare. Oven dry the filter at 160 ±5 °C (320 ±10 °F) for 2 to 3 hours, cool in a desiccator for 2 hours, and weigh. Desiccate to constant weight to obtain the initial tare weight. Use the applicable specifications and techniques of section 8.1.3 of Method 5 for this determination.

8.2 Probe and Filter Temperatures. Maintain the probe outlet and filter temperatures at 160 ±14 °C (320 ±25 °F).

9.0 Quality Control

Same as Method 5, section 9.0.

10.0 Calibration and Standardization

Same as Method 5, section 10.0.

11.0 Analytical Procedure

Same as Method 5, section 11.0, except replace section

11.2.2 With the following:

11.1 Container No. 2. Note the level of liquid in the container, and confirm on the analysis sheet whether leakage occurred during transport. If a noticeable amount of leakage has occurred, either void the sample or use methods, subject to the approval of the Administrator, to correct the final results. Measure the liquid in this container either volumetrically to ±1 ml or gravimetrically to ±0.5 g. Transfer the contents to a tared 250 ml beaker, and evaporate to dryness at ambient temperature and pressure. Then oven dry the probe and filter samples at a temperature of 160 ±5 °C (320 ±10 °F) for 6 hours. Cool in a desiccator for 2 hours, and weigh to constant weight. Report the results to the nearest 0.1 mg.

12.0 Data Analysis and Calculations

Same as in Method 5, section 12.0.

13.0 Method Performance [Reserved]

14.0 Pollution Prevention [Reserved]

15.0 Waste Management [Reserved]

16.0 References

Same as Method 5, section 17.0.

17.0 Tables, Diagrams, Flowcharts, and Validation Data [Reserved]

METHOD 5C [RESERVED]

METHOD 5D—DETERMINATION OF PARTICULATE MATTER EMISSIONS FROM POSITIVE PRESSURE FABRIC FILTERS

NOTE: This method does not include all of the specifications (*e.g.*, equipment and supplies) and procedures (*e.g.*, sampling and analytical) essential to its performance. Some material is incorporated by reference from other methods in this part. Therefore, to obtain reliable results, persons using this method should have a thorough knowledge of at least the following additional test methods: Method 1, Method 2, Method 3, Method 5, Method 17.

1.0 Scope and Application

1.1 Analyte. Particulate matter (PM). No CAS number assigned.

1.2 Applicability.

1.2.1 This method is applicable for the determination of PM emissions from positive pressure fabric filters. Emissions are determined in terms of concentration (mg/m^3 or gr/ft^3) and emission rate (kg/hr or lb/hr).

1.2.2 The General Provisions of 40 CFR part 60, §60.8(e), require that the owner or operator of an affected facility shall provide performance testing facilities. Such performance testing facilities include sampling ports, safe sampling platforms, safe access to sampling sites, and utilities for testing. It is intended that affected facilities also provide sampling locations that meet the specification for adequate stack length and minimal flow disturbances as described in Method 1. Provisions for testing are often overlooked factors in designing fabric filters or are extremely costly. The purpose of this procedure is to identify appropriate alternative locations and procedures for sampling the emissions from positive pressure fabric filters. The requirements that the affected facility owner or operator provide adequate access to performance testing facilities remain in effect.

1.3 Data Quality Objectives. Adherence to the requirements of this method will enhance the quality of the data obtained from air pollutant sampling methods.

2.0 Summary of Method

2.1 Particulate matter is withdrawn isokinetically from the source and collected on a glass fiber filter maintained at a temperature at or above the exhaust gas temperature up to a nominal 120 °C (248 ±25 °F). The particulate mass, which includes any material that condenses at or above the filtration temperature, is determined gravimetrically after the removal of uncombined water.

3.0 Definitions [Reserved]

4.0 Interferences [Reserved]

5.0 Safety

5.1 Disclaimer. This method may involve hazardous materials, operations, and equipment. This test method may not address all of the safety problems associated with its use. It is the responsibility of the user to establish appropriate safety and health practices and to determine the applicability of regulatory limitations prior to performing this test method.

6.0 Equipment and Supplies

Same as section 6.0 of either Method 5 or Method 17.

7.0 Reagents and Standards

Same as section 7.0 of either Method 5 or Method 17.

8.0 Sample Collection, Preservation, Storage, and Transport

Same section 8.0 of either Method 5 or Method 17, except replace section 8.2.1 of Method 5 with the following:

8.1 Determination of Measurement Site. The configuration of positive pressure fabric filter structures frequently are not amenable

to emission testing according to the requirements of Method 1. Following are several alternatives for determining measurement sites for positive pressure fabric filters.

8.1.1 Stacks Meeting Method 1 Criteria. Use a measurement site as specified in Method 1, section 11.1.

8.1.2 Short Stacks Not Meeting Method 1 Criteria. Use stack extensions and the procedures in Method 1. Alternatively, use flow straightening vanes of the "egg-crate" type (see Figure 5D–1). Locate the measurement site downstream of the straightening vanes at a distance equal to or greater than two times the average equivalent diameter of the vane openings and at least one-half of the overall stack diameter upstream of the stack outlet.

8.1.3 Roof Monitor or Monovent. (See Figure 5D–2). For a positive pressure fabric filter equipped with a peaked roof monitor, ridge vent, or other type of monovent, use a measurement site at the base of the monovent. Examples of such locations are shown in Figure 5D–2. The measurement site must be upstream of any exhaust point (*e.g.*, louvered vent).

8.1.4 Compartment Housing. Sample immediately downstream of the filter bags directly above the tops of the bags as shown in the examples in Figure 5D–2. Depending on the housing design, use sampling ports in the housing walls or locate the sampling equipment within the compartment housing.

8.2 Determination of Number and Location of Traverse Points. Locate the traverse points according to Method 1, section 11.3. Because a performance test consists of at least three test runs and because of the varied configurations of positive pressure fabric filters, there are several schemes by which the number of traverse points can be determined and the three test runs can be conducted.

8.2.1 Single Stacks Meeting Method 1 Criteria. Select the number of traverse points according to Method 1. Sample all traverse points for each test run.

8.2.2 Other Single Measurement Sites. For a roof monitor or monovent, single compartment housing, or other stack not meeting Method 1 criteria, use at least 24 traverse points. For example, for a rectangular measurement site, such as a monovent, use a balanced 5 × 5 traverse point matrix. Sample all traverse points for each test run.

8.2.3 Multiple Measurement Sites. Sampling from two or more stacks or measurement sites may be combined for a test run, provided the following guidelines are met:

8.2.3.1 All measurement sites up to 12 must be sampled. For more than 12 measurement sites, conduct sampling on at least 12 sites or 50 percent of the sites, whichever is greater. The measurement sites sampled should be evenly, or nearly evenly, distributed among the available sites; if not, all sites are to be sampled.

8.2.3.2 The same number of measurement sites must be sampled for each test run.

8.2.3.3 The minimum number of traverse points per test run is 24. An exception to the 24-point minimum would be a test combining the sampling from two stacks meeting Method 1 criteria for acceptable stack length, and Method 1 specifies fewer than 12 points per site.

8.2.3.4 As long as the 24 traverse points per test run criterion is met, the number of traverse points per measurement site may be reduced to eight.

8.2.3.5 Alternatively, conduct a test run for each measurement site individually using the criteria in section 8.2.1 or 8.2.2 to determine the number of traverse points. Each test run shall count toward the total of three required for a performance test. If more than three measurement sites are sampled, the number of traverse points per measurement site may be reduced to eight as long as at least 72 traverse points are sampled for all the tests.

8.2.3.6 The following examples demonstrate the procedures for sampling multiple measurement sites.

8.2.3.6.1 Example 1. A source with nine circular measurement sites of equal areas may be tested as follows: For each test run, traverse three measurement sites using four points per diameter (eight points per measurement site). In this manner, test run number 1 will include sampling from sites 1,2, and 3; run 2 will include samples from sites 4, 5, and 6; and run 3 will include sites 7, 8, and 9. Each test area may consist of a separate test of each measurement site using eight points. Use the results from all nine tests in determining the emission average.

8.2.3.6.2 Example 2: A source with 30 rectangular measurement sites of equal areas may be tested as follows: For each of the three test runs, traverse five measurement sites using a 3 × 3 matrix of traverse points for each site. In order to distribute the sampling evenly over all the available measurement sites while sampling only 50 percent of the sites, number the sites consecutively from 1 to 30 and sample all the even numbered (or odd numbered) sites. Alternatively, conduct a separate test of each of 15 measurement sites using section 8.2.1 or 8.2.2 to determine the number and location of traverse points, as appropriate.

8.2.3.6.3 Example 3: A source with two measurement sites of equal areas may be tested as follows: For each test of three test runs, traverse both measurement sites, using section 8.2.3 in determining the number of traverse points. Alternatively, conduct two full emission test runs for each measurement site using the criteria in section 8.2.1 or 8.2.2 to determine the number of traverse points.

8.2.3.7 Other test schemes, such as random determination of traverse points for a large number of measurement sites, may be used with prior approval from the Administrator.

8.3 Velocity Determination.

8.3.1 The velocities of exhaust gases from positive pressure baghouses are often too low to measure accurately with the type S pitot tube specified in Method 2 (*i.e.*, velocity head <1.3 mm H_2O (0.05 in. H_2O)). For these conditions, measure the gas flow rate at the fabric filter inlet following the procedures outlined in Method 2. Calculate the average gas velocity at the measurement site as shown in section 12.2 and use this average velocity in determining and maintaining isokinetic sampling rates.

8.3.2 Velocity determinations to determine and maintain isokinetic rates at measurement sites with gas velocities within the range measurable with the type S pitot tube (*i.e.*, velocity head greater than 1.3 mm H_2O (0.05 in. H_2O)) shall be conducted according to the procedures outlined in Method 2.

8.4 Sampling. Follow the procedures specified in sections 8.1 through 8.6 of Method 5 or sections 8.1 through 8.25 in Method 17 with the exceptions as noted above.

8.5 Sample Recovery. Follow the procedures specified in section 8.7 of Method 5 or section 8.2 of Method 17.

9.0 Quality Control

9.1 Miscellaneous Quality Control Measures.

Section	Quality control measure	Effect
8.0, 10.0	Sampling equipment leak check and calibration.	Ensures accurate measurement of stack gas flow rate, sample volume.

9.2 Volume Metering System Checks. Same as Method 5, section 9.2.

10.0 Calibration and Standardization

Same as section 10.0 of either Method 5 or Method 17.

11.0 Analytical Procedure

Same as section 11.0 of either Method 5 or Method 17.

12.0 Data Analysis and Calculations

Same as section 12.0 of either Method 5 or Method 17 with the following exceptions:

12.1 Nomenclature.

A_o = Measurement site(s) total cross-sectional area, m^2 (ft^2).

$\bar{C}$ or C_{avg} = Average concentration of PM for all n runs, mg/scm (gr/scf).

Q_i = Inlet gas volume flow rate, m^3/sec (ft^3/sec).

m_i = Mass collected for run i of n, mg (gr).

T_o = Average temperature of gas at measurement site, °K (°R).

T_i = Average temperature of gas at inlet, °K (°R).

Vol_i = Sample volume collected for run i of n, scm (scf).

$\bar{v}$ = Average gas velocity at the measurement site(s), m/s (ft/s)

Q_o = Total baghouse exhaust volumetric flow rate, m^3/sec (ft^3/sec).

Q_d = Dilution air flow rate, m^3/sec (ft^3/sec).

Tamb = Ambient Temperature, (°K).

12.2 Average Gas Velocity. When following section 8.3.1, calculate the average gas velocity at the measurement site as follows:

$$\bar{v} = \frac{Q_o}{A_o} \qquad \text{Eq. 5D-1}$$

12.3 Volumetric Flow Rate. Total volumetric flow rate may be determined as follows:

$$Q_o = Q_i + Q_d \qquad \text{Eq. 5D-2}$$

12.4 Dilution Air Flow Rate.

$$Q_d = \frac{Q_i\left(T_i - T_o\right)}{T_o - T_{amb}} \qquad \text{Eq. 5D-3}$$

12.5 Average PM Concentration. For multiple measurement sites, calculate the average PM concentration as follows:

$$C_{avg} \text{ or } \bar{C} = \frac{\sum_{i=1}^{n} m_i}{\sum_{i=1}^{n} Vol_i} \qquad \text{Eq. 5D-4}$$

13.0 Method Performance [Reserved]

14.0 Pollution Prevention [Reserved]

15.0 Waste Management [Reserved]

16.0 References

Same as Method 5, section 17.0.

17.0 Tables, Diagrams, Flowcharts, and Validation Data

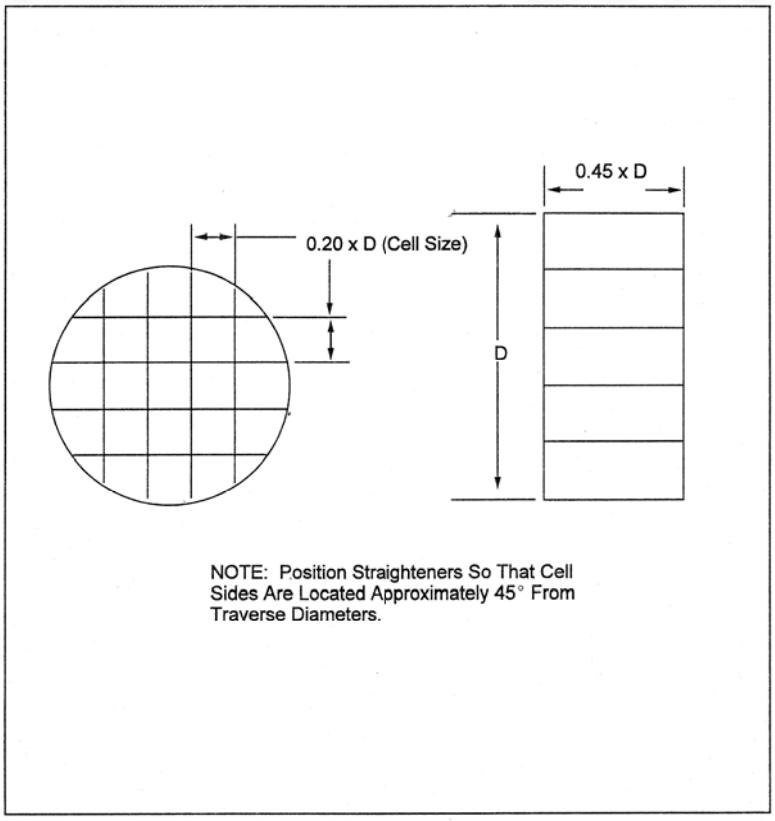


Figure 5D-1. Example of Flow Straightening Vanes.

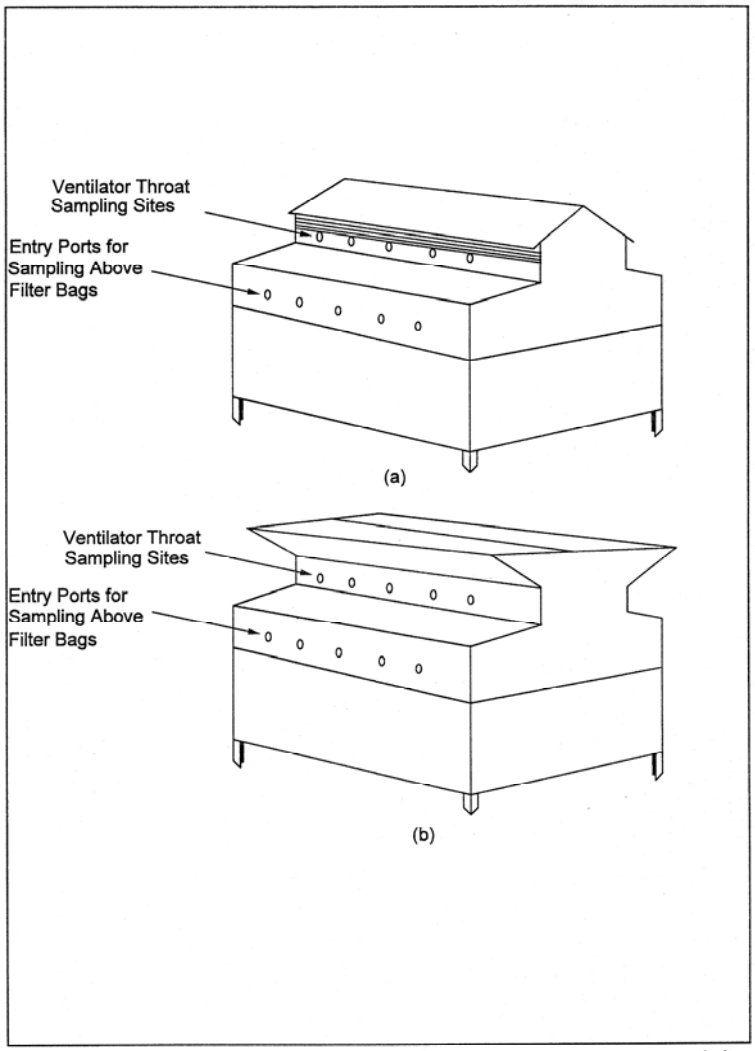


Figure 5D-2. Acceptable Sampling Site Locations for: (a) Peaked Roof; and (b) Ridge Vent Type Fabric Filters

METHOD 5E—DETERMINATION OF PARTICULATE MATTER EMISSIONS FROM THE WOOL FIBERGLASS INSULATION MANUFACTURING INDUSTRY

NOTE: This method does not include all of the specifications (*e.g.*, equipment and supplies) and procedures (*e.g.*, sampling and analytical) essential to its performance. Some material is incorporated by reference from other methods in this part. Therefore, to obtain reliable results, persons using this method should have a thorough knowledge of at least the following additional test methods: Method 1, Method 2, Method 3, and Method 5.

1.0 Scope and Applications

1.1 Analyte. Particulate matter (PM). No CAS number assigned.

1.2 Applicability. This method is applicable for the determination of PM emissions from wool fiberglass insulation manufacturing sources.

2.0 Summary of Method

Particulate matter is withdrawn isokinetically from the source and is collected either on a glass fiber filter maintained at a temperature in the range of 120 ±14 °C (248 ±25 °F) and in impingers in solutions of 0.1 N sodium hydroxide (NaOH). The filtered particulate mass, which includes any material that condenses at or above the filtration temperature, is determined gravimetrically after the removal of uncombined water. The condensed PM collected in the impinger solutions is determined as total organic carbon (TOC) using a nondispersive infrared type of analyzer. The sum of the filtered PM mass and the condensed PM is reported as the total PM mass.

3.0 Definitions [Reserved]

4.0 Interferences [Reserved]

5.0 Safety

5.1 Disclaimer. This method may involve hazardous materials, operations, and equipment. This test method may not address all of the safety problems associated with its use. It is the responsibility of the user of this test method to establish appropriate safety and health practices and to determine the applicability of regulatory limitations prior to performing this test method.

5.2 Corrosive Reagents. The following reagents are hazardous. Personal protective equipment and safe procedures are useful in preventing chemical splashes. If contact occurs, immediately flush with copious amounts of water at least 15 minutes. Remove clothing under shower and decontaminate. Treat residual chemical burn as thermal burn.

5.2.1 Hydrochloric Acid (HCl). Highly toxic. Vapors are highly irritating to eyes, skin, nose, and lungs, causing severe damage. May cause bronchitis, pneumonia, or edema of lungs. Exposure to concentrations of 0.13 to 0.2 percent in air can be lethal in minutes. Will react with metals, producing hydrogen.

5.2.2 Sodium Hydroxide (NaOH). Causes severe damage to eye tissues and to skin. Inhalation causes irritation to nose, throat, and lungs. Reacts exothermically with limited amounts of water.

