



Standard Test Method for Determination of Mass Concentration of Particulate Matter from Stationary Sources at Low Concentrations (Manual Gravimetric Method)¹

This standard is issued under the fixed designation D6331; 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 This test method² covers a method for the measurement of particulate matter (dust) concentration in emission gases in the concentrations below 20 mg/m³ standard conditions, with special emphasis around 5 mg/m³.

1.2 To meet the requirements of this test method, the particulate sample is weighed to a specified level of accuracy. At low dust concentrations, this is achieved by:

1.2.1 Exercising extreme care in weighing,

1.2.2 Extending the sampling time at conventional sampling rates, or

1.2.3 Sampling at higher rates at conventional sampling times (high-volume sampling).

1.3 This test method differs from Test Method [D3685/D3685M](#) by requiring the mass measurement of filter blanks, specifying weighing procedures, and requiring monitoring of the flue gas flow variability over the testing period. It requires that the particulate matter collected on the sample filter have a mass at least five times a positive mass difference on the filter blank. High volume sampling techniques or an extension of the sampling time may be employed to satisfy this requirement.

1.4 This test method may be used for calibration of automated monitoring systems (AMS). If the emission gas contains unstable, reactive, or semi-volatile substances, the measurement will depend on the filtration temperature, and in-stack methods may be more applicable than out-stack methods for the calibration of automated monitoring systems.

1.5 The values stated in SI units are to be regarded as the standard.

1.6 *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:³

[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)⁴

[D3154](#) Test Method for Average Velocity in a Duct (Pitot Tube Method)

[D3631](#) Test Methods for Measuring Surface Atmospheric Pressure

[D3670](#) Guide for Determination of Precision and Bias of Methods of Committee D22

[D3685/D3685M](#) Test Methods for Sampling and Determination of Particulate Matter in Stack Gases

[D3796](#) Practice for Calibration of Type S Pitot Tubes

[E1](#) Specification for ASTM Liquid-in-Glass Thermometers

[E2251](#) Specification for Liquid-in-Glass ASTM Thermometers with Low-Hazard Precision Liquids

2.2 ISO Standards:⁵

[ISO 5725](#) Precision of test methods—Determination of repeatability and reproducibility by inter-laboratory tests

[ISO 9096](#) Stationary source emissions—Determination of concentration and mass flow rate of particulate material in gas-carrying ducts. Manual gravimetric method

¹ 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 June 1, 2014. Published July 2014. Originally approved in 1998. Last previous edition approved in 2013 as D6331 – 13. DOI: 10.1520/D6331-14.

² This test method is based on ISO/CD 12141.3, “Stationary Source Emissions—Determination of Mass Concentration of Particulate Matter (Dust) at Low Concentrations—Manual Gravimetric Method”, available from International Organization for Standardization, Casa Postale 56, CH-1211, Geneva Switzerland.

³ For referenced ASTM standards, visit the ASTM website, www.astm.org, or contact ASTM Customer Service at service@astm.org. For *Annual Book of ASTM Standards* volume information, refer to the standard’s Document Summary page on the ASTM website.

⁴ The last approved version of this historical standard is referenced on www.astm.org.

⁵ Available from International Organization for Standardization, Casa Postals 56, CH-1211, Geneva, Switzerland.

ISO 10780 Stationary source emissions—Measurement of velocity and volume flow rate of gas stream in ducts

3. Terminology

3.1 For definitions of terms used in this test method, refer to Terminology **D1356**.

3.2 *Definitions of Terms Specific to This Standard:*

3.2.1 *filtration temperature*—the temperature of the sampled gas immediately downstream of the filter.

3.2.2 *high volume sampling*—sampling at higher rates than typical in Test Methods **D3685/D3685M** by using larger diameter nozzles and higher flow rates to maintain isokinetic sampling conditions.

3.2.2.1 *Discussion*—Nozzle sizes are typically 20 to 50 mm, with corresponding flow rates from 5 to 50 m³/s.

3.2.3 *hydraulic diameter, d_h*

$$d_h = \frac{4 \times \text{area of sampling plane}}{\text{perimeter of sampling plane}} \quad (1)$$

3.2.4 *in-stack filtration*—filtration in the stack or duct where the filter in its filter housing is placed immediately downstream of the sampling nozzle and collects particulate matter in the flue gas, under flue gas conditions.

3.2.5 *measurement series*—successive measurements carried out at the same sampling plane and at the same process conditions.

3.2.6 *out-stack filtration*—a sampling technique where the filter, in its filter housing, collects particulate matter under controlled temperature conditions outside of the stack or duct.

3.2.7 *overall blank*—the sample taken in a manner identical to the flue gas test samples, except that the sampling duration is shortened to less than 1 min.

3.2.7.1 *Discussion*—The overall blank value is expressed in the same units as the measurement result (for example, mg/m³) using the average sampling volume of the measurement series. The overall blank includes possible deposits on the filter and surfaces upstream of the filter in contact with the sample gas.

3.2.8 *particulate matter (dust)*—solid particles of any shape, structure, or density dispersed in the gas phase at flue gas temperature and pressure conditions.

3.2.8.1 *Discussion*—In accordance with the described test method, all material that may be collected by filtration under specified conditions and that remains upstream of the filter and on the filter after drying under specified conditions are considered to be particulate matter. However, for the purposes of some regulatory standards, the definition of particulate matter may extend to condensibles or reacted materials collected under specified conditions (for example, specified temperature lower than the flue gas temperature).

3.2.9 *sampling line*—the line in the sampling plane along which the sampling points are located bounded by the inner duct wall.

3.2.10 *sampling plane*—the plane normal to the centerline of the duct at the sampling position.

3.2.11 *sampling point*—the specific position on a sampling line at which a sample is extracted.

3.2.12 *weighing control procedures*—quality control procedures utilized for detecting/correcting apparent mass variations due to climatic or environmental changes between pre- and post-sampling weighing series.

3.2.12.1 *Discussion*—In this procedure, control parts, which are identical to those to be weighed for dust measurement and are pretreated under the same conditions, are used. The control parts are kept free from dust contamination.

4. Summary of Test Method

4.1 A sample stream of the gas is extracted for a measured period of time at a controlled flow rate, and the volume of gas collected is subsequently measured. The particulate matter (dust) entrained in the gas sample is separated by a pre-weighed filter, which is then dried and reweighed. Deposits upstream of the filter in the sampling equipment are also recovered and weighed. The increase of mass of the filter and the deposited mass upstream of the filter plus the deposits collected upstream of the filter are attributed to particulate matter collected from the sampled gas. The ratio of the mass of the particulate matter collected to the volume of gas collected allows for the calculation of the flue gas particulate concentration.

4.2 Valid measurements can be achieved only when:

4.2.1 The gas stream in the duct at the sampling plane has a sufficiently steady and identified velocity, a sufficient temperature and pressure, and a sufficiently homogeneous composition;

4.2.2 The flow of the gas is parallel to the centerline of the duct across the whole sampling plane;

4.2.3 Sampling is carried out without disturbance of the gas stream, using a sharp edged nozzle facing into the stream;

4.2.4 Isokinetic sampling conditions are maintained throughout the test;

4.2.5 Samples are taken at a preselected number of stated positions in the sampling plane to obtain a representative sample for a non-uniform distribution of particulate matter in the duct or stack.

