

Environmental Protection Agency

Pt. 60, App. A-3

$$B_w = \frac{P_w}{P_{bar}} \quad 3C-1$$

$$C = \frac{A}{R(1-B_w)} \quad 3C-2$$

$$C = \frac{\frac{P_{tf}}{T_{tf}}}{\frac{P_t - P_{ti}}{T_t - T_{ti}}} \cdot \frac{A}{R(1-B_w)} \quad 3C-3$$

7.3 Measured N₂ Concentration Correction. Calculate the reported N₂ correction for Method 25-C using Eq. 3C-4. If oxygen is determined in place of N₂, substitute the oxygen concentration for the nitrogen concentration in the equation.

$$C_{N_2, Corr} = \frac{\frac{P_{tf}}{T_{tf}}}{\frac{P_t - P_{ti}}{T_t - T_{ti}}} (C_{N_2}) \quad Eq. 3C-4$$

8. Bibliography

1. McNair, H.M., and E.J. Bonnelly, Basic Gas Chromatography. Consolidated Printers, Berkeley, CA. 1969.

[36 FR 24877, Dec. 23, 1971]

EDITORIAL NOTE: For FEDERAL REGISTER citations affecting appendix A-2 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.

APPENDIX A-3 TO PART 60—TEST METHODS 4 THROUGH 5I

- Method 4—Determination of moisture content in stack gases
- Method 5—Determination of particulate matter emissions from stationary sources
- Method 5A—Determination of particulate matter emissions from the asphalt processing and asphalt roofing industry
- Method 5B—Determination of nonsulfuric acid particulate matter emissions from stationary sources
- Method 5C [Reserved]
- Method 5D—Determination of particulate matter emissions from positive pressure fabric filters
- Method 5E—Determination of particulate matter emissions from the wool fiber-glass insulation manufacturing industry

Method 5F—Determination of nonsulfate particulate matter emissions from stationary sources

Method 5G—Determination of particulate matter emissions from wood heaters (dilution tunnel sampling location)

Method 5H—Determination of particulate emissions from wood heaters from a stack location

Method 5I—Determination of Low Level Particulate Matter Emissions From Stationary Sources

The test methods in this appendix are referred to in §60.8 (Performance Tests) and §60.11 (Compliance With Standards and Maintenance Requirements) of 40 CFR part 60, subpart A (General Provisions). Specific uses of these test methods are described in the standards of performance contained in the subparts, beginning with Subpart D.

Within each standard of performance, a section title "Test Methods and Procedures" is provided to: (1) identify the test methods to be used as reference methods to the facility subject to the respective standard and (2) identify any special instructions or conditions to be followed when applying a method to the respective facility. Such instructions (for example, establish sampling rates, volumes, or temperatures) are to be used either in addition to, or as a substitute for procedures in a test method. Similarly, for sources subject to emission monitoring requirements, specific instructions pertaining to any use of a test method as a reference

method are provided in the subpart or in Appendix B.

Inclusion of methods in this appendix is not intended as an endorsement or denial of their applicability to sources that are not subject to standards of performance. The methods are potentially applicable to other sources; however, applicability should be confirmed by careful and appropriate evaluation of the conditions prevalent at such sources.

The approach followed in the formulation of the test methods involves specifications for equipment, procedures, and performance. In concept, a performance specification approach would be preferable in all methods because this allows the greatest flexibility to the user. In practice, however, this approach is impractical in most cases because performance specifications cannot be established. Most of the methods described herein, therefore, involve specific equipment specifications and procedures, and only a few methods in this appendix rely on performance criteria.

Minor changes in the test methods should not necessarily affect the validity of the results and it is recognized that alternative and equivalent methods exist. section 60.8 provides authority for the Administrator to specify or approve (1) equivalent methods, (2) alternative methods, and (3) minor changes in the methodology of the test methods. It should be clearly understood that unless otherwise identified all such methods and changes must have prior approval of the Administrator. An owner employing such methods or deviations from the test methods without obtaining prior approval does so at the risk of subsequent disapproval and retesting with approved methods.

Within the test methods, certain specific equipment or procedures are recognized as being acceptable or potentially acceptable and are specifically identified in the methods. The items identified as acceptable options may be used without approval but must be identified in the test report. The potentially approvable options are cited as "subject to the approval of the Administrator" or as "or equivalent." Such potentially approvable techniques or alternatives may be used at the discretion of the owner without prior approval. However, detailed descriptions for applying these potentially approvable techniques or alternatives are not provided in the test methods. Also, the potentially approvable options are not necessarily acceptable in all applications. Therefore, an owner electing to use such potentially approvable techniques or alternatives is responsible for: (1) assuring that the techniques or alternatives are in fact applicable and are properly executed; (2) including a written description of the alternative method in the test report (the written method must be clear and must be capable of

being performed without additional instruction, and the degree of detail should be similar to the detail contained in the test methods); and (3) providing any rationale or supporting data necessary to show the validity of the alternative in the particular application. Failure to meet these requirements can result in the Administrator's disapproval of the alternative.

METHOD 4—DETERMINATION OF MOISTURE CONTENT IN STACK GASES

NOTE: This method does not include all the specifications (e.g., equipment and supplies) and procedures (e.g., sampling) 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 5, and Method 6.

1.0 Scope and Application

1.1 Analytes.

Analyte	CAS No.	Sensitivity
Water vapor (H ₂ O) ...	7732-18-5	N/A

1.2 Applicability. This method is applicable for the determination of the moisture content of stack gas.

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 A gas sample is extracted at a constant rate from the source; moisture is removed from the sample stream and determined either volumetrically or gravimetrically.

2.2 The method contains two possible procedures: a reference method and an approximation method.

2.2.1 The reference method is used for accurate determinations of moisture content (such as are needed to calculate emission data). The approximation method, provides estimates of percent moisture to aid in setting isokinetic sampling rates prior to a pollutant emission measurement run. The approximation method described herein is only a suggested approach; alternative means for approximating the moisture content (e.g., drying tubes, wet bulb-dry bulb techniques, condensation techniques, stoichiometric calculations, previous experience, etc.) are also acceptable.

2.2.2 The reference method is often conducted simultaneously with a pollutant emission measurement run. When it is, calculation of percent isokinetic, pollutant emission rate, etc., for the run shall be based upon the results of the reference method or its equivalent. These calculations shall not

be based upon the results of the approximation method, unless the approximation method is shown, to the satisfaction of the Administrator, to be capable of yielding results within one percent H₂O of the reference method.

3.0 Definitions [Reserved]

4.0 Interferences

4.1 The moisture content of saturated gas streams or streams that contain water droplets, as measured by the reference method, may be positively biased. Therefore, when these conditions exist or are suspected, a second determination of the moisture content shall be made simultaneously with the reference method, as follows: Assume that the gas stream is saturated. Attach a temperature sensor [capable of measuring to ± 1 °C (2 °F)] to the reference method probe. Measure the stack gas temperature at each traverse point (see section 8.1.1.1) during the reference method traverse, and calculate the average stack gas temperature. Next, determine the moisture percentage, either by: (1) Using a psychrometric chart and making appropriate corrections if the stack pressure is different from that of the chart, or (2) using saturation vapor pressure tables. In cases where the psychrometric chart or the saturation vapor pressure tables are not applicable (based on evaluation of the process), alternative methods, subject to the approval of the Administrator, shall be used.

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 determine the applicability of regulatory limitations prior to performing this test method.

6.0 Equipment and Supplies

6.1 Reference Method. A schematic of the sampling train used in this reference method is shown in Figure 4-1.

6.1.1 Probe. Stainless steel or glass tubing, sufficiently heated to prevent water condensation, and equipped with a filter, either in-stack (*e.g.*, a plug of glass wool inserted into the end of the probe) or heated out-of-stack (*e.g.*, as described in Method 5), to remove particulate matter. When stack conditions permit, other metals or plastic tubing may be used for the probe, subject to the approval of the Administrator.

6.1.2 Condenser. Same as Method 5, section 6.1.1.8.

6.1.3 Cooling System. An ice bath container, crushed ice, and water (or equivalent), to aid in condensing moisture.

6.1.4 Metering System. Same as in Method 5, section 6.1.1.9, except do not use sampling systems designed for flow rates higher than 0.0283 m³/min (1.0 cfm). Other metering systems, capable of maintaining a constant sampling rate to within 10 percent and determining sample gas volume to within 2 percent, may be used, subject to the approval of the Administrator.

6.1.5 Barometer and Graduated Cylinder and/or Balance. Same as Method 5, sections 6.1.2 and 6.2.5, respectively.

6.2. Approximation Method. A schematic of the sampling train used in this approximation method is shown in Figure 4-2.

6.2.1 Probe. Same as section 6.1.1.

6.2.2 Condenser. Two midjet impingers, each with 30-ml capacity, or equivalent.

6.2.3 Cooling System. Ice bath container, crushed ice, and water, to aid in condensing moisture in impingers.

6.2.4 Drying Tube. Tube packed with new or regenerated 6- to 16-mesh indicating-type silica gel (or equivalent desiccant), to dry the sample gas and to protect the meter and pump.

6.2.5 Valve. Needle valve, to regulate the sample gas flow rate.

6.2.6 Pump. Leak-free, diaphragm type, or equivalent, to pull the gas sample through the train.

6.2.7 Volume Meter. Dry gas meter, sufficiently accurate to measure the sample volume to within 2 percent, and calibrated over the range of flow rates and conditions actually encountered during sampling.

6.2.8 Rate Meter. Rotameter, or equivalent, to measure the flow range from 0 to 3 liters/min (0 to 0.11 cfm).

6.2.9 Graduated Cylinder. 25-ml.

6.2.10 Barometer. Same as Method 5, section 6.1.2.

6.2.11 Vacuum Gauge. At least 760-mm (30-in.) Hg gauge, to be used for the sampling leak check.

7.0 Reagents and Standards [Reserved]

8.0 Sample Collection, Preservation, Transport, and Storage

8.1 Reference Method. The following procedure is intended for a condenser system (such as the impinger system described in section 6.1.1.8 of Method 5) incorporating volumetric analysis to measure the condensed moisture, and silica gel and gravimetric analysis to measure the moisture leaving the condenser.

8.1.1 Preliminary Determinations.

8.1.1.1 Unless otherwise specified by the Administrator, a minimum of eight traverse points shall be used for circular stacks having diameters less than 0.61 m (24 in.), a minimum of nine points shall be used for rectangular stacks having equivalent diameters less than 0.61 m (24 in.), and a minimum of twelve traverse points shall be used in all

other cases. The traverse points shall be located according to Method 1. The use of fewer points is subject to the approval of the Administrator. Select a suitable probe and probe length such that all traverse points can be sampled. Consider sampling from opposite sides of the stack (four total sampling ports) for large stacks, to permit use of shorter probe lengths. Mark the probe with heat resistant tape or by some other method to denote the proper distance into the stack or duct for each sampling point.

8.1.1.2 Select a total sampling time such that a minimum total gas volume of 0.60 scm (21 scf) will be collected, at a rate no greater than 0.021 m³/min (0.75 cfm). When both moisture content and pollutant emission rate are to be determined, the moisture determination shall be simultaneous with, and for the same total length of time as, the pollutant emission rate run, unless otherwise specified in an applicable subpart of the standards.

8.1.2 Preparation of Sampling Train.

8.1.2.1 Place known volumes of water in the first two impingers; alternatively, transfer water into the first two impingers and record the weight of each impinger (plus water) to the nearest 0.5 g. Weigh and record the weight of the silica gel to the nearest 0.5 g, and transfer the silica gel to the fourth impinger; alternatively, the silica gel may first be transferred to the impinger, and the weight of the silica gel plus impinger recorded.

8.1.2.2 Set up the sampling train as shown in Figure 4-1. Turn on the probe heater and (if applicable) the filter heating system to temperatures of approximately 120 °C (248 °F), to prevent water condensation ahead of the condenser. Allow time for the temperatures to stabilize. Place crushed ice and water in the ice bath container.

8.1.3 Leak Check Procedures. It is recommended, but not required, that the volume metering system and sampling train be leak-checked as follows:

8.1.3.1 Metering System. Same as Method 5, section 8.4.1.

8.1.3.2 Sampling Train. Disconnect the probe from the first impinger or (if applicable) from the filter holder. Plug the inlet to the first impinger (or filter holder), and pull a 380 mm (15 in.) Hg vacuum. A lower vacuum may be used, provided that it is not exceeded during the test. A leakage rate in excess of 4 percent of the average sampling rate or 0.00057 m³/min (0.020 cfm), whichever is less, is unacceptable. Following the leak check, reconnect the probe to the sampling train.

8.1.4 Sampling Train Operation. During the sampling run, maintain a sampling rate within 10 percent of constant rate, or as specified by the Administrator. For each

run, record the data required on a data sheet similar to that shown in Figure 4-3. Be sure to record the dry gas meter reading at the beginning and end of each sampling time increment and whenever sampling is halted. Take other appropriate readings at each sample point at least once during each time increment.

NOTE: When Method 4 is used concurrently with an isokinetic method (*e.g.*, Method 5) the sampling rate should be maintained at isokinetic conditions rather than 10 percent of constant rate.

8.1.4.1 To begin sampling, position the probe tip at the first traverse point. Immediately start the pump, and adjust the flow to the desired rate. Traverse the cross section, sampling at each traverse point for an equal length of time. Add more ice and, if necessary, salt to maintain a temperature of less than 20 °C (68 °F) at the silica gel outlet.

8.1.4.2 After collecting the sample, disconnect the probe from the first impinger (or from the filter holder), and conduct a leak check (mandatory) of the sampling train as described in section 8.1.3.2. Record the leak rate. If the leakage rate exceeds the allowable rate, either reject the test results or correct the sample volume as in section 12.3 of Method 5.

8.2 Approximation Method.

NOTE: The approximation method described below is presented only as a suggested method (see section 2.0).

8.2.1 Place exactly 5 ml water in each impinger. Leak check the sampling train as follows: Temporarily insert a vacuum gauge at or near the probe inlet. Then, plug the probe inlet and pull a vacuum of at least 250 mm (10 in.) Hg. Note the time rate of change of the dry gas meter dial; alternatively, a rotameter (0 to 40 ml/min) may be temporarily attached to the dry gas meter outlet to determine the leakage rate. A leak rate not in excess of 2 percent of the average sampling rate is acceptable.

NOTE: Release the probe inlet plug slowly before turning off the pump.

8.2.2 Connect the probe, insert it into the stack, and sample at a constant rate of 2 liters/min (0.071 cfm). Continue sampling until the dry gas meter registers about 30 liters (1.1 ft³) or until visible liquid droplets are carried over from the first impinger to the second. Record temperature, pressure, and dry gas meter readings as indicated by Figure 4-4.

9.0 Quality Control

9.1 Miscellaneous Quality Control Measures.

Section	Quality control measure	Effect
Section 8.1.1.4	Leak rate of the sampling system cannot exceed four percent of the average sampling rate or 0.00057 m ³ /min (0.020 cfm).	Ensures the accuracy of the volume of gas sampled. (Reference Method).
Section 8.2.1	Leak rate of the sampling system cannot exceed two percent of the average sampling rate.	Ensures the accuracy of the volume of gas sampled. (Approximation Method).

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

10.0 Calibration and Standardization

NOTE: Maintain a laboratory log of all calibrations.

10.1 Reference Method. Calibrate the metering system, temperature sensors, and barometer according to Method 5, sections 10.3, 10.5, and 10.6, respectively.

10.2 Approximation Method. Calibrate the metering system and the barometer according to Method 6, section 10.1 and Method 5, section 10.6, respectively.

10.3 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.

11.0 Analytical Procedure

11.1 Reference Method. Measure the volume of the moisture condensed in each of the impingers to the nearest ml. Alternatively, if the impingers were weighed prior to sampling, weigh the impingers after sampling and record the difference in weight to the nearest 0.5 g. Determine the increase in weight of the silica gel (or silica gel plus impinger) to the nearest 0.5 g. Record this information (see example data sheet, Figure 4-5), and calculate the moisture content, as described in section 12.0.