6.0 Equipment and Supplies

6.1 Sample Collection. Same as Method 5, section 6.1, with the exception of the following:

6.1.1 Probe Liner. Same as described in section 6.1.1.2 of Method 5 except use only borosilicate or quartz glass liners.

6.1.2 Filter Holder. Same as described in section 6.1.1.5 of Method 5 with the addition of a leak-tight connection in the rear half of the filter holder designed for insertion of a temperature sensor used for measuring the sample gas exit temperature.

6.2 Sample Recovery. Same as Method 5, section 6.2, except three wash bottles are needed instead of two and only glass storage bottles and funnels may be used.

6.3 Sample Analysis. Same as Method 5, section 6.3, with the additional equipment for TOC analysis as described below:

6.3.1 Sample Blender or Homogenizer. Waring type or ultrasonic.

6.3.2 Magnetic Stirrer.

6.3.3 Hypodermic Syringe. 0- to 100-µl capacity.

6.3.4 Total Organic Carbon Analyzer. Rosemount Model 2100A analyzer or equivalent and a recorder.

6.3.5 Beaker. 30-ml.

6.3.6 Water Bath. Temperature controlled.

6.3.7 Volumetric Flasks. 1000-ml and 500-ml.

7.0 Reagents and Standards

Unless otherwise indicated, it is intended that all reagents conform to the specifications established by the Committee on Analytical Reagents of the American Chemical Society, where such specifications are available; otherwise, use the best available grade.

7.1 Sample Collection. Same as Method 5, section 7.1, with the addition of 0.1 N NaOH (Dissolve 4 g of NaOH in water and dilute to 1 liter).

7.2 Sample Recovery. Same as Method 5, section 7.2, with the addition of the following:

7.2.1 Water. Deionized distilled to conform to ASTM Specification D 1193–77 or 91 Type 3 (incorporated by reference—see §60.17). The potassium permanganate ($KMnO_4$) test for oxidizable organic matter may be omitted when high concentrations of organic matter are not expected to be present.

7.2.2 Sodium Hydroxide. Same as described in section 7.1.

7.3 Sample Analysis. Same as Method 5, section 7.3, with the addition of the following:

7.3.1 Carbon Dioxide-Free Water. Distilled or deionized water that has been freshly boiled for 15 minutes and cooled to room temperature while preventing exposure to ambient air by using a cover vented with an Ascarite tube.

7.3.2 Hydrochloric Acid. HCl, concentrated, with a dropper.

7.3.3 Organic Carbon Stock Solution. Dissolve 2.1254 g of dried potassium biphthalate ($HOOCC_6H_4COOK$) in CO_2-free water, and dilute to 1 liter in a volumetric flask. This solution contains 1000 mg/L organic carbon.

7.3.4 Inorganic Carbon Stock Solution. Dissolve 4.404 g anhydrous sodium carbonate

(Na_2CO_3.) in about 500 ml of CO_2-free water in a 1-liter volumetric flask. Add 3.497 g anhydrous sodium bicarbonate ($NaHCO_3$) to the flask, and dilute to 1 liter with CO_2 -free water. This solution contains 1000 mg/L inorganic carbon.

7.3.5 Oxygen Gas. CO_2 -free.

8.0 Sample Collection, Preservation, Storage, and Transport

8.1 Pretest Preparation and Preliminary Determinations. Same as Method 5, sections 8.1 and 8.2, respectively.

8.2 Preparation of Sampling Train. Same as Method 5, section 8.3, except that 0.1 N NaOH is used in place of water in the impingers. The volumes of the solutions are the same as in Method 5.

8.3 Leak-Check Procedures, Sampling Train Operation, Calculation of Percent Isokinetic. Same as Method 5, sections 8.4 through 8.6, respectively.

8.4 Sample Recovery. Same as Method 5, sections 8.7.1 through 8.7.4, with the addition of the following:

8.4.1 Save portions of the water, acetone, and 0.1 N NaOH used for cleanup as blanks. Take 200 ml of each liquid directly from the wash bottles being used, and place in glass sample containers labeled "water blank," "acetone blank," and "NaOH blank," respectively.

8.4.2 Inspect the train prior to and during disassembly, and note any abnormal conditions. Treat the samples as follows:

8.4.2.1 Container No. 1. Same as Method 5, section 8.7.6.1.

8.4.2.2 Container No. 2. Use water to rinse the sample nozzle, probe, and front half of the filter holder three times in the manner described in section 8.7.6.2 of Method 5 except that no brushing is done. Put all the water wash in one container, seal, and label.

8.4.2.3 Container No. 3. Rinse and brush the sample nozzle, probe, and front half of the filter holder with acetone as described for Container No. 2 in section 8.7.6.2 of Method 5.

8.4.2.4 Container No. 4. Place the contents of the silica gel impinger in its original container as described for Container No. 3 in section 8.7.6.3 of Method 5.

8.4.2.5 Container No. 5. Measure the liquid in the first three impingers and record the volume or weight as described for the Impinger Water in section 8.7.6.4 of Method 5. Do not discard this liquid, but place it in a sample container using a glass funnel to aid in the transfer from the impingers or graduated cylinder (if used) to the sample container. Rinse each impinger thoroughly with 0.1 N NaOH three times, as well as the graduated cylinder (if used) and the funnel, and put these rinsings in the same sample container. Seal the container and label to clearly identify its contents.

8.5 Sample Transport. Whenever possible, containers should be shipped in such a way that they remain upright at all times.

9.0 Quality Control.

9.1 Miscellaneous Quality Control Measures.

Section	Quality control measure	Effect
8.3, 10.0	Sampling equipment leak-check and calibration.	Ensures accurate measurement of stack gas flow rate, sample volume.
10.1.2, 11.2.5.3	Repetitive analyses	Ensures precise measurement of total carbon and inorganic carbon concentration of samples, blank, and standards.
10.1.4	TOC analyzer calibration	Ensures linearity of analyzer response to standards.

9.2 Volume Metering System Checks. Same as Method 5, section 9.2.

10.0 Calibration and Standardization

Same as Method 5, section 10.0, with the addition of the following procedures for calibrating the total organic carbon analyzer:

10.1 Preparation of Organic Carbon Standard Curve.

10.1.1 Add 10 ml, 20 ml, 30 ml, 40 ml, and 50 ml of the organic carbon stock solution to a series of five 1000-ml volumetric flasks. Add 30 ml, 40 ml, and 50 ml of the same solution to a series of three 500-ml volumetric flasks. Dilute the contents of each flask to the mark using CO_2-free water. These flasks contain 10, 20, 30, 40, 50, 60, 80, and 100 mg/L organic carbon, respectively.

10.1.2 Use a hypodermic syringe to withdraw a 20- to 50-µl aliquot from the 10 mg/L standard solution and inject it into the total carbon port of the analyzer. Measure the peak height. Repeat the injections until three consecutive peaks are obtained within 10 percent of their arithmetic mean. Repeat this procedure for the remaining organic carbon standard solutions.

10.1.3 Calculate the corrected peak height for each standard by deducting the blank correction (see section 11.2.5.3) as follows:

$$\text{Corrected Peak Height} = A - B \qquad \text{Eq. 5E-1}$$

Where:

A = Peak height of standard or sample, mm or other appropriate unit.

B = Peak height of blank, mm or other appropriate unit.

10.1.4 Prepare a linear regression plot of the arithmetic mean of the three consecutive peak heights obtained for each standard solution against the concentration of that solution. Calculate the calibration factor as the inverse of the slope of this curve. If the product of the arithmetic mean peak height for any standard solution and the calibration factor differs from the actual concentration by more than 5 percent, remake and reanalyze that standard.

10.2 Preparation of Inorganic Carbon Standard Curve. Repeat the procedures outlined in sections 10.1.1 through 10.1.4, substituting the inorganic carbon stock solution for the organic carbon stock solution, and the inorganic carbon port of the analyzer for the total carbon port.

11.0 Analytical Procedure

11.1 Record the data required on a sheet such as the one shown in Figure 5–6 of Method 5.

11.2 Handle each sample container as follows:

11.2.1 Container No. 1. Same as Method 5, section 11.2.1, except that the filters must be dried at 20 ±6 °C (68 ±10 °F) and ambient pressure.

11.2.2 Containers No. 2 and No. 3. Same as Method 5, section 11.2.2, except that evaporation of the samples must be at 20 ±6 °C (68 ±10 °F) and ambient pressure.

11.2.3 Container No. 4. Same as Method 5, section 11.2.3.

11.2.4 "Water Blank" and "Acetone Blank" Containers. Determine the water and acetone blank values following the procedures for the "Acetone Blank" container in section 11.2.4 of Method 5. Evaporate the samples at ambient temperature (20 ±6 °C (68 ±10 °F)) and pressure.

11.2.5 Container No. 5. For the determination of total organic carbon, perform two analyses on successive identical samples, *i.e.*, total carbon and inorganic carbon. The desired quantity is the difference between the two values obtained. Both analyses are based on conversion of sample carbon into carbon dioxide for measurement by a nondispersive infrared analyzer. Results of analyses register as peaks on a strip chart recorder.

11.2.5.1 The principal differences between the operating parameters for the two channels involve the combustion tube packing material and temperature. In the total carbon channel, a high temperature (950 °C (1740 °F)) furnace heats a Hastelloy combustion tube packed with cobalt oxide-impregnated asbestos fiber. The oxygen in the carrier gas, the elevated temperature, and the catalytic effect of the packing result in oxidation of both organic and inorganic carbonaceous material to CO_2, and steam. In the inorganic carbon channel, a low temperature (150 °C (300 °F)) furnace heats a glass tube containing quartz chips wetted with 85 percent phosphoric acid. The acid liberates CO_2 and steam from inorganic carbonates. The operating temperature is below that required to oxidize organic matter. Follow the manufacturer's instructions for assembly, testing, calibration, and operation of the analyzer.

11.2.5.2 As samples collected in 0.1 N NaOH often contain a high measure of inorganic carbon that inhibits repeatable determinations of TOC, sample pretreatment is necessary. Measure and record the liquid volume of each sample (or impinger contents). If the sample contains solids or immiscible liquid matter, homogenize the sample with a blender or ultrasonics until satisfactory repeatability is obtained. Transfer a representative portion of 10 to 15 ml to a 30-ml beaker, and acidify with about 2 drops of concentrated HCl to a pH of 2 or less. Warm the acidified sample at 50 °C (120 °F) in a water bath for 15 minutes.

11.2.5.3 While stirring the sample with a magnetic stirrer, use a hypodermic syringe to withdraw a 20-to 50-µl aliquot from the beaker. Analyze the sample for total carbon and calculate its corrected mean peak height according to the procedures outlined in sections 10.1.2 and 10.1.3. Similarly analyze an aliquot of the sample for inorganic carbon. Repeat the analyses for all the samples and for the 0.1 N NaOH blank.

11.2.5.4 Ascertain the total carbon and inorganic carbon concentrations (C_{TC} and C_{IC}, respectively) of each sample and blank by comparing the corrected mean peak heights for each sample and blank to the appropriate standard curve.

NOTE: If samples must be diluted for analysis, apply an appropriate dilution factor.

12.0 Data Analysis and Calculations

Same as Method 5, section 12.0, with the addition of the following:

12.1 Nomenclature.

C_c = Concentration of condensed particulate matter in stack gas, gas dry basis, corrected to standard conditions, g/dscm (gr/dscf).

C_{IC} = Concentration of condensed TOC in the liquid sample, from section 11.2.5, mg/L.

C_t = Total particulate concentration, dry basis, corrected to standard conditions, g/dscm (gr/dscf).

C_{TC} = Concentration of condensed TOC in the liquid sample, from section 11.2.5, mg/L.

C_{TOC} = Concentration of condensed TOC in the liquid sample, mg/L.

m_{TOC} = Mass of condensed TOC collected in the impingers, mg.

$V_{m(std)}$ = Volume of gas sample measured by the dry gas meter, corrected to standard conditions, from section 12.3 of Method 5, dscm (dscf).

V_s = Total volume of liquid sample, ml.

12.2 Concentration of Condensed TOC in Liquid Sample.

$$C_{TOC} = C_{TC} - C_{IC} \qquad \text{Eq. 5E-2}$$

12.3 Mass of Condensed TOC Collected.

$$m_{TOC} = 0.001\, C_{TOC} V_s \qquad \text{Eq. 5E-3}$$

Where:

0.001 = Liters per milliliter.

12.4 Concentration of Condensed Particulate Material.

$$C_c = K_4 m_{TOC} / V_{m(std)} \qquad \text{Eq. 5E-4}$$

Where:

K_4 = 0.001 g/mg for metric units.
= 0.0154 gr/mg for English units.

12.5 Total Particulate Concentration.

$$C_t = C_s + C_c \qquad \text{Eq. 5E-4}$$

13.0 Method Performance [Reserved]

14.0 Pollution Prevention [Reserved]

15.0 Waste Management [Reserved]

16.0 Alternative Procedures

16.1 Total Organic Carbon Analyzer. Tekmar-Dohrmann analyzers using the single injection technique may be used as an alternative to Rosemount Model 2100A analyzers.

17.0 References.

Same as section 17.0 of Method 5, with the addition of the following:

1. American Public Health Association, American Water Works Association, Water Pollution Control Federation. Standard Methods for the Examination of Water and Wastewater. Fifteenth Edition. Washington, D.C. 1980.

18.0 Tables, Diagrams, Flowcharts, and Validation Data [Reserved]

METHOD 5F—DETERMINATION OF NONSULFATE PARTICULATE MATTER EMISSIONS FROM STATIONARY SOURCES

NOTE: This method does not include all of the specifications (*e.g.*, equipment and supplies) and procedures (*e.g.*, sampling and analytical) essential to its performance. Some material is incorporated by reference from other methods in this part. Therefore, to obtain reliable results, persons using this method should have a thorough knowledge of at least the following additional test methods: Method 1, Method 2, Method 3, and Method 5.

1.0 Scope and Applications

1.1 Analyte. Nonsulfate particulate matter (PM). No CAS number assigned.

1.2 Applicability. This method is applicable for the determination of nonsulfate PM emissions from stationary sources. Use of this method must be specified by an applicable subpart of the standards, or approved by the Administrator for a particular application.

1.3 Data Quality Objectives. Adherence to the requirements of this method will enhance the quality of the data obtained from air pollutant sampling methods.

2.0 Summary of Method

Particulate matter is withdrawn isokinetically from the source and collected on a filter maintained at a temperature in the range 160 ±14 °C (320 ±25 °F). The collected sample is extracted with water. A portion of the extract is analyzed for sulfate content by ion chromatography. The remainder is neutralized with ammonium hydroxide (NH_4OH), dried, and weighed. The weight of sulfate in the sample is calculated as ammonium sulfate ($(NH_4)_2SO_4$), and is subtracted from the total particulate weight; the result is reported as nonsulfate particulate matter.

3.0 Definitions [Reserved]

4.0 Interferences [Reserved]

5.0 Safety

5.1 Disclaimer. This method may involve hazardous materials, operations, and equipment. This test method may not address all of the safety problems associated with its use. It is the responsibility of the user of this test method to establish appropriate safety and health practices and to determine the applicability of regulatory limitations prior to performing this test method.

6.0 Equipment and Supplies

6.1 Sample Collection and Recovery. Same as Method 5, sections 6.1 and 6.2, respectively.

6.2 Sample Analysis. Same as Method 5, section 6.3, with the addition of the following:

6.2.1 Erlenmeyer Flasks. 125-ml, with ground glass joints.

6.2.2 Air Condenser. With ground glass joint compatible with the Erlenmeyer flasks.

6.2.3 Beakers. 600-ml.

6.2.4 Volumetric Flasks. 1 liter, 500 ml (one for each sample), 200-ml, and 50-ml (one for each sample and standard).

6.2.5 Pipet. 5-ml (one for each sample and standard).

6.2.6 Ion Chromatograph. The ion chromatograph should have at least the following components.

6.2.6.1 Columns. An anion separation column or other column capable of resolving the sulfate ion from other species present and a standard anion suppressor column. Suppressor columns are produced as proprietary items; however, one can be produced in the laboratory using the resin available from BioRad Company, 32nd and Griffin Streets, Richmond, California. Other systems which do not use suppressor columns may also be used.

6.2.6.2 Pump. Capable of maintaining a steady flow as required by the system.

6.2.6.3 Flow Gauges. Capable of measuring the specified system flow rate.

6.2.6.4 Conductivity Detector.

6.2.6.5 Recorder. Compatible with the output voltage range of the detector.

7.0 Reagents and Standards

Unless otherwise indicated, it is intended that all reagents conform to the specifications established by the Committee on Analytical Reagents of the American Chemical Society, where such specifications are available; otherwise, use the best available grade.

7.1 Sample Collection. Same as Method 5, section 7.1.

7.2 Sample Recovery. Same as Method 5, section 7.2, with the addition of the following:

7.2.1 Water. Deionized distilled, to conform to ASTM D 1193–77 or 91 Type 3 (incorporated by reference—see §60.17). The potassium permanganate ($KMnO_4$) test for oxidizable organic matter may be omitted when high concentrations of organic matter are not expected to be present.

7.3 Analysis. Same as Method 5, section 7.3, with the addition of the following:

7.3.1 Water. Same as in section 7.2.1.

7.3.2 Stock Standard Solution, 1 mg $(NH_4)_2SO_4$/ml. Dry an adequate amount of primary standard grade ammonium sulfate ($(NH_4)_2SO_4$) at 105 to 110 °C (220 to 230 °F) for a minimum of 2 hours before preparing the standard solution. Then dissolve exactly 1.000 g of dried $(NH_4)_2SO_4$ in water in a 1-liter volumetric flask, and dilute to 1 liter. Mix well.

7.3.3 Working Standard Solution, 25 µg $(NH_4)_2SO_4$/ml. Pipet 5 ml of the stock standard solution into a 200-ml volumetric flask. Dilute to 200 ml with water.

7.3.4 Eluent Solution. Weigh 1.018 g of sodium carbonate (Na_2CO_3) and 1.008 g of sodium bicarbonate ($NaHCO_3$), and dissolve in 4 liters of water. This solution is 0.0024 M Na_2CO_3/0.003 M $NaHCO_3$. Other eluents appropriate to the column type and capable of resolving sulfate ion from other species present may be used.

7.3.5 Ammonium Hydroxide. Concentrated, 14.8 M.

7.3.6 Phenolphthalein Indicator. 3,3-Bis(4-hydroxyphenyl)-1-(3H)-isobenzo-furanone. Dissolve 0.05 g in 50 ml of ethanol and 50 ml of water.

8.0 Sample Collection, Preservation, Storage, and Transport

Same as Method 5, section 8.0, with the exception of the following:

8.1 Sampling Train Operation. Same as Method 5, section 8.5, except that the probe outlet and filter temperatures shall be maintained at 160 ±14 °C (320 ±25 °F).

8.2 Sample Recovery. Same as Method 5, section 8.7, except that the recovery solvent shall be water instead of acetone, and a clean filter from the same lot as those used during testing shall be saved for analysis as a blank.

9.0 Quality Control

9.1 Miscellaneous Quality Control Measures

Section	Quality control measure	Effect
8.3, 10.0	Sampling equipment leak check and calibration.	Ensures accurate measurement of stack gas flow rate, sample volume.
10.1.2, 11.2.5.3	Repetitive analyses	Ensures precise measurement of total carbon and inorganic carbon concentration of samples, blank, and standards.