4.2.6 The sampling train is designed and operated to avoid condensation and to be leak free;

4.2.7 Dust deposits upstream of the filter are recovered or taken into account, or both; and

4.2.8 The sampling and weighing procedures are adapted to the expected dust quantities.

5. Significance and Use

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

5.1.1 These measurements, when approved by federal or 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 to demonstrate conformance with contractual performance specifications.

5.2 The collected residue obtained with this test method 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.

5.3 These measurements also can be used to calibrate continuous particulate emission monitoring systems by correlating the output of the monitoring instruments with the data obtained by using this test method.

5.3.1 This test method is useful in such correlation applications when emissions are less than 20 mg/m³.

5.3.2 The correlation test method is most valid when the monitoring instrumentation samples the particulate matter under the same test conditions as this test method.

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 (SO₂) 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 (CaSO₄) or the reaction with ammonia gas (NH₃) to form ammonium sulfate (NH₄)₂ SO₄ and the potential reaction of hydrogen fluoride (HF) with glass components in the sample train with resultant collection of silicon tetrafluoride (SiF₄) 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 an occurrence would result in a negative interference. See also [Appendix X1](#).

7. Requirements for Sampling Plane and Sampling Points

7.1 Representative sampling is possible when a suitable location that has sufficiently homogeneous gas velocity at the sampling plane is available.

7.1.1 Perform sampling at a sufficient number of sampling points, which are usually located on several sampling lines. Convenient access ports and a working platform are required for the testing. See Test Method [D3685/D3685M](#) for additional criteria.

7.2 Sampling Plane:

7.2.1 The sampling plane shall be situated in a length of straight duct (preferably vertical) with a constant shape and constant cross-sectional area. The sampling shall be conducted

as far downstream and upstream from any obstruction that may cause a disturbance and produce a change in the direction of flow (disturbances can be caused by bends, fans, or control equipment).

7.2.2 Measurements at all the sampling points defined in [7.3](#) shall prove that the gas stream at the sampling plane meets the following requirements:

7.2.2.1 The angle of gas flow is less than 15° with regard to the duct axis (method for estimation is indicated in Annex C of ISO 10780);

7.2.2.2 No local negative flow is present;

7.2.2.3 The minimum velocity is measurable by the test method used (for example, using Test Method [D3154](#), a differential pressure larger than 5 Pa); and

7.2.2.4 The ratio of the highest to lowest local gas velocities is less than 3:1.

7.2.3 If the above requirements cannot be met, the sampling location will not be in compliance with this test method.

7.3 Minimum Number and Location of Sampling Points:

7.3.1 See Test Method [D3154](#), Section 8, Figs. 7 and 8, and Tables 1 and 2.

7.4 Access Ports:

7.4.1 Provide sampling ports for access to the sampling points selected, in accordance with [7.3](#) and Test Method [D3154](#).

7.4.2 Ensure that the port dimensions offer ample space for the insertion and removal of the sampling equipment and associated devices.

8. Velocity and Gas Composition Measurement Apparatus

8.1 See Section 6, Test Method [D3154](#).

9. Sampling Apparatus

9.1 *Sampling Train*—For schematic drawings of the major sampling train components refer to [Fig. 1](#) for the in-stack method and [Fig. 2](#) for the out-of-stack method.

9.1.1 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 that will withstand corrosive or otherwise reactive components 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), 316 stainless steel (up to 800°C), and some resistant

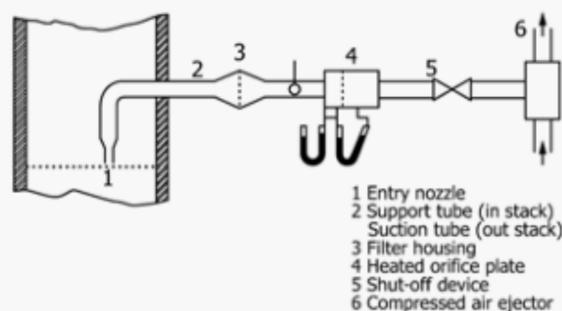


FIG. 1 In Stack Sampling Train—Example of a Dry Basis Measurement System

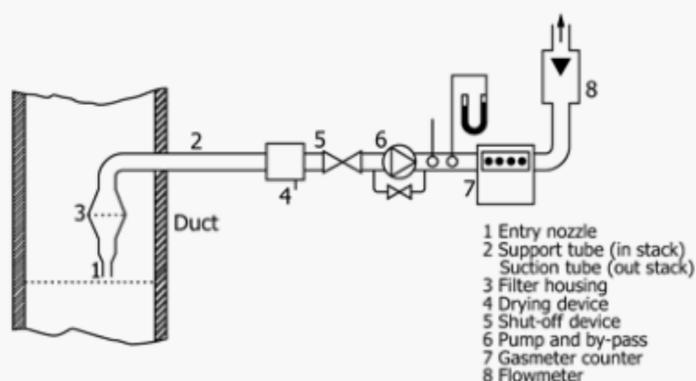


FIG. 2 Out-Stack Sampling Device Example of a Wet Basis Measurement System

silicone materials (up to 150°C). Extreme temperature conditions may require the use of materials such as quartz or a nickel-chromium alloy, or a water-cooled probe may be used.

9.2 *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 following interconnected elements:

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

9.3.1 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 shall be <30° with the taper on the outside to establish a constant inside diameter (ID). The straight length from the nozzle opening to the first bend of the nozzle shall be greater than 30 mm.

9.3.2 A range of nozzle IDs, for example, 3 to 15 mm (0.125 to 0.5 in.), in increments of 1.5 mm are required for isokinetic sampling. Larger nozzle sizes may be required if high volume sampling trains 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 calibrating purposes.

9.4 *Filter Holders:*

9.4.1 *In-Stack Method:*

9.4.1.1 *Thimble Holder*—A stainless steel holder for the porous aluminum oxide thimble (see 10.9). Holders for a glass and glass fiber thimble are somewhat different in design and method of retention. Keep the overall diameter to a minimum to facilitate insertion of the entire holder through a relatively small (75 mm) 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, such as stainless steel or TFE-fluorocarbon ferrules, that will provide an adequate seal at the desired temperature. Such holders and the thimbles can withstand temperatures approaching 550°C. 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. Other designs, such as nozzle/filter holder combinations that are weighed as a single unit, may be used if the performance characteristics of this test method can be met in their use.

9.4.1.2 *Alternate Filter Holder/Backup Filter Holder*—An in-stack filter holder constructed of borosilicate or quartz glass or stainless steel. 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 (but greater than 50 mm from 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 leakless seal.

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

(3) Place the filter holder in an oven (an out-of-stack method filter heater compartment can be used) at about 100°C 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 is unable to pass the leak check procedure at 100°C, reject the holder unless sampling is to be performed only at ambient temperature.

9.4.2 *Out-of-Stack Method Filter Holder*—Refer to 9.4.1 for in-stack method details.

9.4.2.1 *Filter Heating System*—A heating system capable of maintaining the filter holder at 120 ± 15°C during sampling. Other temperatures may be specified for a particular application.

9.4.2.2 *Filter Thermometer*—Monitoring device for measuring temperature of the filter holder to within 3°C during sampling.

9.4.2.3 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.

