



**Designation: ~~D3685/D3685M – 98 (Reapproved 2005)~~ D3685/D3685M – 13**

## Standard Test Methods for Sampling and Determination of Particulate Matter in Stack Gases<sup>1</sup>

This standard is issued under the fixed designation D3685/D3685M; 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 ( $\epsilon$ ) 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.*

<sup>1</sup> This test method is under the jurisdiction of ASTM Committee D22 on Air Quality and is the direct responsibility of Subcommittee D22.03 on Ambient Atmospheres and Source Emissions.

Current edition approved March 1, 2005. Published May 2005. Originally approved in 1978. Last previous edition approved in 1998 as ~~D3685/D3685M – 98~~ D3685/D3685M – 98 (2005). DOI: ~~10.1520/D3685\_D3685M-98R05-10~~ 10.1520/D3685\_D3685M-13.

## 2. Referenced Documents

### 2.1 ASTM Standards:<sup>2</sup>

- [D1071 Test Methods for Volumetric Measurement of Gaseous Fuel Samples](#)
- [D1193 Specification for Reagent Water](#)
- [D1356 Terminology Relating to Sampling and Analysis of Atmospheres](#)
- [D2986 Practice for Evaluation of Air Assay Media by the Monodisperse DOP \(Dioctyl Phthalate\) Smoke Test \(Withdrawn 2004\)<sup>3</sup>](#)
- [D3154 Test Method for Average Velocity in a Duct \(Pitot Tube Method\)](#)
- [D3631 Test Methods for Measuring Surface Atmospheric Pressure](#)
- [D3796 Practice for Calibration of Type S Pitot Tubes](#)
- [D4536 Test Method for High-Volume Sampling for Solid Particulate Matter and Determination of Particulate Emissions \(Withdrawn 2000\)<sup>3</sup>](#)
- [E1 Specification for ASTM Liquid-in-Glass Thermometers](#)
- [E2251 Specification for Liquid-in-Glass ASTM Thermometers with Low-Hazard Precision Liquids](#)

## 3. Terminology

3.1 *Definitions*—For definitions of terms used in these test methods, refer to Terminology [D1356](#).

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, $mg/m^3$ ( $gr/dsft^3$ )
$C'_{P.M.}$	= concentration of particulate matter in stack gas, at actual gas conditions, $mg/m^3$ ( $gr/af t^3$ ).
$C^2_{P.M.}$	= concentration of collected residue in stack gas, dry basis, standard conditions, $mg/m^3$ ( $gr/dsft^3$ ).
$C^2_{pm}$	= concentration of collected residue in stack gas, at actual conditions, $mg/m^3$ ( $gr/af t^3$ ).
$E_{P.M.}$	= 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, $g/g\text{-mol}$ ( $lb/lb\text{-mole}$ ).
$M_{H_2O}$	= molecular weight of water, $18.0 g/g\text{-mol}$ ( $18.0 lb/lb\text{-mole}$ ).
$M_s$	= molecular weight of stack gas, wet basis, $g/g\text{-mol}$ ( $lb/lb\text{-mole}$ ).
$P_{bar}$	= 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).
$Q_{stp-d}$	= stack gas volumetric flow rate, dry basis, standard conditions, $m^3/h$ ( $dsft^3/h$ ).
$R$	= ideal gas constant = $8.32 \times 10^{-3}$ ( $kPa \cdot m^3$ )/( $K \cdot g - mol$ ) for the SI system, and $21.8$ (in. Hg· $ft^3$ )/( $^{\circ}R \cdot lb - mole$ ) 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, $K$ ( $^{\circ}R$ ) (see <a href="#">10.2.2.6</a> and <a href="#">10.2.2.9</a> ).
$T_m$	= absolute average dry gas meter temperature, $K$ ( $^{\circ}R$ ).
$(T_s)_{avg}$	= absolute average stack gas temperature, $K$ ( $^{\circ}R$ ).
$T_{std}$	= absolute temperature at standard conditions, $298 K$ ( $25^{\circ}C$ ) ( $537^{\circ}R$ ).
$T_w$	= temperature of the gas in the wet test meter, $K$ ( $^{\circ}R$ ) (see <a href="#">10.2.2.6</a> and <a href="#">10.2.2.9</a> ).
$V_d$	= gas volume passing through the dry gas meter, $K$ ( $^{\circ}R$ ) (see <a href="#">10.2.2.6</a> and <a href="#">10.2.2.9</a> ).

