



Designation: 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, health, and environmental practices and determine the applicability of regulatory limitations prior to use.*

1.13 *This international standard was developed in accordance with internationally recognized principles on standardization established in the Decision on Principles for the*

<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.

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*Development of International Standards, Guides and Recommendations issued by the World Trade Organization Technical Barriers to Trade (TBT) Committee.*

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

<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).

- $C^2_{pm}$  = concentration of collected residue in stack gas, dry basis, standard conditions,  $mg/m^3$  ( $gr/dsft^3$ ).
- $C^2_{pm.act}$  = 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,  $g/g\text{-mol}$  ( $lb/lb\text{-mole}$ ).
- $M_{H_2O}$  = 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  $21.8 (in. Hg \cdot ft^3)/(^{\circ}R \cdot lb - mole)$  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 [10.2.2.6](#) and [10.2.2.9](#)).
- $T_m$  = 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 [10.2.2.6](#) and [10.2.2.9](#)).
- $V_d$  = gas volume passing through the dry gas meter,  $K$  ( $^{\circ}R$ ) (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,  $m^3$  ( $dft^3$ ).
- $V_{m.act}$  = volume of gas sample through the dry gas meter, corrected to actual gas conditions,  $m^3$  (or  $aft^3$ ).
- $V_{m.std}$  = volume of gas sample through the dry gas meter, corrected to dry standard conditions,  $m^3$  ( $dft^3$ ).
- $(V_s)_{avg}$  = average stack gas velocity,  $m/s$  ( $ft/s$ ).
- $V_{m.std}$  = volume of water vapor in the gas sample, corrected to actual conditions,  $m^3$  ( $dsft^3$ ).
- $V_w$  = gas volume passing through the wet test meter,  $m^3$  ( $aft^3$ ) (see [10.2.2.6](#) and [10.2.2.9](#)).
- $V_{w.std}$  = 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](#)).
- $\theta$  = total sampling or calibration run time,  $min$ .
- $\rho_{H_2O}$  = density of water,  $997 g/m^3$ , 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).

$\Delta P_{\text{avg}}$  = average stack gas velocity head, kPa (in. H<sub>2</sub>O).

NOTE 1—To convert  $\Delta H$  and  $\Delta P_{\text{avg}}$  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 Method

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.

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 <30° with the taper on the outside to establish a constant inside diameter (ID).

