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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 method, an in-situ, inertial microbalance, is based on inertial mass measurement using a hollow tube oscillator. This 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 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 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 Stack temperatures limitation for this test method is approximately 200°C [392°F].~~

~~1.7 This test method may be 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.~~

~~1.8~~

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 the standard. The values given in parentheses are for information only.

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 and health practices and determine the applicability of regulatory limitations prior to use.

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*—~~for solid~~—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 ~~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 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 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 ~~only~~ 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 total emission rates to the atmosphere for regulatory purposes.~~

~~5.2 This test method is particularly well suited for use in performance assessment and optimization of particulate matter control systems, continuous particulate matter emissions monitoring systems and the measurement of low concentration particulate matter laden gas streams in the range of 0.2 mg/m~~

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 method is well suited for measurement of particulate matter-laden gas streams in the range of 0.2 mg/m³ to 50 mg/m³, especially at low concentrations.

5.4 The U.S. EPA has concurred that this 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 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.

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, Rupperecht & Patashnick Co., Inc.

Sample Pneumatic/ Electrical Umbilical Cables (see 6.1.3) Control Unit (see 6.1.4)	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.
Pump / Power Unit (see 6.1.5)	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. 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 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 determination of mass from the measured frequency is shown below in Eq 1 and is detailed in Eq 2-6.

$$f^2 = K_0 / M \quad (1)$$

where:

- f = oscillation frequency of the hollow tube oscillator
- K_0 = 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 ±1.5 % of the absolute minimum 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

