Designation: D 3685/D 3685M – 98

Standard Test Methods for Sampling and Determination of Particulate Matter in Stack Gases[1]

This standard is issued under the fixed designation D 3685/D 3685M; the number immediately following the designation indicates the year of original adoption or, in the case of revision, the year of last revision. A number in parentheses indicates the year of last reapproval. A superscript epsilon (ϵ) indicates an editorial change since the last revision or reapproval.

1. Scope

1.1 These test methods describe procedures to determine the mass emission rates of particulate matter and collected residue in gaseous streams by in-stack test methods (Test Method A) or out-of-stack test methods (Test Method B).

1.2 These test methods are suitable for measuring particulate matter and collected residue concentrations.

1.3 These test methods include a description of equipment and procedures to be used for obtaining samples from effluent ducts and stacks, a description of equipment and procedures for laboratory analysis, and a description of procedures for calculating results.

1.4 These test methods are applicable for sampling particulate matter and collected residue in wet (Test Method A or B) or dry (Test Method A) streams before and after particulate matter control equipment, and for determination of control device particulate matter collection efficiency.

1.5 These test methods are also applicable for determining compliance with regulations and statutes limiting particulate matter existing in stack gases when approved by federal or state agencies.

1.6 The particulate matter and collected residue samples collected by these test methods may be used for subsequent size and chemical analysis.

1.7 These test methods describe the instrumentation, equipment, and operational procedures, including site selection, necessary for sampling and determination of particulate mass emissions. These test methods also include procedures for collection and gravimetric determination of residues collected in an impinger-condenser train. The sampling and analysis of particulate matter may be performed independently or simultaneously with the determination of collected residue.

1.8 These test methods provide for the use of optional filter designs and filter material as necessary to accommodate the wide range of particulate matter loadings to which the test methods are applicable.

1.9 Stack temperatures limitation for Test Method A is approximately 400°C (752°F) and for Test Method B is 815°C (1500°F).

1.10 A known limitation of these test methods concerns the use of collected residue data. Since some collected residues can be formed in the sample train by chemical reaction in addition to condensation, these data should not be used without prior characterization (see 4.4.1).

1.10.1 A second limitation concerns the use of the test methods for sampling gas streams containing fluoride, or ammonia or calcium compounds in the presence of sulfur dioxide and other reactive species having the potential to react within the sample train.

1.10.2 A suspected but unverified limitation of these test methods concerns the possible vaporization and loss of collected particulate organic matter during a sampling run.

1.11 The values stated in either SI units or inch-pound units are to be regarded separately as standard within the text. The inch-pound units are shown in parentheses. The values stated in each system are not exact equivalents; therefore each system shall be used independently of the other. Combining values from the two systems may result in nonconformance to this standard.

1.12 *This standard does not purport to address all of the safety concerns, if any, associated with its use. It is the responsibility of the user of this standard to establish appropriate safety and health practices and determine the applicability of regulatory limitations prior to use.*

2. Referenced Documents

2.1 *ASTM Standards:*

D 1071 Test Methods for Volumetric Measurement of Gaseous Fuel Samples[2]

D 1193 Specification for Reagent Water[3]

D 1356 Terminology Relating to Sampling and Analysis of Atmospheres[4]

D 2986 Practice for Evaluation of Air Assay Media by the

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[1] These test method are under the jurisdiction of ASTM Committee D-22 on Sampling and Analysis of Atmospheres and are the direct responsibility of Subcommittee D22.03 on Ambient Atmospheres and Source Emissions.

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[2] *Annual Book of ASTM Standards*, Vol 05.05.

[3] *Annual Book of ASTM Standards*, Vol 11.01.

[4] *Annual Book of ASTM Standards*, Vol 11.03.

Monodisperse DOP (Dioctyl Phthalate) Smoke Test[4]

D 3154 Test Method for Average Velocity in a Duct (Pitot Tube Method)[4]

D 3631 Test Methods for Measuring Surface Atmospheric Pressure[4]

D 3796 Practice for Calibration of Type S Pitot Tubes[4]

D 4536 Test Method for High-Volume Sampling for Solid Particulate Matter and Determination of Particulate Emissions[4]

E 1 Specification for ASTM Thermometers[5]

3. Terminology

3.1 *Definitions*—For definitions of terms used in these test methods, refer to Terminology D 1356.

3.2 *Definitions of Terms Specific to This Standard:*

3.2.1 *collected residue—for the purpose of these test methods,* solid or liquid matter collected in the impingers employed in these test methods and remaining after solvent removal.

3.2.2 *particulate matter—for the purpose of these test methods,* all gas-borne matter (solid or liquid) collected in the front half of the sample train (probe, nozzle, and front half of filter).

3.3 *Symbols*:

A = internal cross-sectional area of stack, m^2 (ft^2).

A_n = cross-sectional area of nozzle, m^2 (ft^2).

B_{wo} = proportion by volume of water vapor in the gas stream, dimensionless.

C_m = dry gas meter correction factor, dimensionless.

C_p = pitot tube coefficient, dimensionless.

$C'_{P.M.}$ = concentration of particulate matter in stack gas, on the dry basis, standard conditions, mg/m^3 (gr/dsft^3)

$C'_{P.M._{\mathrm{act}}}$ = concentration of particulate matter in stack gas, at actual gas conditions, mg/m^3 (gr/aft^3).

C^2_{pm} = concentration of collected residue in stack gas, dry basis, standard conditions, mg/m^3 (gr/dsft^3).

$C^2_{pm_{\mathrm{act}}}$ = concentration of collected residue in stack gas, at actual conditions, mg/m^3 (gr/aft^3).

$E_{P.M.}$ = emission rate for particulate matter, kg/h (lb/h).

E^2_{pm} = emission rate for collected residue, kg/h (lb/h).

I = percent of isokinetic sampling.

M_d = dry molecular weight of stack gas, g/g-mol (lb/lb-mole).

$M_{\mathrm{H_2O}}$ = molecular weight of water, 18.0 g/g-mol (18.0 lb/lb-mole).

M_s = molecular weight of stack gas, wet basis, g/g-mol (lb/lb-mole).

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

$P.M.$ = total amount of particulate matter collected, mg.

pm = total amount of collected residue, mg.

P_s = absolute stack gas pressure, kPa (in. Hg).

P_{stat} = static stack gas pressure, kPa (in. Hg).

P_{std} = absolute pressure at standard conditions, 101.3 kPa (29.9 in. Hg).

$Q_{\mathrm{stp\text{-}d}}$ = stack gas volumetric flow rate, dry basis, standard conditions, m^3/h (dsft^3/h).

R = ideal gas constant = 8.32×10^{-3}(kPa·m^3)/(K·g − mol) for the SI system, and 21.8 (in. Hg·ft^3)/(°R·lb − mole) for the U.S. customary system.

T_d = average temperature of the gas in the dry gas meter, obtained from the average of the initial and the final temperatures, K (°R) (see 10.2.2.6 and 10.2.2.9).

T_m = absolute average dry gas meter temperature, K (°R).

$(T_s)_{\mathrm{avg}}$ = absolute average stack gas temperature, K (°R).

T_{std} = absolute temperature at standard conditions, 298 K (25°C) (537°R).

T_w = temperature of the gas in the wet test meter, K (°R) (see 10.2.2.6 and 10.2.2.9).

V_d = gas volume passing through the dry gas meter, K (°R) (see 10.2.2.6 and 10.2.2.9).

V_{lc} = total volume of liquid collected in impingers and desiccant, mL.

V_m = volume of gas sample through the dry gas meter, meter conditions, m^3 (dft^3).

$V_{m_{\mathrm{act}}}$ = volume of gas sample through the dry gas meter, corrected to actual gas conditions, m^3 (or aft^3).

$V_{m_{\mathrm{std}}}$ = volume of gas sample through the dry gas meter, corrected to dry standard conditions, m^3 (dft^3).

$(V_s)_{\mathrm{avg}}$ = average stack gas velocity, m/s (ft/s).

$V_{m_{\mathrm{std}}}$ = volume of water vapor in the gas sample, corrected to actual conditions, m^3 (dsft^3).

V_w = gas volume passing through the wet test meter, m^3 (aft^3) (see 10.2.2.6 and 10.2.2.9).

$V_{w\,\mathrm{std}}$ = volume of water vapor in the gas sample, corrected to dry standard conditions, m^3 (dsft^3).

Y = dry gas meter calibration factor.

Y_i = ratio of accuracy of wet test meter to dry gas meter (see 10.2.2.6 and 10.2.2.9).

θ = total sampling or calibration run time, min.

$\rho_{\mathrm{H_2O}}$ = density of water, 997 kg/m^3, at 298 K.

ΔH = average pressure drop across the orifice meter, kPa (in. H_2O).

$\Delta H@$ = average orifice pressure differential that develops 0.021 m^3 (0.75 ft^3) of air at standard conditions for all six calibration runs, kPa (in. H_2O) (see 10.2.2.9).

$\Delta H@_i$ = orifice pressure differential at each flow rate that gives 0.021 m^3(0.75 ft^3) of air at standard conditions for each calibration run, kPa (in. H_2O) (see 10.2.2.9).

ΔP_{avg} = average stack gas velocity head, kPa (in. H_2O).

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[5] *Annual Book of ASTM Standards*, Vol 14.03.

NOTE 1—To convert ΔH and ΔP_{avg} from inches of water to inches of mercury, divide by 13.6, the specific gravity of mercury. To convert from inches of water to kilopascals, multiply by 0.248.

4. Summary of Test Methods

4.1 Test Method A (in-stack) involves a sampling train with a primary and a backup filter located in-stack. (Use of the backup filter is optional.) The sample is withdrawn from the stack isokinetically through the filter system followed by a series of impingers or condensers set in an ice bath, which act as a moisture trap and collect the collected residue. A dry gas meter is used to measure the sample gas volume.

4.1.1 The primary filter may be a thimble type filter or a glass fiber filter. No back-up is required when the primary filter is of the latter type.

4.2 Test Method B (out-of-stack) involves a sampling train with a filter located out-of-stack heated above the moisture-acid dew point in order to prevent filter saturation. Sample is withdrawn from the stack isokinetically through the filter system followed by moisture condensors set in an ice bath. The moisture condensors provide the collection mechanism for collected residue.

4.2.1 The sample gas volume is measured with a dry gas meter.

4.3 Particulate matter mass and collected residue mass are determined gravimetrically. Particulate matter (12.10.1 and collected residue (12.10.2) are calculated separately as mass per volume sampled at standard conditions, dry, and on the actual gas basis.

4.4 The gravimetric analysis procedure is nondestructive and thus both the particulate matter and the collected residue catches are available for further physical and chemical characterization.

4.4.1 Although procedures are not included in these test methods, it is recommended that the collected residues be subjected to chemical analysis or otherwise characterized prior to use of the mass results.

5. Significance and Use

5.1 The measurement of particulate matter and collected residue emission rates is an important test widely used in the practice of air pollution control. Particulate matter measurements after control devices are necessary to determine total emission rates to the atmosphere.

