



Designation: D6831 – 11 (Reapproved 2018)

Standard Test Method for Sampling and Determining Particulate Matter in Stack Gases Using an In-Stack, Inertial Microbalance¹

This standard is issued under the fixed designation D6831; 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 describes the procedures for determining the mass concentration of particulate matter in gaseous streams using an automated, in-stack test method. This test method, an in-situ, inertial microbalance, is based on inertial mass measurement using a hollow tube oscillator. This test method describes the design of the apparatus, operating procedure, and the quality control procedures required to obtain the levels of precision and accuracy stated.

1.2 This test method is suitable for collecting and measuring filterable particulate matter concentrations in the ranges 0.2 mg/m³ and above taken in effluent ducts and stacks.

1.3 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 this test method (and other in-stack methods) may be more applicable than out-stack methods for the calibration of automated monitoring systems.

1.4 This test method can be employed in sources having gas temperature up to 200°C (392°F) and having gas velocities from 3 to 27 m/s.

1.5 This test method includes a description of equipment and methods to be used for obtaining and analyzing samples and a description of the procedure used for calculating the results.

1.6 This test method may also be limited from use in sampling gas streams that contain fluoride, or other reactive species having the potential to react with or within the sample train.

1.7 **Appendix X1** provides procedures for assessment of the spatial variation in particulate matter (PM) concentration within the cross section of a stack or duct test location to

determine whether a particular sampling point or limited number of sampling points can be used to acquire representative PM samples.

1.8 **Appendix X2** provides procedures for reducing the sampling time required to perform calibrations of automated monitoring systems where representative PM samples can be acquired from a single sample point and certain other conditions are met.

1.9 The values stated in SI units are to be regarded as standard. The values given in parentheses are mathematical conversions to inch-pound units that are provided for information only and are not considered standard.

1.10 *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.*

1.11 *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:*²

D1356 Terminology Relating to Sampling and Analysis of Atmospheres

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

D3685/D3685M Test Methods for Sampling and Determination of Particulate Matter in Stack Gases

D3796 Practice for Calibration of Type S Pitot Tubes

D6331 Test Method for Determination of Mass Concentration of Particulate Matter from Stationary Sources at Low Concentrations (Manual Gravimetric Method)

¹ This test method is under the jurisdiction of ASTM Committee D22 on Air Quality and is the direct responsibility of Subcommittee D22.03 on Ambient Atmospheres and Source Emissions.

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² 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.

- 2.2 *EPA Methods from 40 CFR Part 60, Appendix A:*
Method 3A Determination of Oxygen and Carbon Dioxide Concentrations in Emissions from Stationary Sources (Instrumental Analyzer Procedure)
Method 5 Determination of Particulate Emissions from Stationary Sources
Method 17 Determination of Particulate Emissions from Stationary Sources (In-Situ Filtration Method)
- 2.3 *EPA Methods from 40 CFR Part 60, Appendix B:*
Performance Specification 11 Specifications and Test Procedures for Particulate Matter Continuous Emission Monitoring Systems at Stationary Sources
- 2.4 *EPA Methods from 40 CFR Part 63, Appendix A:*
Method 301 Field Validation of Pollutant Measurement Methods from Various Waste Media

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

3.2.1.1 *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. For the purposes of this test method, particulate matter is defined by gas borne matter (solid or liquid) captured on or in the filter after drying and weighing in accordance with this test method.

3.2.2 *in-stack, inertial microbalance*—a mechanical oscillator constructed of a hollow tube of a specific metal alloy and fitted with a filter cartridge that is designed to oscillate at a frequency that is proportional to the mass of the hollow tube oscillator plus the mass of its filter cartridge.

3.2.3 *mass transducer*—the mass transducer is a principle component of an in-stack inertial, microbalance. The mass transducer provides the mechanical structure to support and contain the hollow tube oscillator and to support the sample inlet nozzle fixture, source gas temperature thermocouple, and S-type Pitot tube assembly. Refer to **6.1.1** for a detailed description of this component.

3.2.4 *articulating elbow*—a mechanical component that may be integrated into the sample probe just before the end connector attaching to the mass transducer. This elbow is used control the angle of the mass transducer relative to the sample probe during insertion of the probe and mass transducer into the stack and while positioning the mass transducer inlet nozzle into the gas stream.

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

3.2.5.1 *Discussion*—The temperature of the filter cartridge is maintained at the desired temperature by controlling the temperature of the mass transducer case and cap.