11.2 Approximation Method. Combine the contents of the two impingers, and measure the volume to the nearest 0.5 ml.

12.0 Data Analysis and Calculations

Carry out the following calculations, retaining at least one extra significant figure beyond that of the acquired data. Round off figures after final calculation.

12.1 Reference Method.

12.1.1 Nomenclature.

B_{wes} = Proportion of water vapor, by volume, in the gas stream.

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

P_m = Absolute pressure (for this method, same as barometric pressure) at the dry gas meter, mm Hg (in. Hg).

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

R = Ideal gas constant, 0.06236 (mm Hg)(m³)/(g-mole)(°K) for metric units and 21.85 (in. Hg)(ft³)/(lb-mole)(°R) for English units.

T_m = Absolute temperature at meter, °K (°R).

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

V_f = Final volume of condenser water, ml.

V_i = Initial volume, if any, of condenser water, ml.

V_m = Dry gas volume measured by dry gas meter, dcm (dcf).

V_{m(std)} = Dry gas volume measured by the dry gas meter, corrected to standard conditions, dscm (dscf).

V_{wc(std)} = Volume of water vapor condensed, corrected to standard conditions, scm (scf).

V_{wsg(std)} = Volume of water vapor collected in silica gel, corrected to standard conditions, scm (scf).

W_f = Final weight of silica gel or silica gel plus impinger, g.

W_i = Initial weight of silica gel or silica gel plus impinger, g.

Y = Dry gas meter calibration factor.

ΔV_m = Incremental dry gas volume measured by dry gas meter at each traverse point, dcm (dcf).

ρ_w = Density of water, 0.9982 g/ml (0.002201 lb/ml).

12.1.2 Volume of Water Vapor Condensed.

$$V_{wc(std)} = \frac{(V_f - V_i) \rho_w R T_{std}}{P_{std} M_w} \quad \text{Eq. 4-1}$$

$$= K_1 (V_f - V_i)$$

Where:

K₁ = 0.001333 m³/ml for metric units.
 = 0.04706 ft³/ml for English units.

12.1.3 Volume of Water Collected in Silica Gel.

$$V_{wsg(std)} = \frac{(W_f - W_i) R T_{std}}{P_{std} M_w K_2} \quad \text{Eq. 4-2}$$

$$= K_3 (W_f - W_i)$$

Where:

$K_2 = 1.0$ g/g for metric units,
 = 453.6 g/lb for English units.
 $K_3 = 0.001335$ m³/g for metric units,
 = 0.04715 ft³/g for English units.

12.1.4 Sample Gas Volume.

$$V_{m(std)} = \frac{V_m Y P_m T_{std}}{P_{std} T_m} \quad \text{Eq. 4-3}$$

$$= K_4 Y \frac{V_m P_m}{T_m}$$

$$B_{ws} = \frac{V_{wc(std)} + V_{wsg(std)}}{V_{wc(std)} + V_{wsg(std)} + V_{m(std)}} \quad \text{Eq. 4-4}$$

12.1.6 Verification of Constant Sampling Rate. For each time increment, determine the ΔV_m . Calculate the average. If the value for any time increment differs from the average by more than 10 percent, reject the results, and repeat the run.

12.1.7 In saturated or moisture droplet-laden gas streams, two calculations of the moisture content of the stack gas shall be made, one using a value based upon the saturated conditions (see section 4.1), and another based upon the results of the impinger analysis. The lower of these two values of B_{ws} shall be considered correct.

12.2 Approximation Method. The approximation method presented is designed to estimate the moisture in the stack gas; therefore, other data, which are only necessary for accurate moisture determinations, are not collected. The following equations adequately estimate the moisture content for the purpose of determining isokinetic sampling rate settings.

12.2.1 Nomenclature.

B_{wm} = Approximate proportion by volume of water vapor in the gas stream leaving the second impinger, 0.025.
 B_{ws} = Water vapor in the gas stream, proportion by volume.
 M_w = Molecular weight of water, 18.0 g/g-mole (18.0 lb/lb-mole).
 P_m = Absolute pressure (for this method, same as barometric pressure) at the dry gas meter, mm Hg (in. Hg).
 P_{std} = Standard absolute pressure, 760 mm Hg (29.92 in. Hg).
 R = Ideal gas constant, 0.06236 [(mm Hg)(m³)/[(g-mole)(K)] for metric units and 21.85 [(in. Hg)(ft³)/[(lb-mole)(°R)] for English units].
 T_m = Absolute temperature at meter, °K (°R).
 T_{std} = Standard absolute temperature, 293 °K (528 °R).
 V_f = Final volume of impinger contents, ml.
 V_i = Initial volume of impinger contents, ml.

Where:

$K_4 = 0.3855$ °K/mm Hg for metric units,
 = 17.64 °R/in. Hg for English units.

NOTE: If the post-test leak rate (Section 8.1.4.2) exceeds the allowable rate, correct the value of V_m in Equation 4-3, as described in section 12.3 of Method 5.

12.1.5 Moisture Content.

V_m = Dry gas volume measured by dry gas meter, dcm (dcf).

$V_{m(std)}$ = Dry gas volume measured by dry gas meter, corrected to standard conditions, dscm (dscf).

$V_{wc(std)}$ = Volume of water vapor condensed, corrected to standard conditions, scm (scf).

Y = Dry gas meter calibration factor.

ρ_w = Density of water, 0.99982 g/ml (0.002201 lb/ml).

12.2.2 Volume of Water Vapor Collected.

$$V_{wc(std)} = \frac{(V_f - V_i) \rho_w R T_{std}}{P_{std} M_w} \quad \text{Eq. 4-5}$$

$$= K_5 (V_f - V_i)$$

Where:

$K_5 = 0.001333$ m³/ml for metric units,
 = 0.04706 ft³/ml for English units.

12.2.3 Sample Gas Volume.

$$V_{m(std)} = \frac{V_m Y P_m T_{std}}{P_{std} T_m} \quad \text{Eq. 4-6}$$

$$= K_6 Y \frac{V_m P_m}{T_m}$$

Where:

$K_6 = 0.3855$ °K/mm Hg for metric units,
 = 17.64 °R/in. Hg for English units.

12.2.4 Approximate Moisture Content.

$$B_{ws} = \frac{V_{wc(std)}}{V_{wc(std)} + V_{m(std)}} + B_{wm} \quad \text{Eq. 4-7}$$

$$= \frac{V_{wc(std)}}{V_{wc(std)} + V_{m(std)}} + (0.025)$$

12.2.5 Using F-factors to determine approximate moisture for estimating moisture content where no wet scrubber is being used, for the purpose of determining isokinetic sampling rate settings with no fuel sample, is acceptable using the average F_w or F_d factor from Method 19 (see Method 19, section

12.3.1). If this option is selected, calculate the approximate moisture as follows:

$$B_{ws} = B_H + B_A + B_F$$

Where:

B_A = Mole Fraction of moisture in the ambient air.

$$B_A = \frac{\%RH}{100 * P_{Bar}} * 10 \left[6.6912 - \left(\frac{3144}{T+390.86} \right) \right]$$

B_F = Mole fraction of moisture from free water in the fuel.

$$B_F = \left[\frac{0.0036W^2 + 0.075W}{100} \right] \left[\frac{20.9 - O_2}{20.9} \right]$$

B_H = Mole fraction of moisture from the hydrogen in the fuel.

$$B_H = \left[1 - \frac{F_d}{F_w} \right] \frac{(20.9 - O_2)}{20.9}$$

B_{ws} = Mole fraction of moisture in the stack gas.

F_d = Volume of dry combustion components per unit of heat content at 0 percent oxygen, dscf/10⁶.

Btu (scm/J). See Table 19-2 in Method 19.

F_w = Volume of wet combustion components per unit of heat content at 0 percent oxygen, wet.

scf/10⁶ Btu (scm/J). See Table 19-2 in Method 19.

%RH = Percent relative humidity (calibrated hygrometer acceptable), percent.

P_{Bar} = Barometric pressure, in. Hg.

T = Ambient temperature, °F.

W = Percent free water by weight, percent.

O_2 = Percent oxygen in stack gas, dry basis, percent.

13.0 Method Performance [Reserved]

14.0 Pollution Prevention [Reserved]

15.0 Waste Management [Reserved]

16.0 Alternative Procedures

16.1 The procedure described in Method 5 for determining moisture content is an acceptable alternative to Method 4.

16.2 The procedures in Method 6A for determining moisture is an acceptable alternative to Method 4.

16.3 Method 320 is an acceptable alternative to Method 4 for determining moisture.

16.4 Using F-factors to determine moisture is an acceptable alternative to Method 4 for a combustion stack not using a scrubber, and where a fuel sample is taken during the test run and analyzed for development of an F_d factor (see Method 19, section 12.3.2), and where stack O_2 content is measured by Method 3A or 3B during each test run. If this option is selected, calculate the moisture content as follows:

$$B_{ws} = B_H + B_A + B_F$$

Where:

B_A = Mole fraction of moisture in the ambient air.

$$B_A = \frac{\%RH}{100 P_{Bar}} \left[10^{6.6912 - \left(\frac{3144}{T+390.86} \right)} \right]$$

NOTE: Values of B_A should be between 0.00 and 0.06 with common values being about 0.015.

B_F = Mole fraction of moisture from free water in the fuel.

$$B_F = \left[\frac{0.0036 W^2 + 0.075 W}{100} \right] \left[\frac{20.9 - O_2}{20.9} \right]$$

NOTE: Free water in fuel is minimal for distillate oil and gases, such as propane and natural gas, so this step may be omitted for those fuels.

B_H = Mole fraction of moisture from the hydrogen in the fuel.

$$B_H = \left(1 - \frac{F_d}{F_w} \right) \frac{(20.9 - O_2)}{20.9}$$

B_{ws} = Mole fraction of moisture in the stack gas.

F_d = Volume of dry combustion components per unit of heat content at 0 percent oxygen, dscf/10⁶ Btu (scm/J). Develop a test specific F_d value using an integrated fuel sample from each test run and Equation 19-13 in section 12.3.2 of Method 19.

F_w = Volume of wet combustion components per unit of heat content at 0 percent oxygen, wet scf/10⁶ Btu (scm/J). Develop a test specific F_w value using an integrated fuel sample from each test run and Equation 19-14 in section 12.3.2 of Method 19.

$\%RH$ = Percent relative humidity (calibrated hygrometer acceptable), percent.

P_{Bar} = Barometric pressure, in. Hg.

T = Ambient temperature, °F.

W = Percent free water by weight, percent.

O_2 = Percent oxygen in stack gas, dry basis, percent.

17.0 References

1. Air Pollution Engineering Manual (Second Edition). Danielson, J.A. (ed.). U.S. Environmental Protection Agency, Office of Air Quality Planning and Standards. Research Triangle Park, NC. Publication No. AP-40. 1973.
2. Devorkin, Howard, et al. Air Pollution Source Testing Manual. Air Pollution Control District, Los Angeles, CA. November 1963.
3. Methods for Determination of Velocity, Volume, Dust and Mist Content of Gases. Western Precipitation Division of Joy Manufacturing Co. Los Angeles, CA. Bulletin WP-50. 1968.

18.0 Tables, Diagrams, Flowcharts, and Validation Data

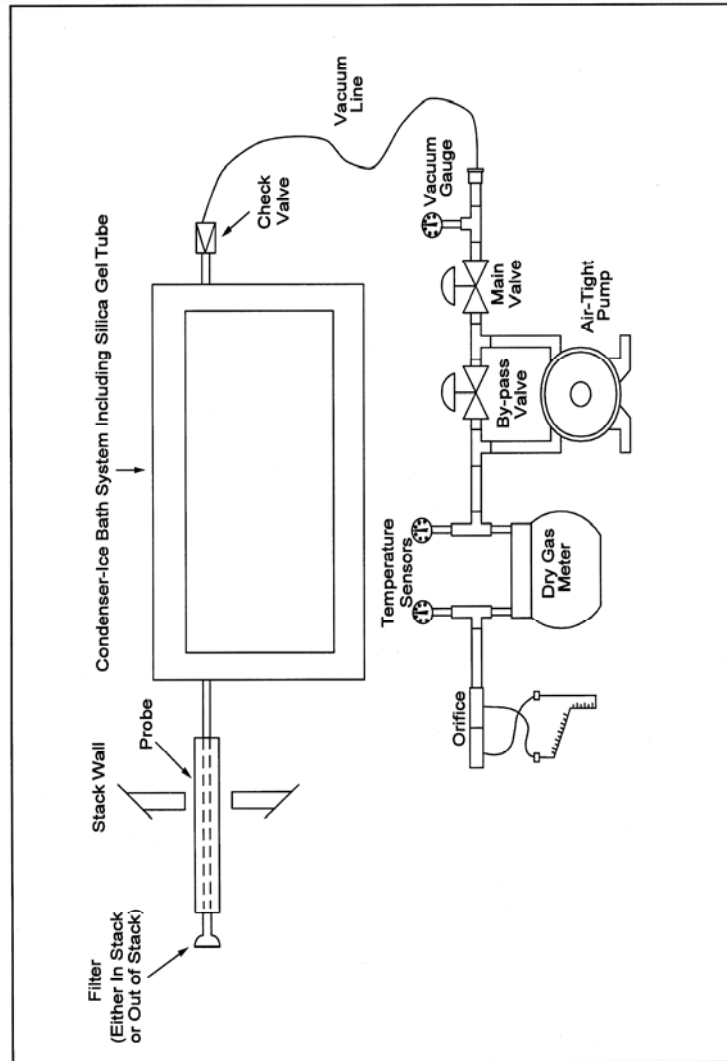


Figure 4-1. Moisture Sampling Train-Reference Method

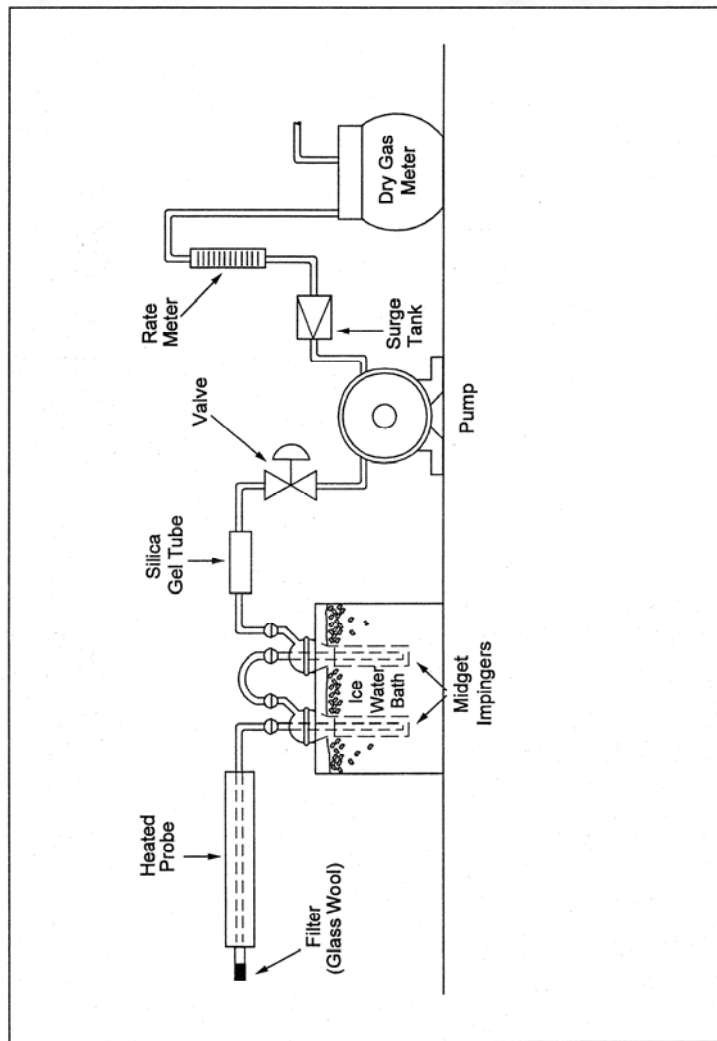


Figure 4-2. Moisture Sampling Train - Approximation Method.