5.1.1 These measurements, when approved by federal and state agencies, are often required for the purpose of determining compliance with regulations and statutes.

5.1.2 The measurements made before and after control devices are often necessary as a means of demonstrating conformance with contractual performance specifications.

5.2 The collected residue obtained with these test methods is also important in characterizing stack emissions. However, the utility of these data is limited unless a chemical analysis of the collected residue is performed.

6. Interferences

6.1 Gaseous species present in-stack gases that are capable of reacting to form particulate matter within the sample train can result in positive interference.

6.1.1 Examples include the potential reaction of sulfur dioxide (SO_2) to an insoluble sulfate compound in the moisture portion of the system (such as with limestone in flue gas following a wet flue gas desulfurization system (FGDS) to form calcium sulfate ($CaSO_4$) or the reaction with ammonia gas (NH_3) to form ammonium sulfate (NH_4SO_4) and the potential reaction of hydrogen fluoride (HF) with glass components in the sample train with resultant collection of silicon tetrafluoride (SiF_4) in the impingers.

6.2 Volatile matter existing in solid or liquid form in the stack gas may vaporize after collection on the sample train filtration material due to continued exposure to the hot sample stream during the sampling period. Such occurrence would result in a negative interference.

7. Apparatus

7.1 *Sampling Train*—For schematic drawings of the major sampling train components refer to Figs. 1 and 2 for Test Method A and Fig. 3 for Test Method B.

7.1.1 The materials of construction of in-stack and certain out-of-stack components (such as the nozzle, probe, unions, filter holder, gaskets, and other seals) shall be constructed of materials which will withstand corrosive or otherwise reactive compounds or properties of the stack or gas stream, or both. Recommended materials for a normal range of stack and sample conditions include PFTE fluoro hydrocarbons (up to 175°C (350°F), 316 stainless steel (up to 800°C (1500°F), and some resistant silicone materials (up to 150°C (300°F). Extreme temperature conditions may require the use of materials such as quartz or nickel-chromium alloy, or a water-cooled probe may be used.

7.2 *Elements of the Sampling Train*— The sampling train for collecting particulate matter and collected residue from a gas stream flowing through a stack consists of the interconnected elements described in 7.3-7.10.

7.3 *Nozzles*—The first part of the sampling equipment to encounter the dust or moisture-laden gas stream, or both, is the nozzle. In order to extract a representative sample of gas and particulate matter, the nozzle used for sampling shall be within a narrow range of inside diameters.

7.3.1 The probe nozzle is provided with a sharp, tapered leading edge and is constructed of either seamless 316 stainless steel tubing or glass, formed in a button-hook or elbow configuration. The tapered angle is <30° with the taper on the outside to establish a constant inside diameter (ID).

7.3.2 A range of nozzle inside diameters, for example, 3 to 15 mm (0.125 to 0.5 in.), in increments of 1.5 mm (0.0625 in.), are required for isokinetic sampling. Larger nozzle sizes may be required if high volume sampling trains (see Test Method D 4536) are used or if very low flows are encountered. Inspect the nozzle before use for roundness and for damage to the tapered edge, such as nicks, dents, and burrs. Check the diameter with a micrometer or other acceptable measuring device. A slight variation from exact sizes should be expected due to machining tolerances. Engrave each nozzle with an identification number for inventory and calibration purposes.

7.3.3 Calibration procedures are described in 10.9.

7.4 *Filter Holders*:

7.4.1 *Test Method A*:

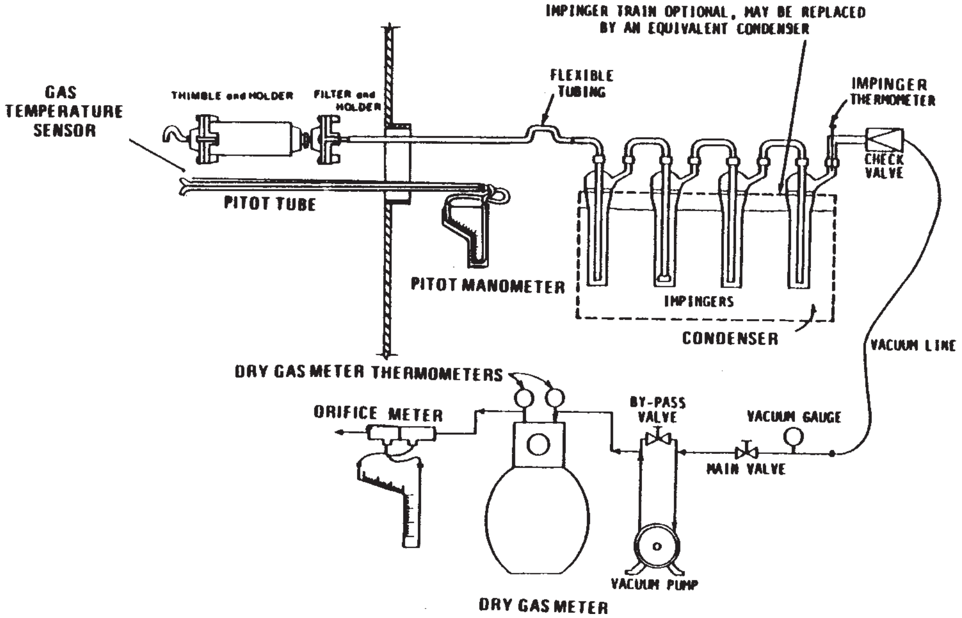

FIG. 1 Test Method A (In Stack) Sampling Train

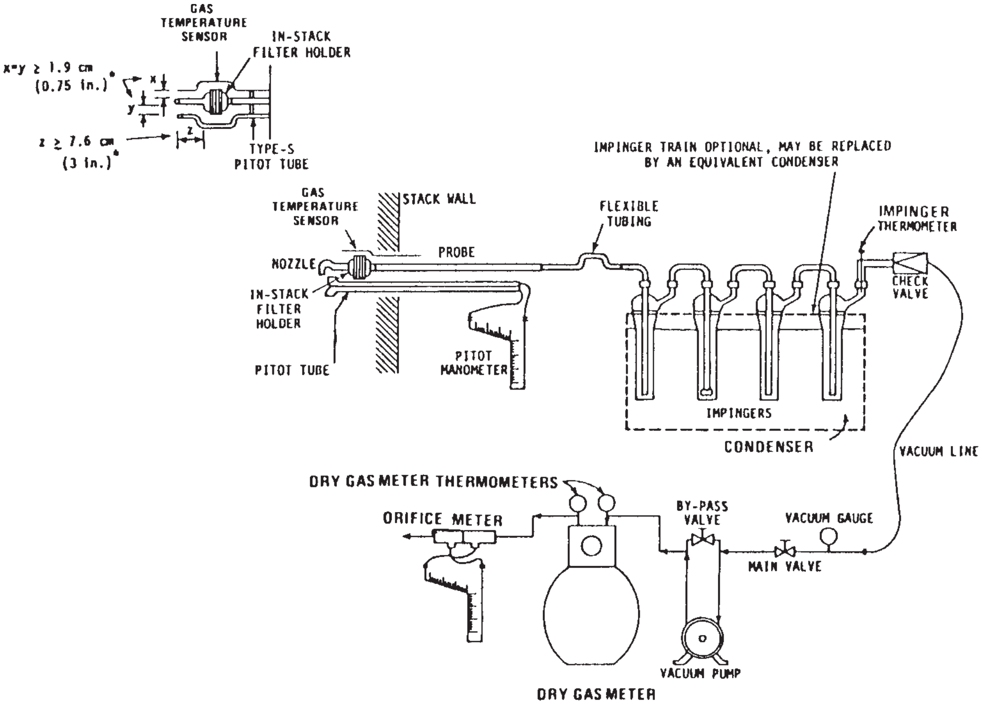

FIG. 2 (Out of Stack) Sampling Train

7.4.1.1 *Thimble Holder*—A stainless steel holder for the porous aluminum oxide thimble is shown in Fig. 4. Holders for a glass and glass-fiber thimbles are somewhat different in design and method of retention. The overall diameter has been kept to a minimum to facilitate insertion of the entire holder through a relatively small (75-mm (3-in.)) sampling port. The holder provides a method for clamping the thimble firmly in position with its lip pressed against a soft gasket. The gasket sealing together the cap and housing of the holder is made of a compressible material that will provide an adequate seal at the desired temperature, such as stainless steel or TFE-fluorocarbon ferrules. Such holders and the thimbles can withstand temperatures approaching 550°C (1000°F). If porous aluminum oxide thimbles are used, take care to avoid any spalling or crushing of the thimble lip in assembling and disassembling the thimble in its holder, as the tare mass is critical to the determination of the test results.

7.4.1.2 *Alternate Filter Holder/Backup Filter Holder*—An in-stack filter holder constructed of borosilicate or quartz glass, or stainless steel is shown in Figs. 5 and 6. Use a silicone rubber, TFE-fluorocarbon, or stainless steel gasket. The holder shall be durable, easy to load, and leak free in normal

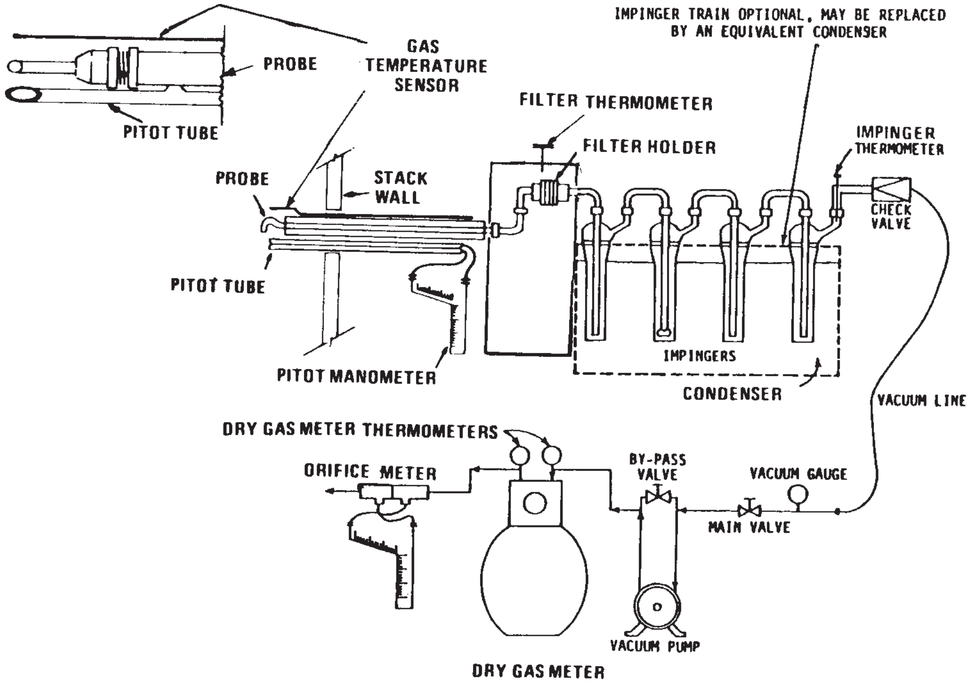

FIG. 3 Test Method A (In Stack Optional) Sampling Train

applications. It is positioned immediately following the nozzle, with the filter placed toward the flow. Perform the following leak check prior to use, to ensure that each filter holder is properly assembled.