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

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

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

3.2.9 *weighing control procedures*—quality control procedures used for verifying the calibration constant for the hollow tube oscillator.

3.2.9.1 *Discussion*—Unlike test methods such as **D6331** or **D3685/D3685M**, this test method does not rely on weighing sample media in a laboratory before and after a test is conducted. The method includes an integrated filter drying mechanism to desiccate the sample collection media in-situ immediately prior to and following each test run. No physical handling of sample collection media takes place prior to the start of a test run through final filter analysis for the test run. Consequently, control filters typically used to characterize the impact of filter/sample handling and transportation are not required with this test method.

4. Summary of Test Method

4.1 The in-stack, inertial microbalance method involves the use of a filter cartridge affixed at one end of a hollow tube oscillator that is housed in a mass transducer housing. The mass transducer is attached to the end of an integrated sample probe and inserted through a port into the stack or duct. A sample is withdrawn isokinetically from the gas stream and directed through the filter cartridge attached to the end of the hollow tube oscillator. Captured particulate matter and any captured moisture is weighed continuously as the sample gases pass through the filter cartridge and hollow tube oscillator. Sample gases then continue through the heated probe and umbilical assemblies and into a gas conditioning/control module where the collected gas sample volume is determined. A calibrated, orifice-based flow meter is used to measure the sample gas volume. In sources where the particulate matter characteristics can result in significant quantity of particulate matter to be trapped on the inlet nozzle walls during sampling, the trapped particulate matter can be recovered after sampling has been completed using a properly sized brush to detach and recover trapped particulate matter from the inlet walls.

4.1.1 *Discussion*—The ability of this mass measurement technique to precisely quantify the mass of the filter and collected particulate matter by correlating mass change to a measured frequency change of the hollow tube oscillator is predicated on the isolation of the oscillator from external vibration sources. To remove the potential for external vibration to interfere with the measurement process, the mass transducer housing must be sufficiently massive so that any energy that it absorbs from external vibrations will result in the mass transducer case oscillating at a resonant frequency that is much lower the hollow tube oscillator. As a result, a massive housing will absorb any external vibrations and prevent those vibrations from affecting the resonance of the hollow tube oscillator.

4.2 The filter media typically used is PTFE coated glass fiber filter media (TX-40 or equivalent) although other filter media can be used if desired. The filter media is mounted in a specially designed filter cartridge housing that is designed to promote a constant face velocity through the entire surface of the filter. The junction of the oscillating element and the base of the filter cartridge is designed to ensure a leak free union.

4.3 The sample gases are dried using a selectively permeable membrane dryer followed by silica gel before the sample volume is measured. An integrated computer-controlled feedback system is used to control the sample flow rate based on stack gas temperature, velocity and gas density measurements, or user input data, to automatically maintain isokinetic sampling conditions.

4.4 To account for source gas density (molecular weight) inputs to set the isokinetic sampling conditions, the user has the option to use manually input data acquired using an Orsat analyzer and moisture determination apparatus, or equivalent methods, or data supplied by an on-board carbon dioxide analyzer, oxygen analyzer and moisture measurement system.

4.5 Valid measurements can be achieved when:

4.5.1 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;

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

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

4.5.4 Isokinetic sampling conditions are maintained throughout the test within $\pm 10\%$;

4.5.5 Samples are taken at a pre-selected 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.

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

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

4.5.8 The sampling and weighing procedures include desiccation of the filter immediately before and after each test run is conducted.

5. Significance and Use

5.1 The measurement of particulate matter is widely performed to characterize emissions from stationary sources in terms of emission concentrations and emission rates to the atmosphere for engineering and regulatory purposes.

5.2 This test method provides near real-time measurement results and is particularly well suited for use in performance assessment and optimization of particulate matter controls achieved by air pollution control devices or process modifications (including fuel, feed, or process operational changes) and performance assessments of particulate matter continuous emissions monitoring systems (PM CEMS)

5.3 This test method is well suited for measurement of particulate matter-laden gas streams in the range of 0.2 mg/m^3 to 50 mg/m^3 , especially at low concentrations.

5.4 The U.S. EPA has concurred that this test method has been demonstrated to meet the Method 301 bias³ and precision criteria for measuring particulate matter from coal fired utility boilers when compared with EPA Method 17 and Method 5 (40CFR60, Appendix A).

5.5 This test method can accurately measure relative particulate matter concentrations over short intervals and can be used to assess the uniformity of particulate concentrations at various points on a measurement traverse within a duct or stack.