9.5 Probes:

9.5.1 *Probe Extension (In-Stack Method)*—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 (see 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 glasslined 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 extenders of nickel-iron-chromium alloy (UNS N08825) (see DS 56I⁶) or equivalent at temperatures greater than 315°C. (Record the probe material selection in the field data sheet.)

9.5.2 *Out-of-Stack Method*—The sampling probe shall be constructed of borosilicate or quartz glass tubing with an outside diameter (OD) of about 16 mm, encased in a stainless steel sheath with an OD of 25 mm. Whenever practical, use borosilicate or quartz glass liners; alternatively, metal seamless liners of 316 stainless steel, a nickel-chromium alloy, nickel-iron-chromium alloy (UNS N08825) (see DS 56I⁶), or other corrosion resistant metals may be used. A heating system that will maintain an exit gas temperature of 120 ± 14°C (250 ± 25°F) during sampling is required. 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. 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 and 1500°C 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:

9.5.2.1 Connect the probe with a nozzle attached to the inlet of the vacuum pump (see 9.10.3).

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

9.5.2.3 Activate the pump and adjust the needle valve until a flow rate of about 20 L/min is achieved.

9.5.2.4 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. Otherwise, reject or repair the probe.

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

9.6.1 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 ID and that extends to within 13 mm of the flask bottom.

9.6.1.1 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).

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

9.6.3 The fourth impinger outlet connection shall allow for insertion of a thermometer (see 9.6.5). 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.

9.6.4 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.

9.6.5 *Impinger Thermometer*—Monitoring device for measuring temperature of gas exiting the fourth impinger (see 9.6.3) within ±1°C of true value in the range from 0 to 25°C.

9.7 *Gas Temperature Sensor*—For measuring gas temperature to within ±1°C. Permanently attach the temperature sensor to either the probe (see 9.5) or the pitot tube (see 9.9 and Figs. 1 and 2).

9.8 *Vacuum Lines*—Locate all components of the sampling system as close together as possible, with direct interconnection between successive components in the system wherever possible. When direct interconnection 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 of withstanding a vacuum of 65 kPa without collapse or leakage.

9.9 *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. 2. 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. Repair or return any pitot tube that does not meet specifications. Calibrate the Type S tube following the procedures given in Practice D3796.

9.10 *Metering System*—The 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 of true value in the range from 0 to 90°C, pressure gage, check valves, and related equipment, as shown

⁶ Metals and Alloys in the Unified Numbering System. Available from ASTM, 100 Barr Harbor Drive, West Conshohocken, PA 19428.

in Figs. 1 and 2. Other metering systems capable of maintaining sampling rates within 10 % of isokinetic and of 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 Test Methods [D3685](#)/[D3685M](#). Any leakage requires repair or replacement of the malfunctioning item. Components include the following:

9.10.1 *Differential Pressure Gage*—Two inclined manometers or the equivalent, as specified in Test Method [D3154](#). One (called the pitot manometer) is utilized to monitor the stack velocity pressure, and the other (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. The gages shall read within 5 % of the gage-oil monometer at each test point. Repair or reject any gage that does not meet these requirements

9.10.2 *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) 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 the correction factor as necessary.

9.10.2.1 *Dry Gas Meter Thermometer*—Two monitoring devices for measuring temperature to within 3°C in the range from 0 to 90°C of the gas entering and exiting from the dry gas meter (see [9.10.2](#)).

9.10.3 *Vacuum Pump*—An airtight, leak-free vacuum pump with coarse and fine flow controls capable of maintaining a flow rate of 20 L/min for a pump inlet vacuum of 50 kPa is used to draw the gas sample.

9.10.4 *Vacuum Gauge*, for measuring pressure at the vacuum pump inlet, capable of measuring ± 3 kPa over the range from 0 to 101 kPa. Check it against a mercury U-tube manometer upon receipt, and yearly thereafter.

9.11 *Nomograph*, to determine the isokinetic sampling. Its function may be applied with a hand-held programmable calculator or laptop computer.

9.12 *Thermometers*—Temperature measuring devices such as RTDs, thermistors, and organic liquid-in-glass thermometers meeting the requirements of specific applications may be used. 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. See Test Methods [D3685](#)/[D3685M](#) for calibration procedures.

9.13 *Barometer*—An aneroid, or other barometer capable of measuring atmospheric pressure to within ± 300 Pa shall be used. Calibrate the barometer against a mercury-in-glass barometer or the equivalent, as described in Test Method [D3631](#).

9.13.1 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.

9.14 *Wet Test Meter*, with a capacity of 3.5 m³/h or 30 L for each revolution with an accuracy of ± 1.0 %, to calibrate the dry test meter.

9.15 *Orsat Gas Analyzer*, stack gas analyzer, as described in Test Method [D3154](#).

9.16 *U-Tube Manometer*, a water manometer or pressure sensor capable of measuring gas pressure to within 0.33 kPa.

9.17 *Sample Recovery Apparatus*:

9.17.1 *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.

9.17.2 *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.

9.17.3 *Sample Storage Container*, 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.

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

9.17.5 *Graduated Cylinder or Triple Beam Balance, or Both*, a graduated cylinder or a triple beam balance 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 ≤ 2 mL. Use a triple beam balance capable of weighing to the nearest 1.0 g.

9.17.6 *Plastic Storage Containers*, several airtight plastic containers for storage of silica gel.

9.17.7 *Funnel and Rubber Policeman*—A 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.

9.17.8 *Desiccator*, used to dry filters before weighing. Use anhydrous CaSO₄ (see [10.5](#)) as the desiccant.

9.17.9 *Laboratory Drying Oven*, capable of heating filters and thimbles to 102°C.

9.17.10 *Laboratory Muffle Furnace*, capable of heating thimbles to 550°C.

9.17.11 *Steam Bath*:

9.18 *Analytical Equipment*:

9.18.1 *Glassware*, borosilicate glass dishes to facilitate filter weighing. Use a 250-mL glass beaker for evaporation of the acetone rinse.

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

10. Reagents and Materials

10.1 *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.⁷

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

NOTE 1—Type IV reagent water is required in **11.4.2**.

10.3 Determine reagent blanks on the acetone and reagent water.

10.4 *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 **10.3**) is >0.001 % of the total acetone mass.

10.5 *Calcium Sulfate, Anhydrous*— CaSO_4 , indicating type, for use in desiccator.

10.6 *Crushed Ice*.

10.7 *Silica Gel*— SiO_2 , indicating-type, 6 to 16-mesh, for use in the fourth impinger in the condenser. Dry at 175°C for at least 2 h prior to use.

10.8 *Gloves*, insulated, heat-resistant.

10.9 *Filter Material*:

10.9.1 *In-Stack Method*—The primary filter is generally a porous aluminum oxide glass, or glass-fiber thimble. For heavy dust loading applications, such as sampling stacks without control equipment or sampling on the inlet side of the control equipment. A secondary filter for back-up may be used (see **10.9.2**). Procedures for filter preparation are given in **11.1.1** and **11.1.2**.

10.9.2 *Alternate In-stack Method A and Out-of-Stack Method*—Use glass fiber filters without organic binders. The filters shall exhibit at least 99.95 % collection efficiency of a 0.3-mm dioctyl phthalate smoke particle, in accordance with Practice **D2986**. The manufacturer's quality control test data are sufficient for validation of efficiency.