9.2 Volume Metering System Checks. Same as Method 5, section 9.2.

10.0 Calibration and Standardization

Same as Method 5, section 10.0, with the addition of the following:

10.1 Determination of Ion Chromatograph Calibration Factor S. Prepare a series of five standards by adding 1.0, 2.0, 4.0, 6.0, and 10.0 ml of working standard solution (25 μg/ml) to a series of five 50-ml volumetric flasks. (The standard masses will equal 25, 50, 100, 150, and 250 μg.) Dilute each flask to the mark with water, and mix well. Analyze each standard according to the chromatograph manufacturer's instructions. Take peak height measurements with symmetrical peaks; in all other cases, calculate peak areas. Prepare or calculate a linear regression plot of the standard masses in μg (x-axis) versus their responses (y-axis). From this line, or equation, determine the slope and calculate its reciprocal which is the calibration factor, S. If any point deviates from the line by more than 7 percent of the concentration at that point, remake and reanalyze that standard. This deviation can be determined by multiplying S times the response for each standard. The resultant concentrations must not differ by more than 7 percent from each known standard mass (*i.e.*, 25, 50, 100, 150, and 250 μg).

10.2 Conductivity Detector. Calibrate according to manufacturer's specifications prior to initial use.

11.0 Analytical Procedure

11.1 Sample Extraction.

11.1.1 Note on the analytical data sheet, the level of the liquid in the container, and whether any sample was lost during shipment. If a noticeable amount of leakage has occurred, either void the sample or use methods, subject to the approval of the Administrator, to correct the final results.

11.1.2 Cut the filter into small pieces, and place it in a 125-ml Erlenmeyer flask with a ground glass joint equipped with an air condenser. Rinse the shipping container with water, and pour the rinse into the flask. Add additional water to the flask until it contains about 75 ml, and place the flask on a hot plate. Gently reflux the contents for 6 to 8 hours. Cool the solution, and transfer it to a 500-ml volumetric flask. Rinse the Erlenmeyer flask with water, and transfer the rinsings to the volumetric flask including the pieces of filter.

11.1.3 Transfer the probe rinse to the same 500-ml volumetric flask with the filter sample. Rinse the sample bottle with water, and add the rinsings to the volumetric flask. Dilute the contents of the flask to the mark with water.

11.1.4 Allow the contents of the flask to settle until all solid material is at the bottom of the flask. If necessary, remove and centrifuge a portion of the sample.

11.1.5 Repeat the procedures outlined in sections 11.1.1 through 11.1.4 for each sample and for the filter blank.

11.2 Sulfate (SO_4) Analysis.

11.2.1 Prepare a standard calibration curve according to the procedures outlined in section 10.1.

11.2.2 Pipet 5 ml of the sample into a 50-ml volumetric flask, and dilute to 50 ml with water. (Alternatively, eluent solution may be used instead of water in all sample, standard, and blank dilutions.) Analyze the set of standards followed by the set of samples, including the filter blank, using the same injection volume used for the standards.

11.2.3 Repeat the analyses of the standards and the samples, with the standard set being done last. The two peak height or peak area responses for each sample must agree within 5 percent of their arithmetic mean for the analysis to be valid. Perform this analysis sequence on the same day. Dilute any sample and the blank with equal volumes of water if the concentration exceeds that of the highest standard.

11.2.4 Document each sample chromatogram by listing the following analytical parameters: injection point, injection volume, sulfate retention time, flow rate, detector sensitivity setting, and recorder chart speed.

11.3 Sample Residue.

11.3.1 Transfer the remaining contents of the volumetric flask to a tared 600-ml beaker or similar container. Rinse the volumetric flask with water, and add the rinsings to the tared beaker. Make certain that all particulate matter is transferred to the beaker. Evaporate the water in an oven at 105 °C (220 °F) until only about 100 ml of water remains. Remove the beakers from the oven, and allow them to cool.

11.3.2 After the beakers have cooled, add five drops of phenolphthalein indicator, and then add concentrated ammonium hydroxide until the solution turns pink. Return the samples to the oven at 105 °C (220 °F), and evaporate the samples to dryness. Cool the samples in a desiccator, and weigh the samples to constant weight.

12.0 Data Analysis and Calculations

Same as Method 5, section 12.0, with the addition of the following:

12.1 Nomenclature.

C_W = Water blank residue concentration, mg/ml.

F = Dilution factor (required only if sample dilution was needed to reduce the concentration into the range of calibration).

H_S = Arithmetic mean response of duplicate sample analyses, mm for height or mm2 for area.

H_b = Arithmetic mean response of duplicate filter blank analyses, mm for height or mm2 for area.
m_b = Mass of beaker used to dry sample, mg.
m_f = Mass of sample filter, mg.
m_n = Mass of nonsulfate particulate matter in the sample as collected, mg.
m_s = Mass of ammonium sulfate in the sample as collected, mg.
m_t = Mass of beaker, filter, and dried sample, mg.
m_w = Mass of residue after evaporation of water blank, mg.
S = Calibration factor, µg/mm.
V_b = Volume of water blank, ml.
V_s = Volume of sample collected, 500 ml.

12.2 Water Blank Concentration.

$$C_w = \frac{m_w}{V_b} \qquad \text{Eq. 5F-1}$$

12.3 Mass of Ammonium Sulfate.

$$m_s = \frac{(99)\, S\, (H_s - H_b)}{(1000)} F \qquad \text{Eq. 5F-2}$$

Where:
100 = Aliquot factor, 495 ml/5 ml
1000 = Constant, µg/mg

12.4 Mass of Nonsulfate Particulate Matter.

$$m_n = m_t - m_b - m_s - m_f - V_s C_w \qquad \text{Eq. 5F-3}$$

13.0 Method Performance [Reserved]

14.0 Pollution Prevention [Reserved]

15.0 Waste Management [Reserved]

16.0 Alternative Procedures

16.1 The following procedure may be used as an alternative to the procedure in section 11.0

16.1.1 Apparatus. Same as for Method 6, sections 6.3.3 to 6.3.6 with the following additions.

16.1.1.1 Beakers. 250-ml, one for each sample, and 600-ml.

16.1.1.2 Oven. Capable of maintaining temperatures of 75 ±5 °C (167 ±9 °F) and 105 ±5 °C (221 ±9 °F).

16.1.1.3 Buchner Funnel.

16.1.1.4 Glass Columns. 25-mm × 305-mm (1-in. × 12-in.) with Teflon stopcock.

16.1.1.5 Volumetric Flasks. 50-ml and 500-ml, one set for each sample, and 100-ml, 200-ml, and 1000-ml.

16.1.1.6 Pipettes. Two 20-ml and one 200-ml, one set for each sample, and 5-ml.

16.1.1.7 Filter Flasks. 500-ml.

16.1.1.8 Polyethylene Bottle. 500-ml, one for each sample.

16.1.2 Reagents. Same as Method 6, sections 7.3.2 to 7.3.5 with the following additions:

16.1.2.1 Water, Ammonium Hydroxide, and Phenolphthalein. Same as sections 7.2.1, 7.3.5, and 7.3.6 of this method, respectively.

16.1.2.2 Filter. Glass fiber to fit Buchner funnel.

16.1.2.3 Hydrochloric Acid (HCl), 1 m. Add 8.3 ml of concentrated HCl (12 M) to 50 ml of water in a 100-ml volumetric flask. Dilute to 100 ml with water.

16.1.2.4 Glass Wool.

16.1.2.5 Ion Exchange Resin. Strong cation exchange resin, hydrogen form, analytical grade.

16.1.2.6 pH Paper. Range of 1 to 7.

16.1.3 Analysis.

16.1.3.1 Ion Exchange Column Preparation. Slurry the resin with 1 M HCl in a 250-ml beaker, and allow to stand overnight. Place 2.5 cm (1 in.) of glass wool in the bottom of the glass column. Rinse the slurried resin twice with water. Resuspend the resin in water, and pour sufficient resin into the column to make a bed 5.1 cm (2 in.) deep. Do not allow air bubbles to become entrapped in the resin or glass wool to avoid channeling, which may produce erratic results. If necessary, stir the resin with a glass rod to remove air bubbles, after the column has been prepared, never let the liquid level fall below the top of the upper glass wool plug. Place a 2.5-cm (1-in.) plug of glass wool on top of the resin. Rinse the column with water until the eluate gives a pH of 5 or greater as measured with pH paper.

16.1.3.2 Sample Extraction. Followup the procedure given in section 11.1.3 except do not dilute the sample to 500 ml.

16.1.3.3 Sample Residue.

16.1.3.3.1 Place at least one clean glass filter for each sample in a Buchner funnel, and rinse the filters with water. Remove the filters from the funnel, and dry them in an oven at 105 ±5 °C (221 ±9 °F); then cool in a desiccator. Weigh each filter to constant weight according to the procedure in Method 5, section 11.0. Record the weight of each filter to the nearest 0.1 mg.

16.1.3.3.2 Assemble the vacuum filter apparatus, and place one of the clean, tared glass fiber filters in the Buchner funnel. Decant the liquid portion of the extracted sample (Section 16.1.3.2) through the tared glass fiber filter into a clean, dry, 500-ml filter flask. Rinse all the particulate matter remaining in the volumetric flask onto the glass fiber filter with water. Rinse the particulate matter with additional water,

Transfer the filtrate to a 500-ml volumetric flask, and dilute to 500 ml with water. Dry the filter overnight at 105 ±5 °C (221 ±9 °F), cool in a desiccator, and weigh to the nearest 0.1 mg.

16.1.3.3.3 Dry a 250-ml beaker at 75 ±5 °C (167 ±9 °F), and cool in a desiccator; then weigh to constant weight to the nearest 0.1 mg. Pipette 200 ml of the filtrate that was saved into a tared 250-ml beaker; add five drops of phenolphthalein indicator and sufficient concentrated ammonium hydroxide to turn the solution pink. Carefully evaporate the contents of the beaker to dryness at 75 ±5 °C (167 ±9 °F). Check for dryness every 30 minutes. Do not continue to bake the sample once it has dried. Cool the sample in a desiccator, and weigh to constant weight to the nearest 0.1 mg.

16.1.3.4 Sulfate Analysis. Adjust the flow rate through the ion exchange column to 3 ml/min. Pipette a 20-ml aliquot of the filtrate onto the top of the ion exchange column, and collect the eluate in a 50-ml volumetric flask. Rinse the column with two 15-ml portions of water. Stop collection of the eluate when the volume in the flask reaches 50-ml. Pipette a 20-ml aliquot of the eluate into a 250-ml Erlenmeyer flask, add 80 ml of 100 percent isopropanol and two to four drops of thorin indicator, and titrate to a pink end point using 0.0100 N barium perchlorate. Repeat and average the titration volumes. Run a blank with each series of samples. Replicate titrations must agree within 1 percent or 0.2 ml, whichever is larger. Perform the ion exchange and titration procedures on duplicate portions of the filtrate. Results should agree within 5 percent. Regenerate or replace the ion exchange resin after 20 sample aliquots have been analyzed or if the end point of the titration becomes unclear.

NOTE: Protect the 0.0100 N barium perchlorate solution from evaporation at all times.

16.1.3.5 Blank Determination. Begin with a sample of water of the same volume as the samples being processed and carry it through the analysis steps described in sections 16.1.3.3 and 16.1.3.4. A blank value larger than 5 mg should not be subtracted from the final particulate matter mass. Causes for large blank values should be investigated and any problems resolved before proceeding with further analyses.

16.1.4 Calibration. Calibrate the barium perchlorate solutions as in Method 6, section 10.5.

16.1.5 Calculations.

16.1.5.1 Nomenclature. Same as section 12.1 with the following additions:

m_a = Mass of clean analytical filter, mg.
m_d = Mass of dissolved particulate matter, mg.
m_e = Mass of beaker and dissolved particulate matter after evaporation of filtrate, mg.
m_p = Mass of insoluble particulate matter, mg.
m_r = Mass of analytical filter, sample filter, and insoluble particulate matter, mg.
m_{bk} = Mass of nonsulfate particulate matter in blank sample, mg.
m_n = Mass of nonsulfate particulate matter, mg.
m_s = Mass of Ammonium sulfate, mg.
N = Normality of $Ba(ClO_4)$ titrant, meq/ml.
V_a = Volume of aliquot taken for titration, 20 ml.
V_c = Volume of titrant used for titration blank, ml.
V_d = Volume of filtrate evaporated, 200 ml.
V_e = Volume of eluate collected, 50 ml.
V_f = Volume of extracted sample, 500 ml.
V_i = Volume of filtrate added to ion exchange column, 20 ml.
V_t = Volume of $Ba(C10_4)_2$ titrant, ml.
W = Equivalent weight of ammonium sulfate, 66.07 mg/meq.

16.1.5.2 Mass of Insoluble Particulate Matter.

$$m_p = m_r - m_a - m_f \qquad \text{Eq. 5F-4}$$

16.1.5.3 Mass of Dissolved Particulate Matter.

$$m_d = \left(m_e - \left(V_f / V_d\right) m_b\right) \qquad \text{Eq. 5F-5}$$

16.1.5.4 Mass of Ammonium Sulfate.

$$m_s = \frac{\left(V_t - V_c\right) N\, W\, V_e V_f}{V_a V_i} \qquad \text{Eq. 5F-6}$$

16.1.5.5 Mass of Nonsulfate Particulate Matter.

$$m_n = m_p + m_d - m_s - m_{bk} \qquad \text{Eq. 5F-7}$$

17.0 References

Same as Method 5, section 17.0, with the addition of the following:

1. Mulik, J.D. and E. Sawicki. Ion Chromatographic Analysis of Environmental Pollutants. Ann Arbor, Ann Arbor Science Publishers, Inc. Vol. 2, 1979.

2. Sawicki, E., J.D. Mulik, and E. Wittgenstein. Ion Chromatographic Analysis of Environmental Pollutants. Ann Arbor, Ann Arbor Science Publishers, Inc. Vol. 1. 1978.

3. Siemer, D.D. Separation of Chloride and Bromide from Complex Matrices Prior to Ion Chromatographic Determination. Analytical Chemistry 52(12): 1874–1877. October 1980.

4. Small, H., T.S. Stevens, and W.C. Bauman. Novel Ion Exchange Chromatographic Method Using

Conductimetric Determination. Analytical Chemistry. 47(11):1801. 1975.

18.0 Tables, Diagrams, Flowcharts, and Validation Data [Reserved]

METHOD 5G—DETERMINATION OF PARTICULATE MATTER EMISSIONS FROM WOOD HEATERS (DILUTION TUNNEL SAMPLING LOCATION)

NOTE: This method does not include all of the specifications (*e.g.*, equipment and supplies) and procedures (*e.g.*, sampling and analytical) essential to its performance. Some material is incorporated by reference from other methods in this part. Therefore, to obtain reliable results, persons using this method should have a thorough knowledge of at least the following additional test methods: Method 1, Method 2, Method 3, Method 4, Method 5, Method 5H, and Method 28.

1.0 Scope and Application

1.1 Analyte. Particulate matter (PM). No CAS number assigned.

1.2 Applicability. This method is applicable for the determination of PM emissions from wood heaters.

1.3 Data Quality Objectives. Adherence to the requirements of this method will enhance the quality of the data obtained from air pollutant sampling methods.

2.0 Summary of Method

2.1 The exhaust from a wood heater is collected with a total collection hood, and is combined with ambient dilution air. Particulate matter is withdrawn proportionally from a single point in a sampling tunnel, and is collected on two glass fiber filters in series. The filters are maintained at a temperature of no greater than 32 °C (90 °F). The particulate mass is determined gravimetrically after the removal of uncombined water.

2.2 There are three sampling train approaches described in this method: (1) One dual-filter dry sampling train operated at about 0.015 m^3/min (0.5 cfm), (2) One dual-filter plus impingers sampling train operated at about 0.015 m^3/min (0.5 cfm), and (3) two dual-filter dry sampling trains operated simultaneously at any flow rate. Options (2) and (3) are referenced in section 16.0 of this method. The dual-filter dry sampling train equipment and operation, option (1), are described in detail in this method.

3.0 Definitions [Reserved]

4.0 Interferences [Reserved]

5.0 Safety

5.1 Disclaimer. This method may involve hazardous materials, operations, and equipment. This test method may not address all of the safety problems associated with its use. It is the responsibility of the user of this test method to establish appropriate safety and health practices and to determine the applicability of regulatory limitations prior to performing this test method.

6.0 Equipment and Supplies

6.1 Sample Collection. The following items are required for sample collection:

6.1.1 Sampling Train. The sampling train configuration is shown in Figure 5G–1 and consists of the following components:

6.1.1.1 Probe. Stainless steel (*e.g.*, 316 or grade more corrosion resistant) or glass about 9.5 mm (3/8 in.) I.D., 0.6 m (24 in.) in length. If made of stainless steel, the probe shall be constructed from seamless tubing.

6.1.1.2 Pitot Tube. Type S, as described in section 6.1 of Method 2. The Type S pitot tube assembly shall have a known coefficient, determined as outlined in Method 2, section 10. Alternatively, a standard pitot may be used as described in Method 2, section 6.1.2.

6.1.1.3 Differential Pressure Gauge. Inclined manometer or equivalent device, as described in Method 2, section 6.2. One manometer shall be used for velocity head (Δp) readings and another (optional) for orifice differential pressure readings (ΔH).

6.1.1.4 Filter Holders. Two each made of borosilicate glass, stainless steel, or Teflon, with a glass frit or stainless steel filter support and a silicone rubber, Teflon, or Viton gasket. The holder design shall provide a positive seal against leakage from the outside or around the filters. The filter holders shall be placed in series with the backup filter holder located 25 to 100 mm (1 to 4 in.) downstream from the primary filter holder. The filter holder shall be capable of holding a filter with a 100 mm (4 in.) diameter, except as noted in section 16.

6.1.1.5 Filter Temperature Monitoring System. A temperature sensor capable of measuring temperature to within ±3 °C (±5 °F). The sensor shall be installed at the exit side of the front filter holder so that the sensing tip of the temperature sensor is in direct contact with the sample gas or in a thermowell as shown in Figure 5G–1. The temperature sensor shall comply with the calibration specifications in Method 2, section 10.3. Alternatively, the sensing tip of the temperature sensor may be installed at the inlet side of the front filter holder.

6.1.1.6 Dryer. Any system capable of removing water from the sample gas to less than 1.5 percent moisture (volume percent) prior to the metering system. The system shall include a temperature sensor for demonstrating that sample gas temperature exiting the dryer is less than 20 °C (68 °F).

6.1.1.7 Metering System. Same as Method 5, section 6.1.1.9.

6.1.2 Barometer. Same as Method 5, section 6.1.2.

6.1.3 Dilution Tunnel Gas Temperature Measurement. A temperature sensor capable

of measuring temperature to within ±3 °C (±5 °F).

6.1.4 Dilution Tunnel. The dilution tunnel apparatus is shown in Figure 5G–2 and consists of the following components:

6.1.4.1 Hood. Constructed of steel with a minimum diameter of 0.3 m (1 ft) on the large end and a standard 0.15 to 0.3 m (0.5 to 1 ft) coupling capable of connecting to standard 0.15 to 0.3 m (0.5 to 1 ft) stove pipe on the small end.

6.1.4.2 90° Elbows. Steel 90° elbows, 0.15 to 0.3 m (0.5 to 1 ft) in diameter for connecting mixing duct, straight duct and optional damper assembly. There shall be at least two 90° elbows upstream of the sampling section (see Figure 5G–2).

6.1.4.3 Straight Duct. Steel, 0.15 to 0.3 m (0.5 to 1 ft) in diameter to provide the ducting for the dilution apparatus upstream of the sampling section. Steel duct, 0.15 m (0.5 ft) in diameter shall be used for the sampling section. In the sampling section, at least 1.2 m (4 ft) downstream of the elbow, shall be two holes (velocity traverse ports) at 90° to each other of sufficient size to allow entry of the pitot for traverse measurements. At least 1.2 m (4 ft) downstream of the velocity traverse ports, shall be one hole (sampling port) of sufficient size to allow entry of the sampling probe. Ducts of larger diameter may be used for the sampling section, provided the specifications for minimum gas velocity and the dilution rate range shown in section 8 are maintained. The length of duct from the hood inlet to the sampling ports shall not exceed 9.1 m (30 ft).

