



Designation: **D6331–98 (Reapproved 2005) D6331 – 13**

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 *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](#)

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 March 1, 2005 April 1, 2013. Published May 2005 April 2013. Originally approved in 1998. Last previous edition approved in 1998 2005 as **D6331–98, D6331 – 98(2005)**. DOI: 10.1520/D6331-98R05-10.1520/D6331-13.

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.

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

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.

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

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 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 end 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

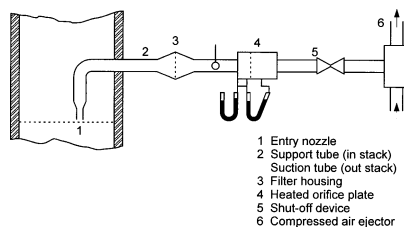


FIG. 1 In Stack Sampling Train—Example of a Dry Basis Measurement System

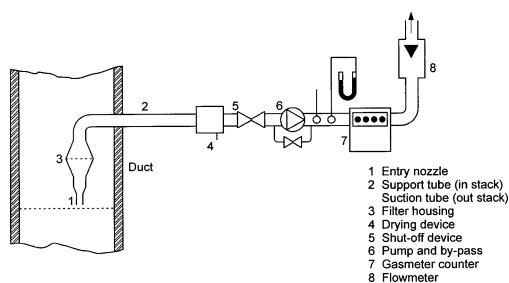


FIG. 2 Out-Stack Sampling Device Example of a Wet Basis Measurement System

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 $\pm 20^{\circ}120 \pm 15^{\circ}\text{C}$ 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 condensable material is present in the stack. Otherwise, the condensed or corroded materials in the probe extension may drain or be back flushed into the filter and contaminate the sample. Use a nonreactive material to prevent contamination of the sample if condensibles are to be retained. Use probe extenders of nickel-iron-chromium alloy (UNS N08825) (see DS 561⁶) 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 561⁶), or other corrosion resistant metals may be used. A heating system that will maintain an exit

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

gas temperature of $120 \pm 14^\circ\text{C}$ ($250 \pm 25^\circ\text{F}$) 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 $\pm 1^\circ\text{C}$ 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 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 $\pm 3^\circ\text{C}$ 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. 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).