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

11. Weighing and Pre-Sampling Procedure

11.1 *General Aspects*—Before carrying out any measurements, discuss the purpose of the sampling and the sampling procedures with the plant personnel concerned. The nature of the plant process, for example, steady state or cyclic, can affect the sampling program. If the process can be

performed in a steady state, it is important that this state is maintained during sampling

11.1.1 Agree upon, with the plant management, the dates, starting times, duration of survey, and sampling periods as well as plant operating conditions during these periods.

11.1.2 Make preliminary calculations on the basis of the expected dust loading to determine the appropriate nozzle size or sampling conditions, or both. Also determine whether the chosen nozzle size and sampling time will result in sufficient matter collected to meet weighing requirements. Longer sampling times or sampling with the use of a larger nozzle and higher sample flow rates may be necessary to obtain the sample filter mass sufficiently greater than the blank filter mass.

11.2 Taking into account the objective of the measurements and the flue gas characteristics:

11.2.1 Choose between an in-stack or an out-stack filtration device. If the flue gas is saturated with water or contains appreciable amounts of SO_3 , use out-stack filtration devices:

11.2.1.1 Choose a relevant temperature for conditioning and drying the filter before and after sampling (see **14.2**); and

11.2.1.2 When using an out-stack filtration device, set the out-stack filter temperature as specified by the regulatory agency or as determined for technical reasons.

11.2.2 Take an overall blank sample after each measurement series and at least after each day of sampling following the sampling procedure described in **12.3**, either without starting the suction device or for a sample duration less than one minute. This blank sample leads to an estimation of the dispersion of results related to the whole procedure as carried out by the operators for a near zero dust concentration, due to contamination of filters and of rinsing solutions during handling on-site, transport, storage, handling in the laboratory, and weighing procedures, and so forth.

11.3 *Weighing Procedures*:

11.3.1 Depending on the kind of sampling device used, the parts to be weighed may be the filter (with or without its support) or may include all parts upstream from the filter. Options include:

11.3.1.1 The filter and particulate matter collected upstream of the filter,

11.3.1.2 The filter and its housing and particulate matter collected upstream of the housing, and

11.3.1.3 The nozzle, the filter and its housing, and any components connecting the nozzle to the housing.

11.3.2 For **11.3.1.2** and **11.3.1.3**, pre- and post-treat all the relevant parts in accordance with **11.4** and weigh them together, without disassembling them.

11.3.3 Depending upon the system used, evaporate the rinsing solutions obtained from components that are rinsed and weigh them in the same container or transfer them to a smaller container for weighing.

11.4 *Pre-Sampling Treatment of Weighed Parts*:

11.4.1 *Flat Filter Media for Primary or Secondary Holder*—Heat each filter (see **10.9.2**) in a laboratory drying oven (see **9.17.9**) on a numbered container for 2 h at the temperature of the sampling. After removal from the oven, cool to room temperature in a desiccator (see **9.17.8**). After cooling,

⁷ *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.

weigh each filter to the nearest 0.1 mg, and record the data. Transport weighed filters in petri dishes (see 9.17.4) to the holders.

11.4.2 *Thimble for Primary Holder*—Sand the lip of the porous aluminum oxide thimble (see 10.9.1), 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 (see 10.2), fire at 550°C in a muffle furnace (see 9.17.10) for 1 h, cool, and store until needed.

11.4.3 When preparing the porous aluminum oxide thimble for use, number the outside of the thimble or the jar with a heat-resistant pencil. Place the thimble in a drying oven (see 9.17.9) and heat overnight at 102°C. Remove thimbles from the oven, immediately desiccate it, and allow it to cool to room temperature. Weigh the thimble to the nearest 0.1 mg, and record the results. Return the thimble to its container.

11.5 *Weighing:*

11.5.1 Weigh the filter on a suitable balance (see 9.18.2) to at least 0.1 mg.

11.5.2 Since the sample mass is determined by calculating the difference between data often obtained at one or two week intervals, special care is required to avoid weighing errors related to balance drift, to insufficient temperature equilibrium of parts to be weighed, and to climatic changes (see examples in Appendix X2). Therefore, before performing any weighing, validate the weighing procedure.

11.5.3 Before each weighing series:

11.5.3.1 Calibrate the balance against standard weights;

11.5.3.2 Perform additional checks by weighing control parts that are identical to the others and pretreated in the same conditions, but kept free from contamination, and;

11.5.3.3 Record the climatic conditions in the room.

11.5.4 When weighing large volume parts (for example, beakers), the temperature and barometric pressure may influence the apparent mass. This influence may be detected using the reference mass of the control parts. In these conditions, apply weighing corrections based on the apparent mass modification of three identical control parts of each type (filter including support, container, and so forth).

11.5.5 Give attention also to weighing artifacts related to:

11.5.5.1 Electrostatic charges, which may have to be discharged or neutralized;

11.5.5.2 Hygroscopic characteristics of the filter material or dusts, or both, which may lead to an increase of mass. Perform weighing within 3 min after removal from the desiccator. Take three readings at 1, 2, and 3 min intervals. If a significant increase is detected, return the parts to the desiccator and desiccate for a minimum period of 8 h. Repeat the weighing procedure. Calculate the dry mass by extrapolating the 1, 2, and 3 min data to time zero; and

11.5.5.3 Small differences in temperature between the part to be weighed and the environment may disturb the balance.

11.6 *Post-Sampling Treatment of Weighed Parts:*

11.6.1 Dry weighed parts in a drying oven for at least 1 h at the temperature of the sampling (see 11.2 for specific cases).

11.6.2 After drying, equilibrate the parts to ambient temperature as described in 11.4.

11.7 *Post-Sampling Treatment of the Rinsing Solutions:*

11.7.1 Take all the rinsing solutions (water and acetone) from all parts upstream of the filter, as described in 12.4, to the laboratory for further treatment. Exercise care that no contamination occurs.

11.7.2 Transfer the solutions quantitatively to the dried and pre-weighed containers (see 11.4). Do not boil the solvent mixtures during the evaporation. Use smaller vessels as the volume of the solution is reduced through the evaporation process.

11.7.3 Two methods have been tested for evaporation:

11.7.3.1 Evaporation in an oven at 120°C at ambient pressure. An efficient ventilation system with clean air or nitrogen is necessary to dilute acetone vapor to a safe level; and

11.7.3.2 Evaporation in a closed system (desiccator). Set the initial temperature to 90°C and reduce the pressure to 40 kPa (absolute). Periodically, increase the temperature as well as the pressure. For the last period, maintain them at 140°C and 20 kPa (absolute).

11.7.4 After the evaporation, place the weighing containers in the drying oven for 2 h at 160°C or at the same temperature chosen for the post sampling treatment of the filters, (see 11.4 – 11.6). Transfer the weighing containers to the desiccator. Weigh the containers, including the deposits, after thermal equilibration in the weighing room has been maintained for at least 4 h or more, depending on the type of weighing container (that is, 12 h for large containers).

11.7.5 Due to the relative large mass and volume of the weighing containers compared to the deposits under investigation, changes of the barometric pressure may influence the weighing. Therefore, weigh at least three empty weighing containers of equal size with each series.

11.7.6 Obtain at least one blank value for each solvent, using the same volume as used in the rinsing.

12. Sampling

12.1 *Preparation:*

12.1.1 Clean (brushed and rinsed), calibrate, and check all the equipment before moving it to the test site. Exercise care not to reuse any part of a sampling train that has previously been used for high dust concentration sampling without dismantling and thorough cleaning.