6.1.4.4 Mixing Baffles. Steel semicircles (two) attached at 90° to the duct axis on opposite sides of the duct midway between the two elbows upstream of sampling section. The space between the baffles shall be about 0.3 m (1 ft).

6.1.4.5 Blower. Squirrel cage or other fan capable of extracting gas from the dilution tunnel of sufficient flow to maintain the velocity and dilution rate specifications in section 8 and exhausting the gas to the atmosphere.

6.2 Sample Recovery. The following items are required for sample recovery: probe brushes, wash bottles, sample storage containers, petri dishes, and funnel. Same as Method 5, sections 6.2.1 through 6.2.4, and 6.2.8, respectively.

6.3 Sample Analysis. The following items are required for sample analysis: glass weighing dishes, desiccator, analytical balance, beakers (250-ml or smaller), hygrometer, and temperature sensor. Same as Method 5, sections 6.3.1 through 6.3.3 and 6.3.5 through 6.3.7, respectively.

7.0 Reagents and Standards

7.1 Sample Collection. The following reagents are required for sample collection:

7.1.1 Filters. Glass fiber filters with a minimum diameter of 100 mm (4 in.), without organic binder, exhibiting at least 99.95 percent efficiency (<0.05 percent penetration) on 0.3-micron dioctyl phthalate smoke particles. Gelman A/E 61631 has been found acceptable for this purpose.

7.1.2 Stopcock Grease. Same as Method 5, section 7.1.5. 7.2 Sample Recovery. Acetone-reagent grade, same as Method 5, section 7.2.

7.3 Sample Analysis. Two reagents are required for the sample analysis:

7.3.1 Acetone. Same as in section 7.2.

7.3.2 Desiccant. Anhydrous calcium sulfate, calcium chloride, or silica gel, indicating type.

8.0 Sample Collection, Preservation, Transport, and Storage

8.1 Dilution Tunnel Assembly and Cleaning. A schematic of a dilution tunnel is shown in Figure 5G–2. The dilution tunnel dimensions and other features are described in section 6.1.4. Assemble the dilution tunnel, sealing joints and seams to prevent air leakage. Clean the dilution tunnel with an appropriately sized wire chimney brush before each certification test.

8.2 Draft Determination. Prepare the wood heater as in Method 28, section 6.2.1. Locate the dilution tunnel hood centrally over the wood heater stack exhaust. Operate the dilution tunnel blower at the flow rate to be used during the test run. Measure the draft imposed on the wood heater by the dilution tunnel (*i.e.*, the difference in draft measured with and without the dilution tunnel operating) as described in Method 28, section 6.2.3. Adjust the distance between the top of the wood heater stack exhaust and the dilution tunnel hood so that the dilution tunnel induced draft is less than 1.25 Pa (0.005 in. H_2O). Have no fire in the wood heater, close the wood heater doors, and open fully the air supply controls during this check and adjustment.

8.3 Pretest Ignition. Same as Method 28, section 8.7.

8.4 Smoke Capture. During the pretest ignition period, operate the dilution tunnel and visually monitor the wood heater stack exhaust. Operate the wood heater with the doors closed and determine that 100 percent of the exhaust gas is collected by the dilution tunnel hood. If less than 100 percent of the wood heater exhaust gas is collected, adjust the distance between the wood heater stack and the dilution tunnel hood until no visible exhaust gas is escaping. Stop the pretest ignition period, and repeat the draft determination procedure described in section 8.2.

8.5 Velocity Measurements. During the pretest ignition period, conduct a velocity traverse to identify the point of average velocity. This single point shall be used for measuring velocity during the test run.

8.5.1 Velocity Traverse. Measure the diameter of the duct at the velocity traverse port location through both ports. Calculate the duct area using the average of the two diameters. A pretest leak-check of pitot lines as in Method 2, section 8.1, is recommended. Place the calibrated pitot tube at the centroid of the stack in either of the velocity traverse ports. Adjust the damper or similar device on the blower inlet until the velocity indicated by the pitot is approximately 220 m/min (720 ft/min). Continue to read the Δp and temperature until the velocity has remained constant (less than 5 percent change) for 1 minute. Once a constant velocity is obtained at the centroid of the duct, perform a velocity traverse as outlined in Method 2, section 8.3 using four points per traverse as outlined in Method 1. Measure the Δp and tunnel temperature at each traverse point and record the readings. Calculate the total gas flow rate using calculations contained in Method 2, section 12. Verify that the flow rate is 4 ±0.40 dscm/min (140 ±14 dscf/min); if not, readjust the damper, and repeat the velocity traverse. The moisture may be assumed to be 4 percent (100 percent relative humidity at 85 °F). Direct moisture measurements (*e.g.*, according to Method 4) are also permissible.

NOTE: If burn rates exceed 3 kg/hr (6.6 lb/hr), dilution tunnel duct flow rates greater than 4 dscm/min (140 dscfm) and sampling section duct diameters larger than 150 mm (6 in.) are allowed. If larger ducts or flow rates are used, the sampling section velocity shall be at least 220 m/min (720 fpm). In order to ensure measurable particulate mass catch, it is recommended that the ratio of the average mass flow rate in the dilution tunnel to the average fuel burn rate be less than 150:1 if larger duct sizes or flow rates are used.

8.5.2 Testing Velocity Measurements. After obtaining velocity traverse results that meet the flow rate requirements, choose a point of average velocity and place the pitot and temperature sensor at that location in the duct. Alternatively, locate the pitot and the temperature sensor at the duct centroid and calculate a velocity correction factor for the centroidal position. Mount the pitot to ensure no movement during the test run and seal the port holes to prevent any air leakage. Align the pitot opening to be parallel with the duct axis at the measurement point. Check that this condition is maintained during the test run (about 30-minute intervals). Monitor the temperature and velocity during the pretest ignition period to ensure that the proper flow rate is maintained. Make adjustments to the dilution tunnel flow rate as necessary.

8.6 Pretest Preparation. Same as Method 5, section 8.1.

8.7 Preparation of Sampling Train. During preparation and assembly of the sampling train, keep all openings where contamination can occur covered until just prior to assembly or until sampling is about to begin.

Using a tweezer or clean disposable surgical gloves, place one labeled (identified) and weighed filter in each of the filter holders. Be sure that each filter is properly centered and that the gasket is properly placed so as to prevent the sample gas stream from circumventing the filter. Check each filter for tears after assembly is completed.

Mark the probe with heat resistant tape or by some other method to denote the proper distance into the stack or duct. Set up the train as shown in Figure 5G–1.

8.8 Leak-Check Procedures.

8.8.1 Leak-Check of Metering System Shown in Figure 5G–1. That portion of the sampling train from the pump to the orifice meter shall be leak-checked prior to initial use and after each certification or audit test. Leakage after the pump will result in less volume being recorded than is actually sampled. Use the procedure described in Method 5, section 8.4.1. Similar leak-checks shall be conducted for other types of metering systems (*i.e.*, without orifice meters).

8.8.2 Pretest Leak-Check. A pretest leak-check of the sampling train is recommended, but not required. If the pretest leak check is conducted, the procedures outlined in Method 5, section 8.4.2 should be used. A vacuum of 130 mm Hg (5 in. Hg) may be used instead of 380 mm Hg (15 in. Hg).

8.8.3 Post-Test Leak-Check. A leak-check of the sampling train is mandatory at the conclusion of each test run. The leak-check shall be performed in accordance with the procedures outlined in Method 5, section 8.4.2. A vacuum of 130 mm Hg (5 in. Hg) or the highest vacuum measured during the test run, whichever is greater, may be used instead of 380 mm Hg (15 in. Hg).

8.9 Preliminary Determinations. Determine the pressure, temperature and the average velocity of the tunnel gases as in section 8.5. Moisture content of diluted tunnel gases is assumed to be 4 percent for making flow rate calculations; the moisture content may be measured directly as in Method 4.

8.10 Sampling Train Operation. Position the probe inlet at the stack centroid, and block off the openings around the probe and porthole to prevent unrepresentative dilution of the gas stream. Be careful not to bump the probe into the stack wall when removing or inserting the probe through the porthole; this minimizes the chance of extracting deposited material.

8.10.1 Begin sampling at the start of the test run as defined in Method 28, section 8.8.1. During the test run, maintain a sample flow rate proportional to the dilution tunnel flow rate (within 10 percent of the initial proportionality ratio) and a filter holder temperature of no greater than 32 °C (90 °F).

The initial sample flow rate shall be approximately 0.015 m^3/min (0.5 cfm).

8.10.2 For each test run, record the data required on a data sheet such as the one shown in Figure 5G–3. Be sure to record the initial dry gas meter reading. Record the dry gas meter readings at the beginning and end of each sampling time increment and when sampling is halted. Take other readings as indicated on Figure 5G–3 at least once each 10 minutes during the test run. Since the manometer level and zero may drift because of vibrations and temperature changes, make periodic checks during the test run.

8.10.3 For the purposes of proportional sampling rate determinations, data from calibrated flow rate devices, such as glass rotameters, may be used in lieu of incremental dry gas meter readings. Proportional rate calculation procedures must be revised, but acceptability limits remain the same.

8.10.4 During the test run, make periodic adjustments to keep the temperature between (or upstream of) the filters at the proper level. Do not change sampling trains during the test run.

8.10.5 At the end of the test run (see Method 28, section 6.4.6), turn off the coarse adjust valve, remove the probe from the stack, turn off the pump, record the final dry gas meter reading, and conduct a post-test leak-check, as outlined in section 8.8.2. Also, leak-check the pitot lines as described in Method 2, section 8.1; the lines must pass this leak-check in order to validate the velocity head data.

8.11 Calculation of Proportional Sampling Rate. Calculate percent proportionality (see section 12.7) to determine whether the run was valid or another test run should be made.

8.12 Sample Recovery. Same as Method 5, section 8.7, with the exception of the following:

8.12.1 An acetone blank volume of about 50-ml or more may be used.

8.12.2 Treat the samples as follows:

8.12.2.1 Container Nos. 1 and 1A. Treat the two filters according to the procedures outlined in Method 5, section 8.7.6.1. The filters may be stored either in a single container or in separate containers. Use the sum of the filter tare weights to determine the sample mass collected.

8.12.2.3 Container No. 2.

8.12.2.3.1 Taking care to see that dust on the outside of the probe or other exterior surfaces does not get into the sample, quantitatively recover particulate matter or any condensate from the probe and filter holders by washing and brushing these components with acetone and placing the wash in a labeled glass container. At least three cycles of brushing and rinsing are required.

8.12.2.3.2 Between sampling runs, keep brushes clean and protected from contamination.

8.12.2.3.3 After all acetone washings and particulate matter have been collected in the sample containers, tighten the lids on the sample containers so that the acetone will not leak out when transferred to the laboratory weighing area. Mark the height of the fluid levels to determine whether leakage occurs during transport. Label the containers clearly to identify contents.

8.13 Sample Transport. Whenever possible, containers should be shipped in such a way that they remain upright at all times.

NOTE: Requirements for capping and transport of sample containers are not applicable if sample recovery and analysis occur in the same room.

9.0 Quality Control

9.1 Miscellaneous Quality Control Measures.

Section	Quality control measure	Effect
8.8, 10.1–10.4	Sampling equipment leak check and calibration.	Ensures accurate measurement of stack gas flow rate, sample volume.
10.5	Analytical balance calibration	Ensure accurate and precise measurement of collected particulate.
16.2.5	Simultaneous, dual-train sample collection.	Ensure precision of measured particulate concentration.

9.2 Volume Metering System Checks. Same as Method 5, section 9.2.

10.0 Calibration and Standardization

NOTE: Maintain a laboratory record of all calibrations.

10.1 Pitot Tube. The Type S pitot tube assembly shall be calibrated according to the procedure outlined in Method 2, section 10.1, prior to the first certification test and checked semiannually, thereafter. A standard pitot need not be calibrated but shall be inspected and cleaned, if necessary, prior to each certification test.

10.2 Volume Metering System.

10.2.1 Initial and Periodic Calibration. Before its initial use and at least semiannually thereafter, calibrate the volume metering system as described in Method 5, section 10.3.1, except that the wet test meter with a capacity of 3.0 liters/rev (0.1 ft^3/rev) may be used. Other liquid displacement systems accurate to within ±1 percent, may be used as calibration standards.

NOTE: Procedures and equipment specified in Method 5, section 16.0, for alternative calibration standards, including calibrated dry gas meters and critical orifices, are allowed for calibrating the dry gas meter in the sampling train. A dry gas meter used as a calibration standard shall be recalibrated at least once annually.

10.2.2 Calibration After Use. After each certification or audit test (four or more test runs conducted on a wood heater at the four burn rates specified in Method 28), check calibration of the metering system by performing three calibration runs at a single, intermediate flow rate as described in Method 5, section 10.3.2.

NOTE: Procedures and equipment specified in Method 5, section 16.0, for alternative calibration standards are allowed for the post-test dry gas meter calibration check.

10.2.3 Acceptable Variation in Calibration. If the dry gas meter coefficient values obtained before and after a certification test differ by more than 5 percent, the certification test shall either be voided and repeated, or calculations for the certification test shall be performed using whichever meter coefficient value (*i.e.*, before or after) gives the lower value of total sample volume.

10.3 Temperature Sensors. Use the procedure in Method 2, section 10.3, to calibrate temperature sensors before the first certification or audit test and at least semiannually, thereafter.

10.4 Barometer. Calibrate against a mercury barometer before the first certification test and at least semiannually, thereafter. If a mercury barometer is used, no calibration is necessary. Follow the manufacturer's instructions for operation.

10.5 Analytical Balance. Perform a multipoint calibration (at least five points spanning the operational range) of the analytical balance before the first certification test and semiannually, thereafter. Before each certification test, audit the balance by weighing at least one calibration weight (class F) that corresponds to 50 to 150 percent of the weight of one filter. If the scale cannot reproduce the value of the calibration weight to within 0.1 mg, conduct the multipoint calibration before use.

11.0 Analytical Procedure

11.1 Record the data required on a sheet such as the one shown in Figure 5G–4. Use the same analytical balance for determining tare weights and final sample weights.

11.2 Handle each sample container as follows:

11.2.1 Container Nos. 1 and 1A. Treat the two filters according to the procedures outlined in Method 5, section 11.2.1.

11.2.2 Container No. 2. Same as Method 5, section 11.2.2, except that the beaker may be smaller than 250 ml.

11.2.3 Acetone Blank Container. Same as Method 5, section 11.2.4, except that the beaker may be smaller than 250 ml.

12.0 Data Analysis and Calculations

Carry out calculations, retaining at least one extra significant figure beyond that of the acquired data. Round off figures after the final calculation. Other forms of the equations may be used as long as they give equivalent results.

12.1 Nomenclature.

B_{ws} = Water vapor in the gas stream, proportion by volume (assumed to be 0.04).

c_s = Concentration of particulate matter in stack gas, dry basis, corrected to standard conditions, g/dscm (gr/dscf).

E = Particulate emission rate, g/hr (lb/hr).

E_{adj} = Adjusted particulate emission rate, g/hr (lb/hr).

L_a = Maximum acceptable leakage rate for either a pretest or post-test leak-check, equal to 0.00057 m^3/min (0.020 cfm) or 4 percent of the average sampling rate, whichever is less.

L_p = Leakage rate observed during the post-test leak-check, m^3/min (cfm).

m_a = Mass of residue of acetone blank after evaporation, mg.

m_{aw} = Mass of residue from acetone wash after evaporation, mg.

m_n = Total amount of particulate matter collected, mg.

M_w = Molecular weight of water, 18.0 g/g-mole (18.0 lb/lb-mole).

P_{bar} = Barometric pressure at the sampling site, mm Hg (in. Hg).

PR = Percent of proportional sampling rate.

P_s = Absolute gas pressure in dilution tunnel, mm Hg (in. Hg).

P_{std} = Standard absolute pressure, 760 mm Hg (29.92 in. Hg).

Q_{sd} = Average gas flow rate in dilution tunnel, calculated as in Method 2, Equation 2–8, dscm/hr (dscf/hr).

T_m = Absolute average dry gas meter temperature (see Figure 5G–3), °K (°R).

T_{mi} = Absolute average dry gas meter temperature during each 10-minute interval, i, of the test run, °K (°R).

T_s = Absolute average gas temperature in the dilution tunnel (see Figure 5G–3), °K (°R).

T_{si} = Absolute average gas temperature in the dilution tunnel during each 10 minute interval, i, of the test run, °K (°R).

T_{std} = Standard absolute temperature, 293 °K (528 °R).

V_a = Volume of acetone blank, ml.

V_{aw} = Volume of acetone used in wash, ml.

V_m = Volume of gas sample as measured by dry gas meter, dcm (dcf).

V_{mi} = Volume of gas sample as measured by dry gas meter during each 10-minute interval, i, of the test run, dcm.

$V_{m(std)}$ = Volume of gas sample measured by the dry gas meter, corrected to standard conditions, dscm (dscf).

V_s = Average gas velocity in the dilution tunnel, calculated by Method 2, Equation 2–7, m/sec (ft/sec). The dilution tunnel dry gas molecular weight may be assumed to be 29 g/g mole (lb/lb mole).

V_{si} = Average gas velocity in dilution tunnel during each 10-minute interval, i, of the test run, calculated by Method 2, Equation 2–7, m/sec (ft/sec).

Y = Dry gas meter calibration factor.

ΔH = Average pressure differential across the orifice meter, if used (see Figure 5G–2), mm H^2O (in. H^2O).

U = Total sampling time, min.

10 = 10 minutes, length of first sampling period.

13.6 = Specific gravity of mercury.

100 = Conversion to percent.

12.2 Dry Gas Volume. Same as Method 5, section 12.2, except that component changes are not allowable.

12.3 Solvent Wash Blank.

$$m_{aw} = \frac{m_a V_{aw}}{V_a} \qquad \text{Eq. 5G-1}$$

12.4 Total Particulate Weight. Determine the total particulate catch, mn, from the sum of the weights obtained from Container Nos. 1, 1A, and 2, less the acetone blank (see Figure 5G–4).

12.5 Particulate Concentration.

$$c_s = K_2 \frac{m_n}{V_{m(std)}} \qquad \text{Eq. 5G-2}$$

Where:

K_2 = 0.001 g/mg for metric units.

= 0.0154 gr/mg for English units.

12.6 Particulate Emission Rate.

$$E = C_s Q_{sd} \qquad \text{Eq. 5G-3}$$

NOTE: Particulate emission rate results produced using the sampling train described in section 6 and shown in Figure 5G–1 shall be adjusted for reporting purposes by the following method adjustment factor:

$$E_{adj} = K_3 E^{0.83} \qquad \text{Eq. 5G-4}$$

Where:

K_3 = constant, 1.82 for metric units.

= constant, 0.643 for English units.

12.7 Proportional Rate Variation. Calculate PR for each 10-minute interval, i, of the test run.

$$PR = \left(\frac{\theta \left(V_{mi} V_s T_m T_{si} \right)}{10 \left(V_m V_{si} T_s T_{mi} \right)} \right) \times 100 \qquad \text{Eq. 5G-5}$$

Alternate calculation procedures for proportional rate variation may be used if other sample flow rate data (e.g., orifice flow meters or rotameters) are monitored to maintain proportional sampling rates. The proportional rate variations shall be calculated for each 10-minute interval by comparing the stack to nozzle velocity ratio for each 10-minute interval to the average stack to nozzle velocity ratio for the test run. Proportional rate variation may be calculated for intervals shorter than 10 minutes with appropriate revisions to Equation 5G–5. If no more than 10 percent of the PR values for all the intervals exceed 90 percent ≤PR ≤110 percent, and if no PR value for any interval exceeds 80 percent ≤PR ≤120 percent, the results are acceptable. If the PR values for the test run are judged to be unacceptable, report the test run emission results, but do not include the results in calculating the weighted average emission rate, and repeat the test run.