(a) Assemble the sample probe, filter holder, and filter with the exception that a steel plug is used in place of the nozzle to provide a leak-less seal.

(b) Perform the standard leak check at 50 kPa (380 mm Hg) vacuum at ambient temperature. A leakage rate of 570 mL/min (0.02 ft^3/min) is allowed; however, under these laboratory conditions the entire train shall be leak-less.

(c) Place the filter holder in an oven (a Test Method B filter heater compartment can be used) at about 100°C (212°F) for about 30 min. Perform the leak check with the filter holder in the oven. The filter holder shall again remain leak-less.

(d) Remove the filter holder from the oven and cool for 30 min. Again run the leak check.

(e) Elevate the temperature of the oven to the maximum temperature expected during the test. Place the filter holder in the oven, and heat it for 30 min. Repeat the leak test.

(f) Remove the filter holder and allow it to cool for 30 min. Run the final leak check. If the filter holder passes these leak check procedures then it is properly designed to remain leak free when properly maintained.

(g) If the filter holder passes the leak checks at the lower temperatures, but not the maximum temperature, replace the filter holder.

(h) If the filter holder fails to pass the leak check procedure at 100°C, reject the holder unless sampling is to be performed only at ambient temperature.

7.4.2 *Test Method B Filter Holder*—Refer to 7.4.1 for Test Method A details.

7.4.2.1 *Filter Heating System*, capable of maintaining the filter holder at 120 ± 15°C (248± 25°F) during sampling. Other temperatures may be specified for a particular application.

7.4.2.2 *Filter Thermometer*—Monitoring device for measuring temperature of the filter holder to within 3°C (5.5°F) during sampling.

7.4.2.3 Before sampling, check the heating system and the temperature monitoring device. It is important that the heating element be easily replaceable in case of a malfunction during sampling.

7.5 *Probes*:

7.5.1 *Probe Extension (Test Method A)*— Any rigid probe extension may be used. Its diameter shall be sufficient to provide adequate stiffness for support at the greatest distance within the stack. Check the probe extension visually for cracks or breaks, and for leaks on a sampling train (Fig. 2). This includes a proper leak-free connection from filter holder to probe. The probe extension shall be constructed of stainless steel when non-corrosive gases are present during testing. Use a heated glass-lined probe when corrosive or condensible material is present in the stack. Otherwise the condensed or corroded materials in the probe extension may drain or be back flushed into the filter and contaminate the sample. Use a nonreactive material to prevent contamination of the sample if condensibles are to be retained. Use probe extendors of nickel-iron-chromium alloy (UNS N08825) (see DS 56F[6]), or equivalent at temperatures greater than 600°F (315°C). (Record probe material selection in the field data sheet.)

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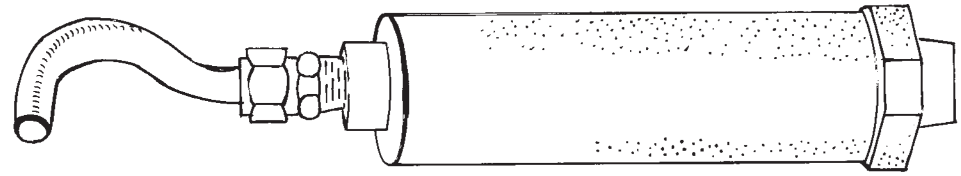
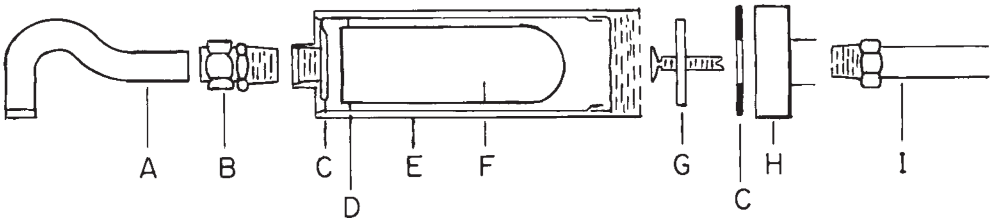
[6] *Metals and Alloys in the Unified Numbering System*, available from ASTM Headquarters.



POROUS ALUMINUM OXIDE THIMBLE PROBE



Note—Code	Part Identification/Function
	Complete assembly, including porous aluminum oxide thimble:
A	Nozzles (3 to 15 mm ID, in 1.5 mm increments, 1 each)
B	Fittings (adapts nozzle to holder)
C	Gaskets
D	Guide ring
E	Holder
F	Porous aluminum oxide
G	Clamp
H	Cap
I	Adapter (holds holder to probe extension)

FIG. 4 Thimble Holder

7.5.2 *Test Method B*—The sampling probe shall be constructed of borosilicate or quartz glass tubing with an outside diameter (OD) of approximately 16 mm (0.625 in.), encased in a stainless steel sheath with an outside diameter of 25 mm (1.0 in.). Whenever practical, every effort should be made to use borosilicate or quartz glass liners; alternatively, metal seamless liners of 316 stainless steel, nickel-chromium alloy, nickel-iron-chromium alloy (UNS N08825) (see DS 56F[6]), or other corrosion-resistant metals may be used. A heating system is required that will maintain an exit gas temperature of 120 ± 14°C (250 ± 25°F) during sampling. Other temperatures may be specified for a particular application. Use either borosilicate or quartz glass liners for stack temperatures up to about 480°C (900°F), but use quartz glass liners from 480 to 900°C (900 to 1650°F). Either type of liner may be used at higher temperatures for short time periods. However, do not exceed the absolute upper limits, that is, the softening temperatures of 820°C (1500°F) and 1500°C (2750°F) for borosilicate and quartz respectively. Visually check new probes for breaks or cracks, and for leaks on a sampling train. This includes a proper nozzle-to-probe connection with a fluoroelastomer O-ring or TFE-fluorocarbon ferrules. Check the probe heating system as follows:

7.5.2.1 Connect the probe with a nozzle attached to the inlet of the vacuum pump (7.10.3).

7.5.2.2 Electrically connect and turn on the probe heater for 2 or 3 min. The probe should become warm to the touch.

7.5.2.3 Activate the pump and adjust the needle valve until a flow rate of approximately 20 L/min (0.75 ft^3/min) is achieved.

7.5.2.4 Be sure the probe remains warm to the touch and the heater is capable of maintaining the exit air temperature at a minimum of 100°C (212°F). Otherwise, reject or repair the probe.

7.6 *Condenser*—Four impingers connected in series and immersed in an ice bath, with leak-free ground-glass fittings or any similar noncontaminating fittings.

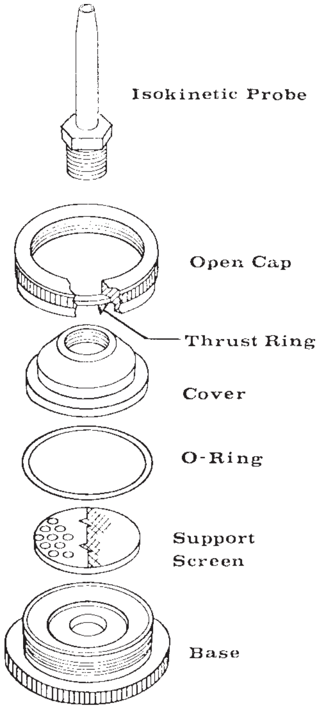


FIG. 5 Exploded Diagram for Flat, Round Filters

7.6.1 The first, third, and fourth impingers shall be the Greenburg-Smith design modified by replacing the inserts with a glass tube that has an unconstricted 13-mm (0.5-in.) inside diameter and that extends to within 13 mm of the flask bottom.

7.6.1.1 If no analysis of the collected residue is to be performed on the impinger catch, use of glass impingers is not required, as long as the gas moisture content is determined by alternate means. See Test Method D 3154.

7.6.2 The second impinger shall be a Greenburg-Smith with the standard tip and plate. Modifications (for example, using flexible connections between impingers, using materials other than glass, or using a flexible vacuum hose to connect the filter holder to the condenser) may be used.

7.6.3 The fourth impinger outlet connection shall allow for insertion of a thermometer (7.6.5). Alternatively, any system that cools the gas stream and allows measurement of the condensed water and the water vapor leaving the condenser, each to within 1 mL or 1 g, may be used.

7.6.4 Test the standard Greenburg-Smith impinger by filling the inner tube with water. If the water does not drain through the orifice in 6 to 8 s or less, replace the impinger tip or enlarge it to prevent an excessive pressure drop in the sampling system. Check each impinger visually for damage, including breaks, cracks, or manufacturing flaws such as poorly shaped connections.

7.6.5 *Impinger Thermometer*—Monitoring device for measuring temperature of gas exiting the fourth impinger (7.6) within ± 1°C (2°F) of true value in the range from 0 to 25°C (32 to 77°F).

7.7 *Gas Temperature Sensor*—For measuring gas temperature to within ± 1°C (2°F). Permanently attach the temperature sensor to either the probe (7.5) or the pitot tube (7.9). (See Figs. 1-3.)

FIG. 6 Typical Holder for Flat, Round Filters

7.8 *Vacuum Lines*—Locate all components of the sampling system as close together as possible and with direct interconnection between successive components in the system. When direct inter-connection is not possible, all vacuum (gas sampling) lines shall be of smooth bore, inert material capable of withstanding internal and external temperatures at the sampling location and a vacuum of 65 kPa (500 mm Hg) without collapse or leakage.

7.9 *Pitot Tube*—The pitot tube, Type S design, meeting the requirements of Test Method D 3154 shall be used. Attach the pitot tube to the probe as shown in Fig. 3. Visually inspect the pitot tube for both vertical and horizontal tip alignments. If the tube is purchased as an integral part of a probe assembly, check the dimensional clearances using forms referenced in 14.1. Repair or return any pitot tube which does not meet specifications.

7.10 *Metering System*, consisting of two vacuum gages, a vacuum pump, a dry gas meter with 2 % accuracy at the required sampling rate, thermometers capable of measuring ± 3°C (5.5°F) of true value in the range from 0 to 90°C (32 to 194°F), pressure gage, check valves, and related equipment as shown in Figs. 1-3. Other metering systems capable of maintaining sampling rates within 10 % of isokinetic and determining sample volumes to within 2 % may be used. Upon receipt or after construction of the equipment, perform both positive and negative pressure leak checks before beginning the system calibration procedure, as described in 10.2.1. Any leakage requires repair or replacement of the malfunctioning item. Components include the following:

7.10.1 *Differential Pressure Gage*—Two inclined manometers or the equivalent as specified in Test Method D 3154. One (also called the pitot manometer) is utilized to monitor the stack velocity pressure, and the other (also called the orifice meter) to measure the orifice pressure differential. Initially, check the gages against a gage-oil manometer at a minimum of three points: 5, 125, and 250 Pa (0.025 in., 0.5 in., and 1.0 in. H_2O). The gages shall read within 5 % of the gage-oil manometer at each test point. Repair or reject any gage which does not meet these requirements.