12.1.2 Depending on the measurement program, prepare the filter and associated parts to be weighed for each sample run. This includes parts for the overall blank tests and additional parts to accommodate process and equipment malfunctions.

12.1.3 Perform the weighing procedures in accordance with 11.3.

12.1.4 Protect all the weighed parts, including the suction tube and the other parts or equipment that will come into contact with the sample (and will be rinsed later) from contamination during transportation and storage.

12.2 *Pre-Sampling Measurements:*

12.2.1 Depending on the dimensions of the duct, which are to be verified using a measuring rod, surveyor's transit, or other means, select the number and location of sampling points, in

accordance with 7.3 and Test Method D3154. Mark on the pitot tube and the sampling tube the distance from the sampling points to the entry point of the access port.

12.2.2 Determine the temperature, pressure, moisture content, and molecular weight of the flue gas at the sampling location in accordance with Test Method D3154, in order to calculate isokinetic sampling conditions and correct emissions data from actual to standard conditions. Monitor these parameters at each sampling point if they vary spatially or temporally during the period of the test.

12.2.3 Taking into account the preliminary calculation (11.1) and the measured velocities, select a suitable nozzle as per the requirements of 9.3 above.

12.3 Sampling:

12.3.1 Assemble the sampling train in the cleanest possible area, and check for possible leaks in accordance with 12.6.2 requirements;

12.3.2 Preheat the relevant parts of the sampling train up to the selected filtration temperature, that is, stack temperature or recommended temperature of $160 \pm 5^\circ\text{C}$, and insert it in the duct at a selected point, avoiding contact of the entry nozzle with any deposits within the duct. Seal the opening of the access port to minimize air in-leakage or exposure of operators to toxic gases;

12.3.3 Estimate the isokinetic sampling rate from preliminary stack velocity, temperature, and dry gas molecular weight data using the methods of Test Methods D3685/D3685M;

12.3.4 Assuming axial flow, turn the sampling train until the entry nozzle is facing upstream within $\pm 10\%$ of the flow. Recalculate, if necessary, the isokinetic sampling rate based upon the flue gas velocity, temperature, and composition (if monitored) at the sampling point. Open the shut off device, start the suction device and adjust the flow rate in order to sample isokinetically within $\pm 10\%$.

12.3.5 The sampling duration at each selected point shall be identical. The total sampling duration shall be at least 30 min.

12.3.6 Check the flue gas velocity and temperature at each test point by using a Pitot tube and thermocouple associated with the sampling equipment. At each test point and at least every 5 min, recalculate the isokinetic sampling rate in accordance with Test Methods D3685/D3685M, based on the new velocity and temperature data, if necessary. Adjust the sample train flow rate so that isokinetic sampling is maintained within $\pm 10\%$;

12.3.7 Do not stop sampling when moving the sampling train to the next sampling point in the same sampling line. If the flue gas velocity or temperature changes at the new sampling point, recalculate the necessary isokinetic sampling rate and adjust the flow rate of the sampling system immediately;

12.3.8 Record the sampling time and sampled volume/flow rate at each sampling point;

12.3.9 After completing sampling at all the selected points of the sampling line, close the shut off device and the suction device, remove the sampling train from the duct and reposition it on the next sampling line;

NOTE 2—For a low dust concentration measurement, use if possible only one filter for a complete measurement (cumulative sampling).

12.3.10 After completing sampling at all selected points: (a) remove the sample train after closing the shut off valve and suction device; (b) leak check the equipment after sampling, before dismantling; and (c) dismantle the sampling equipment and check visually the filter and the filter housing for signs of stains on the filter due to moisture (sampling equipment operated below or too close to the dew point). In such cases, repeat the test.

12.3.11 Place the parts to be weighed in a closed container and transport them to a laboratory for weighing (see 11.3). The container material shall be sufficiently electrically conductive to avoid dust losses from the filter due to electrostatic charge effects.

12.4 Recovery of Deposits Upstream of the Filter:

12.4.1 Rinse all the non-weighed parts upstream of the filter that are in contact with the sample gas rinsed with water to recover the deposits.

12.4.2 A clean environment is recommended for the following procedure. Exercise special care to avoid contamination of the sample if the procedure is performed on-site.

12.4.2.1 Rinse carefully, with water, the internal surfaces of the nozzle, elbow, and the front part of the filter housing. Collect the rinse into a storage bottle and allow no external dust to fall into the bottle. Rinse these same surfaces with acetone and collect the acetone into the same bottle.

12.4.2.2 To rinse the suction tube (sample probe), seal one end and fill with enough water to wet and clean the inner surface (one third to one half of the volume) and then seal the other end. Rotate the along the long axis and tilt the tube several times. Transfer the water to the transport storage container. Repeat the procedure with a second rinsing of water, followed by a rinsing with acetone.

12.4.2.3 Do not mechanically clean surfaces to recover deposits upstream of the filter after sampling. However, it is necessary to mechanically clean and rinse the equipment before the measuring series is initiated.

12.4.3 The purpose of the overall blank is to include a blank rinse of a pre-cleaned sampling system. Obtain the blank rinse following the procedures given above and evaporate and weigh the residue, following the same procedures as for the sample rinse.

12.5 Validation of Results:

12.5.1 Parameters Depending on the Stationary Source:

12.5.1.1 If testing was conducted at an unsuitable location or was carried out under fluctuating plant operating conditions, the representativeness of the sample may be questioned and the measurement results uncertain. In such cases, the test report shall clearly indicate that the test was not conducted in accordance with the specifications of this test method.

12.5.1.2 Included in the test report details of the flow characteristics at the sampling location or on the variations of the flow rate in the duct while sampling, or both.

12.6 Leak Check:

12.6.1 Significant errors may be caused by leaks in the sampling train, specially in parts that are under vacuum.

12.6.2 Therefore, before and after each test, leak test the sample train by sealing the nozzle and starting the suction

device. The flow rate shall be at most 2 % of the normal flow rate at the maximum vacuum reached during sampling, otherwise the measurement is not valid.

NOTE 3—Leaks can be monitored during sampling by measuring continuously the concentration of a relevant gas component (such as CO₂, O₂, and so forth) directly in the duct and at the exhaust of the sampling train. Any difference between those concentrations indicates a leak in the sampling train.

12.6.3 *Isokinetic Rate:*

12.6.3.1 If the actual flow rate through the nozzle differs by more than 10 % from the theoretical isokinetic rate over the duration of the sampling, repeat the test.

12.6.3.2 If this criterion is not fulfilled due to frequent variation of the flow rate in the duct, see 15.1.4.

12.6.4 *Deposits of Dust on Non-weighed Parts Upstream of the Filter:*

12.6.4.1 Add the mass of deposits on non-weighed parts upstream of the filter to the mass of the deposits determined from the filter and weighed parts.

12.6.4.2 This requirement may be disregarded if an in-stack filter is used that has no bends between the nozzle and the filter, provided that it is used in an application having unsaturated gases and has a temperature well above the stack gas dew point and if a validation has been carried out at similar conditions as the process to demonstrate that the deposits do not exceed 10 % of the daily average limit value set for the process.

12.6.5 *Overall Blank:*

12.6.5.1 A measurement series is validated only when the quantity of dust collected during the sampling is at least five times a corresponding positive difference in an overall blank.