6. System Description

6.1 *Major Components*—The in-stack, inertial microbalance measurement system is comprised of five major components that are listed in the following table.

Mass Transducer (see 6.1.1)	An assembly that houses the sample filter and inertial microbalance. Also contains the Pitot tube assembly, stack gas temperature thermocouple, sample inlet nozzle and mass transducer heaters.
Sample Probe and Probe Extensions (see 6.1.2)	A heated support conduit for mass transducer, sample and purge flow lines; electrical supplies for mass transducer and probe heaters; mass transducer electrical signal cables; and the pivoting elbow used for positioning the mass transducer into the source gas flow.
Sample Pneumatic/Electrical Umbilical Cables (see 6.1.3)	A heated, flexible tubing bundle that contains the pneumatic lines for transporting the sample and purge gases from/to the mass transducer; and the electrical supply and signal cabling.
Control Unit (see 6.1.4)	A unit that contains sample and purge supply flow sensors and controllers; stack gas velocity pressure and temperature transducers; sample and purge supply pressure and temperature transducers, data acquisition and instrument control systems; sample and purge gas conditioners; heater relays; and optionally, CO ₂ , O ₂ and moisture measurement systems comprising the real-time molecular weight measurement system.
Pump / Power Unit (see 6.1.5)	Contains the sample vacuum and purge supply pumps and the 24 VDC power supply transformer for the 24 VDC heaters in the probe and mass transducer.

A block diagram of the major components of an in-stack, inertial microbalance system is shown in Fig. 1.

6.1.1 *Mass Transducer*—The mass transducer houses the hollow tube oscillator that is the main component of the inertial microbalance. The mass transducer can also serve as the support structure for the S-type Pitot tube assembly and a thermocouple that are used for measuring stack gas velocity and temperature, respectively. A filter cartridge is mounted at the end of the hollow tube oscillator. As sample gas is drawn through the filter, particulate matter is trapped on the filter and removed from the sample gas stream. The trapped particulate matter on the filter cartridge causes the oscillation frequency of the hollow tube oscillator/filter cartridge system to change. The

³ See October 3, 2002 letter from Conniesue B. Oldham, Group Leader, Source Technology Measurement Group, Office of Air Quality Planning and Standards, U.S. EPA to Edward C. Burgher, Rupprecht & Patashnick Co., Inc.

