



Designation: D6331 – 16

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 Precise and repeatable weighing procedures,

1.2.2 Using low tare weight weighing dishes,

1.2.3 Extending the sampling time at conventional sampling rates, or

1.2.4 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. This test method has tightened requirements on sampling temperature fluctuations and isokinetic sampling deviation. This test method has eliminated the in-stack filtration technique.

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.

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.

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² This test method was originally 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.

1.5 The values stated in SI units are to be regarded as standard. No other units of measurement are included in this 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, health, and environmental practices and determine the applicability of regulatory limitations prior to use.*

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

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.

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

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

2.3 U.S. EPA Documents:⁶

Reference Method 1, 40 CFR 60, Appendix A Sample and velocity traverses for stationary sources

Reference Method 3A, 40 CFR 60, Appendix A Determination of oxygen and carbon dioxide concentrations in emissions from stationary sources (instrumental analyzer procedure)

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

3.2.5 *out-of-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.

3.2.6 *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.

3.2.6.1 *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.

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

3.2.7.1 *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).

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

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

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

3.2.11 *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.

3.2.11.1 *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 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 measurable 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 Available from International Organization for Standardization (ISO), ISO Central Secretariat, BIBC II, Chemin de Blandonnet 8, CP 401, 1214 Vernier, Geneva, Switzerland, <http://www.iso.org>.

⁶ Available from United States Environmental Protection Agency (EPA), William Jefferson Clinton Bldg., 1200 Pennsylvania Ave., NW, Washington, DC 20460, <http://www.epa.gov>.

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 national, state, provincial, or other regional 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 regulatory or 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.

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.

6.1.2 Corrosion residue in rinse of metallic nozzle and metallic probe liner when used in supersaturated, acidic flue gas streams.

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](#).

6.3 Residual material existing in sample nozzle, probe, filter housing, or glassware prior to testing.

6.4 Residue present in solvent and water reagent(s).

6.5 Transient dust and material present at sampling location (platform and port) and cleanup area.

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 U.S. EPA Reference Method 1, 40 CFR 60, Appendix A, or ASTM 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). The sampling plane location shall maximize the distance downstream from a flow disturbance.

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 (0.02 in. H₂O)); 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.

7.4.3 Sample ports must be clean and free of debris, and allow for clean access/egress of the sample nozzle and probe.

8. Velocity and Gas Composition Measurement Apparatus

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

9. Sampling Apparatus and Equipment

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

9.1.1 The materials of construction of components (such as the nozzle, probe, unions, filter holder, gaskets, and other seals) shall be 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 fluoroplastic (up to 175°C), 316 stainless steel (up to 800°C), and some resistant 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.

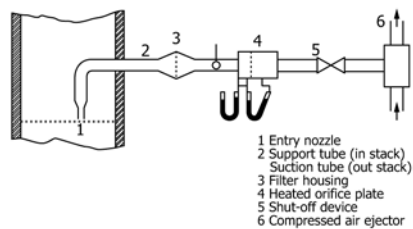


FIG. 1 Out-of-Stack Sampling Train—Example of a Dry Basis Measurement System

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 glass, virgin seamless 316 stainless steel tubing, or other virgin corrosion-resistant metal or material that is appropriate for the temperature of the gas to be sampled formed in a button-hook or elbow configuration. The tapered angle shall be $<30^\circ$ 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. Glass nozzles should be used whenever possible and especially in wet, corrosive gas streams.

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. Do not use a damaged nozzle. 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 and Heating:*

9.4.1 A filter holder constructed of borosilicate or quartz glass. Use a glass/silicone rubber or TFE-fluorocarbon frit to support the filter inside the filter holder. The holder shall be durable, easy to load, and leak free in normal applications. It is recommended to perform the following leak check procedure prior to using a new filter holder assembly to ensure that each filter holder is able to be assembled in a leak-free manner. This leak check procedure is not intended to be used for pretest and posttest leak checks of the sampling system.

- (1) Assemble the filter holder and filter.
- (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 (a 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 *Filter Heating System*—A heating system capable of maintaining the filter holder at the specified filtration temperature $\pm 8^\circ\text{C}$ during sampling.