12.6.5.2 Any emission value determined in the test series that is less than the blank value is not valid.

12.6.5.3 However, when measured concentrations are below 5 mg/m³, it may not be possible to fulfill this requirement. In such a case, extend the sampling time or employ a larger sample nozzle and high volume sampling techniques to collect sufficient particulate matter within the specified sampling period.

12.6.6 *Summary of the Requirements of This Test Method:*

12.6.6.1 A summary of the specifications and requirements of Sections 7, 8, and 9 of this test method is shown in Table 1.

13. Calibration

13.1 Calibrate the velocity and gas composition measurement apparatus in accordance with Test Method D3154.

13.2 Calibrate the particulate sampling apparatus in accordance with Test Methods D3685/D3685M.

14. Additional Aspects

14.1 *Sampling Duration and Detection Limit:*

14.1.1 Some uses or regulatory purposes may require a sampling time limited to 0.5 h. In such a case, the detection limit of the test method is in the range of 2 mg/m³ (see 16.2). For medium-size ducts, such a measurement requires that two teams sample simultaneously, each sampling on one of the two sampling lines.

14.1.2 Where possible, extend the sampling time, which decreases the detection limit. The sampling time should be less than 2 h, to minimize the effect of nonsteady state conditions of the stationary source.

TABLE 1 Summary of the Standard Requirements

Equipment and Sampling Parameters	Value
Nozzle internal diameter	> 6 m
Nozzle: area known better than	± 10 %
Nozzle: constant internal diameter on a length	> 10 mm
Nozzle: change in diameter angle	< 30°
Elbow: radius of the bend	> 1.5 times inside diameter
Straight Length before the first bend	> 30 mm
Nozzle tip: distance to obstacles	> 50 mm
Filter quality (test aerosol 0.3 µm)	> 99.5 %
Filter material (absorption of components)	no reaction and no absorption
Condenser, drying tower: residual gas moisture	< 10 g/m ³
Gas meter volume measurement uncertainty	± 2 %
Absolute pressure	± 1 %
Absolute temperature	± 1 %
Alignment of the nozzle	± 10 %
Isokinetic criteria (average within)	± 10 %
Leak test	< 2 %
Balance resolution (mg)	0.01 to 0.1 mg
Weighing uncertainties	< 5 % of the ALV ^A set for the process. See 13.9.5
Temperature equilibrium duration	> 8 h
Overall blank value	< 10 % of the ALV ^A set for the process.
Sampling Location	
Flow angle	< 15°
Negative flow	none
Pressure difference (Pitot tube)	> 5 Pa
Ratio of max to min velocity	3:1
Straight Length before the sampling plane	See D3685/D3685M
Straight Length after the sampling plane	See D3685/D3685M
Number of sampling points	See D3685/D3685M
Equipment: Flue Gas Characteristics	
Absolute temperature	± 1 %
Flue gas composition	± 0.05 kg/m ³

^A ALV: Average Limit Value

14.2 Thermal Behavior of Particulate Matter:

14.2.1 Emitted particulate matter is generally stable. However, in some processes, the sample gases may contain unstable or semi-volatile compounds (that is, materials that are in particulate form at a low temperature, but in gaseous form at a higher temperature). In such cases the measured concentration will depend on the filtration temperature or on the drying temperature before final weighing, or both.

14.2.2 Such phenomena have been reported in various industries:

14.2.2.1 Power plants equipped with de-sulfurization processes, due to the occurrence of hydrates;

14.2.2.2 Heavy fuel oil power plants or diesel engines due to SO₃ and heavy organic emissions, or both; and

14.2.2.3 Glass furnaces due to the occurrence of semi-volatile boron compounds.

14.2.3 Very high discrepancies (up to a factor of 10) have been reported due to these effects. However, it is possible to obtain reproducible results if unstable or semi-volatile compounds are not trapped or are further evaporated when drying, or both. In such cases, the measured results are only consistent when associated with a specified temperature, such as the highest temperature sustained by the sampled dust before weighing.

14.2.4 Maintain the suction system, filter, and associated parts exposed to the hot flue gas at a temperature of 160°C to minimize trapping of volatile organic compounds and to decompose hydrates. However, because of the extreme variety of the situations that may be encountered, it is not possible to specify a conventional temperature that will be relevant in all cases.

14.2.5 The following requirements apply to all parts of the sampling train that are to be weighed:

14.2.5.1 Condition them at 180°C before pre-weighing;

14.2.5.2 Set them at a temperature equal to or less than 160°C during sampling; and

14.2.5.3 Dry them at 160°C before final weighing.

14.2.6 Other temperatures may be specified depending upon technical or regulatory test objectives.

14.2.7 For other temperatures:

14.2.7.1 Condition the weighed parts to a temperature at least 20°C above the maximum temperature reached during sampling and post-sampling treatment; and

14.2.7.2 Indicate the temperature used while sampling and drying before weighing in the test report.

14.3 Particulate Deposits Upstream of the Filter:

14.3.1 Experimental work has shown that particulate deposits upstream of the filter cannot be neglected when sampling low dust concentrations. It has also been shown that significant errors can be introduced into the measurements if these deposited materials are not recovered carefully.

14.3.2 At concentrations around 5 mg/m³, it has been found that such deposits can constitute between 10 and 30 % of the total particulate mass collected. Higher values have been reported at lower concentrations (> 50 % at 1 mg/m³).

14.3.3 Particulate deposits are dependent upon the design of the sampling equipment and the characteristics of the particulate matter to be sampled. Therefore, check all non-weighed

parts for the deposition of particulate matter, and if present, collect the particulate matter and add it to the catch.

14.3.4 Deposits may be collected by the rinsing procedures in 12.4, either after each test of the measurement series or at least after each measurement series on the same sampling plane and at least once a day. If the material is collected at the end of the measurement series, apportion the recovered mass to the individual tests in proportion to the mass collected on each filter.

14.4 Improvement of the Weighing Procedure:

14.4.1 Experience has shown that weighing uncertainties are not only related to the balance performance but to the whole procedure employed. Therefore, before performing any measurement, the user shall establish and validate a procedure, taking into account the sampling equipment and filters to be used.

14.4.2 Repeated weighing of the same parts, spread over several weeks in various conditions, that is, external temperature humidity, and so forth, will provide an estimation of the actual precision of weighing, including the uncertainties related to the manipulation of the filters, equilibrium time, and so forth.

14.4.3 Use the results as a first estimate of the overall blank value, and provide a means of calculation of the gas volume to be sampled in order to obtain significant data, taking into account the anticipated range of particulate concentrations.

15. Calculation

15.1 Isokinetic Rate:

15.1.1 To perform isokinetic sampling, the sampling system flow rate shall be calculated so that the velocity of sample gas entering the nozzle, v_n , is equal to the flue gas velocity at the sampling point, v_s , or:

$$V_n = V_s \quad (2)$$

15.1.2 The flow rate at the nozzle, Q_a , expressed in the actual conditions at the nozzle, is:

15.1.3 In accordance with Eq 1, isokinetic sampling is conducted when:

$$Q_a = A_n V_n \quad (3)$$

$$Q_s = A_n V_n \quad (4)$$

Where A_n is the area of the nozzle.