Environmental Protection Agency

Pt. 60, App. A-3, Meth. 4

Plant _____	Run No. _____
Location _____	Ambient temperature _____
Operator _____	Barometric pressure _____
Date _____	Probe Length _____

Traverse Pt. No.	Sampling time (Δ), min	Stack temperature °C (°F)	Pressure differential across orifice meter ΔH mm (in.) H ₂ O	Meter reading gas sample volume m ³ (ft ³)	ΔV_m m ³ (ft ³)	Gas sample temperature at dry gas meter		Temperature of gas leaving condenser or last impinger °C (°F)
						Inlet $T_{m,in}$ °C (°F)	Outlet $T_{m,out}$ °C (°F)	
Average								

Location _____ Comments: _____
 Test _____
 Date _____
 Operator _____
 Barometric pressure _____

Figure 4-3. Moisture Determination—Reference Method

Clock time	Gas Volume through meter, (V _m), m ³ (ft ³)	Rate meter setting m ³ /min (ft ³ /min)	Meter temperature °C (°F)

Figure 4-4. Example Moisture Determination Field Data Sheet—Approximation Method

	Impinger volume, ml	Silica gel weight, g
Final		
Initial		
Difference		

Figure 4-5. Analytical Data—Reference Method

METHOD 5—DETERMINATION OF 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.

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 stationary sources.

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 tem-

perature of 120 ±14 °C (248 ±25 °F) or such other temperature as specified by an applicable subpart of the standards or approved by the Administrator for a particular application. The PM 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 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. A schematic of the sampling train used in this method is shown in Figure 5-1 in section 18.0. Complete construction details are given in APTD-0581 (Reference 2 in section 17.0); commercial

models of this train are also available. For changes from APTD-0581 and for allowable modifications of the train shown in Figure 5-1, see the following subsections.

NOTE: The operating and maintenance procedures for the sampling train are described in APTD-0576 (Reference 3 in section 17.0). Since correct usage is important in obtaining valid results, all users should read APTD-0576 and adopt the operating and maintenance procedures outlined in it, unless otherwise specified herein.

6.1.1.1 Probe Nozzle. Stainless steel (316) or glass with a sharp, tapered leading edge. The angle of taper shall be $\leq 30^\circ$, and the taper shall be on the outside to preserve a constant internal diameter. The probe nozzle shall be of the button-hook or elbow design, unless otherwise specified by the Administrator. If made of stainless steel, the nozzle shall be constructed from seamless tubing. Other materials of construction may be used, subject to the approval of the Administrator. A range of nozzle sizes suitable for isokinetic sampling should be available. Typical nozzle sizes range from 0.32 to 1.27 cm ($\frac{1}{8}$ to $\frac{1}{2}$ in) inside diameter (ID) in increments of 0.16 cm ($\frac{1}{16}$ in). Larger nozzle sizes are also available if higher volume sampling trains are used. Each nozzle shall be calibrated, according to the procedures outlined in section 10.1.

6.1.1.2 Probe Liner. Borosilicate or quartz glass tubing with a heating system capable of maintaining a probe gas temperature during sampling of $120 \pm 14^\circ\text{C}$ ($248 \pm 25^\circ\text{F}$), or such other temperature as specified by an applicable subpart of the standards or as approved by the Administrator for a particular application. Since the actual temperature at the outlet of the probe is not usually monitored during sampling, probes constructed according to APTD-0581 and utilizing the calibration curves of APTD-0576 (or calibrated according to the procedure outlined in APTD-0576) will be considered acceptable. Either borosilicate or quartz glass probe liners may be used for stack temperatures up to about 480°C (900°F); quartz glass liners shall be used for temperatures between 480 and 900°C (900 and $1,650^\circ\text{F}$). Both types of liners may be used at higher temperatures than specified for short periods of time, subject to the approval of the Administrator. The softening temperature for borosilicate glass is 820°C (1500°F), and for quartz glass it is 1500°C (2700°F). Whenever practical, every effort should be made to use borosilicate or quartz glass probe liners. Alternatively, metal liners (e.g., 316 stainless steel, Incoloy 825 or other corrosion resistant metals) made of seamless tubing may be used, subject to the approval of the Administrator.

6.1.1.3 Pitot Tube. Type S, as described in section 6.1 of Method 2, or other device approved by the Administrator. The pitot tube shall be attached to the probe (as shown in

Figure 5-1) to allow constant monitoring of the stack gas velocity. The impact (high pressure) opening plane of the pitot tube shall be even with or above the nozzle entry plane (see Method 2, Figure 2-7) during sampling. The Type S pitot tube assembly shall have a known coefficient, determined as outlined in section 10.0 of Method 2.

6.1.1.4 Differential Pressure Gauge. Inclined manometer or equivalent device (two), as described in section 6.2 of Method 2. One manometer shall be used for velocity head (Δp) readings, and the other, for orifice differential pressure readings.

6.1.1.5 Filter Holder. Borosilicate glass, with a glass or Teflon frit filter support and a silicone rubber gasket. Other materials of construction (e.g., stainless steel or Viton) may be used, subject to the approval of the Administrator. The holder design shall provide a positive seal against leakage from the outside or around the filter. The holder shall be attached immediately at the outlet of the probe (or cyclone, if used).

6.1.1.6 Filter Heating System. Any heating system capable of monitoring and maintaining temperature around the filter shall be used to ensure the sample gas temperature exiting the filter of $120 \pm 14^\circ\text{C}$ ($248 \pm 25^\circ\text{F}$) during sampling or such other temperature as specified by an applicable subpart of the standards or approved by the Administrator for a particular application. The monitoring and regulation of the temperature around the filter may be done with the filter temperature sensor or another temperature sensor.

6.1.1.7 Filter Temperature Sensor. A temperature sensor capable of measuring temperature to within $\pm 3^\circ\text{C}$ (5.4°F) shall be installed so that the sensing tip of the temperature sensor is in direct contact with the sample gas exiting the filter. The sensing tip of the sensor may be encased in glass, Teflon, or metal and must protrude at least $\frac{1}{2}$ in. into the sample gas exiting the filter. The filter temperature sensor must be monitored and recorded during sampling to ensure a sample gas temperature exiting the filter of $120 \pm 14^\circ\text{C}$ ($248 \pm 25^\circ\text{F}$), or such other temperature as specified by an applicable subpart of the standards or approved by the Administrator for a particular application.

6.1.1.8 Condenser. The following system shall be used to determine the stack gas moisture content: Four impingers connected in series with leak-free ground glass fittings or any similar leak-free noncontaminating fittings. The first, third, and fourth impingers shall be of the Greenburg-Smith design, modified by replacing the tip with a 1.3 cm ($\frac{1}{2}$ in.) ID glass tube extending to about 1.3 cm ($\frac{1}{2}$ in.) from the bottom of the flask. The second impinger shall be of the Greenburg-Smith design with the standard

tip. Modifications (e.g., using flexible connections between the impingers, using materials other than glass, or using flexible vacuum lines to connect the filter holder to the condenser) may be used, subject to the approval of the Administrator. The first and second impingers shall contain known quantities of water (Section 8.3.1), the third shall be empty, and the fourth shall contain a known weight of silica gel, or equivalent desiccant. A temperature sensor, capable of measuring temperature to within 1 °C (2 °F) shall be placed at the outlet of the fourth impinger for monitoring purposes. Alternatively, any system that cools the sample gas stream and allows measurement of the water condensed and moisture leaving the condenser, each to within 1 ml or 1 g may be used, subject to the approval of the Administrator. An acceptable technique involves the measurement of condensed water either gravimetrically or volumetrically and the determination of the moisture leaving the condenser by: (1) monitoring the temperature and pressure at the exit of the condenser and using Dalton's law of partial pressures; or (2) passing the sample gas stream through a tared silica gel (or equivalent desiccant) trap with exit gases kept below 20 °C (68 °F) and determining the weight gain. If means other than silica gel are used to determine the amount of moisture leaving the condenser, it is recommended that silica gel (or equivalent) still be used between the condenser system and pump to prevent moisture condensation in the pump and metering devices and to avoid the need to make corrections for moisture in the metered volume.

NOTE: If a determination of the PM collected in the impingers is desired in addition to moisture content, the impinger system described above shall be used, without modification. Individual States or control agencies requiring this information shall be contacted as to the sample recovery and analysis of the impinger contents.

6.1.1.9 Metering System. Vacuum gauge, leak-free pump, calibrated temperature sensors, dry gas meter (DGM) capable of measuring volume to within 2 percent, and related equipment, as shown in Figure 5-1. Other metering systems capable of maintaining sampling rates within 10 percent of isokinetic and of determining sample volumes to within 2 percent may be used, subject to the approval of the Administrator. When the metering system is used in conjunction with a pitot tube, the system shall allow periodic checks of isokinetic rates.

6.1.1.10 Sampling trains utilizing metering systems designed for higher flow rates than that described in APTD-0581 or APTD-0576 may be used provided that the specifications of this method are met.

6.1.2 Barometer. Mercury, aneroid, or other barometer capable of measuring atmospheric pressure to within 2.5 mm Hg (0.1 in.).

NOTE: The barometric pressure reading may be obtained from a nearby National Weather Service station. In this case, the station value (which is the absolute barometric pressure) shall be requested and an adjustment for elevation differences between the weather station and sampling point shall be made at a rate of minus 2.5 mm Hg (0.1 in.) per 30 m (100 ft) elevation increase or plus 2.5 mm Hg (0.1 in.) per 30 m (100 ft) elevation decrease.

6.1.3 Gas Density Determination Equipment. Temperature sensor and pressure gauge, as described in sections 6.3 and 6.4 of Method 2, and gas analyzer, if necessary, as described in Method 3. The temperature sensor shall, preferably, be permanently attached to the pitot tube or sampling probe in a fixed configuration, such that the tip of the sensor extends beyond the leading edge of the probe sheath and does not touch any metal. Alternatively, the sensor may be attached just prior to use in the field. Note, however, that if the temperature sensor is attached in the field, the sensor must be placed in an interference-free arrangement with respect to the Type S pitot tube openings (see Method 2, Figure 2-4). As a second alternative, if a difference of not more than 1 percent in the average velocity measurement is to be introduced, the temperature sensor need not be attached to the probe or pitot tube. (This alternative is subject to the approval of the Administrator.)

6.2 Sample Recovery. The following items are required for sample recovery:

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

6.2.2 Wash Bottles. Two Glass wash bottles are recommended. Alternatively, polyethylene wash bottles may be used. It is recommended that acetone not be stored in polyethylene bottles for longer than a month.

6.2.3 Glass Sample Storage Containers. Chemically resistant, borosilicate glass bottles, for acetone washes, 500 ml or 1000 ml. Screw cap liners shall either be rubber-backed Teflon or shall be constructed so as to be leak-free and resistant to chemical attack by acetone. (Narrow mouth glass bottles have been found to be less prone to leakage.) Alternatively, polyethylene bottles may be used.

6.2.4 Petri Dishes. For filter samples; glass or polyethylene, unless otherwise specified by the Administrator.

6.2.5 Graduated Cylinder and/or Balance. To measure condensed water to within 1 ml or 0.5 g. Graduated cylinders shall have subdivisions no greater than 2 ml.

6.2.6 Plastic Storage Containers. Air-tight containers to store silica gel.

6.2.7 Funnel and Rubber Policeman. To aid in transfer of silica gel to container; not necessary if silica gel is weighed in the field.

6.2.8 Funnel. Glass or polyethylene, to aid in sample recovery.

6.3 Sample Analysis. The following equipment is required for sample analysis:

6.3.1 Glass Weighing Dishes.

6.3.2 Desiccator.

6.3.3 Analytical Balance. To measure to within 0.1 mg.

6.3.4 Balance. To measure to within 0.5 g.

6.3.5 Beakers. 250 ml.

6.3.6 Hygrometer. To measure the relative humidity of the laboratory environment.

6.3.7 Temperature Sensor. To measure the temperature of the laboratory environment.

7.0 Reagents and Standards

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

7.1.1 Filters. Glass fiber filters, without organic binder, exhibiting at least 99.95 percent efficiency (<0.05 percent penetration) on 0.3 micron dioctyl phthalate smoke particles. The filter efficiency test shall be conducted in accordance with ASTM Method D 2986-71, 78, or 95a (incorporated by reference—see §60.17). Test data from the supplier's quality control program are sufficient for this purpose. In sources containing SO₂ or SO₃, the filter material must be of a type that is unreactive to SO₂ or SO₃. Reference 10 in section 17.0 may be used to select the appropriate filter.

7.1.2 Silica Gel. Indicating type, 6 to 16 mesh. If previously used, dry at 175 °C (350 °F) for 2 hours. New silica gel may be used as received. Alternatively, other types of desiccants (equivalent or better) may be used, subject to the approval of the Administrator.

7.1.3 Water. When analysis of the material caught in the impingers is required, deionized distilled water [to conform to ASTM D1193-77 or 91 Type 3 (incorporated by reference—see §60.17)] with at least <0.001 percent residue shall be used or as specified in the applicable method requiring analysis of the water. Run reagent blanks prior to field use to eliminate a high blank on test samples.

7.1.4 Crushed Ice.

7.2 Sample Recovery. Acetone, reagent grade, <0.001 percent residue, in glass bottles, is required. 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 shall be run prior to field use and only acetone with low

blank values (<0.001 percent) shall be used. In no case shall a blank value of greater than 0.001 percent of the weight of acetone used be subtracted from the sample weight.

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

7.3.1 Acetone. Same as in section 7.2.

7.3.2 Desiccant. Anhydrous calcium sulfate, indicating type. Alternatively, other types of desiccants may be used, subject to the approval of the Administrator.

8.0 Sample Collection, Preservation, Storage, and Transport

8.1 Pretest Preparation. It is suggested that sampling equipment be maintained according to the procedures described in APTD-0576. Alternative mercury-free thermometers may be used if the thermometers are at a minimum equivalent in terms of performance or suitably effective for the specific temperature measurement application.

8.1.1 Place 200 to 300 g of silica gel in each of several air-tight containers. Weigh each container, including silica gel, to the nearest 0.5 g, and record this weight. As an alternative, the silica gel need not be preweighed, but may be weighed directly in its impinger or sampling holder just prior to train assembly.

8.1.2 Check filters visually against light for irregularities, flaws, or pinhole leaks. Label filters of the proper diameter on the back side near the edge using numbering machine ink. As an alternative, label the shipping containers (glass or polyethylene petri dishes), and keep each filter in its identified container at all times except during sampling.

8.1.3 Desiccate the filters at 20 ±5.6 °C (68 ±10 °F) and ambient pressure for at least 24 hours. Weigh each filter (or filter and shipping container) at intervals of at least 6 hours to a constant weight (*i.e.*, <0.5 mg change from previous weighing). Record results to the nearest 0.1 mg. During each weighing, the period for which the filter is exposed to the laboratory atmosphere shall be less than 2 minutes. Alternatively (unless otherwise specified by the Administrator), the filters may be oven dried at 105 °C (220 °F) for 2 to 3 hours, desiccated for 2 hours, and weighed. Procedures other than those described, which account for relative humidity effects, may be used, subject to the approval of the Administrator.

8.2 Preliminary Determinations.

8.2.1 Select the sampling site and the minimum number of sampling points according to Method 1 or as specified by the Administrator. Determine the stack pressure, temperature, and the range of velocity heads using Method 2; it is recommended that a leak check of the pitot lines (see Method 2, section 8.1) be performed. Determine the moisture content using Approximation Method 4 or its alternatives for the purpose

of making isokinetic sampling rate settings. Determine the stack gas dry molecular weight, as described in Method 2, section 8.6; if integrated Method 3 sampling is used for molecular weight determination, the integrated bag sample shall be taken simultaneously with, and for the same total length of time as, the particulate sample run.

8.2.2 Select a nozzle size based on the range of velocity heads, such that it is not necessary to change the nozzle size in order to maintain isokinetic sampling rates. During the run, do not change the nozzle size. Ensure that the proper differential pressure gauge is chosen for the range of velocity heads encountered (see section 8.3 of Method 2).