13.0 Method Performance [Reserved]

14.0 Pollution Prevention [Reserved]

15.0 Waste Management [Reserved]

16.0 Alternative Procedures

16.1 Method 5H Sampling Train. The sampling train and sample collection, recovery, and analysis procedures described in Method 5H, sections 6.1.1, 7.1, 7.2, 8.1, 8.10, 8.11, and 11.0, respectively, may be used in lieu of similar sections in Method 5G. Operation of the Method 5H sampling train in the dilution tunnel is as described in section 8.10 of this method. Filter temperatures and condenser conditions are as described in Method 5H. No adjustment to the measured particulate matter emission rate (Equation 5G–4, section 12.6) is to be applied to the particulate emission rate measured by this alternative method.

16.2 Dual Sampling Trains. Two sampling trains may be operated simultaneously at sample flow rates other than that specified

in section 8.10, provided that the following specifications are met.

16.2.1 Sampling Train. The sampling train configuration shall be the same as specified in section 6.1.1, except the probe, filter, and filter holder need not be the same sizes as specified in the applicable sections. Filter holders of plastic materials such as Nalgene or polycarbonate materials may be used (the Gelman 1119 filter holder has been found suitable for this purpose). With such materials, it is recommended that solvents not be used in sample recovery. The filter face velocity shall not exceed 150 mm/sec (30 ft/min) during the test run. The dry gas meter shall be calibrated for the same flow rate range as encountered during the test runs. Two separate, complete sampling trains are required for each test run.

16.2.2 Probe Location. Locate the two probes in the dilution tunnel at the same level (see section 6.1.4.3). Two sample ports are necessary. Locate the probe inlets within the 50 mm (2 in.) diameter centroidal area of the dilution tunnel no closer than 25 mm (1 in.) apart.

16.2.3 Sampling Train Operation. Operate the sampling trains as specified in section 8.10, maintaining proportional sampling rates and starting and stopping the two sampling trains simultaneously. The pitot values as described in section 8.5.2 shall be used to adjust sampling rates in both sampling trains.

16.2.4 Recovery and Analysis of Sample. Recover and analyze the samples from the two sampling trains separately, as specified in sections 8.12 and 11.0, respectively.

16.2.4.1 For this alternative procedure, the probe and filter holder assembly may be weighed without sample recovery (use no solvents) described above in order to determine the sample weight gains. For this approach, weigh the clean, dry probe and filter holder assembly upstream of the front filter (without filters) to the nearest 0.1 mg to establish the tare weights. The filter holder section between the front and second filter need not be weighed. At the end of the test run, carefully clean the outside of the probe, cap the ends, and identify the sample (label). Remove the filters from the filter holder assemblies as described for container Nos. 1 and 1A in section 8.12.2.1. Reassemble the filter holder assembly, cap the ends, identify the sample (label), and transfer all the samples to the laboratory weighing area for final weighing. Requirements for capping and transport of sample containers are not applicable if sample recovery and analysis occur in the same room.

16.2.4.2 For this alternative procedure, filters may be weighed directly without a petri dish. If the probe and filter holder assembly are to be weighed to determine the sample weight, rinse the probe with acetone to remove moisture before desiccating prior to the test run. Following the test run, transport the probe and filter holder to the desiccator, and uncap the openings of the probe and the filter holder assembly. Desiccate for 24 hours and weigh to a constant weight. Report the results to the nearest 0.1 mg.

16.2.5 Calculations. Calculate an emission rate (Section 12.6) for the sample from each sampling train separately and determine the average emission rate for the two values. The two emission rates shall not differ by more than 7.5 percent from the average emission rate, or 7.5 percent of the weighted average emission rate limit in the applicable subpart of the regulations, whichever is greater. If this specification is not met, the results are unacceptable. Report the results, but do not include the results in calculating the weighted average emission rate. Repeat the test run until acceptable results are achieved, report the average emission rate for the acceptable test run, and use the average in calculating the weighted average emission rate.

17.0 References

Same as Method 5, section 17.0, References 1 through 11, with the addition of the following:

1. Oregon Department of Environmental Quality. Standard Method for Measuring the Emissions and Efficiencies of Woodstoves. June 8, 1984. Pursuant to Oregon Administrative Rules Chapter 340, Division 21.

2. American Society for Testing and Materials. Proposed Test Methods for Heating Performance and Emissions of Residential Wood-fired Closed Combustion-Chamber Heating Appliances. E–6 Proposal P 180. August 1986.

18.0 Tables, Diagrams, Flowcharts, and Validation Data

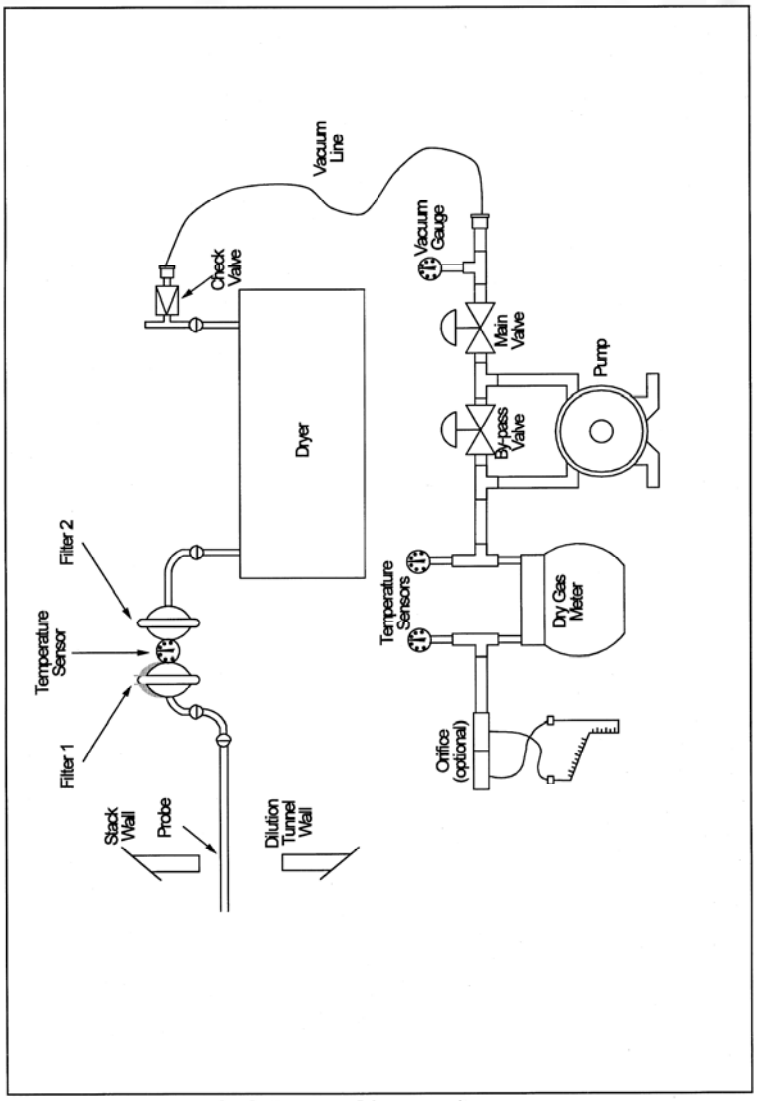


Figure 5G-1. Method 5G Sampling Train.

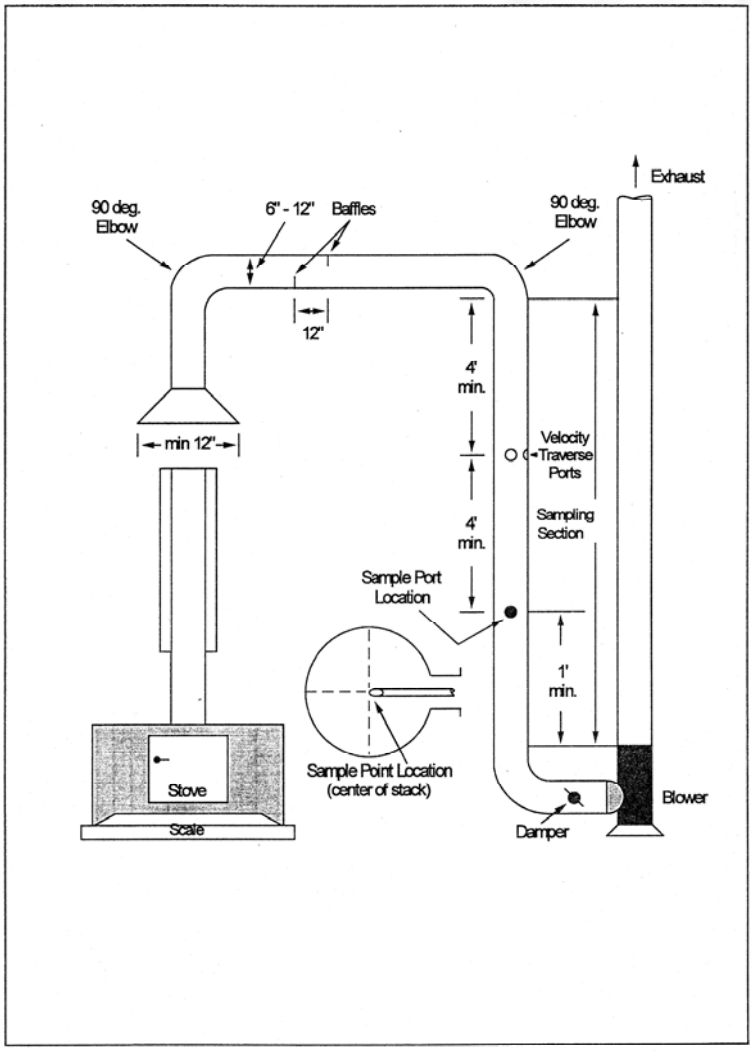
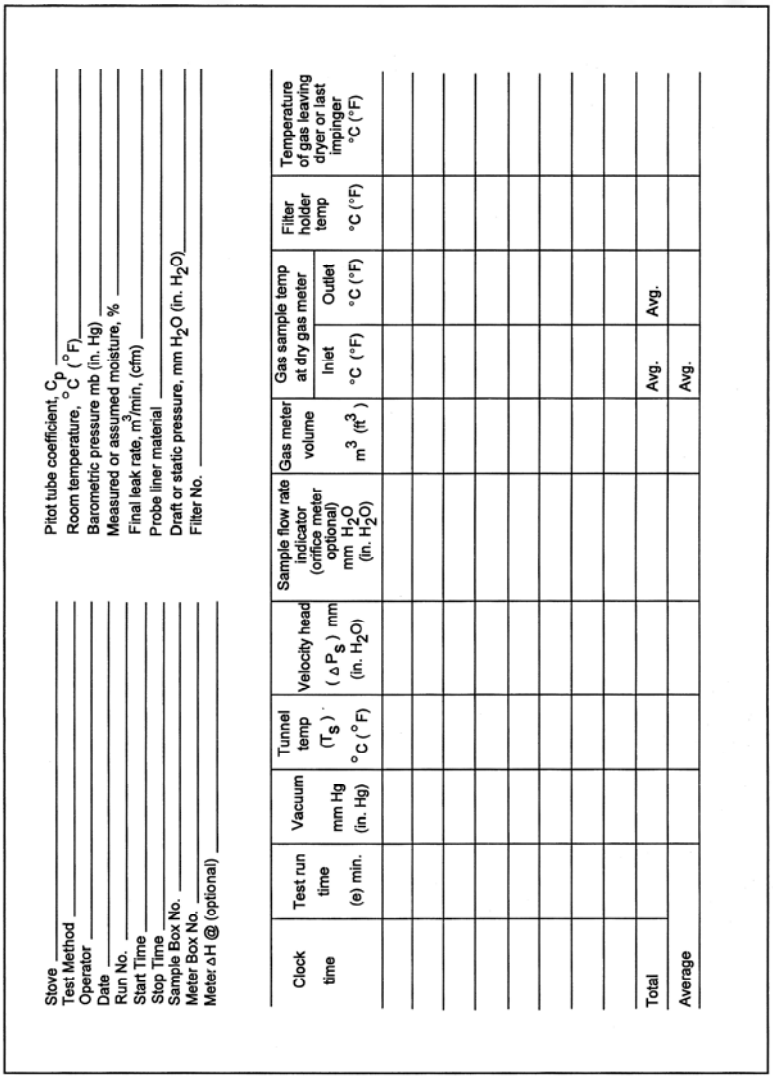


Figure 5G-2. Suggested Construction Details of the Dilution Tunnel.



Stove _____
Test Method _____
Operator _____
Date _____
Run No. _____
Start Time _____
Stop Time _____
Sample Box No. _____
Meter Box No. _____
Meter ΔH @ (optional) _____

Pitot tube coefficient, C_p _____
Room temperature, °C (°F) _____
Barometric pressure mb (in. Hg) _____
Measured or assumed moisture, % _____
Final leak rate, m^3/min, (cfm) _____
Probe liner material _____
Draft or static pressure, mm H_2O (in. H_2O) _____
Filter No. _____

Clock time	Test run time (e) min.	Vacuum mm Hg (in. Hg)	Tunnel temp (T_s) °C (°F)	Velocity head (ΔP_s) mm (in. H_2O)	Sample flow rate indicator (orifice meter optional) mm H_2O (in. H_2O)	Gas meter volume m^3 (ft^3)	Gas sample temp at dry gas meter		Filter holder temp °C (°F)	Temperature of gas leaving dryer or last impinger °C (°F)
							Inlet °C (°F)	Outlet °C (°F)		
Total							Avg.	Avg.		
Average							Avg.			

Figure 5G-3. Sampling Data Sheet.

Stove _____
Date _____
Run No. _____
Filter Nos. _____
Liquid lost during transport, ml _____
Acetone blank volume, ml _____
Acetone wash volume, ml _____
Acetone blank concentration, mg/mg _____
Acetone wash blank, mg _____

Container number	Weight of particulate collected, mg	
	Final weight	Tare weight
1		
2		
3		
Total		
Less acetone blank		
Weight of particulate matter		

Stack Moisture Measurement Data
(Optional)

	Volume of liquid water collected	
	Implinger volume, ml	Silica gel weight, g
Final		
Initial		
Liquid collected		
Total volume collected		g[1] or ml

[1]Convert weight of water to volume by dividing total weight increase by density of water (1 g/ml).

$$\frac{\text{Increase, g}}{\text{(1 g/ml)}} = \text{Volume water, ml}$$

Figure 5G-4. Analysis Data Sheet.

METHOD 5H—DETERMINATION OF PARTICULATE MATTER EMISSIONS FROM WOOD HEATERS FROM A STACK LOCATION

NOTE: This method does not include all of the specifications (*e.g.*, equipment and supplies) and procedures (*e.g.*, sampling and analytical) essential to its performance. Some material is incorporated by reference from other methods in this part. Therefore, to obtain reliable results, persons using this method should have a thorough knowledge of at least the following additional test methods: Method 2, Method 3, Method 5, Method 5G, Method 6, Method 6C, Method 16A, and Method 28.

1.0 Scope and Application

1.1 Analyte. Particulate matter (PM). No CAS number assigned.

1.2 Applicability. This method is applicable for the determination of PM and condensible emissions from wood heaters.

1.3 Data Quality Objectives. Adherence to the requirements of this method will enhance the quality of the data obtained from air pollutant sampling methods.

2.0 Summary of Method

2.1 Particulate matter is withdrawn proportionally from the wood heater exhaust and is collected on two glass fiber filters separated by impingers immersed in an ice water bath. The first filter is maintained at a temperature of no greater than 120 °C (248 °F). The second filter and the impinger system are cooled such that the temperature of the gas exiting the second filter is no greater than 20 °C (68 °F). The particulate mass collected in the probe, on the filters, and in the impingers is determined gravimetrically after the removal of uncombined water.

3.0 Definitions

Same as in Method 6C, section 3.0.

4.0 Interferences [Reserved]

5.0 Safety

5.1 Disclaimer. This method may involve hazardous materials, operations, and equipment. This test method may not address all of the safety problems associated with its use. It is the responsibility of the user of this test method to establish appropriate safety and health practices and to determine the applicability of regulatory limitations prior to performing this test method.

6.0 Equipment and Supplies

6.1 Sample Collection. The following items are required for sample collection:

6.1.1 Sampling Train. The sampling train configuration is shown in Figure 5H–1. Same as Method 5, section 6.1.1, with the exception of the following:

6.1.1.1 Probe Nozzle. The nozzle is optional; a straight sampling probe without a nozzle is an acceptable alternative.

6.1.1.2 Probe Liner. Same as Method 5, section 6.1.1.2, except that the maximum length of the sample probe shall be 0.6 m (2 ft) and probe heating is optional.

6.1.1.3 Filter Holders. Two each of borosilicate glass, with a glass frit or stainless steel filter support and a silicone rubber, Teflon, or Viton gasket. The holder design shall provide a positive seal against leakage from the outside or around the filter. The front filter holder shall be attached immediately at the outlet of the probe and prior to the first impinger. The second filter holder shall be attached on the outlet of the third impinger and prior to the inlet of the fourth (silica gel) impinger.

6.1.2 Barometer. Same as Method 5, section 6.2.

6.1.3 Stack Gas Flow Rate Measurement System. A schematic of an example test system is shown in Figure 5H–2. The flow rate measurement system consists of the following components:

6.1.3.1 Sample Probe. A glass or stainless steel sampling probe.

6.1.3.2 Gas Conditioning System. A high density filter to remove particulate matter and a condenser capable of lowering the dew point of the gas to less than 5 °C (40 °F). Desiccant, such as Drierite, may be used to dry the sample gas. Do not use silica gel.

6.1.3.3 Pump. An inert (*e.g.*, Teflon or stainless steel heads) sampling pump capable of delivering more than the total amount of sample required in the manufacturer's instructions for the individual instruments. A means of controlling the analyzer flow rate and a device for determining proper sample flow rate (*e.g.*, precision rotameter, pressure gauge downstream of all flow controls) shall be provided at the analyzer. The requirements for measuring and controlling the analyzer flow rate are not applicable if data are presented that demonstrate that the analyzer is insensitive to flow variations over the range encountered during the test.

6.1.3.4 Carbon Monoxide (CO) Analyzer. Any analyzer capable of providing a measure of CO in the range of 0 to 10 percent by volume at least once every 10 minutes.

6.1.3.5 Carbon Dioxide (CO_2) Analyzer. Any analyzer capable of providing a measure of CO_2 in the range of 0 to 25 percent by volume at least once every 10 minutes.

NOTE: Analyzers with ranges less than those specified above may be used provided actual concentrations do not exceed the range of the analyzer.

6.1.3.6 Manifold. A sampling tube capable of delivering the sample gas to two analyzers and handling an excess of the total amount used by the analyzers. The excess gas is exhausted through a separate port.

6.1.3.7 Recorders (optional). To provide a permanent record of the analyzer outputs.

6.1.4 Proportional Gas Flow Rate System. To monitor stack flow rate changes and provide a measurement that can be used to adjust and maintain particulate sampling flow rates proportional to the stack gas flow rate. A schematic of the proportional flow rate system is shown in Figure 5H–2 and consists of the following components:

6.1.4.1 Tracer Gas Injection System. To inject a known concentration of sulfur dioxide (SO_2) into the flue. The tracer gas injection system consists of a cylinder of SO_2, a gas cylinder regulator, a stainless steel needle valve or flow controller, a nonreactive (stainless steel and glass) rotameter, and an injection loop to disperse the SO_2 evenly in the flue.