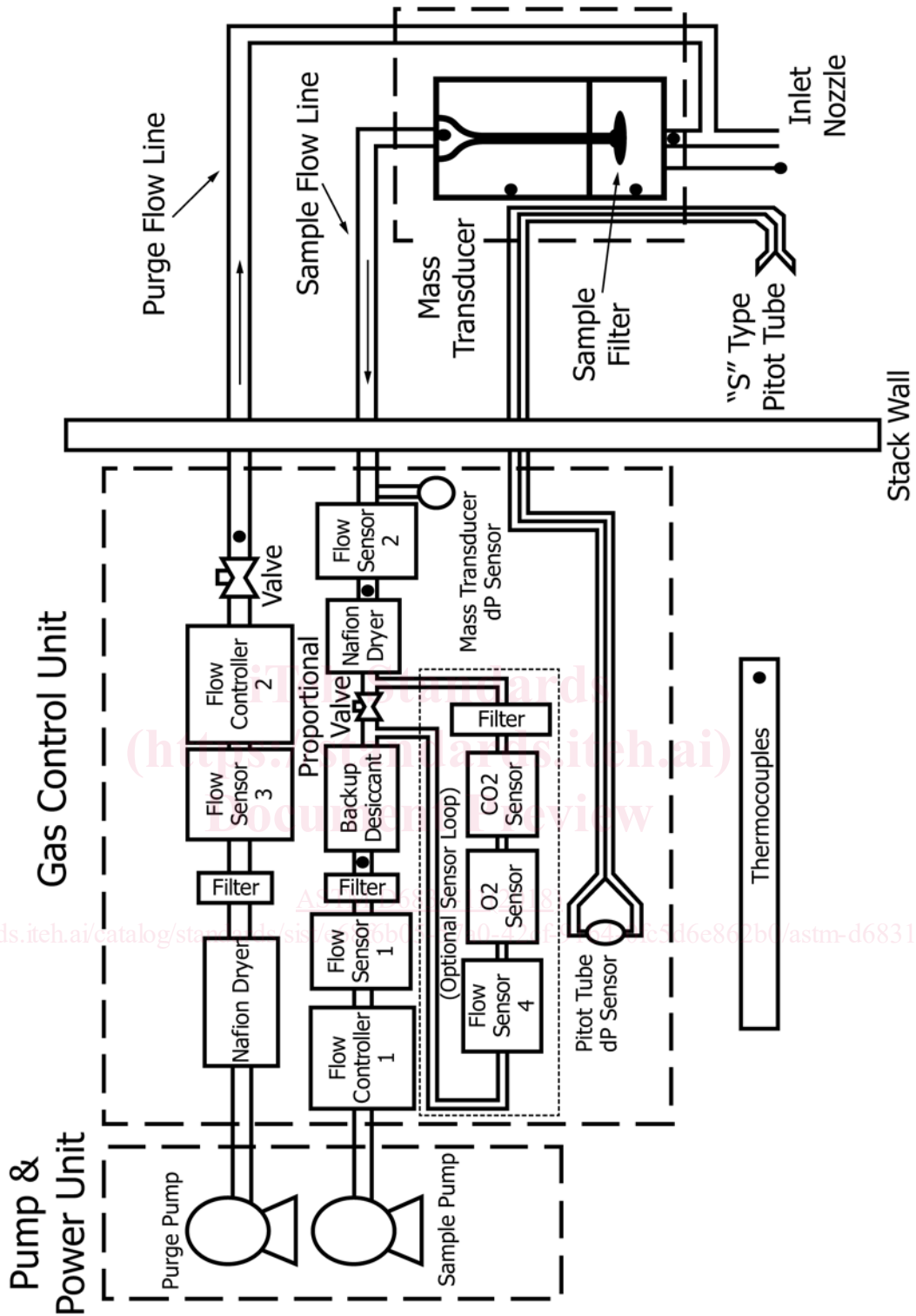


FIG. 1 Block Diagram of In-stack, Inertial Microbalance System

frequency is converted to an electronic signal that is transmitted to an analog to digital frequency converter. The frequency is converted to mass by appropriate computerized calculation software. The firmware computes the mass from the measured frequency approximately once every three seconds. The deter-

mination of mass from the measured frequency is shown below in Eq 1 and is detailed in Eq 2-6.

$$f^2 = K_0/M \tag{1}$$

where:

- f = oscillation frequency of the hollow tube oscillator
 K_o = calibration constant for the hollow tube oscillator, and
 M = mass of filter and collected particulate matter

The mass transducer also combines components for measuring stack gas temperature and velocity, and to provide clean, dry air to desiccate the filter before and after sampling. The components and features of the mass transducer are described in 6.1.1.1 – 6.1.1.4.

6.1.1.1 Main Flow Inlet Nozzle—The main flow inlet nozzle is exchangeable to allow sampling over a wide range of source gas velocity conditions (3 m/s – 27 m/s). Recommended are nozzles having inside diameter ranging from 1.5875 mm (0.0625 in.) to 3.1750 mm (0.125 in.) to allow isokinetic sampling over a range of gas velocity conditions from 3 to 27 m/s. The nozzles are constructed of seamless 316 stainless steel and are designed with a sharp, tapered leading edge. The outside leading edge tapered angle is 30°, and the inside diameter is constant. Verification of the inlet's inside diameter can be performed using precision measuring pins or a micrometer.

6.1.1.2 Purge Flow Supply Line—A separate pneumatic supply line is provided through the mass transducer case to a tubing coil wrapped on the outside of the mass transducer cap and then into a fitting located just downstream of the exchangeable inlet nozzle. This pneumatic line supplies dry, scrubbed air to the inlet nozzle for use in drying the filter before and after sample collection.

6.1.1.3 Impact, Wake and Static Pitot Tubes—An impact and wake Pitot tube assembly is of a type S design and constructed using 316 stainless steel nozzles. A static Pitot tube is oriented perpendicular to the gas flow direction and integrated into the side of the Pitot tube assembly. Initial calibration of the Pitot tube assembly must be performed by attaching the assembly to a mass transducer and dynamically calibrating the system in a wind tunnel. If damage to the Pitot tube assembly occurs or if post-test quality assurance is desired, dimensional checks of the Pitot tube assembly are made using data supplied by the manufacturer or calibration agency. If dimensional checks do not meet specifications, the Pitot tube assembly should be recalibrated or replaced with a calibrated assembly.