8.2.3 Select a suitable probe liner and probe length such that all traverse points can be sampled. For large stacks, consider sampling from opposite sides of the stack to reduce the required probe length.

8.2.4 Select a total sampling time greater than or equal to the minimum total sampling time specified in the test procedures for the specific industry such that (1) the sampling time per point is not less than 2 minutes (or some greater time interval as specified by the Administrator), and (2) the sample volume taken (corrected to standard conditions) will exceed the required minimum total gas sample volume. The latter is based on an approximate average sampling rate.

8.2.5 The sampling time at each point shall be the same. It is recommended that the number of minutes sampled at each point be an integer or an integer plus one-half minute, in order to avoid timekeeping errors.

8.2.6 In some circumstances (*e.g.*, batch cycles) it may be necessary to sample for shorter times at the traverse points and to obtain smaller gas sample volumes. In these cases, the Administrator's approval must first be obtained.

8.3 Preparation of Sampling Train.

8.3.1 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. Place 100 ml of water in each of the first two impingers, leave the third impinger empty, and transfer approximately 200 to 300 g of preweighed silica gel from its container to the fourth impinger. More silica gel may be used, but care should be taken to ensure that it is not entrained and carried out from the impinger during sampling. Place the container in a clean place for later use in the sample recovery. Alternatively, the weight of the silica gel plus impinger may be determined to the nearest 0.5 g and recorded.

8.3.2 Using a tweezer or clean disposable surgical gloves, place a labeled (identified) and weighed filter in the filter holder. Be sure that the filter is properly centered and

the gasket properly placed so as to prevent the sample gas stream from circumventing the filter. Check the filter for tears after assembly is completed.

8.3.3 When glass probe liners are used, install the selected nozzle using a Viton A O-ring when stack temperatures are less than 260 °C (500 °F) or a heat-resistant string gasket when temperatures are higher. See APTD-0576 for details. Other connecting systems using either 316 stainless steel or Teflon ferrules may be used. When metal liners are used, install the nozzle as discussed above or by a leak-free direct mechanical connection. Mark the probe with heat resistant tape or by some other method to denote the proper distance into the stack or duct for each sampling point.

8.3.4 Set up the train as shown in Figure 5-1 ensuring that the connections are leak-tight. Subject to the approval of the Administrator, a glass cyclone may be used between the probe and filter holder when the total particulate catch is expected to exceed 100 mg or when water droplets are present in the stack gas.

8.3.5 Place crushed ice around the impingers.

8.4 Leak-Check Procedures.

8.4.1 Leak Check of Metering System Shown in Figure 5-1. That portion of the sampling train from the pump to the orifice meter should be leak-checked prior to initial use and after each shipment. Leakage after the pump will result in less volume being recorded than is actually sampled. The following procedure is suggested (see Figure 5-2): Close the main valve on the meter box. Insert a one-hole rubber stopper with rubber tubing attached into the orifice exhaust pipe. Disconnect and vent the low side of the orifice manometer. Close off the low side orifice tap. Pressurize the system to 13 to 18 cm (5 to 7 in.) water column by blowing into the rubber tubing. Pinch off the tubing, and observe the manometer for one minute. A loss of pressure on the manometer indicates a leak in the meter box; leaks, if present, must be corrected.

8.4.2 Pretest Leak Check. A pretest leak check of the sampling train is recommended, but not required. If the pretest leak check is conducted, the following procedure should be used.

8.4.2.1 After the sampling train has been assembled, turn on and set the filter and probe heating systems to the desired operating temperatures. Allow time for the temperatures to stabilize. If a Viton A O-ring or other leak-free connection is used in assembling the probe nozzle to the probe liner, leak-check the train at the sampling site by plugging the nozzle and pulling a 380 mm (15 in.) Hg vacuum.

NOTE: A lower vacuum may be used, provided that it is not exceeded during the test.

8.4.2.2 If a heat-resistant string is used, do not connect the probe to the train during the leak check. Instead, leak-check the train by first plugging the inlet to the filter holder (cyclone, if applicable) and pulling a 380 mm (15 in.) Hg vacuum (see note in section 8.4.2.1). Then connect the probe to the train, and leak-check at approximately 25 mm (1 in.) Hg vacuum; alternatively, the probe may be leak-checked with the rest of the sampling train, in one step, at 380 mm (15 in.) Hg vacuum. Leakage rates in excess of 4 percent of the average sampling rate or 0.00057 m³/min (0.020 cfm), whichever is less, are unacceptable.

8.4.2.3 The following leak-check instructions for the sampling train described in APTD-0576 and APTD-0581 may be helpful. Start the pump with the bypass valve fully open and the coarse adjust valve completely closed. Partially open the coarse adjust valve, and slowly close the bypass valve until the desired vacuum is reached. Do not reverse the direction of the bypass valve, as this will cause water to back up into the filter holder. If the desired vacuum is exceeded, either leak-check at this higher vacuum, or end the leak check and start over.

8.4.2.4 When the leak check is completed, first slowly remove the plug from the inlet to the probe, filter holder, or cyclone (if applicable), and immediately turn off the vacuum pump. This prevents the water in the impingers from being forced backward into the filter holder and the silica gel from being entrained backward into the third impinger.

8.4.3 Leak Checks During Sample Run. If, during the sampling run, a component (*e.g.*, filter assembly or impinger) change becomes necessary, a leak check shall be conducted immediately before the change is made. The leak check shall be done according to the procedure outlined in section 8.4.2 above, except that it shall be done at a vacuum equal to or greater than the maximum value recorded up to that point in the test. If the leakage rate is found to be no greater than 0.00057 m³/min (0.020 cfm) or 4 percent of the average sampling rate (whichever is less), the results are acceptable, and no correction will need to be applied to the total volume of dry gas metered; if, however, a higher leakage rate is obtained, either record the leakage rate and plan to correct the sample volume as shown in section 12.3 of this method, or void the sample run.

NOTE: Immediately after component changes, leak checks are optional. If such leak checks are done, the procedure outlined in section 8.4.2 above should be used.

8.4.4 Post-Test Leak Check. A leak check of the sampling train is mandatory at the conclusion of each sampling run. The leak check shall be performed in accordance with the procedures outlined in section 8.4.2, except that it shall be conducted at a vacuum

equal to or greater than the maximum value reached during the sampling run. If the leakage rate is found to be no greater than 0.00057 m³/min (0.020 cfm) or 4 percent of the average sampling rate (whichever is less), the results are acceptable, and no correction need be applied to the total volume of dry gas metered. If, however, a higher leakage rate is obtained, either record the leakage rate and correct the sample volume as shown in section 12.3 of this method, or void the sampling run.

8.5 Sampling Train Operation. During the sampling run, maintain an isokinetic sampling rate (within 10 percent of true isokinetic unless otherwise specified by the Administrator) and a sample gas temperature through the filter of 120 ±14 °C (248 ±25 °F) or such other temperature as specified by an applicable subpart of the standards or approved by the Administrator.

8.5.1 For each run, record the data required on a data sheet such as the one shown in Figure 5-3. Be sure to record the initial DGM reading. Record the DGM 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 indicated by Figure 5-3 at least once at each sample point during each time increment and additional readings when significant changes (20 percent variation in velocity head readings) necessitate additional adjustments in flow rate. Level and zero the manometer. Because the manometer level and zero may drift due to vibrations and temperature changes, make periodic checks during the traverse.

8.5.2 Clean the portholes prior to the test run to minimize the chance of collecting deposited material. To begin sampling, verify that the filter and probe heating systems are up to temperature, remove the nozzle cap, verify that the pitot tube and probe are properly positioned. Position the nozzle at the first traverse point with the tip pointing directly into the gas stream. Immediately start the pump, and adjust the flow to isokinetic conditions. Nomographs are available which aid in the rapid adjustment of the isokinetic sampling rate without excessive computations. These nomographs are designed for use when the Type S pitot tube coefficient (C_p) is 0.85 ±0.02, and the stack gas equivalent density [dry molecular weight (M_d)] is equal to 29 ±4. APTD-0576 details the procedure for using the nomographs. If C_p and M_d are outside the above stated ranges, do not use the nomographs unless appropriate steps (see Reference 7 in section 17.0) are taken to compensate for the deviations.

8.5.3 When the stack is under significant negative pressure (*i.e.*, height of impinger stem), take care to close the coarse adjust valve before inserting the probe into the stack to prevent water from backing into the

filter holder. If necessary, the pump may be turned on with the coarse adjust valve closed.

8.5.4 When the probe is in position, block off the openings around the probe and port-hole to prevent unrepresentative dilution of the gas stream.

8.5.5 Traverse the stack cross-section, as required by Method 1 or as specified by the Administrator, being careful not to bump the probe nozzle into the stack walls when sampling near the walls or when removing or inserting the probe through the portholes; this minimizes the chance of extracting deposited material.

8.5.6 During the test run, make periodic adjustments to keep the temperature around the filter holder at the proper level to maintain the sample gas temperature exiting the filter; add more ice and, if necessary, salt to maintain a temperature of less than 20 °C (68 °F) at the condenser/silica gel outlet. Also, periodically check the level and zero of the manometer.

8.5.7 If the pressure drop across the filter becomes too high, making isokinetic sampling difficult to maintain, the filter may be replaced in the midst of the 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 8.4.3). The total PM weight shall include the summation of the filter assembly catches.

8.5.8 A single train shall be used for the entire sample run, except in cases where simultaneous sampling is required in two or more separate ducts or at two or more different locations within the same duct, or in cases where equipment failure necessitates a change of trains. In all other situations, the use of two or more trains will be subject to the approval of the Administrator.

NOTE: When two or more trains are used, separate analyses of the front-half and (if applicable) impinger catches from each train shall be performed, unless identical nozzle sizes were used on all trains, in which case, the front-half catches from the individual trains may be combined (as may the impinger catches) and one analysis of front-half catch and one analysis of impinger catch may be performed. Consult with the Administrator for details concerning the calculation of results when two or more trains are used.

8.5.9 At the end of the sample run, close the coarse adjust valve, remove the probe and nozzle from the stack, turn off the pump, record the final DGM meter reading, and conduct a post-test leak check, as outlined in section 8.4.4. 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.

8.6 Calculation of Percent Isokinetic. Calculate percent isokinetic (see Calculations, section 12.11) to determine whether the run was valid or another test run should be made. If there was difficulty in maintaining isokinetic rates because of source conditions, consult with the Administrator for possible variance on the isokinetic rates.

8.7 Sample Recovery.

8.7.1 Proper cleanup procedure begins as soon as the probe is removed from the stack at the end of the sampling period. Allow the probe to cool.

8.7.2 When the probe can be safely handled, wipe off all external PM near the tip of the probe nozzle, and place a cap over it to prevent losing or gaining PM. Do not cap off the probe tip tightly while the sampling train is cooling down. This would create a vacuum in the filter holder, thereby drawing water from the impingers into the filter holder.

8.7.3 Before moving the sample train to the cleanup site, remove the probe from the sample train and cap the open outlet of the probe. Be careful not to lose any condensate that might be present. Cap the filter inlet where the probe was fastened. Remove the umbilical cord from the last impinger, and cap the impinger. If a flexible line is used between the first impinger or 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. Cap off the filter holder outlet and impinger inlet. Either ground-glass stoppers, plastic caps, or serum caps may be used to close these openings.

8.7.4 Transfer the probe and filter-impinger assembly to the cleanup area. This area should be clean and protected from the wind so that the chances of contaminating or losing the sample will be minimized.

8.7.5 Save a portion of the acetone used for cleanup as a blank. From each storage container of acetone used for cleanup, save 200 ml and place in a glass sample container labeled "acetone blank." To minimize any particulate contamination, rinse the wash bottle prior to filling from the tested container.

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

8.7.6.1 Container No. 1. Carefully remove the filter from the filter holder, and place it in its identified petri dish container. Use a pair of tweezers and/or clean disposable surgical gloves to handle the filter. If it is necessary to fold the filter, do so such that the PM cake is inside the fold. Using a dry Nylon bristle brush and/or a sharp-edged blade, carefully transfer to the petri dish any PM and/or filter fibers that adhere to the filter holder gasket. Seal the container.

8.7.6.2 Container No. 2. Taking care to see that dust on the outside of the probe or other

exterior surfaces does not get into the sample, quantitatively recover PM or any condensate from the probe nozzle, probe fitting, probe liner, and front half of the filter holder by washing these components with acetone and placing the wash in a glass container. Deionized distilled water may be used instead of acetone when approved by the Administrator and shall be used when specified by the Administrator. In these cases, save a water blank, and follow the Administrator's directions on analysis. Perform the acetone rinse as follows:

8.7.6.2.1 Carefully remove the probe nozzle. Clean the inside surface by rinsing with acetone from a wash bottle and brushing with a Nylon bristle brush. Brush until the acetone rinse shows no visible particles, after which make a final rinse of the inside surface with acetone.

8.7.6.2.2 Brush and rinse the inside parts of the fitting with acetone in a similar way until no visible particles remain.

8.7.6.2.3 Rinse the probe liner with acetone by tilting and rotating the probe while squirting acetone into its upper end so that all inside surfaces will be wetted with acetone. Let the acetone drain from the lower end into the sample container. A funnel (glass or polyethylene) may be used to aid in transferring liquid washes to the container. Follow the acetone rinse with a probe brush. Hold the probe in an inclined position, squirt acetone into the upper end as the probe brush is being pushed with a twisting action through the probe; hold a sample container underneath the lower end of the probe, and catch any acetone and particulate matter that is brushed from the probe. Run the brush through the probe three times or more until no visible PM is carried out with the acetone or until none remains in the probe liner on visual inspection. With stainless steel or other metal probes, run the brush through in the above prescribed manner at least six times since metal probes have small crevices in which particulate matter can be entrapped. Rinse the brush with acetone, and quantitatively collect these washings in the sample container. After the brushing, make a final acetone rinse of the probe.

8.7.6.2.4 It is recommended that two people clean the probe to minimize sample losses. Between sampling runs, keep brushes clean and protected from contamination.

8.7.6.2.5 Clean the inside of the front half of the filter holder by rubbing the surfaces with

a Nylon bristle brush and rinsing with acetone. Rinse each surface three times or more if needed to remove visible particulate. Make a final rinse of the brush and filter holder. Carefully rinse out the glass cyclone, also (if applicable). After all acetone washings and particulate matter have been collected in the sample container, tighten the lid on the sample container so that acetone will not leak out when it is shipped to the laboratory. Mark the height of the fluid level to allow determination of whether leakage occurred during transport. Label the container to clearly identify its contents.

8.7.6.3 Container No. 3. Note the color of the indicating silica gel to determine whether it has been completely spent, and make a notation of its condition. Transfer the silica gel from the fourth impinger to its original container, and seal. A funnel may make it easier to pour the silica gel without spilling. A rubber policeman may be used as an aid in removing the silica gel from the impinger. It is not necessary to remove the small amount of dust particles that may adhere to the impinger wall and are difficult to remove. Since the gain in weight is to be used for moisture calculations, do not use any water or other liquids to transfer the silica gel. If a balance is available in the field, follow the procedure for Container No. 3 in section 11.2.3.

8.7.6.4 Impinger Water. Treat the impingers as follows: Make a notation of any color or film in the liquid catch. Measure the liquid that 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. Record the volume or weight of liquid present. This information is required to calculate the moisture content of the effluent gas. Discard the liquid after measuring and recording the volume or weight, unless analysis of the impinger catch is required (see NOTE, section 6.1.1.8). If a different type of condenser is used, measure the amount of moisture condensed either volumetrically or gravimetrically.

8.8 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
8.4, 10.1-10.6	Sampling equipment leak check and calibration.	Ensures accurate measurement of stack gas flow rate, sample volume.

9.2 Volume Metering System Checks. The following procedures are suggested to check the volume metering system calibration val-

ues at the field test site prior to sample collection. These procedures are optional.