<sup>2</sup> For referenced ASTM standards, visit the ASTM website, [www.astm.org](http://www.astm.org), or contact ASTM Customer Service at [service@astm.org](mailto:service@astm.org). For *Annual Book of ASTM Standards* volume information, refer to the standard's Document Summary page on the ASTM website.

<sup>3</sup> The last approved version of this historical standard is referenced on [www.astm.org](http://www.astm.org).

$V_{lc}$	= 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 <sup>3</sup> ).
$V_{m_{act}}$	= volume of gas sample through the dry gas meter, corrected to actual gas conditions, $m^3$ (or aft <sup>3</sup> ).
$V_{m_{std}}$	= volume of gas sample through the dry gas meter, corrected to dry standard conditions, $m^3$ (dft <sup>3</sup> ).
$(V_s)_{avg}$	= average stack gas velocity, m/s (ft/s).
$V_{m_{std}}$	= volume of water vapor in the gas sample, corrected to actual conditions, $m^3$ (dsft <sup>3</sup> ).
$V_w$	= gas volume passing through the wet test meter, $m^3$ (aft <sup>3</sup> ) (see 10.2.2.6 and 10.2.2.9).
$V_{w_{std}}$	= volume of water vapor in the gas sample, corrected to dry standard conditions, $m^3$ (dsft <sup>3</sup> ).
$Y$	= 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).
$\theta$	= total sampling or calibration run time, min.
$\rho_{H_2O}$	= density of water, 997 kg/m <sup>3</sup> , at 298 K.
$\Delta H$	= average pressure drop across the orifice meter, kPa (in. H <sub>2</sub> O).
$\Delta H@$	= average orifice pressure differential that develops 0.021 m <sup>3</sup> (0.75 ft <sup>3</sup> ) of air at standard conditions for all six calibration runs, kPa (in. H <sub>2</sub> O) (see 10.2.2.9).
$\Delta H@_i$	= orifice pressure differential at each flow rate that gives 0.021 m <sup>3</sup> (0.75 ft <sup>3</sup> ) of air at standard conditions for each calibration run, kPa (in. H <sub>2</sub> O) (see 10.2.2.9).
$\Delta P_{avg}$	= average stack gas velocity head, kPa (in. H <sub>2</sub> O).

NOTE 1—To convert  $\Delta H$  and  $\Delta P_{avg}$  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 condensers set in an ice bath. The moisture condensers 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<sub>2</sub>) 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<sub>4</sub>) or the reaction with ammonia gas (NH<sub>3</sub>) to form ammonium sulfate (NH<sub>4</sub>SO<sub>4</sub>) and the potential reaction of hydrogen fluoride (HF) with glass components in the sample train with resultant collection of silicon tetrafluoride (SiF<sub>4</sub>) in the impingers.

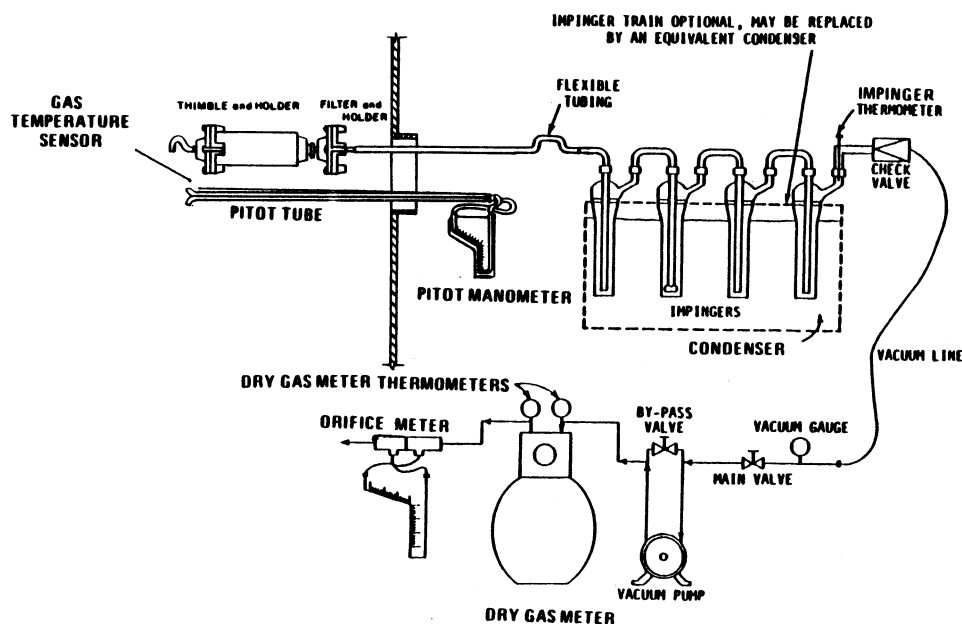


FIG. 1 Test Method A (In Stack) Sampling Train

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 PTFE 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 <math><30^\circ</math> 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 D4536) 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:

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

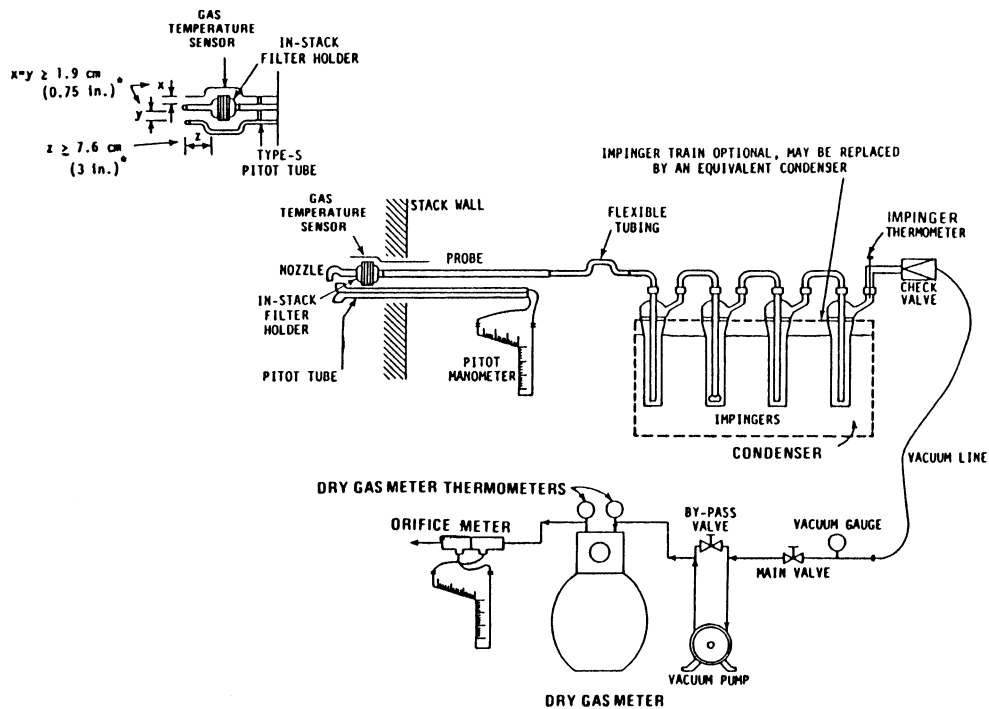


FIG. 2 (Out of Stack) Sampling Train

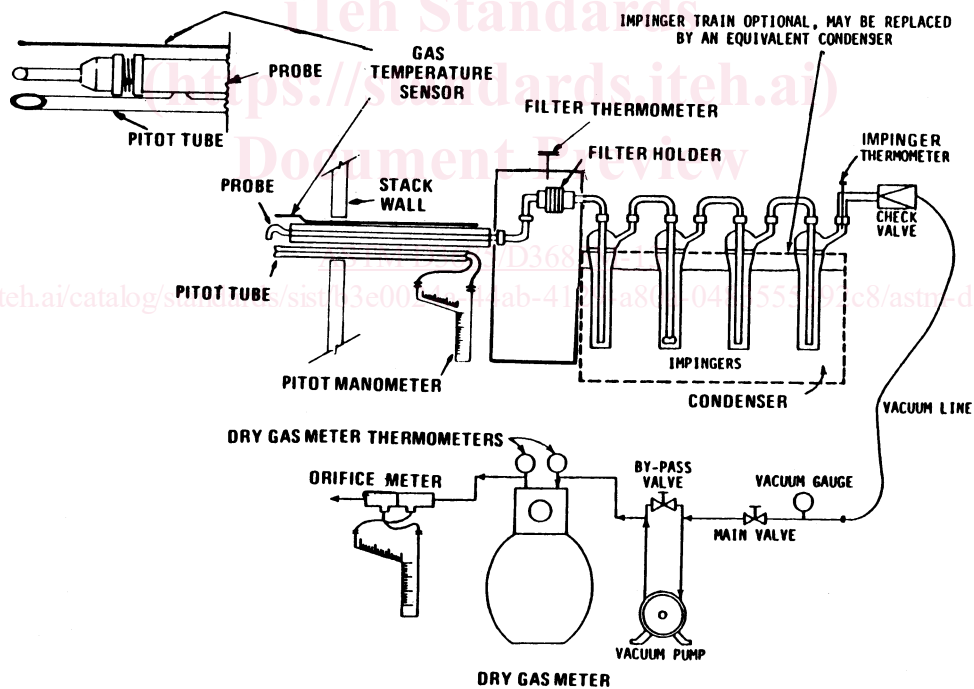


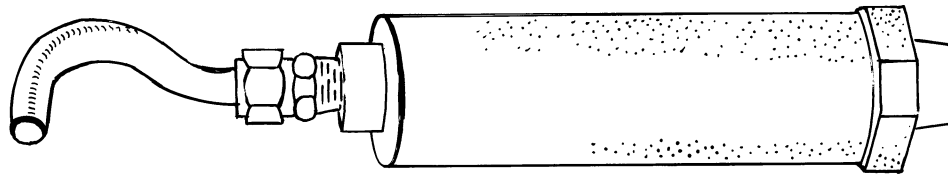
FIG. 3 Test Method A (In Stack Optional) Sampling Train

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.

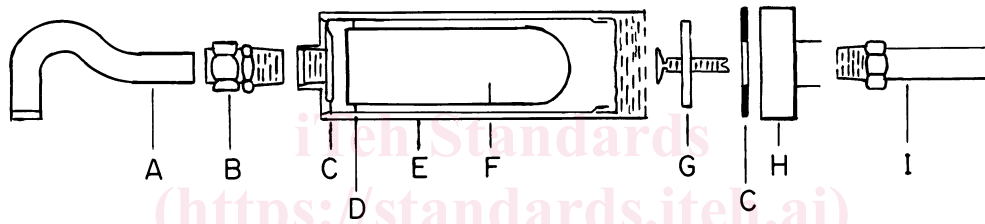
7.4.1.2 *Alternate Filter Holder/Backup Filter Holder*—An in-stack filter holder constructed of borosilicate or quartz glass, or stainless steel is shown in Figs. 5 and 6. Use a silicone rubber, TFE-fluorocarbon, or stainless steel gasket. The holder shall be durable, easy to load, and leak free in normal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

(1) 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.





POROUS ALUMINUM OXIDE THIMBLE PROBE



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

FIG. 4 Thimble Holder

(2) 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<sup>3</sup>/min) is allowed; however, under these laboratory conditions the entire train shall be leak-less.

(3) 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.

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

(5) 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.