15.1.4 Because the sampling flow rate is measured under conditions (temperature, pressure, moisture content) that differ generally from the actual conditions of the gas in the duct, Q_a shall be corrected as follows:

$$Q_m = Q_a \frac{100 - H_a}{100 - H_m} \frac{T_m}{T_s} \frac{P_s}{P_m} \quad (5)$$

where:

- Q_m = the corrected sampling flow rate,
- Q_a = the measured sampling flow rate, expressed in the actual conditions at the sampling point,
- H_m and H_a = the humidity, in % volume, of gases in the measured (m) and actual (a) conditions
- T_m and T_s = the measured and standard temperature of the gas, in kelvin, and

P_m and P_s = the measured and standard absolute pressure of the gas.

15.1.5 Compare the calculated target value of Q_a to the Q_a performed during the test to determine that they agree within $\pm 10\%$ of the isokinetic sampling criteria of this test method.

15.2 *Dust Concentration*—For each test, calculate:

15.2.1 The sampled volume, V , specifying whether on a dry or wet basis and under normal conditions;

15.2.2 The total mass of dust collected m , on the filter and upstream of the filter from the filter rinses; and

15.2.3 The dust concentration, c ;

$$c = \frac{m}{V} \quad (6)$$

15.2.4 It may be necessary to express the particulate concentration relative to a reference O_2 content to correct for dilution effects.

15.2.5 This correction can be achieved using the correction factor:

$$\frac{21 - O_{2ref}}{21 - O_{2meas}} \quad (7)$$

where:

O_{2ref} = the % volume titre of O_2 under the reference conditions, and

O_{2meas} = the measured % volume of O_2 .

15.2.6 See ISO 9096, Section 10.5, and Test Methods D3685/D3685M for other test methods for calculating corrected particulate emissions.

16. Precision and Bias

16.1 *General:*

16.1.1 Because the flue gas composition may vary in the time, it is not possible to determine the repeatability and reproducibility of the test method in accordance with Guide D3670.

16.1.2 However, if one team performs successive parallel sampling tests with two identical sampling systems, such a procedure allows a statistical comparison between paired values x_1 and x_2 to be calculated.

16.1.3 The standard deviation of the differences can be calculated as follows:

$$S = \sqrt{\frac{\sum (x_1 - x_2)^2}{2n}} \quad (8)$$

n , the number of sample pairs, may be used for the calculation of the repeatability confidence interval $r = t\sqrt{2}S$, where t is the Student factor for a 95 % confidence and the degrees of freedom $n-1$.

16.1.4 The repeatability, r , (in accordance with ISO 5725), that is the maximum difference between two measurements by the same team, for a 95 % confidence level is:

$$r = t s \sqrt{2} \quad (9)$$

16.1.5 When data are provided by several independent teams operating together, similar calculations can be conducted and provided as an estimation of:

16.1.5.1 The external uncertainty linked to an individual measurement carried out by any team fulfilling the requirements of the test method; and

16.1.5.2 The reproducibility (in accordance with ISO 5725), that is the maximum difference, R , that can be expected at a 95 % confidence between two measurements by different teams working in accordance with the test method at the process conditions.

16.1.6 When performing measurements at low level concentrations, the detection limit may be estimated:

16.1.6.1 By parallel measurements and calculation of the uncertainty; and

16.1.6.2 By successive measurements at near zero concentration. The detection limit is assumed to be three times the standard deviation.

16.2 *Experimental Data for Sampling for 30 Min at Traditional Flow Rates:*

16.2.1 Validation tests were performed in two municipal waste incinerators equipped with different types of particulate control equipment:^{8, 9}

16.2.1.1 Plant A—a semi-dry process, controlled using a fabric filter and having a flue gas temperature of 140°C.

16.2.1.2 Plant B—controlled using an electrostatic precipitator and a wet scrubber and having a stack gas saturated with water at a temperature of 60°C.

16.2.2 The sampling duration was limited to 30 min. The results are given in Table 2.

16.2.3 The detection limit was estimated from results by one team to be:

16.2.3.1 For dry gases—approximately 0.3 mg/m³ (dust on filter only); and

16.2.3.2 For water saturated gases: approximately 2 mg/m³ (total dust).

16.2.4 During the above tests, some high overall blank values were reported (± 1 mg/m³ or higher) due to weighing uncertainties of the dry extracts from the rinses, use of vessels of improper materials, and so forth. Further investigation showed that these uncertainties can be reduced to less than 0.5 mg/m³.

16.2.5 It was also determined that an increased sampling time of 60 to 90 min will significantly improve the reproducibility of the measurements.

⁸ European Committee for Standardization, “Statistical evaluation of the 3rd intercomparison test for total dust at a waste incinerator in Denmark,” CEN/TC 264/WG N116.

⁹ European Committee for Standardization. “Report of the 4th Validation Measurements: Dust Emissions at Concentrations from 1 mg/m³ to 7 mg/m³ in Wet Stack Gasses,” CEN/TC 264/Wg 5 N127 (revision 1).

TABLE 2 Results of Validation Test for 30 Min Sampling Duration

All Values in Mg/m ³	Plant A		Plant B
	Dust on Filter Only	Total Dust Filter + Rinse	Total Dust
Mean Concentration	4.7	6.4 (2 to 18)	2.5 (0.3 to 6.8)
Repeatability	1.7	2.1	1.9
External uncertainty	2.4	4.0	1.8
Reproducibility	3.4	5.7	2.6

16.3 Precision of the High Volume Test Method:

16.3.1 The percent coefficient of variation of the high volume Test Method D4356 was found to be about 8 %.¹⁰ This value was determined through a collaborative test in which five laboratories each sampled the stack of their choice using two sampling trains with nozzles positioned very close to each other in the stack. Each laboratory collected 6 to 8 sets of paired-train samples from their stack for a total of 12 to 16 samples per laboratory. Stack concentrations encountered ranged from 5.6 to 500 mg/m³.

17. Test Report

17.1 The test report shall refer to this test method and shall include the following information:

17.1.1 Description of the purpose of tests, identification of the site, date of sampling, and participants in the test.

17.1.2 Description of the operating conditions of the plant process and any variation during measurements.

17.1.3 Identification of the sampling location and gas parameters in the duct.

17.1.3.1 Duct dimensions, number and position of sampling lines, and sampling points;

17.1.3.2 Velocity and temperature profile;

17.1.3.3 O₂/CO₂ (if relevant), humidity concentrations, gas volumes; and

17.1.3.4 Compliance with the requirements of 7.2.

17.1.4 Measurement Procedures:

17.1.4.1 Velocity measurement (calibration of S Pitot tubes, and so forth);

17.1.4.2 Characteristics of sampling equipment:

(1) Make (type, manufacturer) of the sampling train;

(2) Nozzle diameter, characteristic of filter (material, size, type);

(3) Calibration of flow rate measurement devices; and

(4) Filtration temperature.

17.1.4.3 Weighing procedures:

(1) Conditioning temperature; and

(2) Correction of the apparent mass.

17.1.5 Test Results:

17.1.5.1 Number, date, duration of tests for each test:

(1) Mass of the particulate matter collected,

(2) Temperature and pressure of the gas in the sampling train,

(3) Gas composition (including moisture content),

(4) Sampling time,

(5) Sampled volume and flow rate,

(6) Any special circumstances or incidents,

(7) All intermediate and final calculation results, and

(8) Raw results and corrected results (standard conditions).