9.2.1 Meter Orifice Check. Using the calibration data obtained during the calibration procedure described in section 10.3, determine the $\Delta H@$ for the metering system orifice. The $\Delta H@$ is the orifice pressure differential in units of in. H₂O that correlates to 0.75 cfm of air at 528 °R and 29.92 in. Hg. The $\Delta H@$ is calculated as follows:

$$\Delta H@ = 0.0319 \Delta H \frac{T_m \theta^2}{P_{\text{bar}} Y^2 V_m^2}$$

Where:

ΔH = Average pressure differential across the orifice meter, in. H₂O.

T_m = Absolute average DGM temperature, °R.

P_{bar} = Barometric pressure, in. Hg.

θ = Total sampling time, min.

Y = DGM calibration factor, dimensionless.

V_m = Volume of gas sample as measured by DGM, dcf.

0.0319 = (0.0567 in. Hg/°R) (0.75 cfm)²

9.2.1.1 Before beginning the field test (a set of three runs usually constitutes a field test), operate the metering system (*i.e.*, pump, volume meter, and orifice) at the $\Delta H@$ pressure differential for 10 minutes. Record the volume collected, the DGM temperature, and the barometric pressure. Calculate a DGM calibration check value, Y_c , as follows:

$$Y_c = \frac{10}{V_m} \left[\frac{0.0319 T_m}{P_{\text{bar}}} \right]^{\frac{1}{2}}$$

where:

Y_c = DGM calibration check value, dimensionless.

10 = Run time, min.

9.2.1.2 Compare the Y_c value with the dry gas meter calibration factor Y to determine that: $0.97Y < Y_c < 1.03Y$. If the Y_c value is not within this range, the volume metering system should be investigated before beginning the test.

9.2.2 Calibrated Critical Orifice. A critical orifice, calibrated against a wet test meter or spirometer and designed to be inserted at the inlet of the sampling meter box, may be used as a check by following the procedure of section 16.2.

10.0 Calibration and Standardization

NOTE: Maintain a laboratory log of all calibrations.

10.1 Probe Nozzle. Probe nozzles shall be calibrated before their initial use in the field. Using a micrometer, measure the ID of the nozzle to the nearest 0.025 mm (0.001 in.). Make three separate measurements using different diameters each time, and obtain the average of the measurements. The difference between the high and low numbers shall not exceed 0.1 mm (0.004 in.). When noz-

zles become nicked, dented, or corroded, they shall be reshaped, sharpened, and recalibrated before use. Each nozzle shall be permanently and uniquely identified.

10.2 Pitot Tube Assembly. The Type S pitot tube assembly shall be calibrated according to the procedure outlined in section 10.1 of Method 2.

10.3 Metering System.

10.3.1 Calibration Prior to Use. Before its initial use in the field, the metering system shall be calibrated as follows: Connect the metering system inlet to the outlet of a wet test meter that is accurate to within 1 percent. Refer to Figure 5-4. The wet test meter should have a capacity of 30 liters/rev (1 ft³/rev). A spirometer of 400 liters (14 ft³) or more capacity, or equivalent, may be used for this calibration, although a wet test meter is usually more practical. The wet test meter should be periodically calibrated with a spirometer or a liquid displacement meter to ensure the accuracy of the wet test meter. Spirometers or wet test meters of other sizes may be used, provided that the specified accuracies of the procedure are maintained. Run the metering system pump for about 15 minutes with the orifice manometer indicating a median reading as expected in field use to allow the pump to warm up and to permit the interior surface of the wet test meter to be thoroughly wetted. Then, at each of a minimum of three orifice manometer settings, pass an exact quantity of gas through the wet test meter and note the gas volume indicated by the DGM. Also note the barometric pressure and the temperatures of the wet test meter, the inlet of the DGM, and the outlet of the DGM. Select the highest and lowest orifice settings to bracket the expected field operating range of the orifice. Use a minimum volume of 0.14 m³ (5 ft³) at all orifice settings. Record all the data on a form similar to Figure 5-5 and calculate Y , the DGM calibration factor, and ΔH , the orifice calibration factor, at each orifice setting as shown on Figure 5-5. Allowable tolerances for individual Y and ΔH values are given in Figure 5-5. Use the average of the Y values in the calculations in section 12.0.

10.3.1.1 Before calibrating the metering system, it is suggested that a leak check be conducted. For metering systems having diaphragm pumps, the normal leak-check procedure will not detect leakages within the pump. For these cases the following leak-check procedure is suggested: make a 10-minute calibration run at 0.00057 m³/min (0.020 cfm). At the end of the run, take the difference of the measured wet test meter and DGM volumes. Divide the difference by 10 to get the leak rate. The leak rate should not exceed 0.00057 m³/min (0.020 cfm).

10.3.2 Calibration After Use. After each field use, the calibration of the metering system shall be checked by performing three calibration runs at a single, intermediate

orifice setting (based on the previous field test), with the vacuum set at the maximum value reached during the test series. To adjust the vacuum, insert a valve between the wet test meter and the inlet of the metering system. Calculate the average value of the DGM calibration factor. If the value has changed by more than 5 percent, recalibrate the meter over the full range of orifice settings, as detailed in section 10.3.1.

NOTE: Alternative procedures (e.g., rechecking the orifice meter coefficient) may be used, subject to the approval of the Administrator.

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

10.4 Probe Heater Calibration. Use a heat source to generate air heated to selected temperatures that approximate those expected to occur in the sources to be sampled. Pass this air through the probe at a typical sample flow rate while measuring the probe inlet and outlet temperatures at various probe heater settings. For each air temperature generated, construct a graph of probe heating system setting versus probe outlet temperature. The procedure outlined in APTD-0576 can also be used. Probes constructed according to APTD-0581 need not be calibrated if the calibration curves in APTD-0576 are used. Also, probes with outlet temperature monitoring capabilities do not require calibration.

NOTE: The probe heating system shall be calibrated before its initial use in the field.

10.5 Temperature Sensors. Use the procedure in Section 10.3 of Method 2 to calibrate in-stack temperature sensors. Dial thermometers, such as are used for the DGM and condenser outlet, shall be calibrated against mercury-in-glass thermometers. An alternative mercury-free NIST-traceable thermometer may be used if the thermometer is, at a minimum, equivalent in terms of performance or suitably effective for the specific temperature measurement application. As an alternative, the following single-point calibration procedure may be used. After each test run series, check the accuracy (and, hence, the calibration) of each thermocouple system at ambient temperature, or any other temperature, within the range specified by the manufacturer, using a reference thermometer (either ASTM reference thermometer or a thermometer that has been calibrated against an ASTM reference thermometer). The temperatures of the thermocouple and reference thermometers shall agree to within ± 2 °F.

10.6 Barometer. Calibrate against a mercury barometer or NIST-traceable barometer prior to the field test. Alternatively, barometric pressure may be obtained from a weather report that has been adjusted for the test point (on the stack) elevation.

10.7 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 ± 0.5 g of the certified mass. If the daily balance calibration check fails, perform corrective measures and repeat the check before using balance.

10.8 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 5-6.

11.2 Handle each sample container as follows:

11.2.1 Container No. 1. Leave the contents in the shipping container or transfer the filter and any loose PM from the sample container to a tared glass weighing dish. Desiccate for 24 hours in a desiccator containing anhydrous calcium sulfate. Weigh to a constant weight, and report the results to the nearest 0.1 mg. For the purposes of this section, the term "constant weight" means a difference of no more than 0.5 mg or 1 percent of total weight less tare weight, whichever is greater, between two consecutive weighings, with no less than 6 hours of desiccation time between weighings. Alternatively, the sample may be oven dried at 104 °C (220 °F) for 2 to 3 hours, cooled in the desiccator, and weighed to a constant weight, unless otherwise specified by the Administrator. The sample may be oven dried at 104 °C (220 °F) for 2 to 3 hours. Once the

sample has cooled, weigh the sample, and use this weight as a final weight.

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

11.2.3 Container No. 3. Weigh the spent silica gel (or silica gel plus impinger) to the nearest 0.5 g using a balance. This step may be conducted in the field.

11.2.4 Acetone Blank Container. Measure the acetone in this container either volumetrically or gravimetrically. Transfer the acetone to a tared 250 ml beaker, and evaporate to dryness at ambient temperature and pressure. Desiccate for 24 hours, and weigh to a constant weight. Report the results to the nearest 0.1 mg.

NOTE: The contents of Container No. 2 as well as the acetone blank container may be evaporated at temperatures higher than ambient. If evaporation is done at an elevated temperature, the temperature must be below the boiling point of the solvent; also, to prevent "bumping," the evaporation process must be closely supervised, and the contents of the beaker must be swirled occasionally to maintain an even temperature. Use extreme care, as acetone is highly flammable and has a low flash point.

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, provided that they give equivalent results.

12.1 Nomenclature.

A_n = Cross-sectional area of nozzle, m^2 (ft^2).
 B_{ws} = Water vapor in the gas stream, proportion by volume.
 C_a = Acetone blank residue concentration, mg/mg.
 c_p = Concentration of particulate matter in stack gas, dry basis, corrected to standard conditions, g/dscm ($gr/dscf$).
 I = Percent of isokinetic sampling.
 L_i = Individual leakage rate observed during the leak-check conducted prior to the first component change, m^3/min (ft^3/min)
 L_m = Maximum acceptable leakage rate for either a pretest leak-check or for a leak-check following a component change; equal to 0.00057 m^3/min (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 prior to the " i^{th} " component change ($i = 1, 2, 3 \dots n$), m^3/min (cfm).
 L_p = Leakage rate observed during the post-test leak-check, m^3/min (cfm).
 m_a = Mass of residue of acetone 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).
 P_s = Absolute stack gas pressure, mm Hg (in. Hg).
 P_{std} = Standard absolute pressure, 760 mm Hg (29.92 in. Hg).
 R = Ideal gas constant, $0.06236 ((mm\ Hg)(m^3))/((K)(g\text{-mole}))$ [$21.85 ((in.\ Hg)\ (ft^3))/((^\circ R)(lb\text{-mole}))$].
 T_m = Absolute average DGM temperature (see Figure 5-3), K ($^\circ R$).
 T_s = Absolute average stack gas temperature (see Figure 5-3), K ($^\circ R$).
 T_{std} = Standard absolute temperature, 293 K (528 $^\circ R$).
 V_a = Volume of acetone blank, ml.
 V_{aw} = Volume of acetone used in wash, ml.
 V_{lc} = Total volume of liquid collected in impingers and silica gel (see Figure 5-6), ml.
 V_m = Volume of gas sample as measured by dry gas meter, dcm ($dscf$).
 $V_{m(std)}$ = Volume of gas sample measured by the dry gas meter, corrected to standard conditions, dscm ($dscf$).
 $V_{w(std)}$ = Volume of water vapor in the gas sample, corrected to standard conditions, scm (scf).
 V_s = Stack gas velocity, calculated by Method 2, Equation 2-7, using data obtained from Method 5, m/sec (ft/sec).
 W_a = Weight of residue in acetone wash, mg.
 Y = Dry gas meter calibration factor.
 ΔH = Average pressure differential across the orifice meter (see Figure 5-4), mm H₂O (in. H₂O).
 ρ_a = Density of acetone, mg/ml (see label on bottle).
 ρ_w = Density of water, 0.9982 g/ml. (0.002201 lb/ml).
 θ = Total sampling time, min.
 θ_1 = Sampling time interval, from the beginning of a run until the first component change, min.
 θ_2 = Sampling time interval, between two successive component changes, beginning with the interval between the first and second changes, min.
 θ_n = Sampling time interval, from the final (n^{th}) component change until the end of the sampling run, min.
13.6 = Specific gravity of mercury.
60 = Sec/min.

100 = Conversion to percent.

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

12.3 Dry Gas Volume. Correct the sample volume measured by the dry gas meter to standard conditions (20 °C, 760 mm Hg or 68 °F, 29.92 in. Hg) by using Equation 5-1.

$$V_{m(std)} = V_m Y \frac{T_{std} \left(P_{bar} + \frac{\Delta H}{13.6} \right)}{T_m P_{std}} \quad \text{Eq. 5-1}$$

$$= K_1 V_m Y \frac{P_{bar} + \left(\frac{\Delta H}{13.6} \right)}{T_m}$$

Where:

$K_1 = 0.3858 \text{ } ^\circ\text{K/mm Hg}$ for metric units, $= 17.64 \text{ } ^\circ\text{R/in. Hg}$ for English units.

NOTE: Equation 5-1 can be used as written unless the leakage rate observed during any of the mandatory leak checks (*i.e.*, the post-test leak check or leak checks conducted prior to component changes) exceeds L_a . If L_p or L_i exceeds L_a , Equation 5-1 must be modified as follows:

(a) Case I. No component changes made during sampling run. In this case, replace V_m in Equation 5-1 with the expression:

$$\left(V_m - (L_p - L_a) \theta \right)$$

(b) Case II. One or more component changes made during the sampling run. In this case, replace V_m in Equation 5-1 by the expression:

$$\left[V_m - (L_1 - L_a) \theta_1 - \sum_{i=2}^n (L_i - L_a) \theta_i - (L_p - L_a) \theta_p \right]$$

and substitute only for those leakage rates (L_i or L_p) which exceed L_a .

12.4 Volume of Water Vapor Condensed.

$$V_{w(std)} = V_{1c} \frac{\rho_w R T_{std}}{M_w P_{std}} \quad \text{Eq. 5-2}$$

$$= K_2 V_{1c}$$

Where:

$K_2 = 0.001333 \text{ m}^3/\text{ml}$ for metric units, $= 0.04706 \text{ ft}^3/\text{ml}$ for English units.

12.5 Moisture Content.

$$B_{ws} = \frac{V_{w(std)}}{V_{m(std)} + V_{w(std)}} \quad \text{Eq. 5-3}$$

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 analysis (Equation 5-3), and a second from the assumption of saturated conditions. The lower of the two values

of B_{ws} , shall be considered correct. The procedure for determining the moisture content based upon the assumption of saturated conditions is given in section 4.0 of Method 4. For the purposes of this method, the average stack gas temperature from Figure 5-3 may be used to make this determination, provided that the accuracy of the in-stack temperature sensor is $\pm 1 \text{ } ^\circ\text{C}$ ($2 \text{ } ^\circ\text{F}$).

12.6 Acetone Blank Concentration.

$$C_a = \frac{m_a}{V_a \rho_a} \quad \text{Eq. 5-4}$$

12.7 Acetone Wash Blank.

$$W_a = C_a V_{3w} \rho_a \quad \text{Eq. 5-5}$$

12.8 Total Particulate Weight. Determine the total particulate matter catch from the sum of the weights obtained from Containers 1 and 2 less the acetone blank (see Figure 5-6).

NOTE: In no case shall a blank value of greater than 0.001 percent of the weight of acetone used be subtracted from the sample weight. Refer to section 8.5.8 to assist in calculation of results involving two or more filter assemblies or two or more sampling trains.

12.9 Particulate Concentration.

$$C_s = \frac{K_3 m_n}{V_{m(std)}} \quad \text{Eq. 5-6}$$

Where:

$K_3 = 0.001 \text{ g/mg}$ for metric units.
 $= 0.0154 \text{ gr/mg}$ for English units.

12.10 Conversion Factors:

From	To	Multiply by
ft ³	m ³	0.02832
gr	mg	64.80004
gr/ft ³	mg/m ³	2288.4
mg	g	0.001
gr	lb	1.429×10^{-4}

12.11 Isokinetic Variation.

12.11.1 Calculation from Raw Data.

$$I = \frac{100 T_s \left[K_4 V_{lc} + \frac{(V_m Y)}{T_m} \left(P_{bar} + \frac{\Delta H}{13.6} \right) \right]}{60 \theta v_s P_s A_n} \quad \text{Eq. 5-7}$$

Where:

$K_4 = 0.003454 \text{ ((mm Hg)(m}^3\text{))/((ml)(}^\circ\text{K))}$ for metric units.
 $= 0.002669 \text{ ((in. Hg)(ft}^3\text{))/((ml)(}^\circ\text{R))}$ for English units.

12.11.2 Calculation from Intermediate Values.

$$I = \frac{T_s V_{m(std)} P_{std} 100}{T_{std} v_s \theta A_n P_s 60 (1 - B_{ws})} \quad \text{Eq. 5-8}$$

$$= K_5 \frac{T_s V_{m(std)}}{P_s v_s A_n \theta (1 - B_{ws})}$$

Where:

$K_5 = 4.320$ for metric units.
 $= 0.09450$ for English units.