9.4.3 *Filter Thermometer*—Monitoring device for measuring temperature inside the filter holder to within 1°C during sampling.

9.4.4 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 The sampling probe liner shall be constructed of corrosion-resistant, seamless tubing with an outside diameter (OD) of about 16 mm, usually encased within a heating system within a stainless steel sheath with an OD of 25 mm. A larger-diameter sheath or over-sheath may be used. 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), titanium or titanium alloy (see DS 5616), or other corrosion resistant metal or material that is appropriate for the temperature of the stack being sampled may be used. A heating system that will maintain an exit gas temperature of $120 \pm 8^\circ\text{C}$ ($250 \pm 15^\circ\text{F}$) during sampling is required except when the temperature of the stack is high enough to maintain such a temperature without a heating system. Other temperatures may be specified for a particular application, but the heating system must maintain $\pm 8^\circ\text{C}$. 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. If metal or other material is used, do not exceed the softening or degradation temperature specific to that material. Metal probe liners should be heat-treated or baked at 350°C or higher before first use to aid in removal of oils used in manufacture, as solvent cleaning will not always remove those oils from the inner surface of the metal tubing. It is recommended that metal liners be of virgin material having never been used on any other source emissions test. Metal probe liners used in wet acidic gas streams have been documented to have a significant contribution of corrosion byproducts in the particulate catch sample. When using metal probe liners, the source emission tester, source owner, or regulatory agency should consider conducting a test to prove the metal liner material will not contribute significantly to the particulate sample under the stack gas conditions. Visually check new probes for breaks or cracks and for leaks on a sampling train. This includes a proper nozzle-to-probe connection. Check the probe heating system prior to conducting a test program as follows:

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

9.5.1.2 After the probe temperature reaches equilibrium record the probe temperature. Rotate the probe one quarter turn inside the sheath and record the probe temperature after one minute. Rotate the probe one quarter turn two more times inside the sheath recording the probe temperature after one minute at each spot. Average the four probe temperature readings. The probe temperature readings taken at each quarter turn shall not vary by more than 5°C from the average probe temperature; otherwise, reject or repair the probe heating system.

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

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

9.5.1.5 Be sure the probe remains warm to the touch and the probe heater is capable of maintaining the exit air temperature at a minimum of 120°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. 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 $\pm 1^\circ\text{C}$ of true value in the range from 0 to 25°C.

9.7 *Gas Temperature Sensor*—For measuring gas temperature to within $\pm 1^\circ\text{C}$. Permanently attach the temperature sensor to either the probe (see 9.5) or the pitot tube (see 9.9 and Fig. 1).

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. 1. 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 $\pm 1^\circ\text{C}$ of true value in the range from 0 to 90°C, pressure gage, check valves, and related equipment, as shown in Fig. 1. Other metering systems capable of maintaining sampling rates within 5 % 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 1°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 or equivalent instrumental analysis of O₂ and CO₂*, stack gas analyzer, as described in Test Method D3154 or U.S. EPA Reference Method 3A, 40 CFR 60, Appendix A.

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 or PTFE/nylon tube handle as long as the probe, and a separate smaller and very flexible

brush for the nozzle may be used. At a minimum use a new probe and nozzle brush for each test program.

9.17.2 *Wash Bottles*, two 500-mL wash bottles for probe and glassware rinsing. Glass bottles or PTFE squeeze bottles are required.

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. Precleaned glass bottles are recommended. 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, polyethylene, styrene, or similar material petri dishes for storage and for transportation of the filter and collected sample.

9.17.5 *Graduated Cylinder, Triple Beam Balance, or Electronic Scale*, a graduated cylinder, a triple beam balance, or electronic scale 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 rinse solution containers to 102°C.

9.18 *Analytical Equipment*:

9.18.1 *Weighing Dishes*, polyethylene petri dish or other low tare weight container to facilitate filter weighing. Use a 250-mL PTFE beaker insert or equivalent low tare weight weighing dish for evaporation of the acetone/water 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.

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.