6.1.4.2 Sample Probe. A glass or stainless steel sampling probe.

6.1.4.3 Gas Conditioning System. A combustor as described in Method 16A, sections 6.1.5 and 6.1.6, followed by a high density filter to remove particulate matter, and a condenser capable of lowering the dew point of the gas to less than 5 °C (40 °F). Desiccant, such as Drierite, may be used to dry the sample gas. Do not use silica gel.

6.1.4.4 Pump. Same as described in section 6.1.3.3.

6.1.4.5 SO_2 Analyzer. Any analyzer capable of providing a measure of the SO_2 concentration in the range of 0 to 1,000 ppm by volume (or other range necessary to measure the SO_2 concentration) at least once every 10 minutes.

6.1.4.6 Recorder (optional). To provide a permanent record of the analyzer outputs.

NOTE: Other tracer gas systems, including helium gas systems, are acceptable for determination of instantaneous proportional sampling rates.

6.2 Sample Recovery. Same as Method 5, section 6.2.

6.3 Sample Analysis. Same as Method 5, section 6.3, with the addition of the following:

6.3.1 Separatory Funnel. Glass or Teflon, 500-ml or greater.

7.0 Reagents and Standards

7.1 Sample Collection. Same as Method 5, section 7.1, including deionized distilled water.

7.2 Sample Recovery. Same as Method 5, section 7.2.

7.3 Sample Analysis. The following reagents and standards are required for sample analysis:

7.3.1 Acetone. Same as Method 5 section 7.2.

7.3.2 Dichloromethane (Methylene Chloride). Reagent grade, <0.001 percent residue in glass bottles.

7.3.3 Desiccant. Anhydrous calcium sulfate, calcium chloride, or silica gel, indicating type.

7.3.4 Cylinder Gases. For the purposes of this procedure, span value is defined as the upper limit of the range specified for each analyzer as described in section 6.1.3.4 or 6.1.3.5. If an analyzer with a range different from that specified in this method is used, the span value shall be equal to the upper limit of the range for the analyzer used (see note in section 6.1.3.5).

7.3.4.1 Calibration Gases. The calibration gases for the CO_2, CO, and SO_2 analyzers shall be CO_2 in nitrogen (N_2), CO in N_2, and SO_2 in N_2, respectively. CO_2 and CO calibration gases may be combined in a single cylinder. Use three calibration gases as specified in Method 6C, sections 7.2.1 through 7.2.3.

7.3.4.2 SO_2 Injection Gas. A known concentration of SO_2 in N_2. The concentration must be at least 2 percent SO_2 with a maximum of 100 percent SO_2.

8.0 Sample Collection, Preservation, Transport, and Storage

8.1 Pretest Preparation. Same as Method 5, section 8.1.

8.2 Calibration Gas and SO_2 Injection Gas Concentration Verification, Sampling System Bias Check, Response Time Test, and Zero and Calibration Drift Tests. Same as Method 6C, sections 8.2.1, 8.2.3, 8.2.4, and 8.5, respectively, except that for verification of CO and CO_2 gas concentrations, substitute Method 3 for Method 6.

8.3 Preliminary Determinations.

8.3.1 Sampling Location. The sampling location for the particulate sampling probe shall be 2.45 ±0.15 m (8 ±0.5 ft) above the platform upon which the wood heater is placed (*i.e.*, the top of the scale).

8.3.2 Sampling Probe and Nozzle. Select a nozzle, if used, sized for the range of velocity heads, such that it is not necessary to change the nozzle size in order to maintain proportional sampling rates. During the run, do not change the nozzle size. Select a suitable probe liner and probe length to effect minimum blockage.

8.4 Preparation of Particulate Sampling Train. Same as Method 5, section 8.3, with the exception of the following:

8.4.1 The train should be assembled as shown in Figure 5H–1.

8.4.2 A glass cyclone may not be used between the probe and filter holder.

8.5 Leak-Check Procedures.

8.5.1 Leak-Check of Metering System Shown in Figure 5H–1. That portion of the sampling train from the pump to the orifice meter shall be leak-checked after each certification or audit test. Use the procedure described in Method 5, section 8.4.1.

8.5.2 Pretest Leak-Check. A pretest leak-check of the sampling train is recommended, but not required. If the pretest leak-check is conducted, the procedures outlined in Method 5, section 8.5.2 should be used. A vacuum of 130 mm Hg (5 in. Hg) may be used instead of 380 mm Hg (15 in. Hg).

8.5.2 Leak-Checks During Sample Run. If, during the sampling run, a component (*e.g.*, filter assembly or impinger) change becomes necessary, conduct a leak-check as described in Method 5, section 8.4.3.

8.5.3 Post-Test Leak-Check. A leak-check is mandatory at the conclusion of each sampling run. The leak-check shall be performed in accordance with the procedures outlined in Method 5, section 8.4.4, except that a vacuum of 130 mm Hg (5 in. Hg) or the greatest vacuum measured during the test run, whichever is greater, may be used instead of 380 mm Hg (15 in. Hg).

8.6 Tracer Gas Procedure. A schematic of the tracer gas injection and sampling systems is shown in Figure 5H–2.

8.6.1 SO_2 Injection Probe. Install the SO_2 injection probe and dispersion loop in the stack at a location 2.9 ±0.15 m (9.5 ±0.5 ft) above the sampling platform.

8.6.2 SO_2 Sampling Probe. Install the SO_2 sampling probe at the centroid of the stack at a location 4.1 ±0.15 m (13.5 ±0.5 ft) above the sampling platform.

8.7 Flow Rate Measurement System. A schematic of the flow rate measurement system is shown in Figure 5H–2. Locate the flow rate measurement sampling probe at the centroid of the stack at a location 2.3 ±0.3 m (7.5 ±1 ft) above the sampling platform.

8.8 Tracer Gas Procedure. Within 1 minute after closing the wood heater door at the start of the test run (as defined in Method 28, section 8.8.1), meter a known concentration of SO_2 tracer gas at a constant flow rate into the wood heater stack. Monitor the SO_2 concentration in the stack, and record the SO_2 concentrations at 10-minute intervals or more often. Adjust the particulate sampling flow rate proportionally to the SO_2 concentration changes using Equation 5H–6 (*e.g.*, the SO_2 concentration at the first 10-minute reading is measured to be 100 ppm; the next 10 minute SO_2 concentration is measured to be 75 ppm: the particulate sample flow rate is adjusted from the initial 0.15 cfm to 0.20 cfm). A check for proportional rate variation shall be made at the completion of the test run using Equation 5H–10.

8.9 Volumetric Flow Rate Procedure. Apply stoichiometric relationships to the wood combustion process in determining the exhaust gas flow rate as follows:

8.9.1 Test Fuel Charge Weight. Record the test fuel charge weight (wet) as specified in Method 28, section 8.8.2. The wood is assumed to have the following weight percent composition: 51 percent carbon, 7.3 percent hydrogen, 41 percent oxygen. Record the wood moisture for each fuel charge as described in Method 28, section 8.6.5. The ash is assumed to have negligible effect on associated C, H, and O concentrations after the test burn.

8.9.2 Measured Values. Record the CO and CO_2 concentrations in the stack on a dry basis every 10 minutes during the test run or more often. Average these values for the test run. Use as a mole fraction (*e.g.*, 10 percent CO_2 is recorded as 0.10) in the calculations to express total flow (see Equation 5H–6).

8.10 Sampling Train Operation.

8.10.1 For each run, record the data required on a data sheet such as the one shown in Figure 5H–3. Be sure to record the initial dry gas meter reading. Record the dry gas meter readings at the beginning and end of each sampling time increment, when changes in flow rates are made, before and after each leak-check, and when sampling is halted. Take other readings as indicated on Figure 5H–3 at least once each 10 minutes during the test run.

8.10.2 Remove the nozzle cap, verify that the filter and probe heating systems are up to temperature, and that the probe is properly positioned. Position the nozzle, if used, facing into gas stream, or the probe tip in the 50 mm (2 in.) centroidal area of the stack.

8.10.3 Be careful not to bump the probe tip into the stack wall when removing or inserting the probe through the porthole; this minimizes the chance of extracting deposited material.

8.10.4 When the probe is in position, block off the openings around the probe and porthole to prevent unrepresentative dilution of the gas stream.

8.10.5 Begin sampling at the start of the test run as defined in Method 28, section 8.8.1, start the sample pump, and adjust the sample flow rate to between 0.003 and 0.014 m^3/min (0.1 and 0.5 cfm). Adjust the sample flow rate proportionally to the stack gas flow during the test run according to the procedures outlined in section 8. Maintain a proportional sampling rate (within 10 percent of the desired value) and a filter holder temperature no greater than 120 °C (248 °F).

8.10.6 During the test run, make periodic adjustments to keep the temperature around the filter holder at the proper level. Add more ice to the impinger box and, if necessary, salt to maintain a temperature of less than 20 °C (68 °F) at the condenser/silica gel outlet.

8.10.7 If the pressure drop across the filter becomes too high, making proportional sampling difficult to maintain, either filter may be replaced during a sample run. It is recommended that another complete filter assembly be used rather than attempting to change the filter itself. Before a new filter assembly is installed, conduct a leak-check (see section 8.5.2). The total particulate weight shall include the summation of all filter assembly catches. The total time for changing sample train components shall not exceed 10 minutes. No more than one component change is allowed for any test run.

8.10.8 At the end of the test run, turn off the coarse adjust valve, remove the probe and nozzle from the stack, turn off the pump, record the final dry gas meter reading, and conduct a post-test leak-check, as outlined in section 8.5.3.

8.11 Sample Recovery. Same as Method 5, section 8.7, with the exception of the following:

8.11.1 Blanks. The volume of the acetone blank may be about 50-ml, rather than 200-ml; a 200-ml water blank shall also be saved for analysis.

8.11.2 Samples.

8.11.2.1 Container Nos. 1 and 1A. Treat the two filters according to the procedures outlined in Method 5, section 8.7.6.1. The filters

may be stored either in a single container or in separate containers.

8.11.2.2 Container No. 2. Same as Method 5, section 8.7.6.2, except that the container should not be sealed until the impinger rinse solution is added (see section 8.10.2.4).

8.11.2.3 Container No. 3. Treat the impingers as follows: Measure the liquid which is in the first three impingers to within 1-ml by using a graduated cylinder or by weighing it to within 0.5 g by using a balance (if one is available). Record the volume or weight of liquid present. This information is required to calculate the moisture content of the effluent gas. Transfer the water from the first, second, and third impingers to a glass container. Tighten the lid on the sample container so that water will not leak out.

8.11.2.4 Rinse impingers and graduated cylinder, if used, with acetone three times or more. Avoid direct contact between the acetone and any stopcock grease or collection of any stopcock grease in the rinse solutions. Add these rinse solutions to sample Container No. 2.

8.11.2.5 Container No. 4. Same as Method 5, section 8.7.6.3

8.12 Sample Transport. Whenever possible, containers should be transferred in such a way that they remain upright at all times.

NOTE: Requirements for capping and transport of sample containers are not applicable if sample recovery and analysis occur in the same room.

9.0 Quality Control

9.1 Miscellaneous Quality Control Measures.

Section	Quality control measure	Effect
8.2	Sampling system bias check	Ensures that bias introduced by measurement system, minus analyzer, is no greater than 3 percent of span.
8.2	Analyzer zero and calibration drift tests	Ensures that bias introduced by drift in the measurement system output during the run is no greater than 3 percent of span.
8.5, 10.1, 12.13	Sampling equipment leak-check and calibration; proportional sampling rate verification.	Ensures accurate measurement of stack gas flow rate, sample volume.
10.1	Analytical balance calibration	Ensure accurate and precise measurement of collected particulate.
10.3	Analyzer calibration error check	Ensures that bias introduced by analyzer calibration error is no greater than 2 percent of span.

9.2 Volume Metering System Checks. Same as Method 5, section 9.2.

10.0 Calibration and Standardization

NOTE: Maintain a laboratory record of all calibrations.

10.1 Volume Metering System, Temperature Sensors, Barometer, and Analytical Balance. Same as Method 5G, sections 10.2 through 10.5, respectively.

10.2 SO_2 Injection Rotameter. Calibrate the SO_2 injection rotameter system with a soap film flowmeter or similar direct volume measuring device with an accuracy of 2 percent. Operate the rotameter at a single reading for at least three calibration runs for 10 minutes each. When three consecutive calibration flow rates agree within 5 percent, average the three flow rates, mark the rotameter at the calibrated setting, and use the calibration flow rate as the SO_2 injection flow rate during the test run. Repeat the rotameter calibration before the first certification test and semiannually thereafter.

10.3 Gas Analyzers. Same as Method 6C, section 10.0.

10.4 Field Balance Calibration Check. Check the calibration of the balance used to weigh impingers with a weight that is at least 500g or within 50g of a loaded impinger. The weight must be ASTM E617–13 "Standard Specification for Laboratory Weights and Precision Mass Standards" (incorporated by reference—see 40 CFR 60.17) Class 6 (or better). Daily before use, the field balance must measure the weight within ± 0.5g of the certified mass. If the daily balance calibration check fails, perform corrective measures and repeat the check before using balance.

10.5 Analytical Balance Calibration. Perform a multipoint calibration (at least five points spanning the operational range) of the analytical balance before the first use, and semiannually thereafter. The calibration of the analytical balance must be conducted using ASTM E617–13 "Standard Specification for Laboratory Weights and Precision Mass Standards" (incorporated by reference—see 40 CFR 60.17) Class 2 (or better) tolerance weights. Audit the balance each day it is used for gravimetric measurements by weighing at least one ASTM E617–13 Class 2 tolerance (or better) calibration weight that corresponds to 50 to 150 percent of the weight of one filter or between 1g and 5g. If the scale cannot reproduce the value of the calibration weight to within 0.5 mg of the certified mass, perform corrective measures, and conduct the multipoint calibration before use.

11.0 Analytical Procedure

11.1 Record the data required on a sheet such as the one shown in Figure 5H–4.

11.2 Handle each sample container as follows:

11.2.1 Container Nos. 1 and 1A. Treat the two filters according to the procedures outlined in Method 5, section 11.2.1.

11.2.2 Container No. 2. Same as Method 5, section 11.2.2, except that the beaker may be smaller than 250-ml.

11.2.3 Container No. 3. Note the level of liquid in the container and confirm on the analysis sheet whether leakage occurred during transport. If a noticeable amount of leakage has occurred, either void the sample or use methods, subject to the approval of the Administrator, to correct the final results. Determination of sample leakage is not applicable if sample recovery and analysis occur in the same room. Measure the liquid in this container either volumetrically to within 1-ml or gravimetrically to within 0.5 g. Transfer the contents to a 500-ml or larger separatory funnel. Rinse the container with water, and add to the separatory funnel. Add 25-ml of dichloromethane to the separatory funnel, stopper and vigorously shake 1 minute, let separate and transfer the dichloromethane (lower layer) into a tared beaker or evaporating dish. Repeat twice more. It is necessary to rinse Container No. 3 with dichloromethane. This rinse is added to the impinger extract container. Transfer the remaining water from the separatory funnel to a tared beaker or evaporating dish and evaporate to dryness at 104 °C (220 °F). Desiccate and weigh to a constant weight. Evaporate the combined impinger water extracts at ambient temperature and pressure. Desiccate and weigh to a constant weight. Report both results to the nearest 0.1 mg.

11.2.4 Container No. 4. Weigh the spent silica gel (or silica gel plus impinger) to the nearest 0.5 g using a balance.

11.2.5 Acetone Blank Container. Same as Method 5, section 11.2.4, except that the beaker may be smaller than 250 ml.

11.2.6 Dichloromethane Blank Container. Treat the same as the acetone blank.

11.2.7 Water Blank Container. Transfer the water to a tared 250 ml beaker and evaporate to dryness at 104 °C (220 °F). Desiccate and weigh to a constant weight.

12.0 Data Analysis and Calculations

Carry out calculations, retaining at least one extra significant figure beyond that of the acquired data. Round off figures after the final calculation. Other forms of the equations may be used as long as they give equivalent results.

12.1 Nomenclature.

A = Sample flow rate adjustment factor.
BR = Dry wood burn rate, kg/hr (lb/hr), from Method 28, Section 8.3.
B_{ws} = Water vapor in the gas stream, proportion by volume.
C_i = Tracer gas concentration at inlet, ppmv.
C_o = Tracer gas concentration at outlet, ppmv.
C_s = Concentration of particulate matter in stack gas, dry basis, corrected to standard conditions, g/dscm (g/dscf).
E = Particulate emission rate, g/hr (lb/hr).
ΔH = Average pressure differential across the orifice meter (see Figure 5H–1), mm H_2O (in. H_2O).
L_a = Maximum acceptable leakage rate for either a post-test leak-check or for a leak-check following a component change; equal to 0.00057 cmm (0.020 cfm) or 4 percent of the average sampling rate, whichever is less.
L_i = Individual leakage rate observed during the leak-check conducted before a component change, cmm (cfm).
L_p = Leakage rate observed during the post-test leak-check, cmm (cfm).
m_n = Total amount of particulate matter collected, mg.
M_a = Mass of residue of solvent after evaporation, mg.
N_C = Grams of carbon/gram of dry fuel (lb/lb), equal to 0.0425.
N_T = Total dry moles of exhaust gas/kg of dry wood burned, g-moles/kg (lb-moles/lb).
PR = Percent of proportional sampling rate.
P_{bar} = Barometric pressure at the sampling site, mm Hg (in.Hg).
P_{std} = Standard absolute pressure, 760 mm Hg (29.92 in.Hg).
Q_i = Gas volumetric flow rate at inlet, cfm (l/min).
Q_o = Gas volumetric flow rate at outlet, cfm (l/min).

12.2 Average Dry Gas Meter Temperature and Average Orifice Pressure Drop. See data sheet (Figure 5H–3).

12.3 Dry Gas Volume. Same as Method 5, section 12.3.

12.4 Volume of Water Vapor.

$$V_{w(std)} = K_2 V_{lc} \qquad \text{Eq. 5H-1}$$

Where:

K_2 = 0.001333 m^3/ml for metric units.
K_2 = 0.04707 ft^3/ml for English units.

12.5 Moisture Content.

$$B_{ws} = \frac{V_{w(std)}}{V_{m(std)} + V_{w(std)}} \qquad \text{Eq. 5H-2}$$

12.6 Solvent Wash Blank.

$$W_a = \frac{M_a V_{aw}}{V_a} \qquad \text{Eq. 5H-3}$$

12.7 Total Particulate Weight. Determine the total particulate catch from the sum of the weights obtained from containers 1, 2, 3,

and 4 less the appropriate solvent blanks (see Figure 5H–4).

NOTE: Refer to Method 5, section 8.5 to assist in calculation of results involving two filter assemblies.

12.8 Particulate Concentration.

$$C_s = \frac{0.001g}{mg} \frac{m_n}{V_{m(std)}} \quad \text{Eq. 5H-4}$$

12.9 Sample Flow Rate Adjustment.

$$a = \frac{S_1}{S_i} \quad \text{Eq. 5H-5}$$

12.10 Carbon Balance for Total Moles of Exhaust Gas (dry)/kg of Wood Burned in the Exhaust Gas.

$$N_T = \frac{K_3 N_C}{Y_{CO_2} + Y_{CO} + Y_{HC}} \quad \text{Eq. 5H-6}$$

Where:

K_3 = 1000 g/kg for metric units.
K_3 = 1.0 lb/lb for English units.