(6) 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.

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

(8) 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.

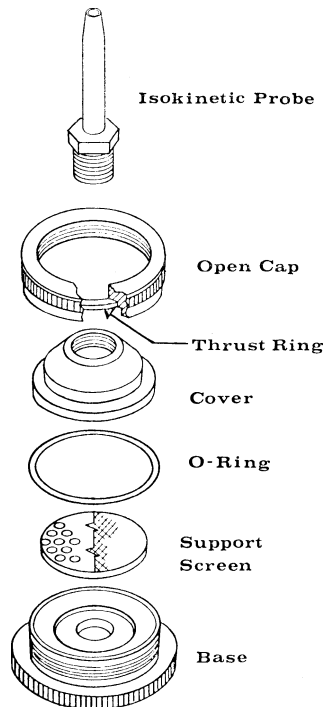


FIG. 5 Exploded Diagram for Flat, Round Filters

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 condensable 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 condensables are to be retained. Use probe extenders of nickel-iron-chromium alloy (UNS N08825) (see DS 561<sup>4</sup>), or equivalent at temperatures greater than 600°F (315°C). (Record probe material selection in the field data sheet.)

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 561<sup>4</sup>), 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<sup>3</sup>/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.

<sup>4</sup> *Metals and Alloys in the Unified Numbering System*, available from ASTM Headquarters.



FIG. 6 Typical Holder 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 unstricted 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 [D3154](#).

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  $\pm 1^\circ\text{C}$  ( $2^\circ\text{F}$ ) of true value in the range from 0 to  $25^\circ\text{C}$  (32 to  $77^\circ\text{F}$ ).