7.10.2 *Dry Gas Meter*—A volume meter is required for measuring the total sample flow for each test. A calibrated dry gas test meter (2 % accuracy at a flow rate of 20 L/min (0.75 ft^3/min)) is the most satisfactory totalizing volume meter available for source test work. Calibrate the meter in the laboratory prior to use with a positive displacement liquid meter and determine a meter correction factor (C_m) as necessary.

7.10.2.1 *Dry Gas Meter Thermometer*—Two monitoring devices for measuring temperature to within 3°C (5.5°F) in the range from 0 to 90°C (32 to 194°F) of the gas entering and exiting from the dry gas meter (7.10.2).

7.10.3 *Vacuum Pump*—An airtight leak-free vacuum pump with coarse and fine flow controls, capable of maintaining a flow rate of 20 L/min (0.75 ft^3/min) for a pump inlet vacuum of 50 kPa (15 in. Hg), is used to draw the gas sample.

7.10.4 *Vacuum Gage*, for measuring pressure at the vacuum pump inlet, capable of measuring ± 3 kPa (1 in. Hg) over the range from 0 to 101 kPa (30 in. Hg). Check it against a mercury U-Tube manometer upon receipt, and yearly thereafter.

7.11 *Nomograph*, to determine the isokinetic sampling rate in accordance with APTD-0576 **(1)**.[7] Its function may be applied with a hand-held programmable calculator as described in 9.2.1.3.

7.12 *Thermometers*, conforming to Specification E 1, for calibration of sample train thermometers/thermocouples, as follows:

Thermometer	Section
3C or F	10.6
59C or F	10.5 and 11.4.6
63C or F	10.4
113C or F	10.3

7.13 *Barometer*—A mercury, aneroid, or other barometer capable of measuring atmospheric pressure to within ± 300 Pa (25 mm Hg) shall be used. Calibrate the barometer against a mercury-in-glass barometer or the equivalent, as described in Test Methods D 3631.

7.13.1 Alternatively, the absolute barometric pressure may be obtained from a nearby weather service station and adjusted for elevation difference between the station and the sampling point. Either subtract 10 Pa/m from the station value for an elevation increase or add the same for an elevation decrease. Replace the barometer if it cannot be adjusted to agree within 300 Pa of the reference barometric pressure.

7.14 *Wet Test Meter*, with a capacity of 3.5 m^3/h (120 ft^3/h) or 30 L for each revolution (1 ft^3/rev) with an accuracy of ± 1.0 %, shall be used to calibrate the dry test meter.

7.15 *Orsat Gas Analyzer*—Stack gas analyzer as described by Test Method D 3154 shall be used.

7.16 *U-Tube Manometer*—A water or mercury instrument capable of measuring gas pressure to within 0.33 kPa (2.5 mm Hg or 0.001 in. H_2O).

7.17 *Sample Recovery Apparatus*:

7.17.1 *Probe Liner and Nozzle Brushes*— Nylon bristle brush with a stainless steel wire handle as long as the probe, and a separate, smaller, and very flexible brush for the nozzle may be used.

7.17.2 *Wash Bottles*—Two 500-mL wash bottles for probe and glassware rinsing. Glass bottles are preferred, but polyethylene is acceptable; however, if polyethylene is used, do not store the acetone in polyethylene wash bottles for longer than a month.

7.17.3 *Sample Storage Containers*—500- or 1000-mL chemically resistant, borosilicate glass bottles for storage of acetone rinses, with leak-proof screw caps with leak-proof, rubber-backed TFE-fluorocarbon cap liners. Wide-mouthed bottles are easiest to use, but narrow-mouth bottles are less prone to leakage. As an alternative to glass, polyethylene bottles may be used, if the storage time is short. Inspect the cap seals and the bottle cap seating surfaces for chips, cuts, cracks, and manufacturing deformities that would permit leakage.

7.17.4 *Petri Dishes*, glass or polyethylene, for storage and for transportation of the filter and collected sample.

7.17.5 *Graduated Cylinder or Triple Beam Balance, or Both*, to measure the water condensed in the impingers during sampling. The graduated cylinder may be used to measure water initially placed in the first and second impingers. In either case, the required accuracy is 1 mL or 1 g; therefore, use a cylinder with subdivisions of $\leq$2 mL. Use a triple beam balance capable of weighing to the nearest 1.0 g.

7.17.6 *Plastic Storage Containers*—Several airtight plastic containers for storage of silica gel.

7.17.7 *Funnel and Rubber Policeman*, to transfer the used silica gel from the impinger to a storage container unless silica gel is weighed in the field after the test.

7.17.8 *Desiccator*, used to dry filters before weighing. Use anhydrous $CaSO_4$ (8.5) as the desiccant.

7.17.9 *Laboratory Drying Oven*, capable of heating filters and thimbles to 102°C (215°F).

7.17.10 *Laboratory Muffle Furnace*, capable of heating thimbles to 550°C (1000°F).

7.17.11 *Steam Bath*.

7.18 *Analytical Equipment*:

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[7] The boldface numbers in parentheses refer to the list of references at the end of this standard.

7.18.1 *Glassware*—Borosilicate glass dishes to facilitate filter weighing. Use a 250-mL glass beaker for evaporation of the acetone rinse.

7.18.2 *Balance*, analytical grade, capable of weighing the filter and the sample beaker to within ±0.1 mg.

8. Reagents and Materials

8.1 *Purity of Reagents*—Reagent grade chemicals shall be used in all tests. All reagents shall conform to the specifications of the committee on Analytical Reagents of the American Chemical Society, where such specifications are available.[8]

8.2 *Purity of Water*—Unless otherwise specified, water shall be Type III reagent water conforming to Specification D 1193.

NOTE 2—Type IV reagent water is required in 11.1.2.

8.3 Determine reagent blanks on the acetone, toluene, and reagent water (see 11.7.5 and 11.8.3).

8.4 *Acetone*—Reagent ACS grade acetone with ≤0.001 % residue in glass bottles. Acetone supplied in metal containers is unacceptable due to the prevalently high residue levels. Reject the acetone if blank residue mass (see 8.3) is >0.001 % of the total acetone mass.

8.5 *Calcium Sulfate*, *Anhydrous* ($CaSO_4$), indicating type, for use in desiccator (7.17.8).

8.6 *Crushed Ice*.

8.7 *Silica Gel* (SiO_2), indicating-type, 6 to 16-mesh, for use in the fourth impinger in the condenser (7.6). Dry at 175°C (350°F) for at least 2 h prior to use.

8.8 *Gloves*, insulated, heat-resistant.

8.9 *Graphite Compound*, high-temperature type, for testing above 260°C (500°F).

8.10 *Packing Material*—A suitable temperature-resistant material for sealing the port during sampling.

8.11 *Stopcock Grease*—An acetone-insoluble, heat-stable, silicone grease for the sealing of ground-glass connections. This is not necessary if screw-on connectors with TFE-fluorocarbon sleeves are used.

8.12 *TFE-Fluorocarbon Tape*, 6.25 mm (¼ in.) wide.

8.13 *Toluene*.

8.14 *Boiling Chips*, used to prevent water heated to boiling from "bumping."

8.15 *Filter Material*:

8.15.1 *Test Method A*—The primary filter is generally an porous aluminum oxide glass, or glass-fiber thimble for heavy dust loading with sampling stacks without control equipment or on the inlet side of the control equipment, with a secondary filter for back-up (8.15.2). Procedures for filter preparation are given in 11.1.1 and 11.1.2.

8.15.2 *Back-Up or Alternate Test Methods A and B*—Use glass-fiber filters without organic binders. The filters shall exhibit at least 99.95 % collection efficiency of a 0.3-µm dioctyl phthalate smoke particle, in accordance with Practice D 2986. Manufacturer's quality control test data are sufficient for validation of efficiency.

8.15.2.1 Check the filters for irregularities, flaws, or pinholes by holding them up against a light source.

9. Sampling

9.1 Select a sampling site in accordance with the criteria of Test Method D 3154.

9.2 *Stack Parameters*—Check the sampling site for cyclonic or nonparallel flow as described in Test Method D 3154. Determine the stack pressure (using the U-tube manometer (7.16)), temperature (using the gas temperature sensor (7.7)), and the range of velocity heads encountered, in accordance with Test Method D 3154.

9.2.1 Determine the moisture content as described in Test Method D 3154 for the purpose of establishing the isokinetic sampling rate. If the particular source has been tested before or if a good estimate of the moisture is available, this data is sufficient.

9.2.1.1 If the stack is saturated with moisture or has water droplets, determine the moisture content by the partial pressure method described in Test Method D 3154.

9.2.1.2 Determine the dry molecular weight of the stack gas, as described in Test Method D 3154. Using the stack parameters obtained by these preliminary measurements, prepare a nomograph (see 7.11) as outlined in APTD-0576 **(1)**.

9.2.1.3 Alternately, the calculation may be performed by the nomograph (see 7.11) using a calculator, as described in 9.2.2.

9.2.2 Select a nozzle size based on the range of velocity heads, so that it will not be necessary to change the size to maintain isokinetic sampling rates during a run. Install the selected nozzle using a fluoroelastomer O-ring.

9.2.3 Mark the probe with heat-resistant tape or crayon to denote the proper distance into the stack or duct for each sampling point.

9.2.4 Select a total sampling time greater than or equal to the minimum total sampling time to ensure the following:

9.2.4.1 The sampling time per point is ≥2 min.

9.2.4.2 The sample volume corrected to standard conditions exceeds the required minimum total gas sample volume, based on an approximate average sampling rate.

9.2.5 Sample at each point for either a whole number of minutes or odd half minutes to avoid timekeeping errors.

9.2.6 In some circumstances (for example, batch cycles), it may be necessary to sample for shorter times at the traverse points and to obtain smaller gas sample volumes.

9.3 Determine the atmospheric pressure as described in Test Methods D 3631, using the calibrated barometer (7.13).

10. Calibration and Set-Up

METERING SYSTEM

10.1 *Wet Test Meter* (see 7.14)—Wet test meters are originally calibrated by the manufacturer with an accuracy of +0.5 %. Obtain a calibration certificate upon purchase.

10.1.1 Verify the calibration of the wet test meter initially upon receipt and yearly thereafter by one of the following methods:

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[8] *Reagent Chemicals, American Chemical Society Specifications*, American Chemical Society, Washington, DC. For suggestions on the testing of reagents not listed by the American Chemical Society, see *Analar Standards for Laboratory Chemicals*, BDH Ltd., Poole, Dorset, U.K., and the *United States Pharmacopeia and National Formulary*, U.S. Pharmacopeial Convention, Inc. (USPC), Rockville, MD.

10.1.1.1 Certification from the manufacturer that the wet test meter is within +1 % of the certified value at the wet test meter discharge, so that only a leak check of the system is then required.

10.1.1.2 Calibration in accordance with Test Methods D 1071, provided that the wet test meter is displaced at least one complete revolution.