NOTE: The NO_X/SO_X portion of the gas is assumed to be negligible.

12.11 Total Stack Gas Flow Rate.

$$Q_{sd} = K_4 N_T BR \quad \text{Eq. 5H-7}$$

Where:

K_4 = 0.02406 dscm/g-mole for metric units.
K_4 = 384.8 dscf/lb-mole for English units.

12.12 Particulate Emission Rate.

$$E = C_s Q_{sd} \quad \text{Eq. 5H-8}$$

12.13 Proportional Rate Variation. Calculate PR for each 10-minute interval, i, of the test run.

$$PR = \frac{\theta S_i V_{mi(std)}}{10 \sum_{i=1}^{N} \left[S_i V_{mi(std)} \right]} \times 100 \quad \text{Eq. 5H-9}$$

12.14 Acceptable Results. If no more than 15 percent of the PR values for all the intervals fall outside the range 90 percent ≤PR ≤110 percent, and if no PR value for any interval falls outside the range 75 ≤PR ≤125 percent, the results are acceptable. If the PR values for the test runs are judged to be unacceptable, report the test run emission results, but do not include the test run results in calculating the weighted average emission rate, and repeat the test.

12.15 Alternative Tracer Gas Flow Rate Determination.

$$Q_o = \frac{Q_i \times C_i}{C_o} \quad Eq.\,5H-10$$

NOTE: This gives Q for a single instance only. Repeated multiple determinations are needed to track temporal variations. Very small variations in Q_i, C_i, or C_o may give very large variations in Q_o.

13.0 Method Performance [Reserved]

14.0 Pollution Prevention [Reserved]

15.0 Waste Management [Reserved]

16.0 Alternative Procedures

16.1 Alternative Stack Gas Volumetric Flow Rate Determination (Tracer Gas).

16.1.1 Apparatus.

16.1.1.1 Tracer Gas Injector System. This is to inject a known concentration of tracer gas into the stack. This system consists of a cylinder of tracer gas, a gas cylinder regulator, a stainless steel needle valve or a flow controller, a nonreactive (stainless steel or glass) rotameter, and an injection loop to disperse the tracer gas evenly in the stack.

16.1.1.2 Tracer Gas Probe. A glass or stainless steel sampling probe.

16.1.1.3 Gas Conditioning System. A gas conditioning system is suitable for delivering a cleaned sample to the analyzer consisting of a filter to remove particulate and a condenser capable of lowering the dew point of the sample gas to less than 5 °C (40 °F). A desiccant such as anhydrous calcium sulfate may be used to dry the sample gas. Desiccants which react or absorb tracer gas or stack gas may not be used, e.g. silica gel absorbs CO_2.

16.1.1.4 Pump. An inert (*i.e.*, stainless steel or Teflon head) pump to deliver more than the total sample required by the manufacturer's specifications for the analyzer used to measure the downstream tracer gas concentration.

16.1.1.5 Gas Analyzer. A gas analyzer is any analyzer capable of measuring the tracer gas concentration in the range necessary at least every 10 minutes. A means of controlling the analyzer flow rate and a device for determining proper sample flow rate shall be provided unless data is provided to show that the analyzer is insensitive to flow variations over the range encountered during the test.

The gas analyzer needs to meet or exceed the following performance specifications:

Linearity	±1 percent of full scale.
Calibration Error	≤2 percent of span.
Response Time	≤10 seconds.
Zero Drift (24 hour)	≤2 percent of full scale.
Span Drift (24 hour)	≤2 percent of full scale.
Resolution	≤0.5 percent of span.

16.1.1.6 Recorder (optional). To provide a permanent record of the analyzer output.

16.1.2 Reagents.

16.1.2.1 Tracer Gas. The tracer gas is sulfur hexafluoride in an appropriate concentration for accurate analyzer measurement or pure sulfur dioxide. The gas used must be nonreactive with the stack effluent and give minimal (<3 percent) interference to measurement by the gas analyzer.

16.1.3 Procedure. Select upstream and downstream locations in the stack or duct for introducing the tracer gas and delivering the sampled gas to the analyzer. The inlet location should be 8 or more duct diameters beyond any upstream flow disturbance. The outlet should be 8 or more undisturbed duct diameters from the inlet and 2 or more duct diameters from the duct exit. After installing the apparatus, meter a known concentration of the tracer gas into the stack at the inlet location. Use the gas sample probe and analyzer to show that no stratification of the tracer gas is found in the stack at the measurement locations. Monitor the tracer gas concentration from the outlet location and record the concentration at 10-minute intervals or more often at the option of the tester. A minimum of three measured intervals is recommended to determine the stack gas volumetric flow rate. Other statistical procedures may be applied for complete flow characterization and additional QA/QC.

17.0 References

Same as Method 5G, section 17.0.

18.0 Tables, Diagrams, Flowcharts, and Validation Data

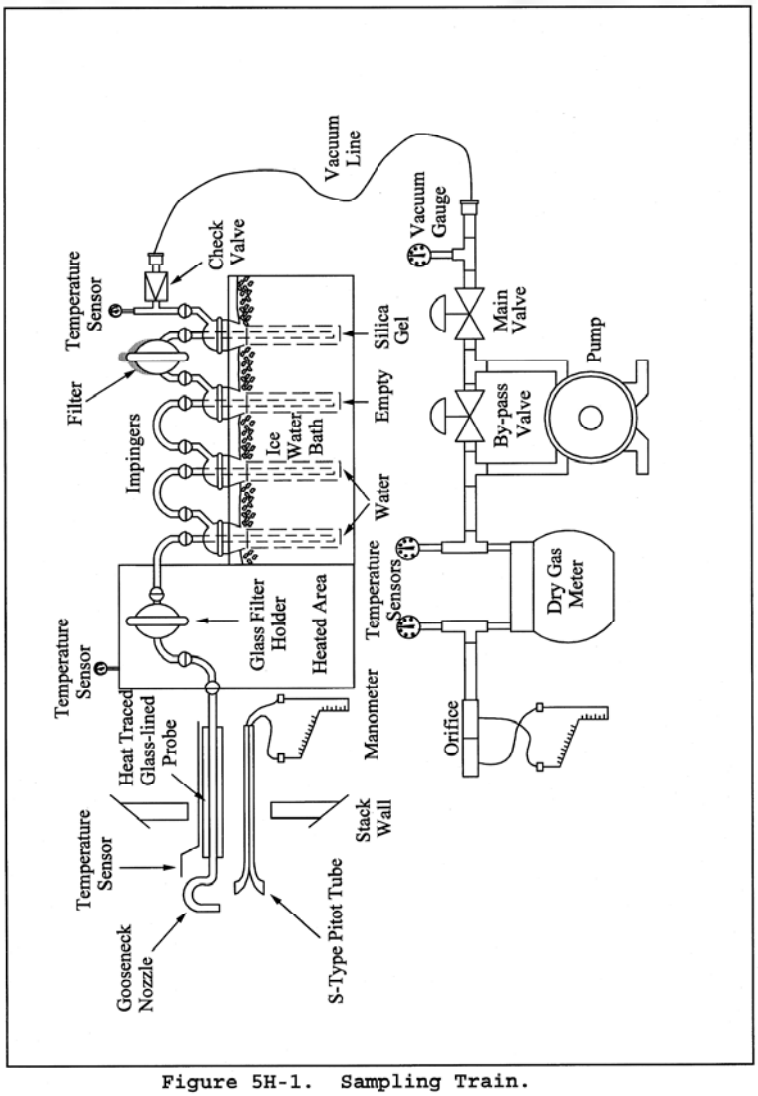


Figure 5H-1. Sampling Train.

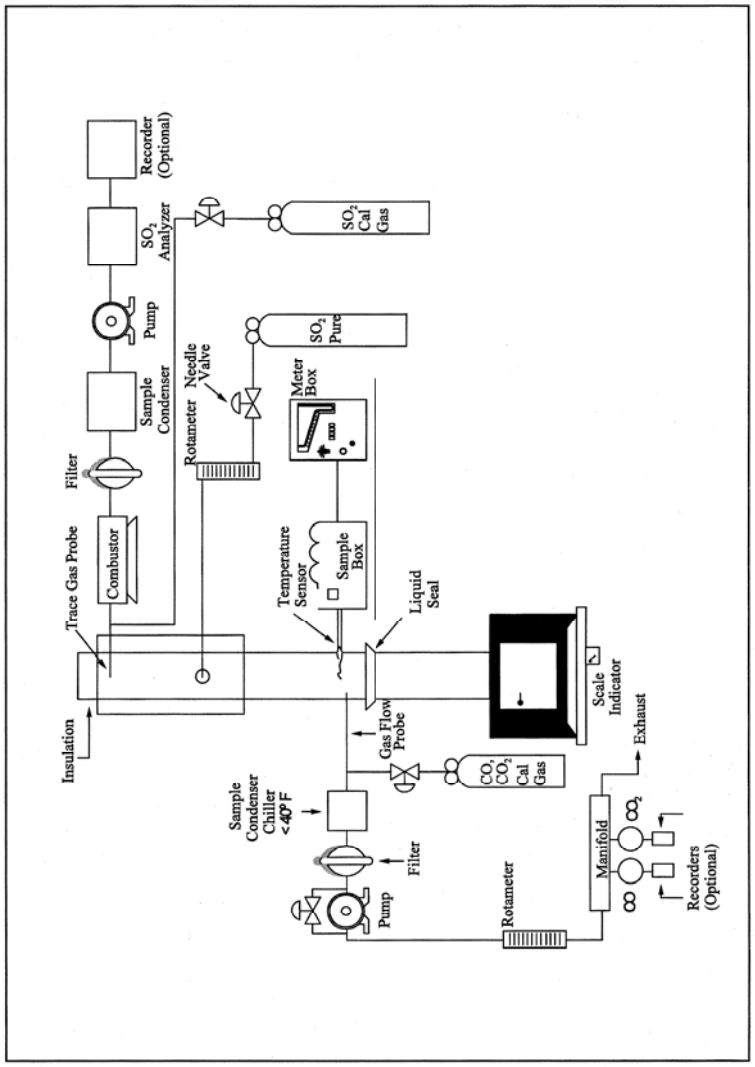


Figure 5H-2. Test System Schematic.

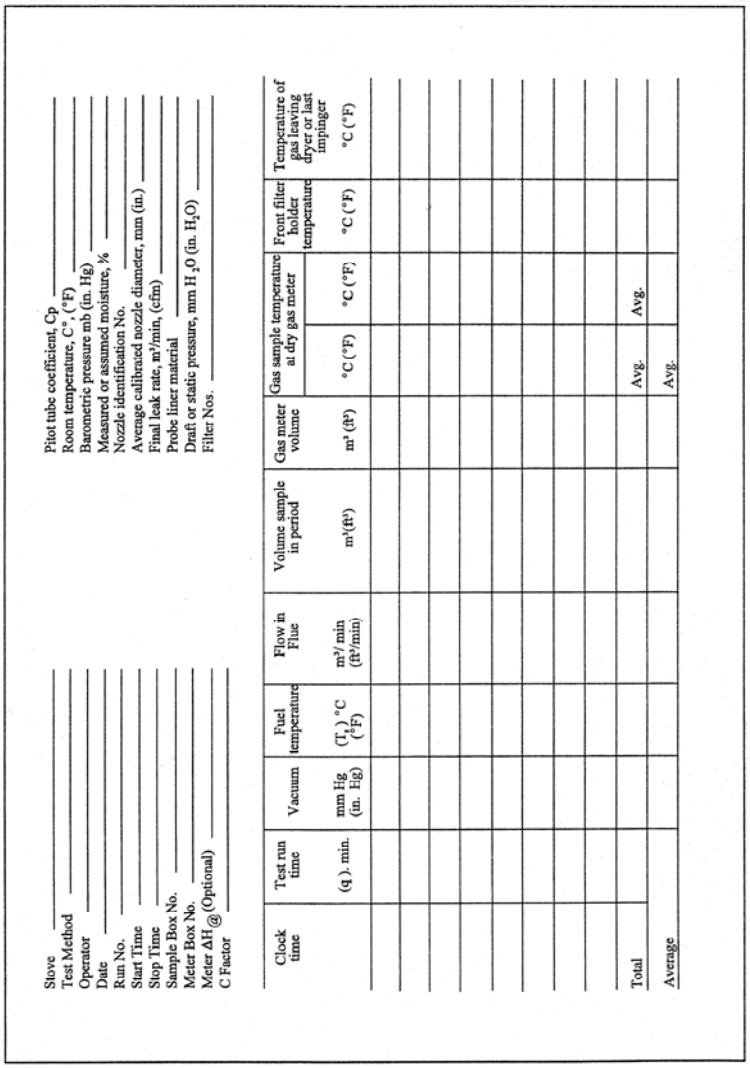


Figure 5H-3. Sampling Data Sheet.

Stove _____
Date _____
Run No. _____
Filter Nos. _____
Amount liquid lost during transport, ml _____
Acetone blank volume, ml _____
Acetone wash volume, ml _____
Acetone blank concentration, mg/ml _____
Acetone wash blank, mg _____

Dichloromethane blank volume, ml _____
Dichloromethane wash volume, ml _____
Dichloromethane blank concentration, mg/ml _____
Dichloromethane wash blank, mg _____
Water blank volume, ml _____
Waster wash volume, ml _____
Water blank concentration, mg/ml _____
Water wash blank, mg _____

Container number	Weight of particulate collected, mg		
	Final weight	Tare weight	Weight gain
1 _____			
2 _____			
3 _____			
4 _____			
5 _____			
Total			
Less acetone blank			
Less dichloromethane blank			
Less water blank			
Weight of particulate matter			

	Volume of liquid water collected	
	Impinger volume, ml	Silica gel weight, g
Final _____		
Initial _____		
Liquid collected _____		
Total volume collected _____		g or ml

* Convert weight of water to volume by dividing total weight increase by density of water (1 g/ml).

$$\frac{\text{Increase, g}}{\text{(1 g/ml)}} = \text{Volume water, ml}$$

Figure 5H-4. Analysis data sheet.

Figure 5H-4. Analysis Data Sheet.

METHOD 5I—DETERMINATION OF LOW LEVEL PARTICULATE MATTER EMISSIONS FROM STATIONARY SOURCES

NOTE: This method does not include all of the specifications (*e.g.*, equipment and supplies) and procedures (*e.g.*, sampling and analytical) essential to its performance. Certain information is contained in other EPA procedures found in this part. Therefore, to obtain reliable results, persons using this method should have experience with and a thorough knowledge of the following Methods: Methods 1. 2. 3. 4 and 5.

1. Scope and Application.

1.1 Analyte. Particulate matter (PM). No CAS number assigned.

1.2 Applicability. This method is applicable for the determination of low level particulate matter (PM) emissions from stationary sources. The method is most effective for total PM catches of 50 mg or less. This method was initially developed for performing correlation of manual PM measurements to PM continuous emission monitoring systems (CEMS), however it is also useful for other low particulate concentration applications.

1.3 Data Quality Objectives. Adherence to the requirements of this method will enhance the quality of the data obtained from air pollutant sampling methods. Method 5I requires the use of paired trains. Acceptance criteria for the identification of data quality outliers from the paired trains are provided in section 12.2 of this Method.

2. Summary of Method.

2.1. Description. The system setup and operation is essentially identical to Method 5. Particulate is withdrawn isokinetically from the source and collected on a 47 mm glass fiber filter maintained at a temperature of 120 ±14 °C (248 ±25 °F). The PM mass is determined by gravimetric analysis after the removal of uncombined water. Specific measures in this procedure designed to improve system performance at low particulate levels include:

1. Improved sample handling procedures
2 Light weight sample filter assembly
3. Use of low residue grade acetone

Accuracy is improved through the minimization of systemic errors associated with sample handling and weighing procedures. High purity reagents, all glass, grease free, sample train components, and light weight filter assemblies and beakers, each contribute to the overall objective of improved precision and accuracy at low particulate concentrations.

2.2 Paired Trains. This method must be performed using a paired train configuration. These trains may be operated as co-located trains (to trains operating collecting from one port) or as simultaneous trains (separate trains operating from different ports at the same time). Procedures for calculating precision of the paired trains are provided in section 12.

2.3 Detection Limit. a. Typical detection limit for manual particulate testing is 0.5 mg. This mass is also cited as the accepted weight variability limit in determination of "constant weight" as cited in section 8.1.2 of this Method. EPA has performed studies to provide guidance on minimum PM catch. The minimum detection limit (MDL) is the minimum concentration or amount of an analyte that can be determined with a specified degree of confidence to be different from zero. We have defined the minimum or target catch as a concentration or amount sufficiently larger than the MDL to ensure that the results are reliable and repeatable. The particulate matter catch is the product of the average particulate matter concentration on a mass per volume basis and the volume of gas collected by the sample train. The tester can generally control the volume of gas collected by increasing the sampling time or to a lesser extent by increasing the rate at which sample is collected. If the tester has a reasonable estimate of the PM concentration from the source, the tester can ensure that the target catch is collected by sampling the appropriate gas volume.

b. However, if the source has a very low particulate matter concentration in the stack, the volume of gas sampled may need to be very large which leads to unacceptably long sampling times. When determining compliance with an emission limit, EPA guidance has been that the tester does not always have to collect the target catch. Instead, we have suggested that the tester sample enough stack gas, that if the source were exactly at the level of the emission standard, the sample catch would equal the target catch. Thus, if at the end of the test the catch were smaller than the target, we could still conclude that the source is in compliance though we might not know the exact emission level. This volume of gas becomes a target volume that can be translated into a target sampling time by assuming an average sampling rate. Because the MDL forms the basis for our guidance on target sampling times, EPA has conducted a systematic laboratory study to define what is the MDL for Method 5 and determined the Method to have a calculated practical quantitation limit (PQL) of 3 mg of PM and an MDL of 1 mg.

c. Based on these results, the EPA has concluded that for PM testing, the target catch must be no less than 3 mg. Those sample catches between 1 mg and 3 mg are between the detection limit and the limit of quantitation. If a tester uses the target catch to estimate a target sampling time that results in sample catches that are less than 3 mg, you should not automatically reject the results. If the tester calculated the target sampling time as described above by assuming that the source was at the level of the emission limit, the results would still be valid for determining that the source was in compliance. For purposes other than determining compliance, results should be divided into two categories—those that fall between 3 mg and 1 mg and those that are below 1 mg. A sample catch between 1 and 3 mg may be used for such purposes as calculating emission rates with the understanding that the resulting emission rates can have a high degree of uncertainty. Results of less than 1 mg should not be used for calculating emission rates or pollutant concentrations.

d. When collecting small catches such as 3 mg, bias becomes an important issue. Source testers must use extreme caution to reach the PQL of 3 mg by assuring that sampling

probes are very clean (perhaps confirmed by low blank weights) before use in the field. They should also use low tare weight sample containers, and establish a well-controlled balance room to weigh the samples.

3. Definitions.

3.1 *Light Weight Filter Housing.* A smaller housing that allows the entire filtering system to be weighed before and after sample collection. (See. 6.1.3)

3.2 *Paired Train.* Sample systems trains may be operated as co-located trains (two sample probes attached to each other in the same port) or as simultaneous trains (two separate trains operating from different ports at the same time).