6.1.1.4 Thermocouple—A type K thermocouple is used for measuring stack gas temperature. The thermocouple is integrated into the Pitot tube assembly and protrudes about two mm above Pitot tube assembly surface on the end of the assembly first impacted by the gas stream when the mass transducer is in the sampling position. A thermocouple should be used that can measure the source gas temperature to within $\pm 1.5\%$ of the absolute stack gas temperature.

6.1.2 Sample Probe and Extensions—The mass transducer is mounted at the end of the sample probe allowing extension of the mass transducer into the source being sampled. An optional probe support assembly can be used with the probe to assist with handling and inserting the sample probe into the source being sampled. A sample probe and extensions are used to position the mass transducer in the desired sampling location. When attached to the probe, the mass transducer inlet

nozzle must be positioned such that the inlet nozzle points directly into the gas stream being sampled. An articulating elbow can be installed just downstream of the connection to the mass transducer to allow the mass transducer to be inserted into a three inch diameter (minimum) sample port, then pivoted in order to properly orient the sample inlet nozzle during sampling. After the probe and mass transducer are introduced through the sample port into the gas stream, the pivoting elbow can be maneuvered to orient the inlet nozzle such that it points directly into the source gas stream.

6.1.2.1 The probe also contains a number of heated (temperature controlled), 316 stainless steel and PTFE pneumatic tubing to supply sample vacuum to the inlet nozzle, carry sample gas from the mass transducer to the flow sensors and controllers, supply purge air to the inlet nozzle and pneumatically connect the Pitot tubes to pressure sensors located in the control unit. Electrical cabling in the probe and the sample umbilical is used to carry signals from mass transducer and thermocouples to the control unit. Power to the probe and mass transducer heater elements is transmitted from the control unit to the probe and mass transducer through a separate electrical umbilical line.

6.1.2.2 The sample probe is inserted into the stack through a bearing mechanism that is attached to a sample port. The bearing mechanism allows the sample probe to be rotated through a complete 360-degree arc about the probe axis. The mechanism must incorporate a locking cam mechanism to ensure the mass transducer remains in the desired position. The bearing mechanism and probe design allows the system operator to position the mass transducer inlet nozzle so that when sampling, the inlet nozzle is pointed directly into the gas stream. It also permits the operator to rotate the probe while observing the pressure drop across the Pitot tubes to determine presence of cyclonic flow or to determine the proper orientation of the probe in the gas stream being sampled.

6.1.3 Umbilical Lines—Flexible umbilical lines are used to connect the pneumatic and electrical assemblies in the probe to a control unit and to connect the sample vacuum, purge supply and heater electrical supply from a pump/power unit to the control unit. A pneumatic umbilical contains PTFE tubing to carry the sample gas to the Control Unit. The pneumatic umbilical also contains PTFE tubing for the Pitot tube system and to convey clean, dry purge air for filter drying from a gas conditioning system to the mass transducer inlet. To prevent condensation of moisture and acid gases that may be present in sample gases, the pneumatic umbilical should be heated to at least 150°C . An electrical umbilical contains electrical wiring to carry signals to/from the mass transducer and electrical power to the heaters located in the mass transducer and the sample probe.

6.1.4 Control Unit—A control unit houses the hardware and electronic assemblies needed to operate the system. This includes the computerized instrument data acquisition and control system, flowmeters and controllers to measure and control source gas sampling rates, control sample umbilical and probe heaters, control the filter purge gases, perform real-time moisture measurements and (optional) CO_2 and O_2 measurements of the sample gases, and store data collected

during sampling. The computer, hard drive, flow sensors and controllers, CO₂ and O₂ analyzers and heater relays are all mounted on an internal chassis that is secured by anti-shock mounts to the Control Unit cabinet to prevent damage during installation or removal of the system from the sampling platform. The system should have active heating and cooling to allow for its use in a wide range of ambient temperatures conditions (–30°C to +50°C). All external electrical and pneumatic connections to the control unit are made using modular connectors that are designed to be weather-tight.

6.1.5 *Pump/Power Unit*—A pump/power unit is configured with two pumps and a power supply to supply sample vacuum, a purge air supply and a power supply. The unit is actively heated or cooled for use in a wide range of ambient temperatures (–30°C to +50°C).

7. System Calibration

7.1 *Mass Transducer—Hollow Tube Oscillator K₀ Verification:*

7.1.1 A Mass Verification Filter Cartridge is prepared for checking the calibration constant of the hollow tube oscillator by conditioning in a desiccator to a dry, constant weight. The gravimetric balance used for weighing the Mass Verification Filter Cartridges must be accurate to within ±1 µg. Proper care must be taken when handling the Mass Verification Filter Cartridges to prevent weighing errors due to contamination and/or absorption of moisture by the filter cartridge.