10.1.1.3 Calibration against a smaller wet gas test meter that has been previously calibrated against a primary air or liquid displacement method.

10.1.1.4 Calibration against a dry gas meter that has been previously calibrated against a primary air or liquid displacement method.

10.1.2 Check the calibration of the wet test meter annually. The calibration check can be made by the same method as that of the original calibration; however, the comparison method need not be recalibrated if the calibration check is within +1 % of the certified value. If this agreement is not obtained, recalibrate the c omparison method or wet test meter against a primary air or liquid displacement method.

10.2 *Calibration of Metering System*— Initially calibrate the metering system (7.10)—consisting of the vacuum pump (7.10.3), vacuum gage (7.10.4), valves, orifice meter (7.10.1), and dry gas meter (7.10.2)—by stringent laboratory methods before it is used in the field. Recheck the calibration after the initial acceptance and after each field test series. This recheck is designed to provide the tester with a method that can be used more often and with less effort to ensure that the calibration has not changed. When a quick check shows that the calibration factor has changed, perform a complete laboratory procedure to obtain the new calibration factor. After recalibration, multiply the metered sample volume by either the initial or the recalibrated calibration factor—that is, the one that yields the lower gas volume for each test run. Conduct a leak test before initial calibration of the metering system, to determine if the meter system is leak free. Perform both positive (pressure) and negative (vacuum) leak checks.

10.2.1 *Pressure Leak-Check*—Following is a pressure leak-check procedure that will check the metering system from the quick disconnect inlet to the orifice outlet and will check the orifice meter:

10.2.1.1 Disconnect the orifice meter line from the downstream orifice pressure tap (the one closest to the exhaust of the orifice), and plug this tap.

10.2.1.2 Vent the negative side of the inclined manometer to the atmosphere. If the inclined manometer is equipped with a three-way valve, this step can be performed by merely turning the three-way valve that is on the negative side of the orifice-inclined manometer to the vent position.

10.2.1.3 Place a one-hole rubber stopper with a tube through its hole in the exit of the orifice, and connect a piece of rubber or plastic tubing to the tube.

10.2.1.4 Open the positive side of the orifice meter to the "reading" position; if the meter is equipped with a three-way valve, this will be the line position.

10.2.1.5 Plug the inlet to the vacuum pump (7.10.3). If a quick disconnect with a leak-free check valve is used on the control module, the inlet will not have to be plugged.

10.2.1.6 Open the main valve and the bypass valve.

10.2.1.7 Blow into the tubing connected to the end of the orifice meter until a pressure of 15 to 25 kPa (5 to 7.5 in. H_2O) has built up in the system.

10.2.1.8 Plug or crimp the tubing to maintain this pressure.

10.2.1.9 Observe the pressure reading for a 1-min period. No noticeable movement in the manometer fluid level should occur. If the metering system has a leak, a bubbling-type leak-check solution may aid in locating the leak(s).

10.2.1.10 After the metering system is determined to be leak-free by the positive leak-check procedure, check the vacuum system up to and including the pump by plugging the air inlet to the metering system. If a quick disconnect with a leak-free stopper system is presently on the metering system, the inlet will not have to be plugged.

10.2.1.11 Activate the pump, develop a vacuum within 10 kPa (75 mm Hg) of absolute zero, and observe the dry gas meter (7.10.2). If the leakage exceeds 150 mL/min (0.005 ft^3/min), seek and remove or minimize the leak(s) until the above specifications are satisfied.

NOTE 3—For metering systems having diaphragm pumps, the normal leak-check procedure described in this section will not detect leakages within the pump. For these cases, perform the following leak-check procedure: make a 10-min calibration run at 570 mL/min (0.02 ft^3/min); at the end of the run, take the difference between the measured wet test meter and the dry gas meter volumes; divide the difference by 10 to determine the leak rate. If the leak rate exceeds 570 mL/min (0.02 ft^3/min), seek and remove the leak.

10.2.2 *Initial Calibration*—Calibrate the dry gas meter (7.10.2) simultaneously with the orifice meter (7.10.1) when first purchased and at any time the post-test check yields a Y value (dry gas meter correction value) outside the range of the calibration factor $Y \pm 0.05\,Y$, using a calibrated wet test meter (7.14) (properly sized, with ± 1 % accuracy) as follows:

10.2.2.1 Leak check the metering system. Eliminate leaks, if present, before proceeding:

10.2.2.2 Assemble the apparatus with the wet test meter (7.14) replacing the probe and impingers, with the outlet of the wet test meter connected to a needle valve that is connected to the inlet side of the metering system.

10.2.2.3 Activate the vacuum pump (7.10.3) for 15 min with the orifice meter (7.10.1) differential (ΔH) set at 1.7 kPa (0.5 in. H_2O) to allow the pump to warm up and to permit the interior surface of the wet test meter to be wetted.

10.2.2.4 Adjust the needle valve so that the vacuum gage (7.10.4) reads between 5 and 10 kPa (50 and 100 mm Hg) during calibration.

10.2.2.5 Collect the information as required in the forms referenced in 14.1. Use sample volumes as shown in that reference.

10.2.2.6 Determine Y_i for six runs at ΔH settings of 0.5, 1.0, 2.0, 5.0, 10, and 15 kPa (0.15, 0.30, 0.60, 1.5, 3.0, and 4.5 in H_2O). Record the results on the form in the space provided, using the form referenced in 14.1, as follows:

$$Y_i = \frac{V_w P_{\text{bar}} T_d}{Vd(P_{\text{bar}} + \Delta H)(T_w)} \qquad (1)$$

for SI, and

$$Y_i = \frac{V_w P_{\text{bar}} T_d}{Vd\left(P_{\text{bar}} + \dfrac{\Delta H}{13.6}\right) T_w} \tag{2}$$

for the U.S. customary system.

10.2.2.7 Calculate the average Y (dry gas meter calibration factor) for the six runs using (Eq 3):

$$Y = \frac{Y_1 + Y_2 + Y_3 + Y_4 + Y_5 + Y_6}{6} \tag{3}$$

Record the average on the form referenced in 14.1 in the space provided.

10.2.2.8 Clean, adjust, and recalibrate, or reject the dry gas meter if one or more values of Y fall outside the interval $Y \pm 0.02\ Y$, Otherwise, the average Y is acceptable. Use the calculated value for future checks and subsequent test runs.

10.2.2.9 Determine $\Delta H@_i$ for each of the six runs and record on the form referenced in 14.1, as follows:

$$\Delta H@_i = \frac{0.00117\ \Delta H}{P_{\text{bar}} T_d}\left(\frac{T_w \theta}{V_w}\right)^2 \tag{4}$$

for SI, and

$$\Delta H@_i = \frac{0.0317\ \Delta H}{P_{\text{bar}} T_d}\left(\frac{T_w \theta}{V_w}\right)^2 \tag{5}$$

for The U.S. customary system.

10.2.2.10 Calculate the average $\Delta H@$ for the six runs using (Eq 6):

$$\Delta H@ = \frac{\Delta H@_1 + \Delta H@_2 + \Delta H@_3 + \Delta H@_4 + \Delta H@_5 + \Delta H@_6}{6} \tag{6}$$

Record the average.

10.2.2.11 Adjust the orifice meter or reject it if $\Delta H@_i$ varies by more than ± 0.5 kPa (0.025 in. H_2O) over the range from 8 to 80 Pa (0.06 to 0.6 in. H_2O). Otherwise, the average $\Delta H@_i$ is acceptable. Use the calculated value for subsequent test runs.

10.2.3 *Post-Test Calibration Check*—After each field test series, conduct a calibration check of the metering system, except for the following variations:

10.2.3.1 Three calibration runs at a single intermediate orifice meter setting may be used with the vacuum set at the maximum value reached during the test series. The single intermediate orifice meter setting shall be based on the previous field test. Insert a valve between the wet test meter (see 7.14) and the inlet of the metering system (7.10) to adjust the vacuum.

10.2.3.2 If a temperature-compensating dry gas meter is used, use a calibration temperature for the dry gas meter of ± 6°C (10.8°F) of the average meter temperature during the test series.

10.2.4 Record the required information:

10.2.4.1 If the calibration factor Y deviates by <5 % from the initial calibration factor Y, then the dry gas meter volumes obtained during the test series are acceptable.

10.2.4.2 If Y deviates by >5 %, recalibrate the metering system, and use the meter coefficient (initial or recalibrated) that yields the lowest gas volume for each test run.

TEMPERATURE GAGES

10.3 *Filter Thermometers*—Initially compare the thermometer (7.4.2.2) used to measure the filter temperature with a mercury-in-glass thermometer (7.12) conforming to Specification E 1 (No. 113C or F), as above, using a similar procedure.

10.3.1 Place the dial type or equivalent thermometer and the mercury-in-glass thermometer in a boiling water bath, 100°C (212°F). Compare readings after thermometers stabilize.

10.3.2 Allow both thermometers to come to room temperature. Compare readings after thermometers stabilize.

10.3.3 Accept the dial type or equivalent thermometer if the values agree within 3°C (5.4°F) at both points or if the temperature differentials at both points are within ± 3°C (5.4°F). Tape the temperature differential to the thermometer and record the value on the pretest sampling check form.

10.4 *Impinger Thermometer*—Initially compare the thermometer (7.6.5) used to measure temperature of the gas leaving the impinger train with a mercury-in-glass thermometer (7.12) that meets Specification E 1 (No. 63C or 63F) as follows:

10.4.1 Place both the reference thermometer and the test thermometer in an ice bath. Compare readings after they stabilize.

10.4.2 Remove the thermometers from the bath and allow both to come to room temperature. Again, compare readings after they stabilize.

10.4.3 Accept the test thermometer if its reading agrees within 1°C (2°F) of the reference thermometer reading at both temperatures. If the difference is greater than ± 1°C (2°F), adjust and recalibrate the thermometer until the criteria are met, or reject it.

10.5 *Dry Gas Meter Thermometers*—Initially compare the thermometers (7.10.2.1) used to measure the metered gas sample temperature with a mercury-in-glass thermometer (7.12) conforming to Specification E 1 (No. 59C or 59F), as above, using a similar procedure.

10.5.1 Place the dry gas meter thermometer and the mercury-in-glass thermometer in a hot water bath, 40 to 50°C (104 to 122°F). Compare the readings after the bath temperature stabilizes.

10.5.2 Allow both thermometers to come to room temperature. Compare readings after thermometers stabilize.

10.5.3 Accept the dry gas meter thermometer if the values agree within 3°C (5.4°F) at both points or if the temperature differentials at both points are within ± 3°C (5.4°F). Tape the temperature differential to the thermometer and record the value on the pretest sampling check form.

10.6 *Gas Temperature Sensor*—Calibrate the gas temperature sensor (7.7) upon receipt or before field use. Uniquely mark each sensor for identification.

10.6.1 Perform a calibration at three points and then extrapolate over the range of temperatures anticipated during actual sampling. Use a mercury-in-glass thermometer (7.12) conforming to Specification E 1 (No. 3C or 3F) for the three-point calibration.