4. Interferences.

a. There are numerous potential interferents that may be encountered during performance of Method 5I sampling and analyses. This Method should be considered more sensitive to the normal interferents typically encountered during particulate testing because of the low level concentrations of the flue gas stream being sampled.

b. Care must be taken to minimize field contamination, especially to the filter housing since the entire unit is weighed (not just the filter media). Care must also be taken to ensure that no sample is lost during the sampling process (such as during port changes, removal of the filter assemblies from the probes, etc.).

c. Balance room conditions are a source of concern for analysis of the low level samples. Relative humidity, ambient temperatures variations, air draft, vibrations and even barometric pressure can affect consistent reproducible measurements of the sample media. Ideally, the same analyst who performs the tare weights should perform the final weights to minimize the effects of procedural differences specific to the analysts.

d. Attention must also be provided to weighing artifacts caused by electrostatic charges which may have to be discharged or neutralized prior to sample analysis. Static charge can affect consistent and reliable gravimetric readings in low humidity environments. Method 5I recommends a relative humidity of less than 50 percent in the weighing room environment used for sample analyses. However, lower humidity may be encountered or required to address sample precision problems. Low humidity conditions can increase the effects of static charge.

e. Other interferences associated with typical Method 5 testing (sulfates, acid gases, etc.) are also applicable to Method 5I.

5. Safety.

Disclaimer. This method may involve hazardous materials, operations, and equipment. This test method may not address all of the safety concerns associated with its use. It is the responsibility of the user to establish appropriate safety and health practices and to determine the applicability and observe all regulatory limitations before using this method.

6. Equipment and Supplies.

6.1 Sample Collection Equipment and Supplies. The sample train is nearly identical in configuration to the train depicted in Figure 5–1 of Method 5. The primary difference in the sample trains is the lightweight Method 5I filter assembly that attaches directly to the exit to the probe. Other exceptions and additions specific to Method 5I include:

6.1.1 Probe Nozzle. Same as Method 5, with the exception that it must be constructed of borosilicate or quartz glass tubing.

6.1.2 Probe Liner. Same as Method 5, with the exception that it must be constructed of borosilicate or quartz glass tubing.

6.1.3 Filter Holder. The filter holder is constructed of borosilicate or quartz glass front cover designed to hold a 47-mm glass fiber filter, with a wafer thin stainless steel (SS) filter support, a silicone rubber or Viton O-ring, and Teflon tape seal. This holder design will provide a positive seal against leakage from the outside or around the filter. The filter holder assembly fits into a SS filter holder and attaches directly to the outlet of the probe. The tare weight of the filter, borosilicate or quartz glass holder, SS filter support, O-ring and Teflon tape seal generally will not exceed approximately 35 grams. The filter holder is designed to use a 47-mm glass fiber filter meeting the quality criteria in of Method 5. These units are commercially available from several source testing equipment vendors. Once the filter holder has been assembled, desiccated and tared, protect it from external sources of contamination by covering the front socket with a ground glass plug. Secure the plug with an impinger clamp or other item that will ensure a leak-free fitting.

6.2 Sample Recovery Equipment and Supplies. Same as Method 5, with the following exceptions:

6.2.1 Probe-Liner and Probe-Nozzle Brushes. Teflon or nylon bristle brushes with stainless steel wire handles, should be used to clean the probe. The probe brush must have extensions (at least as long as the probe) of Teflon, nylon or similarly inert material. The brushes must be properly sized and shaped for brushing out the probe liner and nozzle.

6.2.2 Wash Bottles. Two Teflon wash bottles are recommended however, polyethylene wash bottles may be used at the option of the tester. Acetone should not be stored in polyethylene bottles for longer than one month.

6.2.3 Filter Assembly Transport. A system should be employed to minimize contamination of the filter assemblies during transport to and from the field test location. A carrying case or packet with clean compartments of sufficient size to accommodate each filter assembly can be used. This system should have an air tight seal to further minimize contamination during transport to and from the field.

6.3 Analysis Equipment and Supplies. Same as Method 5, with the following exception:

6.3.1 Lightweight Beaker Liner. Teflon or other lightweight beaker liners are used for the analysis of the probe and nozzle rinses. These light weight liners are used in place of the borosilicate glass beakers typically used for the Method 5 weighings in order to improve sample analytical precision.

6.3.2 Anti-static Treatment. Commercially available gaseous anti-static rinses are recommended for low humidity situations that contribute to static charge problems.

7. Reagents and Standards.

7.1 Sampling Reagents. The reagents used in sampling are the same as Method 5 with the following exceptions:

7.1.1 Filters. The quality specifications for the filters are identical to those cited for Method 5. The only difference is the filter diameter of 47 millimeters.

7.1.2 Stopcock Grease. Stopcock grease cannot be used with this sampling train. We recommend that the sampling train be assembled with glass joints containing O-ring seals or screw-on connectors, or similar.

7.1.3 Acetone. Low residue type acetone, ≤0.001 percent residue, purchased in glass bottles is used for the recovery of particulate matter from the probe and nozzle. Acetone from metal containers generally has a high residue blank and should not be used. Sometimes, suppliers transfer acetone to glass bottles from metal containers; thus, acetone blanks must be run prior to field use and only acetone with low blank values (≤0.001 percent residue, as specified by the manufacturer) must be used. Acetone blank correction is not allowed for this method; therefore, it is critical that high purity reagents be purchased and verified prior to use.

7.1.4 Gloves. Disposable, powder-free, latex surgical gloves, or their equivalent are used at all times when handling the filter housings or performing sample recovery.

7.2 Standards. There are no applicable standards commercially available for Method 5I analyses.

8. Sample Collection, Preservation, Storage, and Transport.

8.1 Pretest Preparation. Same as Method 5 with several exceptions specific to filter assembly and weighing.

8.1.1 Filter Assembly. Uniquely identify each filter support before loading filters into the holder assembly. This can be done with an engraving tool or a permanent marker. Use powder free latex surgical gloves whenever handling the filter holder assemblies. Place the O-ring on the back of the filter housing in the O-ring groove. Place a 47 mm glass fiber filter on the O-ring with the face down. Place a stainless steel filter holder against the back of the filter. Carefully wrap 5 mm (¼ inch) wide Teflon" tape one timearound the outside of the filter holder overlapping the stainless steel filter support by approximately 2.5 mm (⅛ inch). Gently brush the Teflon tape down on the back of the stainless steel filter support. Store the filter assemblies in their transport case until time for weighing or field use.

8.1.2 Filter Weighing Procedures. a. Desiccate the entire filter holder assemblies at 20 ±5.6 °C (68 ±10 °F) and ambient pressure for at least 24 hours. Weigh at intervals of at least 6 hours to a constant weight, *i.e.*, 0.5 mg change from previous weighing. Record the results to the nearest 0.1 mg. During each weighing, the filter holder assemblies must not be exposed to the laboratory atmosphere for a period greater than 2 minutes and a relative humidity above 50 percent. Lower relative humidity may be required in order to improve analytical precision. However, low humidity conditions increase static charge to the sample media.

b. Alternatively (unless otherwise specified by the Administrator), the filters holder assemblies may be oven dried at 105 °C (220 °F) for a minimum of 2 hours, desiccated for 2 hours, and weighed. The procedure used for the tare weigh must also be used for the final weight determination.

c. Experience has shown that weighing uncertainties are not only related to the balance performance but to the entire weighing procedure. Therefore, before performing any measurement, establish and follow standard operating procedures, taking into account the sampling equipment and filters to be used.

8.2 Preliminary Determinations. Select the sampling site, traverse points, probe nozzle, and probe length as specified in Method 5.

8.3 Preparation of Sampling Train. Same as Method 5, section 8.3, with the following exception: During preparation and assembly of the sampling train, keep all openings where contamination can occur covered until justbefore assembly or until sampling is about to begin. Using gloves, place a labeled (identified) and weighed filter holder assembly into the stainless steel holder. Then place this whole unit in the Method 5 hot box, and attach it to the probe. Do not use stopcock grease.

8.4 Leak-Check Procedures. Same as Method 5.

8.5 Sampling Train Operation.

8.5.1. Operation. Operate the sampling train in a manner consistent with those described in Methods 1, 2, 4 and 5 in terms of the number of sample points and minimum time per point. The sample rate and total gas volume should be adjusted based on estimated grain loading of the source being characterized. The total sampling time must be a function of the estimated mass of particulate to be collected for the run. Targeted mass to be collected in a typical Method 5I sample train should be on the order of 10 to 20 mg. Method 5I is most appropriate for total collected masses of less than 50 milligrams, however, there is not an exact particulate loading cutoff, and it is likely that some runs may exceed 50 mg. Exceeding 50 mg (or less than 10 mg) for the sample mass does not necessarily justify invalidating a sample run if all other Method criteria are met.

8.5.2 Paired Train. This Method requires PM samples be collected with paired trains.

8.5.2.1 It is important that the systems be operated truly simultaneously. This implies that both sample systems start and stop at the same times. This also means that if one sample system is stopped during the run, the other sample systems must also be stopped until the cause has been corrected.

8.5.2.2 Care should be taken to maintain the filter box temperature of the paired trains as close as possible to the Method required temperature of 120 ±14 °C (248 ±25 °F). If separate ovens are being used for simultaneously operated trains, it is recommended that the oven temperature of each train be maintained within ±14 °C (±25 °F) of each other.

8.5.2.3 The nozzles for paired trains need not be identically sized.

8.5.2.4 Co-located sample nozzles must be within the same plane perpendicular to the gas flow. Co-located nozzles and pitot assemblies should be within a 6.0 cm × 6.0 cm square (as cited for a quadruple train in Reference Method 301).

8.5.3 Duplicate gas samples for molecular weight determination need not be collected.

8.6 Sample Recovery. Same as Method 5 with several exceptions specific to the filter housing.

8.6.1 Before moving the sampling train to the cleanup site, remove the probe from the train and seal the nozzle inlet and outlet of the probe. Be careful not to lose any condensate that might be present. Cap the filter inlet using a standard ground glass plug and secure the cap with an impinger clamp. Remove the umbilical cord from the last impinger and cap the impinger. If a flexible line is used between the first impinger condenser and the filter holder, disconnect the line at the filter holder and let any condensed water or liquid drain into the impingers or condenser.

8.6.2 Transfer the probe and filter-impinger assembly to the cleanup area. This area must be clean and protected from the wind so that the possibility of losing any of the sample will be minimized.

8.6.3 Inspect the train prior to and during disassembly and note any abnormal conditions such as particulate color, filter loading, impinger liquid color, etc.

8.6.4 Container No. 1, Filter Assembly. Carefully remove the cooled filter holder assembly from the Method 5 hot box and place it in the transport case. Use a pair of clean gloves to handle the filter holder assembly.

8.6.5 Container No. 2, Probe Nozzle and Probe Liner Rinse. Rinse the probe and nozzle components with acetone. Be certain that the probe and nozzle brushes have been thoroughly rinsed prior to use as they can be a source of contamination.

8.6.6 All Other Train Components. (Impingers) Same as Method 5.

8.7 Sample Storage and Transport. Whenever possible, containers should be shipped in such a way that they remain upright at all times. All appropriate dangerous goods shipping requirements must be observed since acetone is a flammable liquid.

9. Quality Control.

9.1 Miscellaneous Field Quality Control Measures.

9.1.1 A quality control (QC) check of the volume metering system at the field site is suggested before collecting the sample using the procedures in Method 5, section 4.4.1.

9.1.2 All other quality control checks outlined in Methods 1, 2, 4 and 5 also apply to Method 5I. This includes procedures such as leak-checks, equipment calibration checks, and independent checks of field data sheets for reasonableness and completeness.

9.2 Quality Control Samples.

9.2.1 Required QC Sample. A laboratory reagent blank must be collected and analyzed for each lot of acetone used for a field program to confirm that it is of suitable purity. The particulate samples cannot be blank corrected.

9.2.2 Recommended QC Samples. These samples may be collected and archived for future analyses.

9.2.2.1 A field reagent blank is a recommended QC sample collected from a portion of the acetone used for cleanup of the probe and nozzle. Take 100 ml of this acetone directly from the wash bottle being used and place it in a glass sample container labeled "field acetone reagent blank." At least one field reagent blank is recommended for every five runs completed. The field reagent blank samples demonstrate the purity of the acetone was maintained throughout the program.

9.2.2.2 A field bias blank train is a recommended QC sample. This sample is collected by recovering a probe and filter assembly that has been assembled, taken to the sample location, leak checked, heated, allowed to sit at the sample location for a similar duration of time as a regular sample run, leak-checked again, and then recovered in the same manner as a regular sample. Field bias blanks are not a Method requirement, however, they are recommended and are very useful for identifying sources of contamination in emission testing samples. Field bias blank train results greater than 5 times the method detection limit may be considered problematic.

10. Calibration and Standardization Same as Method 5, section 5.

10.1 Field Balance Calibration Check. Check the calibration of the balance used to weigh impingers with a weight that is at least 500g or within 50g of a loaded impinger. The weight must be ASTM E617–13 ''Standard Specification for Laboratory Weights and Precision Mass Standards'' (incorporated by reference—see 40 CFR 60.17) Class 6 (or better). Daily, before use, the field balance must measure the weight within ±0.5g of the certified mass. If the daily balance calibration check fails, perform corrective measures and repeat the check before using balance.

10.2 Analytical Balance Calibration. Perform a multipoint calibration (at least five points spanning the operational range) of the analytical balance before the first use, and semiannually thereafter. The calibration of the analytical balance must be conducted using ASTM E617–13 ''Standard Specification for Laboratory Weights and Precision Mass Standards'' (incorporated by reference—see 40 CFR 60.17) Class 2 (or better) tolerance weights. Audit the balance each day it is used for gravimetric measurements by weighing at least one ASTM E617–13 Class 2 tolerance (or better) calibration weight that corresponds to 50 to 150 percent of the weight of one filter or between 1g and 5g. If the scale cannot reproduce the value of the calibration weight to within 0.5 mg of the certified mass, perform corrective measures and conduct the multipoint calibration before use.

11. Analytical Procedures.

11.1 Analysis. Same as Method 5, sections 11.1–11.2.4, with the following exceptions:

11.1.1 Container No. 1. Same as Method 5, section 11.2.1, with the following exception: Use disposable gloves to remove each of the filter holder assemblies from the desiccator, transport container, or sample oven (after appropriate cooling).

11.1.2 Container No. 2. Same as Method 5, section 11.2.2, with the following exception: It is recommended that the contents of Container No. 2 be transferred to a 250 ml beaker with a Teflon liner or similar container that has a minimal tare weight before bringing to dryness.

12. Data Analysis and Calculations.

12.1 Particulate Emissions. The analytical results cannot be blank corrected for residual acetone found in any of the blanks. All other sample calculations are identical to Method 5.

12.2 Paired Trains Outliers. a. Outliers are identified through the determination of precision and any systemic bias of the paired trains. Data that do not meet this criteria should be flagged as a data quality problem. The primary reason for performing dual train sampling is to generate information to quantify the precision of the Reference Method data. The relative standard deviation (RSD) of paired data is the parameter used to quantify data precision. RSD for two simultaneously gathered data points is determined according to:

$$RSD = 100\% * \left|(C_a - C_b)\right| / (C_a + C_b)$$

where, Ca and Cb are concentration values determined from trains A and B respectively. For RSD calculation, the concentration units are unimportant so long as they are consistent.

b. A minimum precision criteria for Reference Method PM data is that RSD for any data pair must be less than 10% as long as the mean PM concentration is greater than 10 mg/dscm. If the mean PM concentration is less than 10 mg/dscm higher RSD values are acceptable. At mean PM concentration of 1 mg/dscm acceptable RSD for paired trains is 25%. Between 1 and 10 mg/dscm acceptable RSD criteria should be linearly scaled from 25% to 10%. Pairs of manual method data exceeding these RSD criteria should be eliminated from the data set used to develop a PM CEMS correlation or to assess RCA. If the mean PM concentration is less than 1 mg/dscm, RSD does not apply and the mean result is acceptable.

13. Method Performance [Reserved]

14. Pollution Prevention [Reserved]

15. Waste Management [Reserved]

16. Alternative Procedures. Same as Method 5.

17. Bibliography. Same as Method 5.

18. Tables, Diagrams, Flowcharts and Validation Data. Figure 5I–1 is a schematic of the sample train.

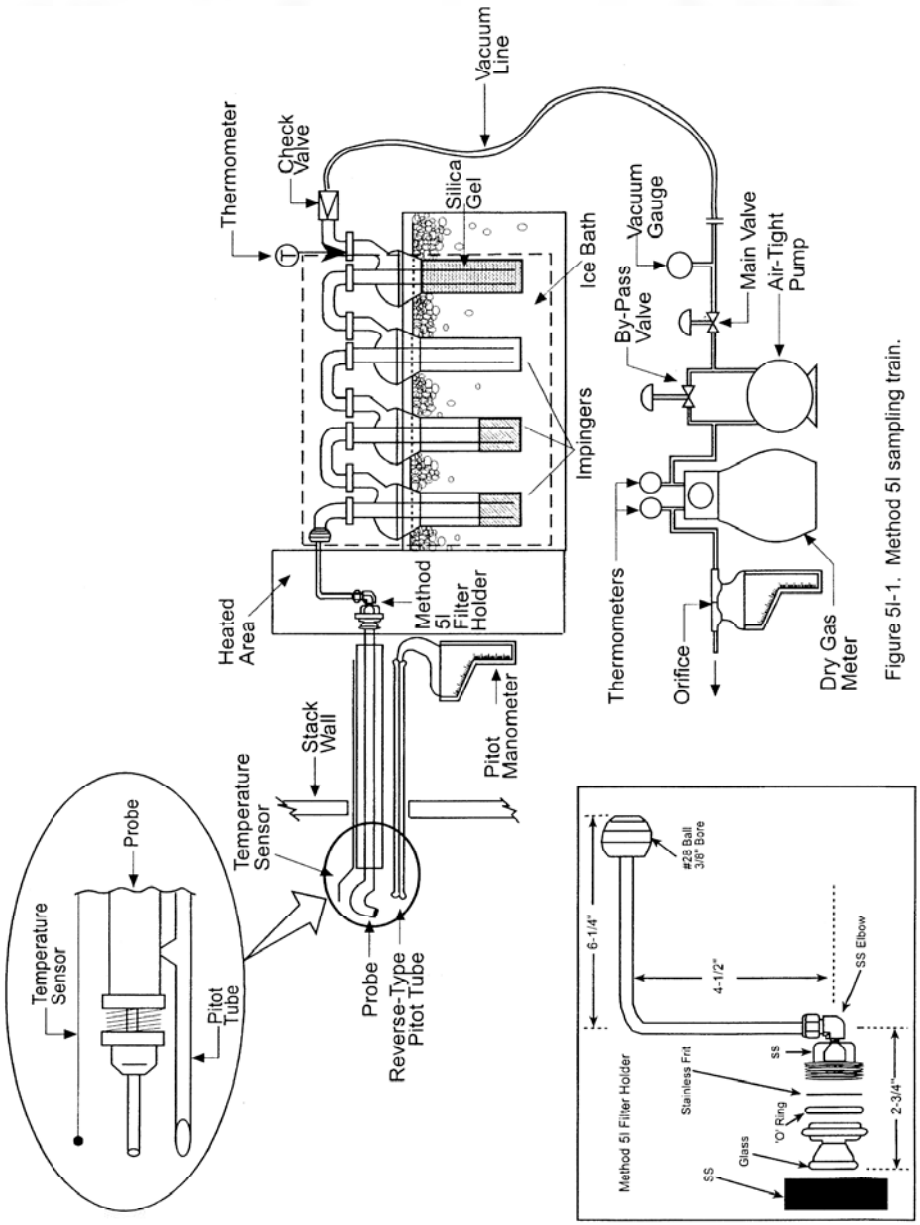


Figure 5I-1. Method 5I sampling train.

[36 FR 24877, Dec. 23, 1971]

EDITORIAL NOTE: For FEDERAL REGISTER citations affecting appendix A–3 to part 60, see the List of CFR sections Affected, which appears in the Finding Aids section of the printed volume and at *www.fdsys.gov*.