Designation: ~~D6331 – 14~~ 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 method[2] covers a method for the measurement of particulate matter (dust) concentration in emission gases in the concentrations below 20 mg/m^3 standard conditions, with special emphasis around 5 mg/m^3.

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,~~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, and in-stack methods may be more applicable than out-stack methods for the calibration of automated monitoring systems.~~temperature.

1.5 The values stated in SI units are to be regarded as ~~the~~ 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 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 (Dioctyl Phthalate) Smoke Test (Withdrawn 2004)[4]

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

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[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 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.~~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.

[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.

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
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 m^3/s.

~~3.2.3 *hydraulic diameter,* d_h~~

$$d_h = \frac{4 \times area\ of\ sampling\ plane}{perimeter\ of\ sampling\ plane} \tag{1}$$

3.2.3 ~~*in-stack filtration*—~~*hydraulic diameter,* d_h~~filtration~~

$$d_h = \frac{4 \times area\ of\ sampling\ plane}{perimeter\ of\ sampling\ plane} \tag{1}$$

~~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.~~

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

3.2.5 ~~*out-stack*~~*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/m^3) 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*—

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[5] Available from International Organization for ~~Standardization, Casa Postals 56, CH-1211, Geneva, Switzerland.~~Standardization (ISO), ISO Central Secretariat, BIBC II, Chemin de Blandonnet 8, CP 401, 1214 Vernier, Geneva, Switzerland, http://www.iso.org.

[6] ~~Metals and Alloys in the Unified Numbering System. Available from ASTM, 100 Barr Harbor Drive, West Conshohocken, PA 19428.~~Available from United States Environmental Protection Agency (EPA), William Jefferson Clinton Bldg., 1200 Pennsylvania Ave., NW, Washington, DC 20460, http://www.epa.gov.

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 ~~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~~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. 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~~ 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.

~~5.3.1 This test method is useful in such correlation applications when emissions are less than 20 mg/m~~^{3}~~.~~

~~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 (SO_2) to an insoluble sulfate compound in the moisture portion of the system (such as with limestone in flue gas following a wet flue gas desulfurization system (FGDS) to form calcium sulfate

($CaSO_4$) or the reaction with ammonia gas (NH_3) to form ammonium sulfate $(NH_4)_2SO_4$ and the potential reaction of hydrogen fluoride (HF) with glass components in the sample train with resultant collection of silicon tetrafluoride (SiF_4) in the impingers.

6.1.2 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);~~ Pa (0.02 in. H_2O)); 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 in-stack method and Fig. 2~~for the out-of-stack method.

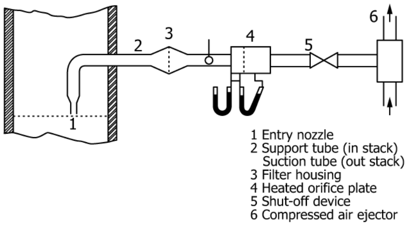

FIG. 1 ~~In Stack~~ Out-of-Stack Sampling Train—Example of a Dry Basis Measurement System

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 ~~PFTE fluoro hydrocarbons~~ 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.

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 glass~~ 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°C~~<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. 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 ~~m~~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:~~ Holders and Heating:*

9.4.1 ~~*In-Stack Method:*~~ 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.

~~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.~~

(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.

~~9.4.1.2 *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 *Filter Heating System*—A heating system capable of maintaining the filter holder at the specified filtration temperature ±8°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 ~~*Out-of-Stack Method Filter Holder*—Refer to~~Before sampling, ~~9.4.1 for in-stack method details.~~ 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.

~~9.4.2.1 *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 56I[6]) or equivalent at temperatures greater than 315°C. (Record the probe material selection in the field data sheet.)~~

9.5.1 ~~*Out-of-Stack Method*—~~The sampling probe liner shall be constructed of ~~borosilicate or quartz glass~~ corrosion-resistant, seamless tubing with an outside diameter (OD) of about 16 mm, usually encased ~~in a~~ 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) (see DS 56I~~N08825), titanium or titanium alloy (see DS 56I6), or other corrosion resistant ~~metals~~ 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 ± ~~14°C~~8°C (250 ± ~~25°F)~~15°F) during sampling is ~~required.~~ 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.~~ application, but the heating system must maintain ±8°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 with a fluoroelastomer O-ring or TFE-fluorocarbon ferrules. Check the probe heating system~~ connection. Check the probe heating system prior to conducting a test program as follows:

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

9.5.1.1 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 ~~100°C.~~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 unconstricted 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).

~~9.6.1.1 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~~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. ~~2~~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 ~~±3°C~~±1°C of true value in the range from 0 to 90°C, pressure gage, check valves, and related equipment, as shown in ~~Figs. 1 and 2~~Fig. 1. Other metering systems capable of maintaining sampling rates within ~~10 %~~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 ~~3°C~~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 ~~rthe~~the requirements of specific applications may be used. ASTM thermometers, S59C and S63C as identified in Specification E2251, may be substituted for thermomenters 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 m^3/h or 30 L for each revolution with an accuracy of ±1.0 %, to calibrate the dry test meter.