7.7 *Gas Temperature Sensor*—For measuring gas temperature to within  $\pm 1^\circ\text{C}$  ( $2^\circ\text{F}$ ). Permanently attach the temperature sensor to either the probe ([7.5](#)) or the pitot tube ([7.9](#)). (See [Figs. 1-3](#).)

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 **D3154** 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, gauges,~~ a vacuum pump, a dry gas meter with 2 % accuracy at the required sampling rate, thermometers capable of measuring  $\pm 3^{\circ}\text{C}$  ( $5.5^{\circ}\text{F}$ ) of true value in the range from 0 to  $90^{\circ}\text{C}$  ( $32$  to  $194^{\circ}\text{F}$ ), pressure ~~gage, gauge,~~ 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—Gauge*—Two inclined manometers or the equivalent as specified in Test Method **D3154**. 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, gauges~~ against a ~~gage-oil gauge-oil~~ manometer at a minimum of three points: 5, 125, and 250 Pa (0.025 in., 0.5 in., and 1.0 in.  $\text{H}_2\text{O}$ ). The ~~gages, gauges~~ shall read within 5 % of the ~~gage-oil gauge-oil~~ manometer at each test point. Repair or reject any ~~gage, gauge~~ 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 ( $0.75\text{ ft}^3/\text{min}$ )) 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  $3^{\circ}\text{C}$  ( $5.5^{\circ}\text{F}$ ) in the range from 0 to  $90^{\circ}\text{C}$  ( $32$  to  $194^{\circ}\text{F}$ ) 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 ( $0.75\text{ ft}^3/\text{min}$ ) for a pump inlet vacuum of 50 kPa (15 in. Hg), is used to draw the gas sample.

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

7.11 *Nomograph*, to determine the isokinetic sampling rate in accordance with APTD-0576 **(1)**.<sup>5</sup> Its function may be applied with a hand-held programmable calculator as described in **9.2.1.3**.

7.12 *Thermometers—Thermometers*—Thermometers conforming to Specification **E1**, for calibration of sample train thermometers/thermocouples, as follows:

Thermometer	Section
3C or F	10.6
<del>59C or F</del>	<del>10.5 and 11.4.6</del>
S59C or F	10.5 and 11.4.6
<del>63C or F</del>	<del>10.4</del>
S63C or F	10.4
113C or F	10.3

ASTM thermometers, S59C and S63C as identified in Specification **E2251** may be substituted for thermometers 59C and 63C directly. In addition, precision digital thermometers based on resistance temperature detectors (RTDs), thermistors, or thermocouples, or organic liquid-in-glass thermometers with equivalent or better accuracy and precision in the appropriate temperature range may be used.

7.13 *Barometer—A mercury, aneroid, An aneroid or other barometer capable of measuring atmospheric pressure to within  $\pm 300$  Pa (25 mm Hg) shall be used. Calibrate the barometer ~~against a mercury-in-glass barometer or the equivalent,~~ as described in Test Methods **D3631**.*

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  $\text{m}^3/\text{h}$  ( $120\text{ ft}^3/\text{h}$ ) or 30 L for each revolution ( $1\text{ ft}^3/\text{rev}$ ) with an accuracy of  $\pm 1.0\%$ , shall be used to calibrate the dry test meter.

7.15 *Orsat Gas Analyzer*—Stack gas analyzer as described by Test Method **D3154** shall be used.

7.16 *U-Tube Manometer*—A water manometer or mercury instrument pressure sensor capable of measuring gas pressure to within 0.33 kPa (2.5 mm Hg or 0.001 in.  $\text{H}_2\text{O}$ ).

<sup>5</sup> The boldface numbers in parentheses refer to the list of references at the end of this standard.

### 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  $\text{CaSO}_4$  (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:*

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  $\pm 0.1$  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.<sup>6</sup>

8.2 *Purity of Water*—Unless otherwise specified, water shall be Type III reagent water conforming to Specification **D1193**.

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  $\leq 0.001$  % 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* ( $\text{CaSO}_4$ ), indicating type, for use in desiccator (7.17.8).

8.6 *Crushed Ice*.

8.7 *Silica Gel* ( $\text{SiO}_2$ ), 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 ( $\frac{1}{4}$  in.) wide.

8.13 *Toluene*.

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

8.15 *Filter Material:*

<sup>6</sup> *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.