7.1.2 Weight determinations of a Mass Verification Filter Cartridge are performed in triplicate until less than 3 % difference between the individual readings is obtained.

7.1.3 The hollow tube oscillator calibration constant (K₀) is determined using a Mass Verification Filter. The calibration constant is deemed acceptable when the deviation from the mean of the individual K₀ measured for each filter is less than or equal to ±1.5 %.

7.2 *Sample Inlet Nozzles*—The inside diameter of inlet nozzles are visually checked prior to each use in a test or when damage is suspected. If damage is suspected, precision measuring pins or a micrometer can be used to verify the inside diameter. The inlet diameter must be determined within ±0.025 mm (0.001 in.). If the requirement is not met, the nozzle is rejected, reshaped or resharpened or both, then re-checked relative to acceptable tolerance before use, or returned to the manufacturer.

7.3 *Flow Sensors*—All flow sensors (main, moisture and the purge flow sensors) are pre-calibrated by the factory prior to the initial shipment or whenever a flow sensor fails a flow audit.

7.3.1 Flow sensor calibrations are performed at minimum of seven evenly spaced points over the range of the sensor.

7.3.2 Flow sensor calibrations are performed using primary flow standards or transfer standards traceable to recognized primary standards (for example, NIST traceable flow standards) accurate to within ±1 %.

7.3.3 Maximum allowable flow sensor calibration error is ±2.0 % of the sensor range.

7.4 *Moisture Measurement Flow Sensor Calibration:*

NOTE 1—This section is only applicable if isokinetic sampling conditions are determined using an onboard moisture measurement system that determines the moisture content of the sample gas by comparing the sample gas volume determined under “hot, wet” conditions to the same volume determined under “cool, dry” conditions.

NOTE 2—The main flow sensor must meet the calibration acceptability limits given in 7.3.3.

7.4.1 Configure the two flow sensors used for the moisture measurement (identified as Q₁ and Q₂ in 12.6) in series.

7.4.2 Connect appropriate gas dryer (for example, silica gel) and filtering devices to the inlet of the upstream sensor and a vacuum source to the outlet of the downstream flow sensor. A flow control valve should be placed in the vacuum line just upstream of the vacuum pump.

7.4.3 Perform flow rate measurements at a minimum of five points equally spaced over the measurement range of the main and moisture flow rate sensor.

7.4.4 Prepare a calibration curve for flow sensor Q₂ using flow sensor Q₁ as the reference.

7.4.5 Enter the calibration equation into the moisture measurement software.

7.4.6 Calibration of the moisture measurement system should be performed in accordance with 7.3.1.

7.5 *Pitot Tubes*—Calibration of the Pitot Tube Assembly is performed in accordance with Practice D3796. When calibrating the Pitot tube assembly in a wind tunnel, the assembly shall be attached to the mass transducer and the mass transducer shall be affixed to a sample probe.

7.5.1 Calibration of the Pitot tube assembly must be performed in a wind tunnel that is at least 0.9144 m by 0.9144 m (4 ft by 4 ft) in cross-section and have laminar flow at the sensor location.

7.5.2 Pitot tube calibration is conducted at a minimum five points over a velocity range of 3 m/s (10 ft/s) to 27 m/s (90 ft/s).

7.5.3 At least four velocity readings are made at each velocity test point to form an average coefficient for each velocity. The average coefficients are then averaged to form the final Pitot tube coefficient. The standard deviation of the average coefficients must be less than or equal to 0.1.

7.6 *Barometric Pressure Sensor*—Calibration of the pressure sensor shall be performed by comparison to mercury, aneroid or other barometer accurate to ±2.5 mm Hg (0.1 in. Hg). The pressure sensor is accurate to within ±2.5 mm Hg (0.1 in. Hg).

7.7 *Stack Gas Temperature Thermocouple*—The stack gas thermocouple is accurate within 4°C and is calibrated against mercury-in-glass or equivalent thermometer.

7.8 *CO₂ and O₂ Gas Sensors*—Three calibration gas cylinders are needed to perform carbon dioxide (CO₂) and oxygen (O₂) gas sensor calibrations or audits.

7.8.1 The cylinder gas mixtures (certified Protocol) are as follows:

7.8.1.1 *Zero:* Nitrogen – 100 %. Residual CO₂ and O₂ must be less than 0.25 % of the CO₂ and O₂ sensor span.