9.15 *Orsat Gas* ~~*Analyzer,*~~ *Analyzer or equivalent instrumental analysis of O_2 and CO_2,* 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 ~~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.~~ 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. ~~As an alternative to glass, polyethylene bottles may be used, if the storage time is short.~~ 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 or polyethylene~~ glass, polyethylene, styrene, or similar material petri dishes for storage and for transportation of the filter and collected sample.

9.17.5 *Graduated* ~~*Cylinder or*~~ *Cylinder, Triple Beam Balance, or* ~~*Both,*~~ *Electronic Scale,* a graduated ~~cylinder or~~ cylinder, a triple beam ~~balance~~ 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 $CaSO_4$ (see 10.5) as the desiccant.

9.17.9 *Laboratory Drying Oven,* capable of heating filters and ~~thimbles~~ rinse solution containers 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,*~~ *Weighing Dishes,* ~~borosilicate glass dishes~~ polyethylene petri dish or other low tare weight container to facilitate filter weighing. Use a 250-mL ~~glass beaker~~ PTFE beaker insert or equivalent low tare weight weighing dish for evaporation of the ~~acetone~~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.

~~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 %~~ ≤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 ~~was~~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.~~

10.9.1 ~~*Alternate In-stack Method A and Out-of-Stack Method*—~~Use glass fiber filters without organic binders. Quartz glass fiber filter are recommended for use in most applications (especially in gas streams with SO_2 or other acid gases). However other filter materials (for example, PTFE, polycarbonate, ceramic, or cellulose) may be used provided the stack tester or source owner can demonstrate the filter material will not bias the results due to chemical reaction between the filter material and the effluent gas matrix. 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~~manufacturer's quality control test data are sufficient for validation of efficiency. Check the filters for irregularities, flaws, or pinholes by holding them up against a light source.

~~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

NOTE 1—There may be regulatory requirements for the state of plant operations and you may wish to consult with regulatory personnel as well.

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 particulate 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.

11.1.3 *Discussion*—To obtain results with 10 percent uncertainty at 0.99 confidence and a reported lab detection limit (DL) of 0.15 mg, one needs to collect 5.0 mg in the sampling system:

$$(DL * 100)/3$$

At a particulate concentration of 5 mg/scm and sampling at 1.0 standard cubic foot per minute (0.028 standard cubic metres per minute), the sampling time needs to be approximately 36 minutes.[8]

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~~a relevant temperature for conditioning and drying the filter before and after sampling (see 14.2~~or contains appreciable amounts of SO~~); and~~$_3$, 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~~

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[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.

[8] *Filterable Particulate Matter Stack Test Methods: Performance Characteristics and Potential Improvements*, EPRI, Palo Alto, CA: 2013, Report No. 3002000975.

11.2.1.1 ~~When using an out-stack filtration device, set the out-stack~~Set the out-of-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 if the measurement series covers multiple days 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. The overall blank can also evaluate the effectiveness of the rinse procedures.

11.3 *Weighing Procedures:*

11.3.1 ~~Depending on the kind of sampling device used, the~~ 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:~~are the filter and the rinse solution container.

~~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~~Pre- ~~11.3.1.2~~and ~~11.3.1.3~~, ~~pre- and~~ post-treat all the ~~relevant parts~~ filters and rinse solution containers in accordance with 11.4 and ~~weigh~~11.6 ~~them together, without disassembling them.~~following the procedures detailed in 11.5.

~~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~~10.9.1) on a numbered container and rinse solution container (see 9.18.1) in a laboratory drying ~~over~~oven (see 9.17.9) ~~on a numbered container~~ for 2 ~~h~~hours at the temperature of the ~~sampling.~~ sampling (see 11.2). After removal from the oven, cool to room temperature in a desiccator (see 9.17.8). After cooling, weigh each filter and rinse solution container to the nearest 0.1 mg, and record the data. Return the filter and rinse solution container to the desiccator. After at least 6 hours of desiccation, weigh each filter and rinse solution container to the nearest 0.1 mg, and record the data. When two consecutive weights have a difference of no more than 0.3 mg, a constant weight is achieved. Average the two weights and report the average as the pre-sampling weight. If the consecutive weights have a difference of more than 0.3 mg, continue to weigh the part at no less than 6 hours of desiccation time between weightings until two consecutive weights have a difference of no more than 0.3 mg. 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 and rinse solution container 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).~~

11.5.4 Give attention also to weighing artifacts related to:

11.5.4.1 Electrostatic charges, which may have to be discharged or neutralized; insert an antistatic device inside the balance enclosure; recommend blowing off outside of PTFE beaker insert while moving it from the desiccator to the balance to eliminate static attraction of dust onto the beaker.