10.6.2 Use the following procedure for calibrating gas temperature sensors (thermocouples and thermometers) for field use.

10.6.2.1 For the ice point calibration, form a slush from crushed ice (8.6) and liquid reagent water in an insulated vessel such as a Dewar flask. Taking care that they do not touch the sides of the flask, insert the gas temperature sensor into the slush to a depth of at least 50 mm. Wait 1 min to achieve thermal equilibrium, and record the readout on the potentiometer. Obtain three readings taken at 1-min intervals. (Longer times may be required to attain thermal equilibrium with thick-sheathed thermocouples.)

10.6.2.2 Fill a large beaker with water to a depth ± 100 mm. Place several boiling chips (8.14) in the water, and bring the water to a full boil, using a hot plate as the heat source. Insert the gas temperature sensor(s) in the boiling water to a depth of at least 50 mm, taking care not to touch the sides or bottom of the beaker. Place a thermometer (7.12) conforming to Specification E 1 (No. 3C or 3F) alongside the sensor(s). If the entire length of the mercury shaft in the thermometer cannot be immersed, make a temperature correction to give the correct reference temperature. After 3 min, both instruments will attain thermal equilibrium. Simultaneously record temperatures from the reference thermometer and the gas temperature sensor three times at 1-min intervals.

10.6.2.3 For thermocouples, repeat 10.6.2.2 with a liquid that has a boiling point (such as cooking oil) in the range from 150 to 250°C (300 to 500°F). Record the data. For thermometers other than thermocouples, repeat 10.6.2.2 with a liquid that boils at the maximum temperature at which the thermometer is to be used, or place the gas thermometer and reference thermometer in a furnace (7.17.10) or other device to reach the required temperature.

NOTE 4—If the thermometer is to be used at temperatures higher than the reference thermometers will record, calibrate the gas thermometer with a thermocouple previously calibrated with the above procedure.

10.6.2.4 If the absolute values of the reference thermometer and thermocouple(s) agree within ± 1.5 % at each of the three calibration points, plot the data on linear graph paper and draw the best-fit line to the three points or calculate the constants of the linear equation using the least-squares method. The data may be extrapolated above and below the calibration points and span the entire manufacturer's suggested range for the thermocouple. For the portion of the plot or equation that agrees within 1.5 % of the absolute reference temperature, no correction need be made. For all other portions that do not agree within ± 1.5 % use the plot or equation to adjust the data. If the absolute values of the reference thermometer and stack temperature sensor (other than the thermocouple) agree within ± 1.5 % at each of the three points, the thermometer may be used over the range of calibration points for testing without applying a correction factor. Do not extrapolate the data outside the calibration points.

10.7 *Probe Heater Thermometer*—Calibrate the probe heater temperature measuring system prior to field use as described in 10.6.

10.8 *Barometer* (see 7.13)—Calibrate the barometer in accordance with Test Methods D 3631.

10.9 *Probe Nozzle*—Calibrate probe nozzles (7.3) before initial use in the field. Using a micrometer, measure the inside diameter of the nozzle to the nearest 0.025 mm (0.001 in.). Take three measurements using different diameters each time, and compute the average.

10.9.1 The difference between the high and the low numbers shall not exceed 0.1 mm (0.004 in.). Reshape, sharpen, and recalibrate the nozzles when they become nicked, dented, or corroded before use. Permanently and uniquely identify each nozzle.

10.10 *Pitot Tube* (7.9)—Calibrate the Type S pitot tube assembly in accordance with Practice D 3796.

10.11 *Triple Beam Balance*—Calibrate the triple beam balance (7.17.5) using Class-S standard weights. The readings shall be within ± 0.5 g of the standard mass. Adjust or reject the balance if limits are not met.

10.12 *Analytical Balance*—Check the analytical balance (7.18.2) initially with Class-S weights, and record the data on an analytical balance calibration log or on a similar form. Adjust the balance to agree within ± 2 mg of the Class-S weight, or reject it.

11. Procedures

11.1 *Laboratory Preparation of Filter Material Prior to Sampling*:

11.1.1 *Flat Filter Material for Primary or Secondary Holder*—Heat each filter (8.15.2) in a laboratory drying oven (7.17.9) on a numbered container for 2 h at 102°C (215°F). After removal from the oven, cool to room temperature in a desiccator using a desiccant. After cooling, weigh each filter to the nearest 0.1 mg and record the data. Transport the weighed flat filters in Petri dishes (7.17.4) to the holders.

11.1.2 *Porous Aluminum Oxide Thimble for Primary Holder*—Sand the lip of the porous aluminum oxide thimble (8.15.1) using fine sandpaper. Take care to sand the edge to remove loose material and to make a flat surface in one plane normal to the long axis of the thimble. If high points or thin edges are created, the seat against the gasket will be uneven and when the holder is tightened, the thimble may crack. Wash in Type IV or better reagent water (8.2), fire at 550°C (1000°F) in a muffle furnace (7.17.10) for 1 h, cool, and store until needed.

11.1.3 When preparing the porous aluminum oxide thimbles for use, number the outside of the porous aluminum oxide thimbles or the jar with a heat-resistant pencil. Place the thimbles in a drying oven (7.17.9) and heat overnight at 102°C (215°F). Remove the thimbles from the oven, immediately desiccate them, and allow them to cool to room temperature. Weigh the thimbles to the nearest 0.1 mg. Return the thimbles to their containers.

11.2 *Preparation for Test*:

11.2.1 *Preparation of Sampling Train*— After the proper nozzle and particulate matter filters have been selected, assemble the major sampling train components (see Fig. 1, Fig. 2, or Fig. 3). Use TFE-fluorocarbon tape (8.12) to lubricate the threads on all connections in the train for stack temperature up to 260°C (500°F). Use a high-temperature graphite compound (8.9) when the gas temperature is above 260°C. Place laboratory-dried, weighed filters (or thimbles) of proper diameter in holders. Place a minimum of 100 mL of reagent water in each of the first two impingers, and record the volume or mass. Leave the third impinger empty. Place approximately

300 g of silica gel (8.7) from a previously tared container in the fourth impinger, and record the mass. Set up the train with the probe (7.5).

11.2.2 Leak check the sampling train as follows:

11.2.2.1 Activate the pump (7.10.3) with the bypass valve fully open and the coarse adjust valve closed.

11.2.2.2 Open the coarse adjust valve and then slowly close the bypass valve until the desired vacuum is reached. Do not reverse the direction of the bypass valve; this will cause distilled water to back up from the impingers into the filter holder.

11.2.2.3 If the desired vacuum is exceeded, either leak check at this higher vacuum or end the leak check as described as follows and start over.

11.2.2.4 When the leak check is complete, first slowly remove the plug from the inlet to the probe or the filter holder and then close the coarse adjust valve and immediately turn off the vacuum pump. (This prevents the water in the impingers from being forced back into the filter holder and prevents the silica gel from being forced back into the third impinger.) Visually check to be sure water did not contact the filter and that the filter has no tears before beginning the test.

11.2.2.5 If the leak rate exceeds 570 mL/min (0.02 ft ^{3}/min) at a vacuum of 50 kPa (15 in. Hg), seek and remove the leak. After observing the leakage rate, gradually unplug the nozzle, and finally deactivate the pump.

11.2.3 Place a crushed ice (8.6) and water mixture around the impingers and add more ice during the run to keep the temperature of the gases leaving the last impinger as low as possible and preferably at 20°C (68°F) or less. Temperatures above 20°C (68°F) may result in damage to the dry gas meter from either moisture condensation or excessive heat.

11.3 *Performing the Test*:

11.3.1 *Prior to Sampling*—Assemble and leak test the sampling train (7.1).

11.3.2 When using Test Method A, preheat the filter holder assembly (containing filters) and retighten. For Test Method B, heat the probe and filter holder to 120°C (248°F) before beginning the test (or above the acid dew point).

11.3.3 Scrape the inside of the sampling port free of dust before each test. Insert the probe into the stack to the first traverse point, with the sampling nozzle pointed in the direction of gas flow. Use packing material (8.10) to seal the sampling port.

11.3.4 When sampling hot gases, start at the traverse point farthest away from the port and move the probe out of the stack as the test progresses. This allows at least part of the probe to cool and makes handling of the probe at the conclusion of the test more convenient. Use insulated heatresistant gloves (8.6) when handling hot probes and pitot tubes. Preheat the nozzle, particulate matter filter, and holders so that moisture present in the gases does not condense in the filter during the initial part of the sampling. In very hot flues, the thimble holder can expand while the gaskets can shrink, thus causing the thimble to loosen and allow the gases to bypass the thimble. In such cases, preheat the thimble holder in the stack, remove it, and retighten the thimble.

11.3.5 *Sampling Time and Volume*—The total sampling time is equal to the number of points to be sampled multiplied by the sample duration at each point. Use the same points for sampling that were used for the velocity traverse. The minimum sample duration is 2 min at each point. If conditions permit, use a sampling duration longer than 2 min and a total sampling time of at least 1 h because of the improved reliability of the test result. The minimum sampling volume shall be 1.25 m^3 (45 ft^3) corrected to standard conditions on a dry basis.

11.4 *Particulate Matter Train Operation*:

11.4.1 For each run, record the data required on the sample form referenced in 14.1. Take readings at each sampling point. Make additional adjustments in flow rate when significant changes in stack conditions occur.

11.4.2 When starting the test, turn the probe so that the nozzle is facing in the upstream direction, turn on the pump, open the control valve, and start the stop watch. Read the pitot manometer or gage (7.10.1) (ΔP), determine the isokinetic sampling rate from the nomograph (7.11) and adjust the control valve until the isokinetic sampling rate is obtained. Maintain the isokinetic rate by continuous observations and adjustment at all points throughout the sampling period.

11.4.3 When sampling at one point has been completed, move the probe to the next point as quickly as possible. Shut the pump off when transferring the probe from one sample port to another. Exclude the time required to transfer the probe from one port to another from the total sampling time.

11.4.4 At the completion of the test, turn off the pump, remove the probe carefully from the stack, making certain that the nozzle does not scrape dust from the inside of the port and record final meter reading. Keep the nozzle elevated at all times so that none of the sample is lost.

11.4.5 Perform a leak check at the conclusion of each sampling run. The leak check should be in accordance with the procedures in 11.2.2, and at a vacuum equal to or greater than the maximum value reached during the sampling run. If the leakage rate is $\leq$570 mL/min (0.02 ft^3/min) or 4 % 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, void the sample run.

NOTE 5—Be sure to record the dry gas meter (7.10.2) reading before and after performing the leak check so that the sample volume can be corrected.

11.4.6 Compare the dry gas meter thermometers (7.10.2) with the thermometer (7.12) conforming to Specification E 1 (No. 59C or 59F) at room temperature. If the two readings agree within 6°C (10.8°F), they are acceptable; if not, recalibrate the thermometer according to 10.4 after the post-test check of the dry gas meter. For calculations, use the dry gas meter thermometer readings (field or recalibration values) that give the higher temperatures. That is, if the field readings are higher, no correction is necessary, but if the recalibration value is higher, add the difference in the two readings to the average dry gas meter temperature reading.