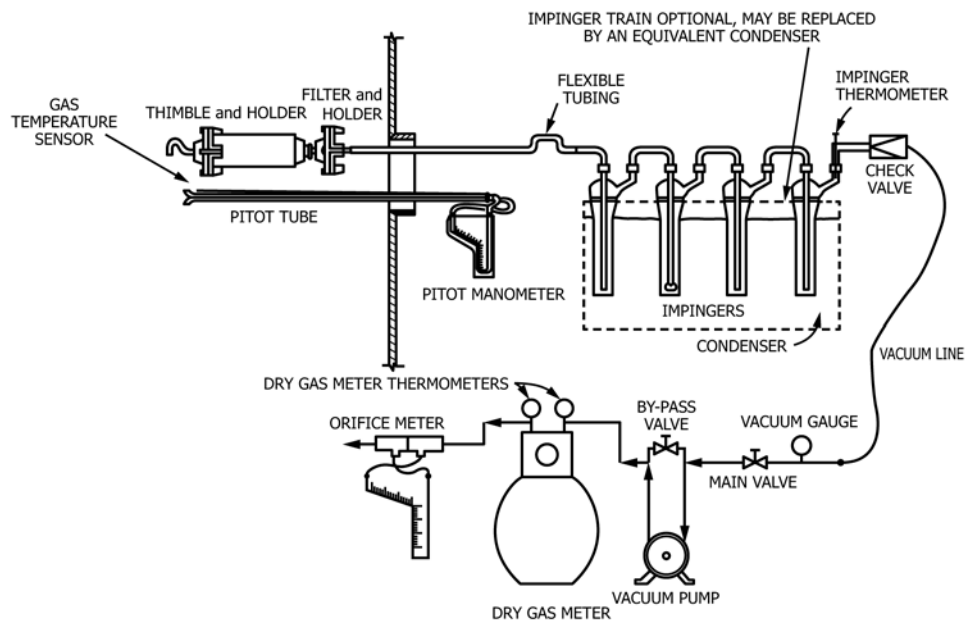


FIG. 1 Test Method A (In Stack) Sampling Train

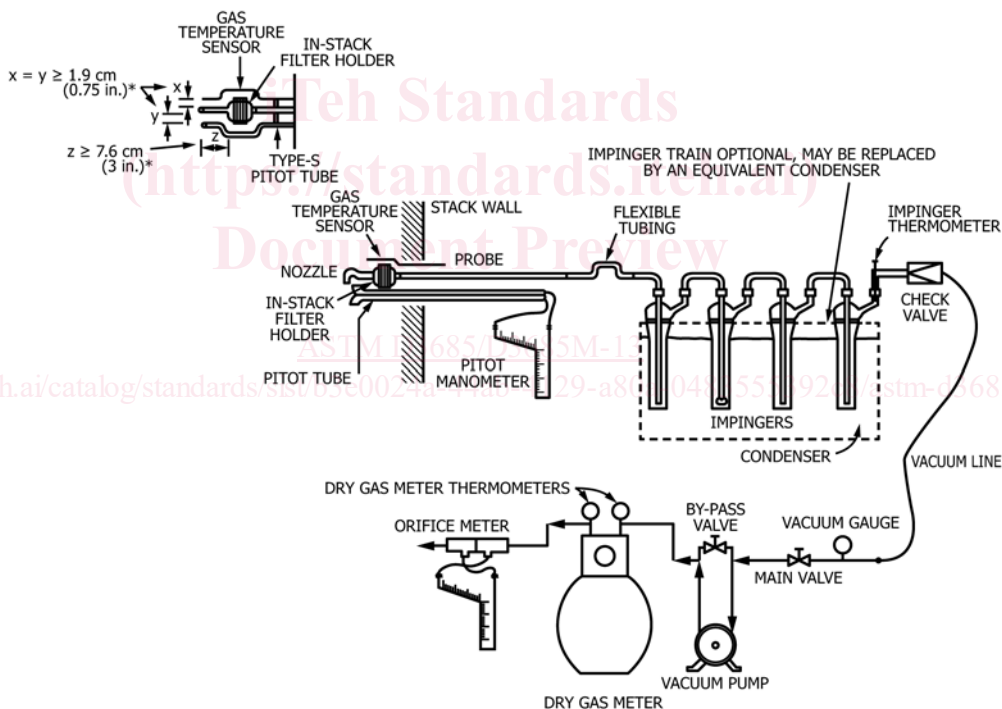


FIG. 2 (Out of Stack) Sampling Train

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



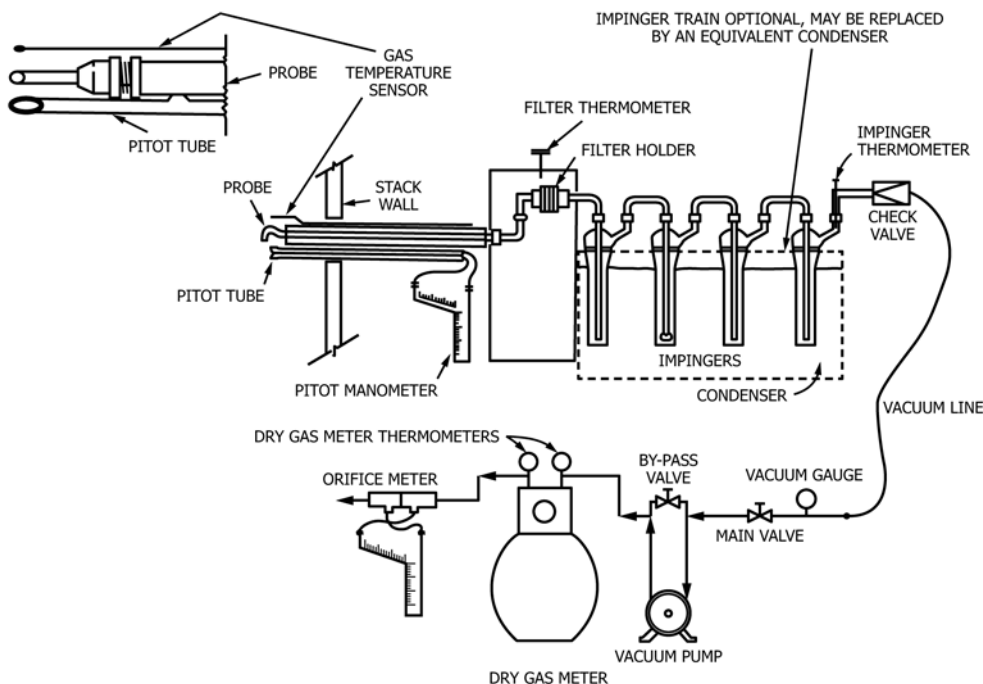


FIG. 3 Test Method A (In Stack Optional) Sampling Train

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 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.

(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.

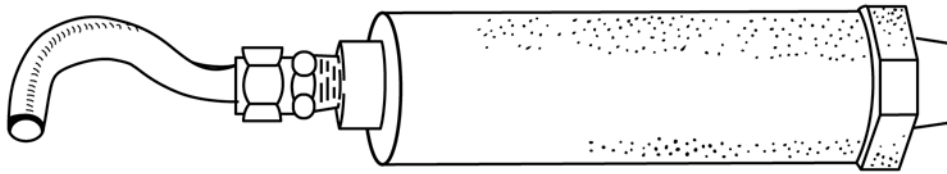
7.4.2.1 *Filter Heating System*, capable of maintaining the filter holder at 120 ± 15°C (248 ± 25°F) during sampling. Other temperatures may be specified for a particular application.

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

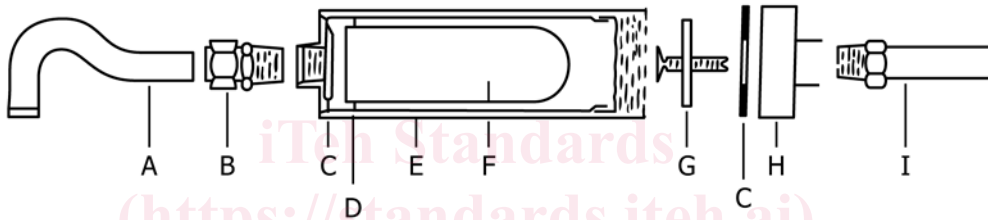
7.4.2.3 Before sampling, check the heating system and the temperature monitoring device. It is important that the heating element be easily replaceable in case of a malfunction during sampling.

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



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
G	Porous aluminum oxide
H	Clamp
I	Cap
	Adapter (holds holder to probe extension)

FIG. 4 Thimble Holder

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 condensibles are to be retained. Use probe extendors of nickel-iron-chromium alloy (UNS N08825) (see DS 56I<sup>4</sup>), or equivalent at temperatures greater than 600°F (315°C). (Record probe material selection in the field data sheet.)

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

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 56I<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