12.11.3 Acceptable Results. If 90 percent $\leq I \leq 110$ percent, the results are acceptable. If the PM results are low in comparison to the standard, and 'I' is over 110 percent or less than 90 percent, the Administrator may opt to accept the results. Reference 4 in section 17.0 may be used to make acceptability judgments. If 'I' is judged to be unacceptable, reject the results, and repeat the sampling run.

12.12 Stack Gas Velocity and Volumetric Flow Rate. Calculate the average stack gas velocity and volumetric flow rate, if needed, using data obtained in this method and the equations in sections 12.3 and 12.4 of Method 2.

13.0 Method Performance [Reserved]

14.0 Pollution Prevention [Reserved]

15.0 Waste Management [Reserved]

16.0 Alternative Procedures

16.1 Dry Gas Meter as a Calibration Standard. A DGM may be used as a calibration standard for volume measurements in place of the wet test meter specified in section 10.3, provided that it is calibrated initially and recalibrated periodically as follows:

16.1.1 Standard Dry Gas Meter Calibration.

16.1.1.1. The DGM to be calibrated and used as a secondary reference meter should be of high quality and have an appropriately sized capacity (e.g., 3 liters/rev (0.1 ft³/rev)). A spirometer (400 liters (14 ft³) or more capacity), or equivalent, may be used for this calibration, although a wet test meter is usually more practical. The wet test meter should have a capacity of 30 liters/rev (1 ft³/rev) and capable of measuring volume to within 1.0 percent. Wet test meters should be checked against a spirometer or a liquid displacement meter to ensure the accuracy of the wet test meter. Spirometers or wet test meters of other sizes may be used, provided that the specified accuracies of the procedure are maintained.

16.1.1.2 Set up the components as shown in Figure 5-7, A spirometer, or equivalent, may be used in place of the wet test meter in the system. Run the pump for at least 5 minutes at a flow rate of about 10 liters/min (0.35 cfm) to condition the interior surface of the wet test meter. The pressure drop indicated by the manometer at the inlet side of the DGM should be minimized (no greater than 100 mm

H₂O (4 in. H₂O) at a flow rate of 30 liters/min (1 cfm)). This can be accomplished by using large diameter tubing connections and straight pipe fittings.

16.1.1.3 Collect the data as shown in the example data sheet (see Figure 5-8). Make triplicate runs at each of the flow rates and at no less than five different flow rates. The range of flow rates should be between 10 and 34 liters/min (0.35 and 1.2 cfm) or over the expected operating range.

16.1.1.4 Calculate flow rate, Q, for each run using the wet test meter volume, V_w, and the run time, θ. Calculate the DGM coefficient, Y_{ds}, for each run. These calculations are as follows:

$$Q = K_1 \frac{P_{\text{bar}} V_w}{(T_w + T_{\text{std}}) \theta} \quad \text{Eq. 5-9}$$

$$Y_{\text{ds}} = \frac{V_w (T_{\text{ds}} + T_{\text{std}}) P_{\text{bar}}}{V_{\text{ds}} (T_w + T_{\text{std}}) \left(P_{\text{bar}} + \frac{\Delta p}{13.6} \right)} \quad \text{Eq. 5-10}$$

Where:

K₁ = 0.3858 °C/mm Hg for metric units = 17.64 °F/in. Hg for English units.

V_w = Wet test meter volume, liter (ft³).

V_{ds} = Dry gas meter volume, liter (ft³).

T_{ds} = Average dry gas meter temperature, °C (°F).

T_{adj} = 273 °C for metric units = 460 °F for English units.

T_w = Average wet test meter temperature, °C (°F)

P_{bar} = Barometric pressure, mm Hg (in. Hg).

Δp = Dry gas meter inlet differential pressure, mm H₂O (in. H₂O).

θ = Run time, min.

16.1.1.5 Compare the three Y_{ds} values at each of the flow rates and determine the maximum and minimum values. The difference between the maximum and minimum values at each flow rate should be no greater than 0.030. Extra sets of triplicate runs may be made in order to complete this requirement. In addition, the meter coefficients should be between 0.95 and 1.05. If these specifications cannot be met in three sets of successive triplicate runs, the meter is not suitable as a calibration standard and should not be used as such. If these specifications are met, average the three Y_{ds} values at each flow rate resulting in no less than five average meter coefficients, Y_{ds}.

16.1.1.6 Prepare a curve of meter coefficient, Y_{ds}, versus flow rate, Q, for the DGM. This curve shall be used as a reference when the meter is used to calibrate other DGMs and to determine whether recalibration is required.

16.1.2 Standard Dry Gas Meter Recalibration.

16.1.2.1 Recalibrate the standard DGM against a wet test meter or spirometer annually or after every 200 hours of operation, whichever comes first. This requirement is valid provided the standard DGM is kept in

a laboratory and, if transported, cared for as any other laboratory instrument. Abuse to the standard meter may cause a change in the calibration and will require more frequent recalibrations.

16.1.2.2 As an alternative to full recalibration, a two-point calibration check may be made. Follow the same procedure and equipment arrangement as for a full recalibration, but run the meter at only two flow rates [suggested rates are 14 and 30 liters/min (0.5 and 1.0 cfm)]. Calculate the meter coefficients for these two points, and compare the values with the meter calibration curve. If the two coefficients are within 1.5 percent of the calibration curve values at the same flow rates, the meter need not be recalibrated until the next date for a recalibration check.

16.2 Critical Orifices As Calibration Standards. Critical orifices may be used as calibration standards in place of the wet test meter specified in section 16.1, provided that they are selected, calibrated, and used as follows:

16.2.1 Selection of Critical Orifices.

16.2.1.1 The procedure that follows describes the use of hypodermic needles or stainless steel needle tubings which have been found suitable for use as critical orifices. Other materials and critical orifice designs may be used provided the orifices act as true critical orifices (*i.e.*, a critical vacuum can be obtained, as described in section 16.2.2.3). Select five critical orifices that are appropriately sized to cover the range of flow rates between 10 and 34 liters/min (0.35 and 1.2 cfm) or the expected operating range. Two of the critical orifices should bracket the expected operating range. A minimum of three critical orifices will be needed to calibrate a Method 5 DGM; the other two critical

orifices can serve as spares and provide better selection for bracketing the range of operating flow rates. The needle sizes and tubing lengths shown in Table 5-1 in section 18.0 give the approximate flow rates.

16.2.1.2 These needles can be adapted to a Method 5 type sampling train as follows: Insert a serum bottle stopper, 13 by 20 mm sleeve type, into a 1/2-inch Swagelok (or equivalent) quick connect. Insert the needle into the stopper as shown in Figure 5-9.

16.2.2 Critical Orifice Calibration. The procedure described in this section uses the Method 5 meter box configuration with a DGM as described in section 6.1.1.9 to calibrate the critical orifices. Other schemes may be used, subject to the approval of the Administrator.

16.2.2.1 Calibration of Meter Box. The critical orifices must be calibrated in the same configuration as they will be used (*i.e.*, there should be no connections to the inlet of the orifice).

16.2.2.1.1 Before calibrating the meter box, leak check the system as follows: Fully open the coarse adjust valve, and completely close the by-pass valve. Plug the inlet. Then turn on the pump, and determine whether there is any leakage. The leakage rate shall be zero (*i.e.*, no detectable movement of the DGM dial shall be seen for 1 minute).

16.2.2.1.2 Check also for leakages in that portion of the sampling train between the pump and the orifice meter. See section 8.4.1 for the procedure; make any corrections, if necessary. If leakage is detected, check for cracked gaskets, loose fittings, worn O-rings, etc., and make the necessary repairs.

16.2.2.1.3 After determining that the meter box is leakless, calibrate the meter box according to the procedure given in section 10.3. Make sure that the wet test meter

meets the requirements stated in section 16.1.1.1. Check the water level in the wet test meter. Record the DGM calibration factor, Y .

16.2.2.2 Calibration of Critical Orifices. Set up the apparatus as shown in Figure 5-10.

16.2.2.2.1 Allow a warm-up time of 15 minutes. This step is important to equilibrate the temperature conditions through the DGM.

16.2.2.2.2 Leak check the system as in section 16.2.2.1.1. The leakage rate shall be zero.

16.2.2.2.3 Before calibrating the critical orifice, determine its suitability and the appropriate operating vacuum as follows: Turn on the pump, fully open the coarse adjust valve, and adjust the by-pass valve to give a vacuum reading corresponding to about half of atmospheric pressure. Observe the meter box orifice manometer reading, ΔH . Slowly increase the vacuum reading until a stable reading is obtained on the meter box orifice manometer. Record the critical vacuum for each orifice. Orifices that do not reach a critical value shall not be used.

16.2.2.2.4 Obtain the barometric pressure using a barometer as described in section 6.1.2. Record the barometric pressure, P_{bar} , in mm Hg (in. Hg).

16.2.2.2.5 Conduct duplicate runs at a vacuum of 25 to 50 mm Hg (1 to 2 in. Hg) above the critical vacuum. The runs shall be at least 5 minutes each. The DGM volume readings shall be in increments of complete revolutions of the DGM. As a guideline, the times should not differ by more than 3.0 seconds (this includes allowance for changes in the DGM temperatures) to achieve ± 0.5 percent in K' (see Eq. 5-11). Record the information listed in Figure 5-11.

16.2.2.2.6 Calculate K' using Equation 5-11.

$$K' = \frac{K_i V_m Y \left(P_{\text{bar}} + \frac{\Delta H}{13.6} \right) T_{\text{amb}}^{1/2}}{P_{\text{bar}} T_m \theta} \quad \text{Eq. 5-11}$$

Where:

K' = Critical orifice coefficient,

$[(\text{m}^3)(^\circ\text{K})^{1/2}]$

$\{[(\text{mm Hg})(\text{min})] \{[(\text{ft}^3)(^\circ\text{R})^{1/2}]\} [(\text{in. Hg})(\text{min})]\}$

T_{amb} = Absolute ambient temperature, $^\circ\text{K}$ ($^\circ\text{R}$).

Calculate the arithmetic mean of the K' values. The individual K' values should not differ by more than ± 0.5 percent from the mean value.

16.2.3 Using the Critical Orifices as Calibration Standards.

16.2.3.1 Record the barometric pressure.

16.2.3.2 Calibrate the metering system according to the procedure outlined in section 16.2.2. Record the information listed in Figure 5-12.

16.2.3.3 Calculate the standard volumes of air passed through the DGM and the critical orifices, and calculate the DGM calibration factor, Y , using the equations below:

$$V_{m(std)} = \frac{K_1 V_m \left[P_{bar} + \left(\frac{\Delta H}{13.6} \right) \right]}{T_m} \quad \text{Eq. 5-12}$$

$$V_{cr(std)} = K' \frac{P_{bar} \theta}{\sqrt{T_{amb}}} \quad \text{Eq. 5-13}$$

$$Y = \frac{V_{cr(std)}}{V_{m(std)}} \quad \text{Eq. 5-14}$$

Where:

$V_{cr(std)}$ = Volume of gas sample passed through the critical orifice, corrected to standard conditions, dscm (dscf).

K_1 = 0.3858 K/mm Hg for metric units
= 17.64 °R/in. Hg for English units.

16.2.3.4 Average the DGM calibration values for each of the flow rates. The calibration factor, Y , at each of the flow rates should not differ by more than ± 2 percent from the average.

16.2.3.5 To determine the need for recalibrating the critical orifices, compare the DGM Y factors obtained from two adjacent orifices each time a DGM is calibrated; for example, when checking orifice 13/2.5, use orifices 12/10.2 and 13/5.1. If any critical orifice yields a DGM Y factor differing by more than 2 percent from the others, recalibrate the critical orifice according to section 16.2.2.

16.3 Alternative Post-Test Metering System Calibration. The following procedure

may be used as an alternative to the post-test calibration described in Section 10.3.2. This alternative procedure does not detect leakages between the inlet of the metering system and the dry gas meter. Therefore, two steps must be included to make it an equivalent alternative:

(1) The metering system must pass the post-test leak-check from either the inlet of the sampling train or the inlet of the metering system. Therefore, if the train fails the former leak-check, another leak-check from the inlet of the metering system must be conducted;

(2) The metering system must pass the leak-check of that portion of the train from the pump to the orifice meter as described in Section 8.4.1.

16.3.1 After each test run, do the following:

16.3.1.1 Ensure that the metering system has passed the post-test leak-check. If not, conduct a leak-check of the metering system from its inlet.

16.3.1.2 Conduct the leak-check of that portion of the train from the pump to the orifice meter as described in Section 10.3.1.1.

16.3.1.3 Calculate Y_{qa} for each test run using the following equation:

$$Y_{qa} = \frac{\theta}{V_m} \sqrt{\frac{0.0319 T_m}{\Delta H @ \left(P_{bar} + \frac{\Delta H_{avg}}{13.6} \right)}} \left(\frac{29}{M_d} \right) (\sqrt{\Delta H})_{avg} \quad \text{Eq. 5-15}$$

Where:

Y_{qa} = Dry gas meter calibration check value, dimensionless.

0.0319 = (29.92/528) (0.75)² (in. Hg/°R) cfm².

$\Delta H @$ = Orifice meter calibration coefficient, in. H₂O.

M_d = Dry molecular weight of stack gas, lb/lb-mole.

29 = Dry molecular weight of air, lb/lb-mole.

16.3.2 After each test run series, do the following:

16.3.2.1 Average the three or more Y_{qa} 's obtained from the test run series and compare this average Y_{qa} with the dry gas meter calibration factor Y . The average Y_{qa} must be within 5 percent of Y .

16.3.2.2 If the average Y_{qa} does not meet the 5 percent criterion, recalibrate the meter

over the full range of orifice settings as detailed in Section 10.3.1. Then follow the procedure in Section 10.3.3.

17.0 References.

1. Addendum to Specifications for Incinerator Testing at Federal Facilities. PHS, NCAPC, December 6, 1967.
2. Martin, Robert M. Construction Details of Isokinetic Source-Sampling Equipment. Environmental Protection Agency, Research Triangle Park, NC, APTD-0581, April 1971.
3. Rom, Jerome J. Maintenance, Calibration, and Operation of Isokinetic Source Sampling Equipment. Environmental Protection Agency, Research Triangle Park, NC, APTD-0576, March 1972.
4. Smith, W.S., R.T. Shigehara, and W.F. Todd. A Method of Interpreting Stack Sampling Data. Paper Presented at the 63rd Annual Meeting of the Air Pollution Control Association, St. Louis, MO, June 14-19, 1970.
5. Smith, W.S., et al. Stack Gas Sampling Improved and Simplified With New Equipment. APCA Paper No. 67-119, 1967.
6. Specifications for Incinerator Testing at Federal Facilities. PHS, NCAPC, 1967.
7. Shigehara, R.T. Adjustment in the EPA Nomograph for Different Pitot Tube Coefficients and Dry Molecular Weights. Stack Sampling News 2:4-11, October 1974.
8. Vollaro, R.F. A Survey of Commercially Available Instrumentation for the Measure-

ment of Low-Range Gas Velocities. U.S. Environmental Protection Agency, Emission Measurement Branch, Research Triangle Park, NC, November 1976 (unpublished paper).

9. Annual Book of ASTM Standards, Part 26. Gaseous Fuels; Coal and Coke; Atmospheric Analysis. American Society for Testing and Materials, Philadelphia, PA, 1974, pp. 617-622.
10. Felix, L.G., G.I. Clinard, G.E. Lacy, and J.D. McCain. Inertial Cascade Impactor Substrate Media for Flue Gas Sampling. U.S. Environmental Protection Agency, Research Triangle Park, NC 27711, Publication No. EPA-600/7-77-060, June 1977, 83 pp.
11. Westlin, P.R. and R.T. Shigehara. Procedure for Calibrating and Using Dry Gas Volume Meters as Calibration Standards. Source Evaluation Society Newsletter, 3(1):17-30, February 1978.
12. Lodge, J.P., Jr., J.B. Pate, B.E. Ammons, and G.A. Swanson. The Use of Hypodermic Needles as Critical Orifices in Air Sampling. J. Air Pollution Control Association, 16:197-200, 1966.
13. Shigehara, Roger T., P.G. Royals, and E.W. Steward. "Alternative Method 5 Post-Test Calibration." Entropy Incorporated, Research Triangle Park, NC 27709.