11.5 *Sample Recovery*:

11.5.1 Take extreme care that none of the sample is lost or contaminated. Seal the probe and filter assembly at the nozzle and downstream end of the probe. If the impinger-condenser section is included in the train, seal it at both ends. Remove all sampling train components carefully to the field laboratory for sample recovery. When a vacuum hose is used for connection of train elements, seal it at both ends.

11.5.2 *Container No. 1 (Primary Filter)*— Carefully remove the primary filter from its holder and place in the original container. If primary is a flat filter, place it sample up in the original container and seal. If the stack being sampled contains very hot gases, allow the filter to cool in the holder before it is transferred to containers. Take care that none of the collected material or filter material is lost.

11.5.3 *Container No. 2 (Secondary Filter)*— Carefully remove the secondary filter from its holder and scrapings of filter material from gasket and place in the original Petri dish (7.17.4) container sample side up. Seal the container.

11.5.4 *Container No. 3 (Acetone Wash of Nozzle and Connecting Fittings)*—Simultaneously wash and brush the nozzle (probe in Test Method B) and connecting fittings between filters with acetone (8.4) directly into a marked container. Mark the liquid level on the container.

11.5.5 *Container No. 4 (Additional Wash of Nozzle and Connecting Fittings)*—If the emission particulate matter is not removed with acetone, then wash surfaces indicated in 11.5.4 with an appropriate solution (that is, water or toluene (8.13)).

11.5.6 *Moisture*—Measure the volume or weigh the liquid collected in the impinger-condensers, transfer the desiccant to its preweighed container, and record the data required by the form reference in 14.1.

11.6 *Recovery of Collected Residue*— When this procedure is used to determine the quantity of collected residue, the following two steps are applied:

11.6.1 *Container No. 5 (Impinger-Condenser Liquid and Water Wash)*—Transfer the liquid from the moisture trap to Container No. 5. Wash impinger-condensers and all internal surfaces between secondary filter and impingers with reagent water. Measure the volume of liquid and add to Container No. 5.

11.6.2 *Container No. 6 (Acetone Wash of Impinger-Condensers)*—Wash the same surfaces as in 11.6.1 with acetone (8.4); determine the liquid volume, and place in clean labeled bottle (Container No. 6).

11.7 *Laboratory Particulate Matter Analysis*—Perform the remaining analysis after the samples have been transported back to the laboratory.

11.7.1 *Container No. 1 (Primary Filter)*— Heat and reweigh the filter material using the procedures described in 11.1.2.

11.7.2 *Container No. 2 (Secondary Filter)*— Heat and reweigh flat filters using the procedures described in 11.1.1.

11.7.3 *Container No. 3 (Acetone Wash of Nozzle and Connecting Fittings)*—Evaporate the solvent rinse liquid from the dry filtration system (Container No. 3) at room temperature, 25 ± 5°C (77 ± 9°F) in normal atmosphere, desiccate to a constant mass, and weigh to the nearest 0.1 mg.

11.7.4 *Container No. 4 (Additional Wash of Nozzle and Connecting Fittings)*—Evaporate the additional wash solution from the dry filtration system, if any (Container No. 4), in a steam bath (7.17.11) to dryness, desiccate to a constant mass, and weigh to the nearest 0.1 mg.

11.7.5 *Blanks on Wash Liquids*—Run blanks on all solvents, solutions, acetone, toluene, and reagent water used in 11.5.4, 11.5.5, 11.6.1, and 11.6.2. Adjust weights in 11.7.3 and 11.7.4 to remove any mass contributed by liquids.

11.8 *Laboratory-Collected Residue Analysis*—Perform the remaining analysis after the samples have been transported back to the laboratory.

11.8.1 *Container No. 5 (Impinger-Condenser Liquid and Water Wash)*—Transfer the liquid from 11.6.1 (Container No. 5) to a tared container and evaporate in a steam bath (7.17.11) to dryness. Desiccate to a constant mass and weigh to the nearest 0.1 mg.

11.8.2 *Container No. 6 (Acetone Wash of Impinger-Condensers)*—Transfer the rinse liquid from 11.6.2 (Container No. 6) to a tared dish, evaporate at room temperature (25 ± 5°C (77 ± 9°F)), desiccate to a constant mass, and weigh to the nearest 0.1 mg.

11.8.3 *Blanks and Wash Liquids*—Run blanks on all solvents, solutions, and distilled water used in 11.8.1 and 11.8.2 to remove any mass contributed by liquids.

11.9 *Sample Mass Determination*:

11.9.1 *Particulate Matter Sample Mass*— Determine the particulate matter sample mass by subtracting the tare mass from the final mass for the primary and secondary filters and sum up the mass gain from Containers Nos. 1, 2, 3, and 4, using the form referenced in 14.1.

11.9.2 *Collected Residue Sample Mass*— Determine the collected residue mass by adding the residue from Containers Nos. 5 and 6 using the form referenced in 14.1.

12. Calculation

12.1 Average the dry gas meter temperatures and the orifice pressure drops.

12.2 *Dry Gas Volume*—Correct the sample volume measured by the dry gas meter (7.10.2) to standard conditions [298 K (537°R) and 101.3 kPa (29.9 in. Hg)] using (Eq 7):

$$\begin{aligned} V_{m_{\text{std}}} &= V_m C_m \left(\frac{T_{\text{std}}}{T_m}\right)\left(\frac{P_{\text{bar}} + \Delta H}{P_{\text{std}}}\right) \\ &= K_1 V_m C_m \left(\frac{P_{\text{bar}} + \Delta H}{T_m}\right) \end{aligned} \quad (7)$$

where:

K_1 = 2.95 K/kPa for SI units, and
= 17.9°R/in. Hg for U.S. customary units.

12.2.1 *Actual Gas Volume*—Correct the sample volume measured by the dry gas meter (7.10.2) to actual gas sample conditions using (Eq 8):

$$V_{m_{\text{act}}} = (V_m + V_{w_{\text{act}}}) C_m \left(\frac{(T_s)_{\text{avg}}}{T_m}\right)\left(\frac{P_{\text{bar}} + \Delta H}{P_s}\right) \quad (8)$$

12.3 *Volume of Water Vapor*:

12.3.1 *Corrected to Standard Conditions*:

$$\begin{aligned} V_{w_{\text{std}}} &= V_{lc}\left(\frac{\rho_{H_2O}}{M_{H_2O}}\right)\left(\frac{RT_{\text{std}}}{P_{\text{std}}}\right), \text{ thus} \\ V_{w_{\text{std}}} &= K_2 V_{lc} \end{aligned} \quad (9)$$

TABLE 1 Particulate Collectors Recommended for Use in Sampling Conditions Indicated

Condition No.	Expected Sample Weight		Flue Temperature			Liquid Droplets Present		Collector Recommended (The Figure in Parentheses is the Minimum Sample Weight Recommended)
	100 mg	100 mg	200°F	200 to 300°F	300 to 1000°F	Yes	No	
1	X		X	X		X	X	Versapor (24 mg)
2	X		X	X	X	X	X	Packed tube (20 mg)
3			X	X	X			Glass fiber (11 mg)
4		X	X	X	X		X	Alundum thimble (100 mg)
5	X		X	X		X	X	Quartz filter Pallflex QAO-2400 or Equal

where:

K_2 = 1.36×10^{-3} m^3/mL for SI units, and
= 4.76×10^{-2} ft^3/mL for U.S. customary units.

12.3.2 *Corrected to Actual Conditions*:

$$V_{w_{\mathrm{act}}} = V_{lc} \left(\frac{\rho_{H_2O}}{M_{H_2O}} \right) \left(\frac{R\,T_s}{P_s} \right) \tag{10}$$

12.4 *Moisture Content*:

$$B_{wo} = \frac{V_{w_{\mathrm{std}}}}{V_{m_{\mathrm{std}}} + V_{w_{\mathrm{std}}}} \tag{11}$$

12.5 *Dry Molecular Weight (Test Method D 3154)*—Use (Eq 12) to calculate dry molecular weight and average the runs. Report the results to the nearest tenth.

$$M_d = 0.440\,(\%\,CO_2) + 0.320\,(\%\,O_2) + 0.280\,(\%\,N_2 + \%\,CO) \tag{12}$$

where:

$\%\,CO_2$ = percent carbon dioxide by volume, dry basis,
$\%\,O_2$ = percent oxygen by volume, dry basis,
$\%\,N_2$ = percent nitrogen by volume, dry basis,
0.440 = molecular weight of carbon dioxide, divided by 100,
0.320 = molecular weight of oxygen, divided by 100, and
0.280 = molecular weight of nitrogen or carbon monoxide, divided by 100.

12.6 *Stack Gas Molecular Weight (Wet Basis) (Test Method D 3154)*:

$$M_s = M_d\,(1 - B_{WO}) + 18.0(B_{WO}) \tag{13}$$

12.7 *Stack Gas Pressure*:

$$P_s = P_{\mathrm{bar}} \pm P_{stat} \tag{14}$$

12.8 *Stack Gas Velocity (Test Method D 3154)*—Use (Eq 15) to calculate the stack gas velocity, as follows:

$$(V_s)_{\mathrm{avg}} = K_p\,C_p\,(\sqrt{\Delta P_{\mathrm{avg}}})\,\sqrt{\frac{(T_s)_{\mathrm{avg}}}{P_s M_s}} \tag{15}$$

where:

K_p, SI constant = 129 m/s [g/g-mol-K]$^{1/2}$ and
K_p, U.S. customary constant = 314 ft/s [lb/lb-mole °R]$^{1/2}$.

12.9 *Stack Gas Volumetric Flow Rate*— Use (Eq 16) to calculate the stack gas volumetric flow rate, as follows:

$$Q_{\mathrm{stp-d}} = [3.60 \times 10^3]\,[(1 - B_{WO})(V_s)_{\mathrm{avg}}]A \left(\frac{T_{\mathrm{std}}}{(T_s)_{\mathrm{avg}}} \right) \left(\frac{P_s}{P_{\mathrm{std}}} \right) \tag{16}$$

12.10 *Concentration*:

12.10.1 *Concentration of Particulate Matter*:

12.10.1.1 For SI:

$$C'_{P.M.} = \frac{(P.M.)}{V_{m_{\mathrm{st}}}} \tag{17}$$

on the dry basis, and

$$C'_{P.M._{\mathrm{act}}} = \frac{(P.M.)}{V_{m_{\mathrm{act}}}} \tag{18}$$

at actual gas conditions.

12.10.1.2 For the U.S. customary system:

$$C'_{P.M.} = (1.54 \times 10^{-2}\ \mathrm{g/mg}) \left(\frac{(P.M.)}{V_{m_{\mathrm{st}}}} \right) \tag{19}$$

on the dry basis, and

$$C'_{P.M._{\mathrm{act}}} = (1.54 \times 10^{-2}\ \mathrm{g/mg}) \left(\frac{(P.M.)}{V_{m_{\mathrm{act}}}} \right) \tag{20}$$

at actual gas conditions.