17.1.6 Quality Assurance:

17.1.6.1 Leak tests results,

17.1.6.2 Overall blank value,

17.1.6.3 Isokinetic criteria, and

17.1.6.4 Deposits of dust upstream of the filter.

17.1.7 Comments:

17.1.7.1 Indicate in the test results, the value of the ratio of the measured values (mg of dust) to the overall blank value (mg of dust).

17.1.7.2 Also indicate in the test report any special circumstances that may have influenced the results and any information concerning the uncertainty of the results.

17.1.7.3 If it has been necessary to modify the test method for any reason, report this modification.

18. Keywords

18.1 high volume stack sampling; isokinetic sampling; particulate matter; particulate matter sampling; stack sampling

¹⁰ Mitchell, W. J. and Suggs, J. C., "Collaborative Evaluation of Proposed ASTM High-Volume Sampling Method for Measuring Particulate in Stack Gases," *EPA Report*, 1980.

APPENDIXES

(Nonmandatory Information)

X1. MEASUREMENT OF PARTICULATE MATTER UNDER DEFINED CONDITIONS—THERMAL BEHAVIOR OF DUSTS

X1.1 Emitted dusts are generally thermally stable. However, gases to be sampled may contain unstable or semi-volatile compounds (in particulate form at low temperature, in gaseous form at higher temperature). In such cases, the measured concentration depends upon the filtration temperature or upon the drying temperature, or both, before final weighing.

X1.2 Particulate matter may be defined in environmental control agency standards in terms of a specified standard temperature (that is, 125°C, 15°C). In such cases, design the sampling system so as to collect pollutant materials that exist as particulate matter (solid phase or liquid droplet (aerosol)) at the specified temperature. This may be done by installing the filter and filter assembly externally to the probe, into a temperature controlled chamber.

X1.3 If materials that are condensible in ambient air are incorporated into the national standard for particulate matter, it may be necessary to include materials in a cooled impinger assembly added after the filter. Suitable extractive methods for organic or other materials must then be defined in the national standard. This test method does not address such procedures; however, it is noted that care shall be taken that any procedures applied shall not result in the introduction of artifacts non-representative of actual emissions.

X1.4 Industries where condensible materials may occur include the following:

X1.4.1 Power plant equipped with desulfurization processes, resulting in the formation of unstable hydrates;

X1.4.2 Heavy fuel oil power plants or diesel engines, causing the formation of SO₃ or organic emissions, or both; and

X1.4.3 Glass furnaces, causing the occurrence of semi-volatile boron compounds.

X1.5 In some cases, very high discrepancies (up to a factor of 10) have been reported, and it is therefore clear that, in such a case, the measured result makes sense only when associated with conventional temperature, which is the highest temperature sustained by the sampled dust before weighing.

X1.6 Because of the extreme variety of the situations that may be encountered, it is not possible to find a conventional temperature that could be relevant in all the cases.

X1.7 However, since the complete trapping of volatile compounds would necessitate a very low filtration temperature and special care during sampling, more reproducible results may be achieved if these compounds are not trapped or are further evaporated when drying. For this reason, a conventional temperature of 160°C, which avoids trapping of most volatile

compounds and decomposes most of the hydrates, is generally convenient.

X1.8 In accordance with this convention, components of the sampling train to be weighed shall therefore be:

X1.8.1 Conditioned at 180°C before preweighing;

X1.8.2 Set at any temperature less than 160°C during sampling; and

X1.8.3 Dried at 160°C before final weighing.

X1.9 Depending on regulatory requirements, plant authorization, the specific effluent, or on specific measurement objectives, other temperatures can be adopted.

X1.10 In any case:

X1.10.1 The weighed parts shall be pre-sampling treated (see 11.4) at a temperature at least 20°C above the maximum temperature reached during sampling and post-sampling treatment.

X1.10.2 Indicate the temperature used while sampling and drying before weighing in the test report.

X2. EXAMPLES OF WEIGHING ARTIFACTS

X2.1 Examples of Weighing Artifacts

X2.1.1 Weighing artifacts related to insufficient temperature equilibrium and to climatic changes between pre- and post-sampling weighings, are illustrated in the following example.

X2.1.2 In this example, the filter is placed in a closed glass petri box, mass 25 g, inside air volume 40 mL. The balance is calibrated against a standard mass 25 g (volumetric mass 8 g/mL). Volumetric mass of glass 2 g/mL, of air, 1.2 mg/mL.

X2.2 Effect of Insufficient Temperature Equilibrium

X2.2.1 Because of too low equilibrium time after drying, the inside air of the petri box is supposed to have a temperature 2 K higher than that of the room balance (300 K). Due to modification of air buoyancy, this difference of air temperature leads to an apparent mass variation of: $(40 \times 1.2 \times 2)/300 = 0.3$ mg.

X2.3 Effect of Temperature Variations

X2.3.1 The room balance temperature is supposed to be 15°C when weighing before sampling and 25°C when weighing after sampling.

X2.3.2 The difference between the volume of air displaced by the standard mass (25 g, volume 3.1 mL) and by the petri box (25 g, volume 12.5 mL) is 9.4 mL.

X2.3.3 Due to the temperature change (10 K) this air volume leads to an apparent mass modification of $9.4 \times 1.2 \times 10/300 = 0.4$ mg.

X2.4 Effect of Barometric Pressure Variations

X2.4.1 The barometric pressure is supposed to be:

X2.4.1.1 When weighing before sampling, 98.5 kPa, and

X2.4.1.2 When weighing after sampling, 104 kPa; therefore, a relative variation of 5.5 %.

X2.4.2 Due to this relative variation, the 9.4 mL air volume leads to an apparent mass modification of $9.4 \times 1.2 \times 0.055 = 0.6$ mg.

X2.5 Conclusions

X2.5.1 Especially when weighing parts with large internal volume, it is mandatory to wait for temperature equilibrium before weighing.

X2.5.2 There is of course no need of correction of temperature effects if the room balance is thermally controlled. But it remains necessary to take into account the effect of barometric pressure variations, specially if the volumetric mass of parts to be weighed is very different from those of standard masses used for calibration. The required correction may be done by weighing the control parts, as indicated in 11.5.

ASTM International takes no position respecting the validity of any patent rights asserted in connection with any item mentioned in this standard. Users of this standard are expressly advised that determination of the validity of any such patent rights, and the risk of infringement of such rights, are entirely their own responsibility.

This standard is subject to revision at any time by the responsible technical committee and must be reviewed every five years and if not revised, either reapproved or withdrawn. Your comments are invited either for revision of this standard or for additional standards and should be addressed to ASTM International Headquarters. Your comments will receive careful consideration at a meeting of the responsible technical committee, which you may attend. If you feel that your comments have not received a fair hearing you should make your views known to the ASTM Committee on Standards, at the address shown below.

This standard is copyrighted by ASTM International, 100 Barr Harbor Drive, PO Box C700, West Conshohocken, PA 19428-2959, United States. Individual reprints (single or multiple copies) of this standard may be obtained by contacting ASTM at the above address or at 610-832-9585 (phone), 610-832-9555 (fax), or service@astm.org (e-mail); or through the ASTM website (www.astm.org). Permission rights to photocopy the standard may also be secured from the Copyright Clearance Center, 222 Rosewood Drive, Danvers, MA 01923, Tel: (978) 646-2600; <http://www.copyright.com/>