18.0 Tables, Diagrams, Flowcharts, and Validation Data

TABLE 5-1 FLOR RATES FOR VARIOUS NEEDLE SIZES AND TUBE LENGTHS

Gauge/cm	Flow rate liters/min.	Gauge/cm	Flow rate liters/min.
12/7.6	32.56	14/2.5	19.54
12/10.2	30.02	14/5.1	17.27
13/2.5	25.77	14/7.6	16.14
13/5.1	23.50	15/3.2	14.16
13/7.6	22.37	15/7.6	11.61
13/10.2	20.67	15/10.2	10.48

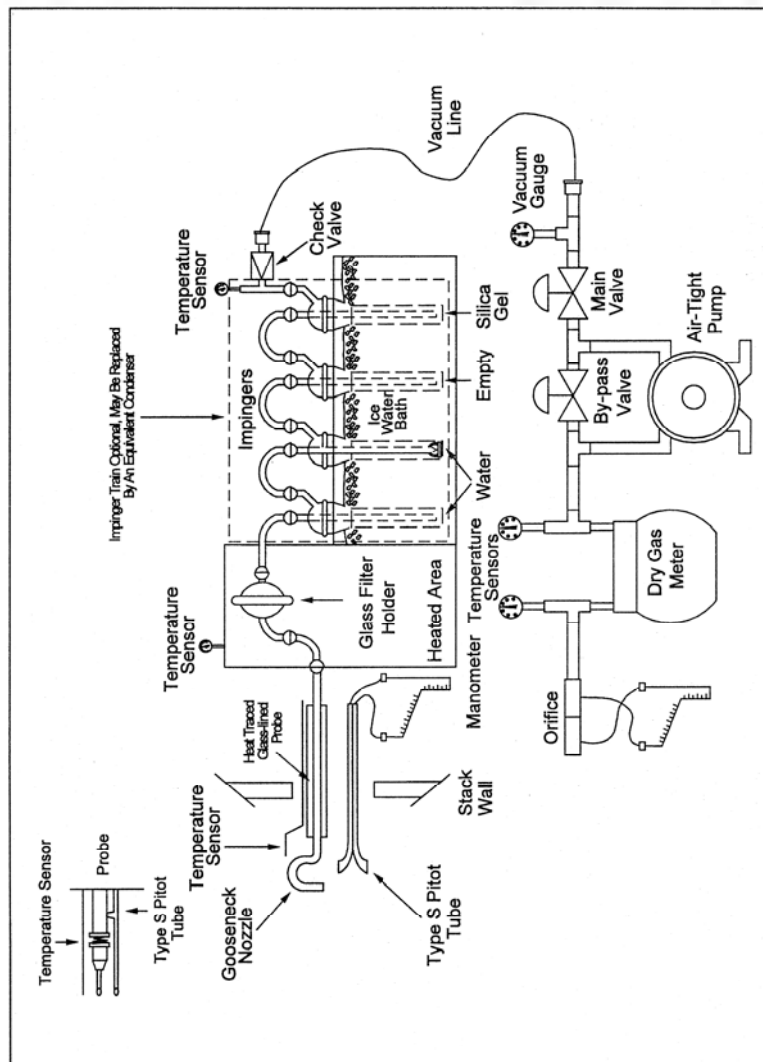


Figure 5-1. Particulate Sampling Train.

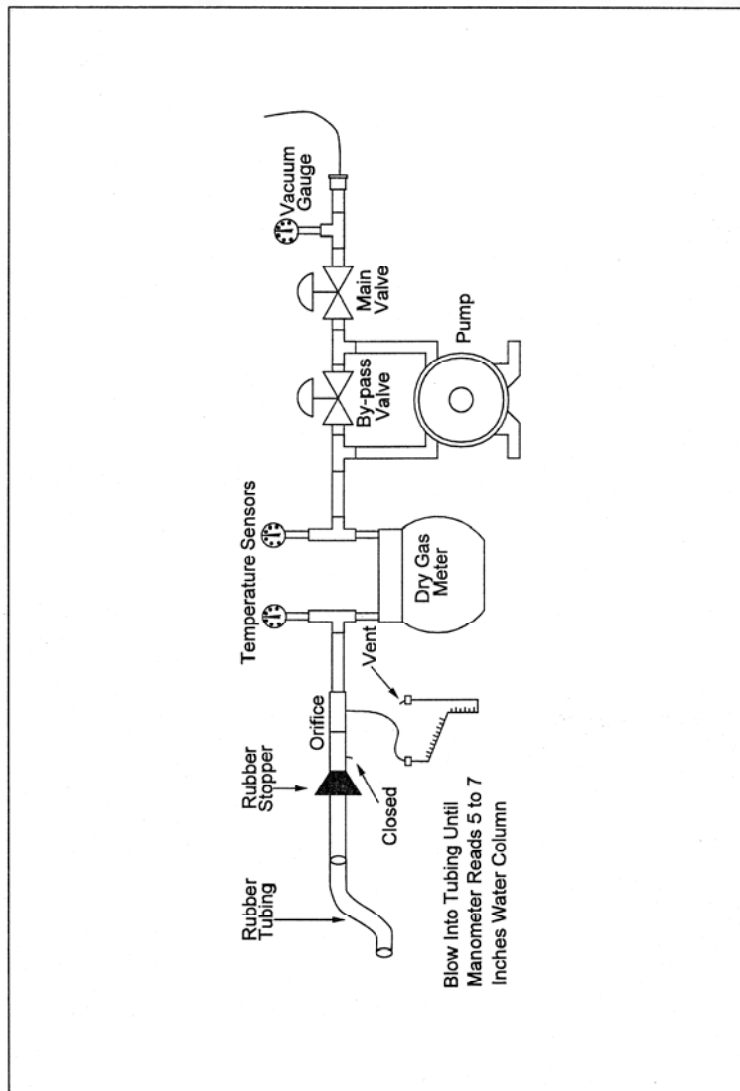


Figure 5-2. Leak Check of Meter Box.

Plant _____ Location _____ Operator _____ Date _____ Run No. _____ Sample No. _____ Meter size No. _____ Meter JAD _____ C factor _____ Filter tube coefficient, Cp _____	<div style="border: 1px solid black; width: 100px; height: 100px; margin: 5px;"></div>	Ambient temperature _____ Barometric pressure _____ Assumed moisture % _____ Probe length, (ft) _____ Probe diameter, (in) _____ Average calibrated nozzle diameter, (in) _____ Probe heater setting _____ Leak rate, (dm) _____ Static pressure, (in. Hg) _____ Filter No. _____							
SCHEMATIC OF STACK CROSS SECTION									
Traverse point number	Sampling time	Vacuum (in. Hg)	Stack temperature (°F)	Velocity head (in. Hg)	Pressure differential across orifice meter (in. H ₂ O)	Gas meter reading (ft ³)	Gas sample temperature at dry gas meter		Temperature of gas leaving condenser or last impinger (°F)
	min.		(°F)	ΔP_v (in. Hg)			Inlet (°F)	Outlet (°F)	(°F)
Total							Aug	Aug	
Average							Aug	Aug	

Figure 5-3. Particulate Field Data.

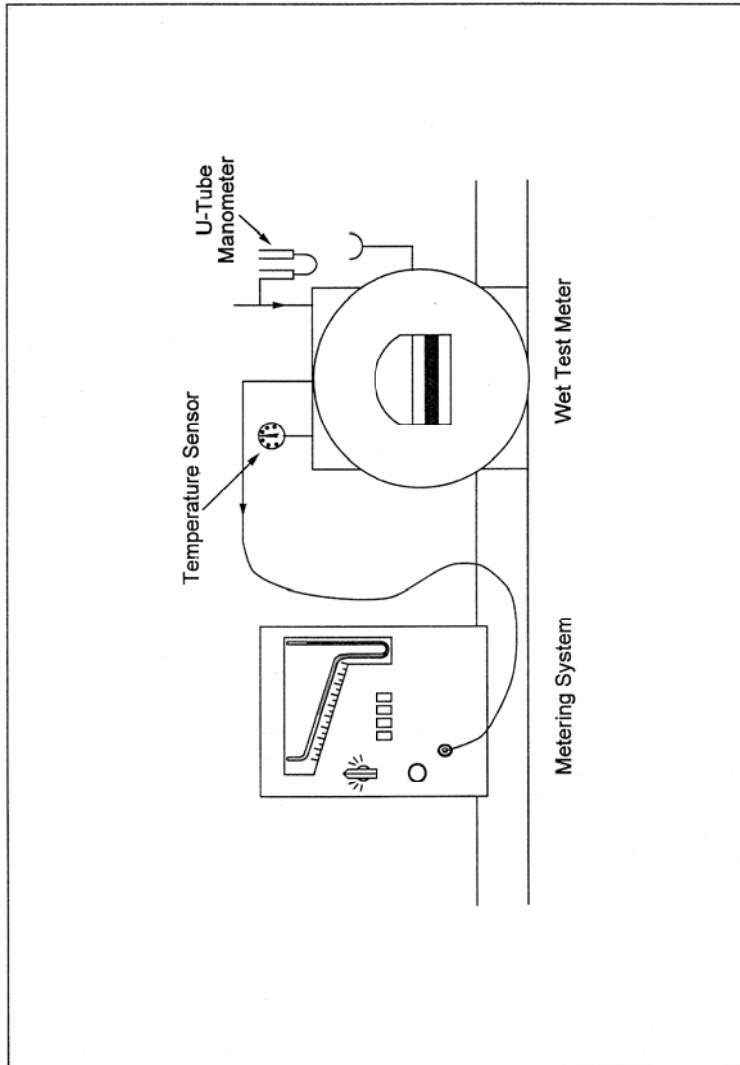


Figure 5-4. Equipment arrangement for metering system calibration.

Date: _____ Metering System Identification: _____							
Barometric pressure, P_b , = _____ in. Hg							
Orifice manometer setting Δh in. H ₂ O	Spirometer (wet meter) gas volume, V_w , ft ³	Dry gas meter volume V_d , ft ³	Spirometer (wet meter) T_w , °F	Temperatures			Time θ , min
				Inlet T_i , °F	Outlet T_o , °F	Average T_a , °F	
Calculations							
Δh in H ₂ O	Y			$\Delta h\theta$			
Average							
Y =	Ratio of reading of wet test meter to dry test meter; tolerance for individual values ± 0.02 from average.						
$\Delta h\theta$ =	Orifice pressure differential that equates to 0.75 cfm of air @ 68°F and 29.92 inches of mercury, in. H ₂ O; tolerance for individual values ± 0.20 from average.						

Figure 5-5. Example Data Sheet for Calibration of Metering System (English Units).

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, 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).

Figure 5-6. Analytical Data Sheet

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

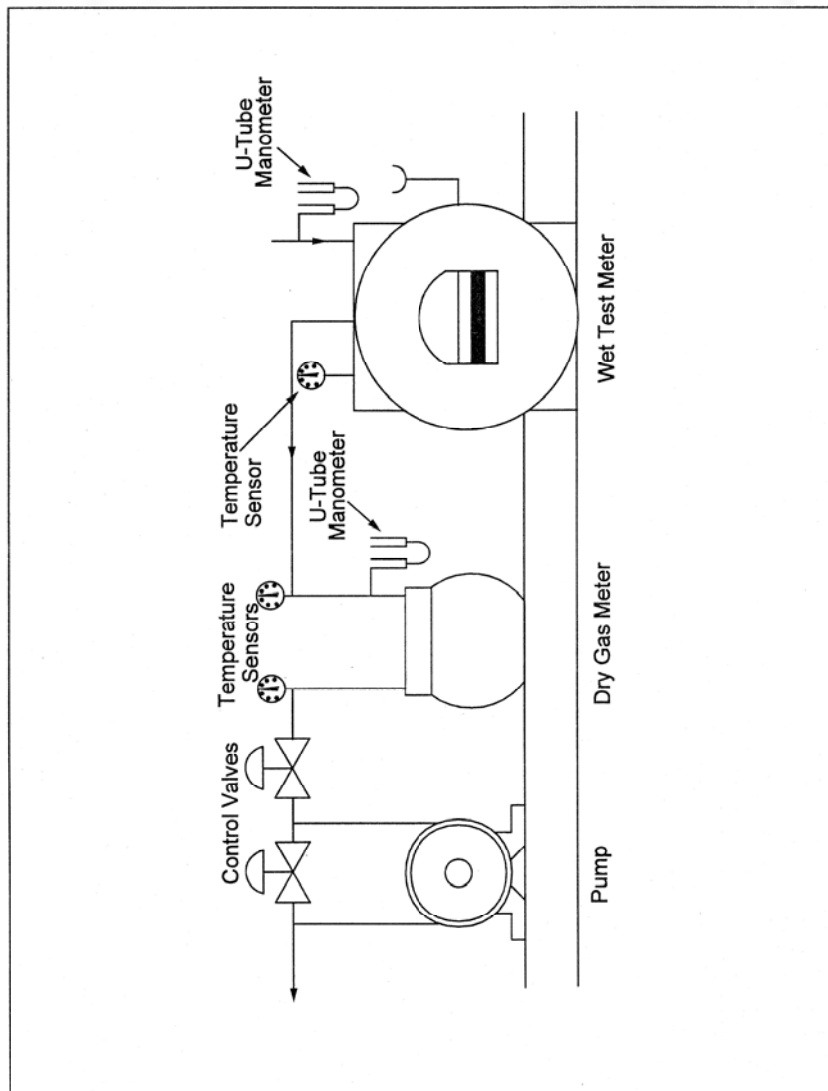


Figure 5-7. Equipment Arrangement for Dry Gas Meter Calibration.

Date: _____
 Dry Gas Meter Identification: _____
 Barometric Pressure (P_{bar}): _____ in. Hg

Approximate Flow Rate (Q) cfm	Spirometer (Wet Meter) Gas Volume (V _g) ft ³	Spirometer (Wet Meter) Volume (V _w) ft ³	Temperatures			Dry Gas Meter Pressure (Δp) in. H ₂ O	Time (t) ft ³	Flow Rate (Q) cfm	Meter Coefficient (V _{ds})	Average Meter Coefficient (V _{da})
			Spirometer (Wet Meter) (t _s) °F	Dry Gas Meter Inlet (t _i) °F	Dry Gas Meter Outlet (t _o) °F					
0.40										
0.60										
0.80										
1.00										
1.20										

$$Q = K_1 \frac{P_{bar} V_w}{(T_w + T_{std} Q)}$$

$$Y_{ds} = \frac{V_w (T_{ds} + T_{std}) P_{bar}}{V_{ds} (T_w + T_{std}) (P_{bar} + \Delta P/13.6)}$$

Figure 5-8. Example Data Sheet for Calibration of a Standard Dry Gas Meter for Method 5 Sampling Equipment (English units).