12.10.2 *Concentration of Collected Residue*:

12.10.2.1 For SI:

$$C^2_{pm} = \frac{(pm)}{V_{m_{\mathrm{st}}}} \tag{21}$$

on the dry basis, and

$$C^2_{pm_{\mathrm{act}}} = \frac{(pm)}{V_{m_{\mathrm{act}}}} \tag{22}$$

at actual gas conditions.

12.10.2.2 For the U.S. customary system:

$$C^2_{pm} = (1.54 \times 10^{-2}\ \mathrm{g/mg})\,\frac{(pm)}{V_{m_{\mathrm{st}}}} \tag{23}$$

on the dry basis, and

$$C^2_{pm_{\mathrm{act}}} = (1.54 \times 10^{-2}\ \mathrm{g/mg})\,\frac{(pm)}{V_{m_{\mathrm{st}}}} \tag{24}$$

at actual gas conditions.

12.11 *Emission Rates*:

12.11.1 *Particulate Matter Emission Rate*:

12.11.1.1 For SI:

$$E_{P.M.} = (1.00 \times 10^{-6}\ \text{kg/mg})C'_{P.M.}(Q_{\text{stp}-\text{d}}) \tag{25}$$

12.11.1.2 For the U.S. customary system:

$$E_{P.M.} = \frac{C'_{P.M.}(Q_{\text{stp}-\text{d}})}{7.00 \times 10^{3}} \tag{26}$$

where:

7.00×10^{3} = grains/lb.

12.11.2 *Collected Residue Emission Rate*:

12.11.2.1 For SI:

$$E^{2}_{pm} = (1.00 \times 10^{-6}\ \text{kg/mg})(C^{2}_{pm})(Q_{\text{stp}-\text{d}}) \tag{27}$$

12.11.2.2 For the U.S. customary system:

$$E^{2}_{pm} = \frac{C^{2}_{pm}(Q_{\text{stp}-\text{d}})}{7.00 \times 10^{3}} \tag{28}$$

where:

7.00×10^{3} = grains/lb.

12.12 *Variation From Isokinetic*:

$$I = \frac{T_s\left[\dfrac{(V_{lc})(\rho_{H_2O})(R)}{M_{H_2O}} + (V_m/T_m)(P_{\text{bar}} + \Delta H)\right]}{(60.0)(\theta)(V_s)_{\text{avg}}\,P_s A_n} \times 100 \tag{29}$$

$$= \frac{T_s(1.67\ \text{min/s})[(K_3V_{lc}) + (V_m/T_m)(P_{\text{bar}} + \Delta H)]}{(\theta)(V_s)_{\text{avg}}\,P_s A_n}$$

12.12.1 *From Raw Data*:

$$I = \frac{100\ T_s\left[K_3V_{lc} + \left(\dfrac{V_m}{T_m}\right)\left(P_{\text{bar}} + \dfrac{\Delta H}{13.6}\right)\right]}{60\ \theta\ V_s P_s A_n} \tag{30}$$

where:

K_3 = 4.63×10^{-4}(kPa·m^3/mL·K) for SI, and
= 2.676×10^{-3} (in. Hg·ft^3/mL·°R) for the U.S. customary system.

12.12.2 *From Intermediate Values*:

$$I = \frac{100\ T_s V_{m(\text{std})} P_{\text{std}}}{60\ \theta\ V_s P_s A_n\ T_{\text{std}}(1 - B_{ws})} \tag{31}$$

$$= K_4 \frac{T_s V_{m(\text{std})}}{\theta\ V_s P_s A_n\,(1 - B_{ws})}$$

where:

K_4 = 0.566 kPa/K for SI, and
= 0.0944 in. Hg/°R.

12.12.3 *Acceptable Results*—The following range sets the limit on acceptable isokinetic sampling:

12.12.3.1 If 90 % $\leq$ I $\leq$ 110 %, the results are acceptable; otherwise, reject the results and repeat the test.

13. Quality Assurance Procedures

13.1 The EPA QA Manuals EPA-600/9-76-005 **(2)** and EPA-600/4-77-0276 **(3)** contain useful quality assurance criteria for performing these test methods.

14. Records

14.1 The EPA QA Manual (EPA-600/4-77-027b) **(3)** contains data recording forms that may be used to document calibration and testing of equipment, and for recording field test data.

15. Precision and Bias (4-94–9)

15.1 These test methods, as presented, have not been subjected to collaborative testing or other types of replicate testing. The test methods do, however, involve basic equipment and procedures that are common to other particulate matter sampling methods that have undergone collaborative testing and the data from these tests are considered indicative of the precision attainable with these test methods.

15.1.1 The Test Method A train is similar to the train tested in Project Threshold.[9] The alternative train is similar to EPA Reference Method 17, and the Test Method B train is similar to EPA Reference Method 5, both of which have been collaboratively tested by the Environmental Protection Agency. The collected residue data obtained in Project Threshold are more limited than the particulate matter data and, as described in 15.5, may not be representative of the capabilities of the test method in the hands of a competent test team.

15.2 The precision of particulate and collected residue sampling methods data is potentially influenced by many factors. These include source variables, procedural variables, and equipment variables. Strict adherence to these methods is necessary to reduce the effect of these variables. Failure to (*1*) ensure a leak-free system, (*2*) calibrate accurately all indicated system components, (*3*) select a proper sampling location, and (*4*) follow prescribed sample recovery and analysis procedures can, for example, each seriously effect the precision of the results.

15.3 Collaborative testing of particulate matter sampling methods has been undertaken under varying degrees of control ranging from tests in which teams of varying experience performed tests and reported data without supervision by the collaborative test director, to one test where careful control was exercised to ensure adherence to the test methods (**4**).

15.3.1 Each collaborative test involved multiple runs by four to eight test teams sampling simultaneously. In the former tests, the participants were noted to deviate from the prescribed test methods. Therefore, as expected, the precision attained during this latter controlled test was much better than that attained at other tests where deviations from the test methods were known to have occurred. The data obtained during these collaborative tests and reported in Table 2 show that the particulate matter sampling method, conducted by a competent test team, is capable of a between-laboratory coefficient of variation of 10 to 12 % for single samples in the range from 30 to 170 mg/m^3. For three runs, as is common in particulate sampling, this coefficient of variation reduces to 6 to 7 % of a mean.

15.3.2 Failure to adhere precisely to the method can result in much larger coefficients of variation **(5)**. Although the precision data reported here and in Table 2 are expressed in terms of coefficient of variation, the user is cautioned that the data are limited and that the concentration dependence of the precision data is not clearly established. An examination of the types of

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[9] The results of Project Threshold, a comprehensive program to validate ASTM methods of measuring various atmospheric contaminants, are available as a series of publications by ASTM.

TABLE 2 Results of Collaborative Tests

Site	Mean Concentration mg/dscm	C. V.
Oil-fired power plant:		
Project Threshold **(3)**	30	10.0
Coal-fired power plant:		
Project Threshold **(3)**	330	7.6
EPA **(4)**	200	36.7
Foundry:		
Project Threshold **(3)**	3.5	45.1
Cement kiln:		
Project Threshold **(3)**	13 000	6.0
EPA **(5)**	50	58.4
Municipal incinerator:		
EPA **(6)**	125	38.7
EPA **(1)**	170	12.1

errors which may affect precision in particulate matter sampling indicates that certain types of errors, for example, calibration errors and weighing errors, are largely independent of concentration. Other errors, for example, contamination errors and errors resulting from loss of sample, may be a function of concentration. Both types of errors may significantly affect the precision of the test methods. This suggests that the standard deviation may approach a constant non-zero lower limit as the concentration approaches zero. Similarly, the coefficient of variation may tend to decrease at higher concentration. While the data are not sufficient to quantify this, the Project Threshold foundry data and cement kiln data indicate this trend.

15.4 Results of eight particulate matter sampling collaborative tests are described in Table 2.

15.5 Higher variability exists in the data for collected residue. The coefficient of variation estimated from collected residue data obtained in Project Threshold ranged from 25 to 93 %. For the oil-fired power plant, collected residue averaged 20 mg/m^3, with CV of 58.4 %; variability drops to under 50 % when data from one of the four teams (with a faulty thimble) is excluded **(2)**. CV for combined dry catch and collected residue was not calculated for Project Threshold. However, analysis of some of the data for the two power plant tests shows less deviation (between-laboratory CV) in the combined-mass result than in either catch reported separately. This is apparently because the deviations tended to cancel each other out. This might be expected, for instance, in the case of a faulty filter seal cutting the dry catch and increasing the load deposited into the impingers.

16. Keywords

16.1 EPA Method 5; EPA Method 17; isokinetic sampling; particulate matter; particulate matter sampling; stack sampling

REFERENCES

(**1**) APTD-0576 “Maintenance, Calibration and Operation of Isokinetic Source Sampling Equipment,” Superintendent of Documents, U.S. Government Printing Office, Washington, DC 20402.

(**2**) EPA-600/9-76-005 “Quality Assurance Handbook for Air Pollution Measurement System. Vol I. Principles,” Superintendent of Documents, U.S. Government Printing Office, Washington, DC 20402.

(**3**) EPA-600/4-77-027b “Quality Assurance Handbook for Air Pollution Measurement Systems. Vol III. Stationary Source Specific Measurements,” Superintendent of Documents, U.S. Government Printing Office, Washington, DC 20402.

(**4**) Hamil, H. F., and Thomas, R. E., “Collaborative Study of Particulate Emissions Measurements by EPA Methods 2, 3, and 5 Using Paired Particulate Sampling Trains (Municipal Incinerators),” U.S. Environmental Protection Agency (EPA), Research Triangle Park, NC, Report No. EPA-600/4-76-014, 1976[5].

(**5**) Midgett, M. R., “The EPA Program for the Standardization of Stationary Source Emission Test Methodology—A Review,” U.S. Environmental Protection Agency (EPA), Research Triangle Park, NC, Report No. EPA-600/4-76-044, August 1976[5].

(**6**) Howes, J. E., Pesut, R. N., and Foster, J. P., “Interlaboratory Cooperative Study of the Precision of Sampling Stacks for Particulate and Collected Residue,” *ASTM DS 55-56, American Society of Testing and Materials*, 1975.

(**7**) Hamil, H. F., and Thomas, R. E., “Collaborative Study of Method for the Determination of Particulate Matter Emissions from Stationary Sources (Fossil Fuel-Fired Steam Generators),” U.S. Environmental Protection Agency (EPA), Research Triangle Park, NC, Report No. EPA-650/4-74-021, 1974[5].

(**8**) Hamil, H. F., and Camann, D. E., “Collaborative Study of Method for the Determination of Particulate Matter Emissions from Stationary Sources (Portland Cement Plants),” U.S. Environmental Protection Agency (EPA), Research Triangle Park, NC, Report No. EPA-650/4-74-029, 1974[5].

(**9**) Hamil, H. F., and Thomas, R. E., “Collaborative Study of Method for the Determination of Particulate Matter Emissions from Stationary Sources (Municipal Incinerators),” U.S. Environmental Protection Agency (EPA), Research Triangle Park, NC, Report No. EPA-650/4-74-022, 1974[5].