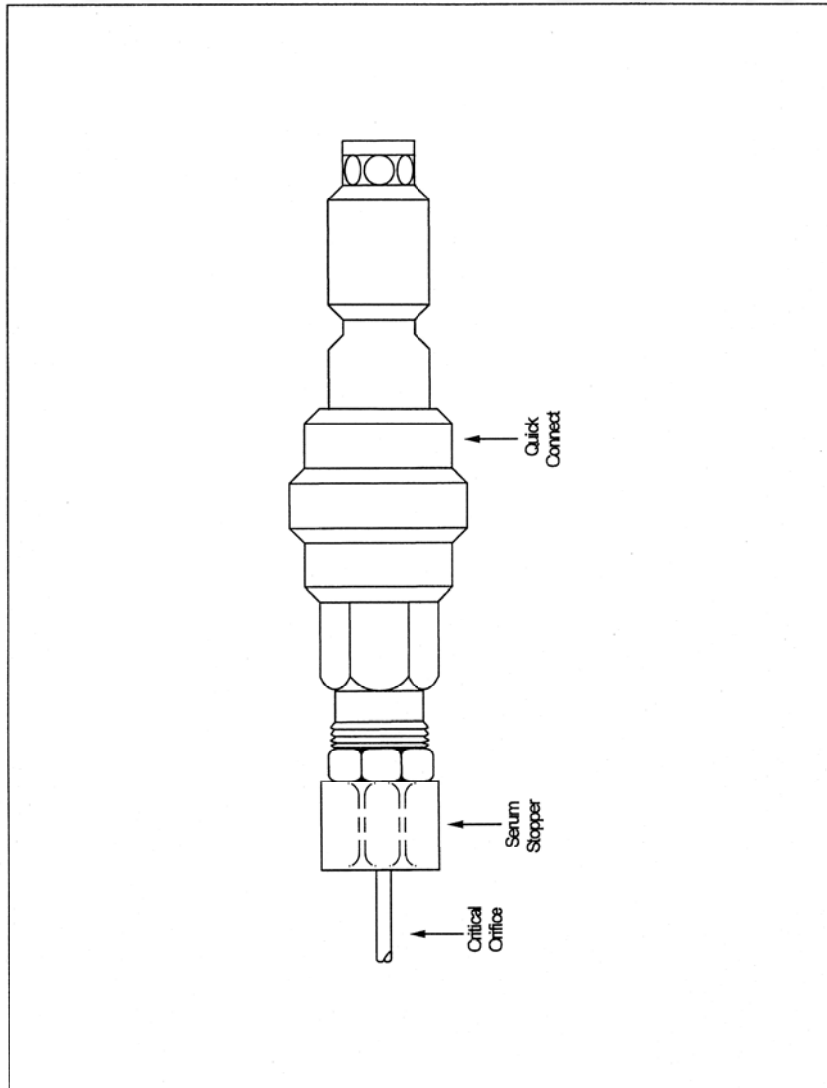


Figure 5-9. Critical Orifice Adaptation to Method 5 Metering System.

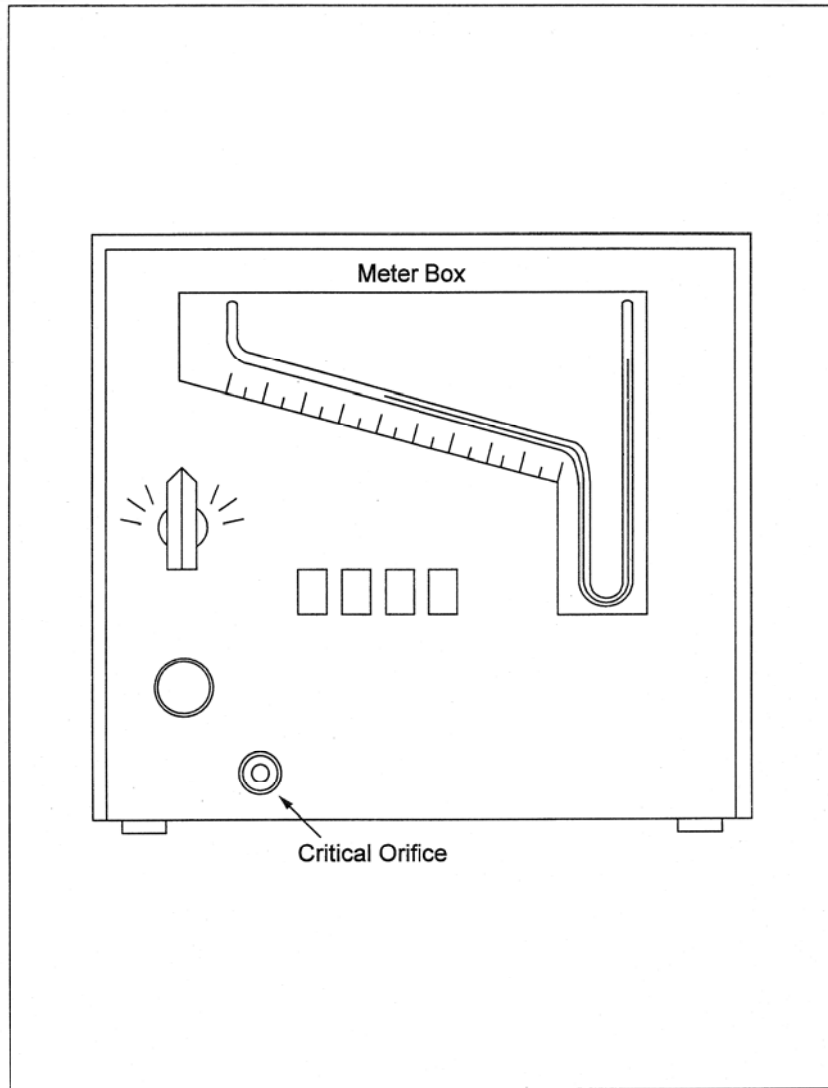


Figure 5-10. Apparatus Setup.

Date _____ DGM cal. factor _____
 Train ID _____ Critical orifice ID _____

Dry gas meter		Run No.	
		1	2
Final reading	m ³ (ft ³)
Initial reading	m ³ (ft ³)
Difference, V ^m	m ³ (ft ³)
Inlet/Outlet

Dry gas meter		Run No.	
		1	2
Temperatures:	°C (°F)	/	/
Initial	°C (°F)	/	/
Final	min/sec	/	/
Avg. Temperature, t_m	min		
Time, θ			
Orifice man. rdg., ΔH	mm (in.) H ₂		
Bar. pressure, P^{bar}	mm (in.) Hg		
Ambient temperature, t_{amb}	mm (in.) Hg		
Pump vacuum			
K' factor			
Average			

Figure 5-11. Data sheet of determining K' factor.

Train ID _____
 Critical orifice ID _____
 Critical orifice K' factor _____

Date _____

Dry gas meter		Run No.	
		1	2
Final reading	m ³ (ft ³)		
Initial reading	m ³ (ft ³)		
Difference, V_m	m ³ (ft ³)		
Inlet/outlet temperatures	°C (°F)	/	/
Initial	°C (°F)	/	/
Final	°C (°F)		
Avg. Temperature, t_m	min/sec	/	/
Time, θ	min		
Orifice man. rdg., ΔH	min		
Bar. pressure, P^{bar}	mm (in.) H ₂ O		
Ambient temperature, t_{amb}	mm (in.) Hg		
Pump vacuum	°C (°F)		
$V_{m(Std)}$	mm (in.) Hg		
$V_{c(Std)}$	m ³ (ft ³)		
DGM cal. factor, Y	m ³ (ft ³)		

Figure 5-12. Data Sheet for Determining DGM Y Factor

METHOD 5A—DETERMINATION OF PARTICULATE MATTER EMISSIONS FROM THE ASPHALT PROCESSING AND ASPHALT ROOFING 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 asphalt roofing industry process saturators, blowing stills, and other sources as specified in the regulations.

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 42 ±10 °C (108 ±18 °F). The PM 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 of this test method to establish appropriate safety and health practices and to determine the

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applicability of regulatory limitations prior to performing this test method.

6.0 Equipment and Supplies

6.1 Sample Collection. Same as Method 5, section 6.1, with the following exceptions and additions:

6.1.1 Probe Liner. Same as Method 5, section 6.1.1.2, with the note that at high stack gas temperatures greater than 250 °C (480 °F), water-cooled probes may be required to control the probe exit temperature to 42 ±10 °C (108 ±18 °F).

6.1.2 Precollector Cyclone. Borosilicate glass following the construction details shown in Air Pollution Technical Document (APTD)-0581, "Construction Details of Isokinetic Source-Sampling Equipment" (Reference 2 in Method 5, section 17.0).

NOTE: The cyclone shall be used when the stack gas moisture is greater than 10 percent, and shall not be used otherwise.

6.1.3 Filter Heating System. Any heating (or cooling) system capable of maintaining a sample gas temperature at the exit end of the filter holder during sampling at 42 ±10 °C (108 ±18 °F).

6.2 Sample Recovery. The following items are required for sample recovery:

6.2.1 Probe-Liner and Probe-Nozzle Brushes, Graduated Cylinder and/or Balance, Plastic Storage Containers, and Funnel and Rubber Policeman. Same as in Method 5, sections 6.2.1, 6.2.5, 6.2.6, and 6.2.7, respectively.

6.2.2 Wash Bottles. Glass.

6.2.3 Sample Storage Containers. Chemically resistant 500-ml or 1,000-ml borosilicate glass bottles, with rubber-backed Teflon screw cap liners or caps that are constructed so as to be leak-free, and resistant to chemical attack by 1,1,1-trichloroethane (TCE). (Narrow-mouth glass bottles have been found to be less prone to leakage.)

6.2.4 Petri Dishes. Glass, unless otherwise specified by the Administrator.

6.2.5 Funnel. Glass.

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

6.3.1 Beakers. Glass, 250-ml and 500-ml.

6.3.2 Separatory Funnel, 100-ml or greater.

7.0. Reagents and Standards

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

7.1.1 Filters, Silica Gel, Water, and Crushed Ice. Same as in Method 5, sections 7.1.1, 7.1.2, 7.1.3, and 7.1.4, respectively.

7.1.2 Stopcock Grease. TCE-insoluble, heat-stable grease (if needed). This is not necessary if screw-on connectors with Teflon sleeves, or similar, are used.

7.2 Sample Recovery. Reagent grade TCE, ≤0.001 percent residue and stored in glass bottles. Run TCE blanks before field use, and use only TCE with low blank values (≤0.001 percent). 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.

7.3 Analysis. Two reagents are required for the analysis:

7.3.1 TCE. Same as in section 7.2.

7.3.2 Desiccant. Same as in Method 5, section 7.3.2.

8.0. Sample Collection, Preservation, Storage, and Transport

8.1 Pretest Preparation. Unless otherwise specified, maintain and calibrate all components according to the procedure described in APTD-0576, "Maintenance, Calibration, and Operation of Isokinetic Source-Sampling Equipment" (Reference 3 in Method 5, Section 17.0). Alternative mercury-free thermometers may be used if the thermometers are, at a minimum, equivalent in terms of performance or suitably effective for the specific temperature measurement application.

8.1.1 Prepare probe liners and sampling nozzles as needed for use. Thoroughly clean each component with soap and water followed by a minimum of three TCE rinses. Use the probe and nozzle brushes during at least one of the TCE rinses (refer to section 8.7 for rinsing techniques). Cap or seal the open ends of the probe liners and nozzles to prevent contamination during shipping.

8.1.2 Prepare silica gel portions and glass filters as specified in Method 5, section 8.1.

8.2 Preliminary Determinations. Select the sampling site, probe nozzle, and probe length as specified in Method 5, section 8.2. Select a total sampling time greater than or equal to the minimum total sampling time specified in the "Test Methods and Procedures" section of the applicable subpart of the regulations. Follow the guidelines outlined in Method 5, section 8.2 for sampling time per point and total sample volume collected.

8.3 Preparation of Sampling Train. Prepare the sampling train as specified in Method 5, section 8.3, with the addition of the precollector cyclone, if used, between the probe and filter holder. The temperature of the precollector cyclone, if used, should be maintained in the same range as that of the filter, i.e., 42 ±10 °C (108 ±18 °F). Use no stopcock grease on ground glass joints unless grease is insoluble in TCE.

8.4 Leak-Check Procedures. Same as Method 5, section 8.4.

8.5 Sampling Train Operation. Operate the sampling train as described in Method 5, section 8.5, except maintain the temperature of the gas exiting the filter holder at 42 ±10 °C (108 ±18 °F).

8.6 Calculation of Percent Isokinetic. Same as Method 5, section 8.6.

8.7 Sample Recovery. Same as Method 5, section 8.7.1 through 8.7.6.1, with the addition of the following:

8.7.1 Container No. 2 (Probe to Filter Holder).

8.7.1.1 Taking care to see that material on the outside of the probe or other exterior surfaces does not get into the sample, quantitatively recover PM or any condensate from the probe nozzle, probe fitting, probe liner, precollector cyclone and collector flask (if used), and front half of the filter holder by washing these components with TCE and placing the wash in a glass container. Carefully measure the total amount of TCE used in the rinses. Perform the TCE rinses as described in Method 5, section 8.7.6.2, using TCE instead of acetone.

8.7.1.2 Brush and rinse the inside of the cyclone, cyclone collection flask, and the front half of the filter holder. Brush and rinse each

surface three times or more, if necessary, to remove visible PM.

8.7.2 Container No. 3 (Silica Gel). Same as in Method 5, section 8.7.6.3.

8.7.3 Impinger Water. Same as Method 5, section 8.7.6.4.

8.8 Blank. Save a portion of the TCE used for cleanup as a blank. Take 200 ml of this TCE directly from the wash bottle being used, and place it in a glass sample container labeled "TCE Blank."

9.0 Quality Control

9.1 Miscellaneous Quality Control Measures.

Section	Quality control measure	Effect
8.4, 10.0	Sampling equipment leak check and calibration.	Ensures accurate measurement of stack gas flow rate, sample volume.

9.2 A quality control (QC) check of the volume metering system at the field site is suggested before collecting the sample. Use the procedure outlined in Method 5, section 9.2.

10.0 Calibration and Standardization

Same as Method 5, section 10.0.

11.0 Analytical Procedures

11.1 Analysis. Record the data required on a sheet such as the one shown in Figure 5A-1. Handle each sample container as follows:

11.1.1 Container No. 1 (Filter). Transfer the filter from the sample container to a tared glass weighing dish, and desiccate for 24 hours in a desiccator containing anhydrous calcium sulfate. Rinse Container No. 1 with a measured amount of TCE, and analyze this rinse with the contents of Container No. 2. Weigh the filter to a constant weight. For the purpose of this analysis, the term "constant weight" means a difference of no more than 10 percent of the net filter weight or 2 mg (whichever is greater) between two consecutive weighings made 24 hours apart. Report the "final weight" to the nearest 0.1 mg as the average of these two values.

11.1.2 Container No. 2 (Probe to Filter Holder).

11.1.2.1 Before adding the rinse from Container No. 1 to Container No. 2, note the level of liquid in Container No. 2, and confirm on the analysis sheet whether leakage occurred during transport. If noticeable leakage occurred, either void the sample or take steps, subject to the approval of the Administrator, to correct the final results.

11.1.2.2 Add the rinse from Container No. 1 to Container No. 2 and measure the liquid in this container either volumetrically to ±1 ml or gravimetrically to ±0.5 g. Check to see whether there is any appreciable quantity of condensed water present in the TCE rinse

(look for a boundary layer or phase separation). If the volume of condensed water appears larger than 5 ml, separate the oil-TCE fraction from the water fraction using a separatory funnel. Measure the volume of the water phase to the nearest ml; adjust the stack gas moisture content, if necessary (see sections 12.3 and 12.4). Next, extract the water phase with several 25-ml portions of TCE until, by visual observation, the TCE does not remove any additional organic material. Transfer the remaining water fraction to a tared beaker and evaporate to dryness at 93 °C (200 °F), desiccate for 24 hours, and weigh to the nearest 0.1 mg.

11.1.2.3 Treat the total TCE fraction (including TCE from the filter container rinse and water phase extractions) as follows: Transfer the TCE and oil to a tared beaker, and evaporate at ambient temperature and pressure. The evaporation of TCE from the solution may take several days. Do not desiccate the sample until the solution reaches an apparent constant volume or until the odor of TCE is not detected. When it appears that the TCE has evaporated, desiccate the sample, and weigh it at 24-hour intervals to obtain a "constant weight" (as defined for Container No. 1 above). The "total weight" for Container No. 2 is the sum of the evaporated PM weight of the TCE-oil and water phase fractions. Report the results to the nearest 0.1 mg.

11.1.3 Container No. 3 (Silica Gel). This step may be conducted in the field. Weigh the spent silica gel (or silica gel plus impinger) to the nearest 0.5 g using a balance.

11.1.4 "TCE Blank" Container. Measure TCE in this container either volumetrically or gravimetrically. Transfer the TCE to a tared 250-ml beaker, and evaporate to dryness at ambient temperature and pressure.