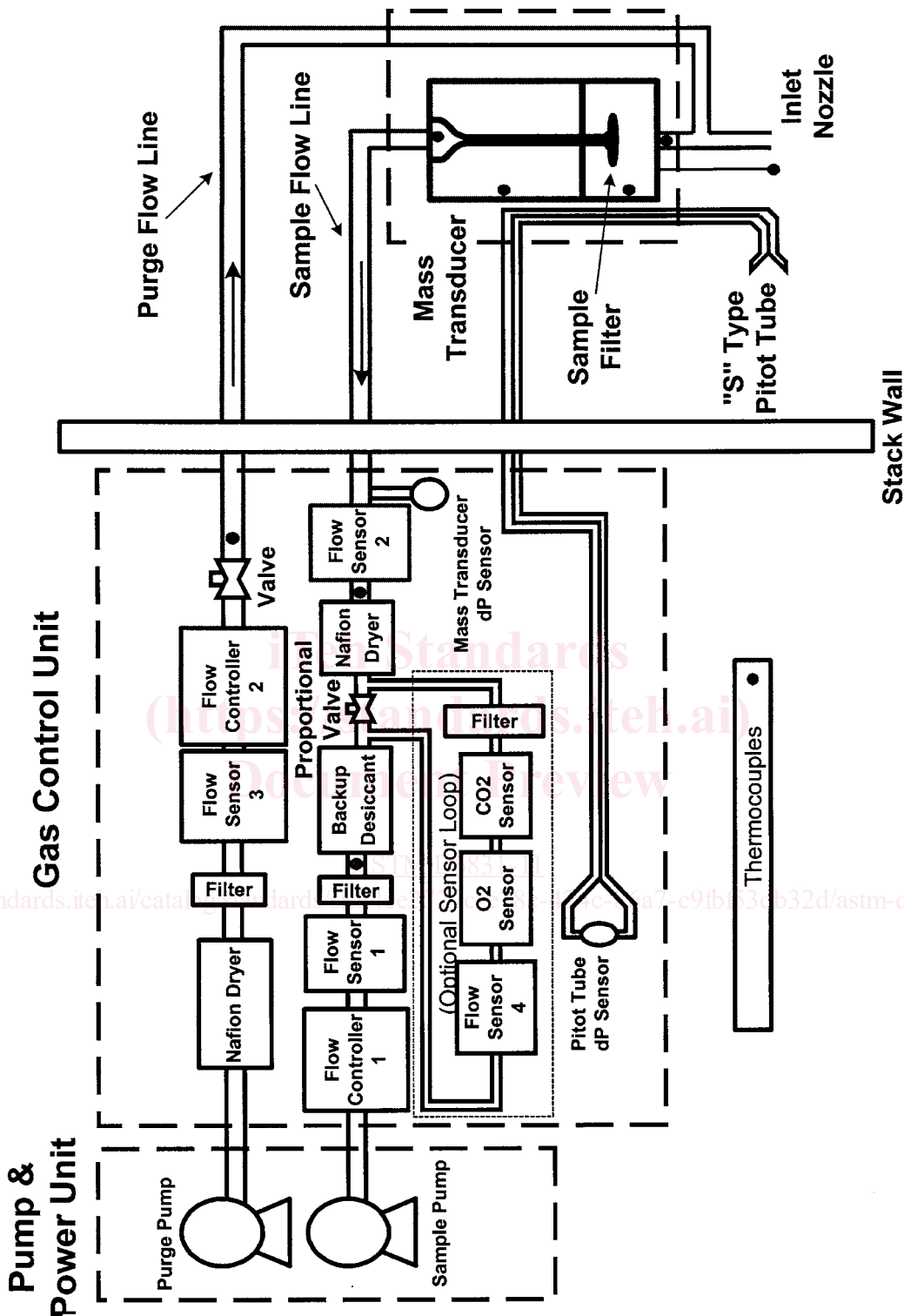


FIG. 1 Block Diagram of In-stack, Inertial Microbalance System

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) CO₂ and O₂ 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 ~~is~~ 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.} (0.001 in.). If the requirement is not met, the nozzle is rejected, resharpened or reshaped, 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 Q_2 using flow sensor Q_1 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(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} (10 ft/s) to 27 m/s {90 ft/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} (Hg). The pressure sensor is accurate to within ± 2.5 mm Hg {0.1 in. Hg} (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.

7.8.1.2 *High*: CO

7.8.1.2 *High*: CO₂ and O₂ in N₂ balance - 80 to 100 % of CO₂ sensor span, 80 to 100 % of O₂ sensor span, remainder N₂.

7.8.1.3 *Mid*: CO

7.8.1.3 *Mid*: CO₂ and O₂ in N₂ balance - 40 to 60 % of CO₂ sensor span, 40 to 60 % of O₂ sensor span, remainder N₂.

7.8.2 Gas sensor calibration and bias checks must be performed with the gas standards' regulator pressure set to instrument manufacturer's specifications.

7.8.3 Prior to performing gas sensor calibration, a leak check of the sampling system must be performed. Leak checks must be performed with all system components (mass transducer, probe, pneumatic umbilical, and control unit) warmed to their normal operating temperature.

7.8.4 Acceptance criteria for gas sensor calibration check error is $\leq \pm 2$ % of the gas sensor measurement range (span) for both the CO₂ and O₂ sensors.

7.8.5 Acceptance criteria for gas sensor bias is $\leq \pm 5$ % of the gas sensor measurement range.

8. System Installation and Configuration

NOTE 3—Before setting up the instrument, the system operator must read the training and operations manual or be fully familiar with its operation to fully understand the procedures given below. Only general descriptions of the instrument setup and operation are provided in this document. Also, users should be familiar with stack testing methods and safety procedures.

NOTE 4—It is important that all system power be turned off or disconnected during assembly and disassembly.

8.1 *Bearing Mechanism and Probe Support Assembly*—Attach a bearing mechanism to the sample port flange. If desired, attach a probe support assembly to the bearing mechanism.

8.2 *Mass Transducer, Probe and Sample/Electrical Umbilicals*—Join the first and second sections of the sample probe together then connect the electrical/pneumatic umbilicals and the mass transducer to the proper ends of the probe assembly. Make sure that all connections are secure.

8.3 *Pump/Power Unit*—Connect the following components to the proper connectors on the right hand side of a pump/power unit:

8.3.1 Pneumatic sample line.

8.3.2 Pneumatic purge supply line.

8.3.3 Power supply cord for sample line heater.

8.3.4 Probe heater power supply umbilical cord.

8.4 *Control Module*—Locate the proper connectors and make the connections for the following components:

8.4.1 Computer and operator electronic interface communication cables.

8.4.2 Ambient Temperature Sensor.

8.4.3 Electrical Umbilical Cable.

- 8.4.4 Probe heater power cable from the pump box.
- 8.4.5 Sample and purge pneumatic lines from the pump/power unit.
- 8.4.6 Junction box for sample/electrical umbilical lines.
- 8.4.7 Power cable for sample line umbilical heater.

8.5 *Verify All Connections*—Make sure all electrical and pneumatic connections are secure then turn on the power switches for the control and pump/power units.

9. Requirements for Sampling Plane and Sampling Points

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

9.2 Perform sampling at a sufficient number of sampling points. Sample points are usually located on two or more sampling lines.

9.3 Convenient access ports and a working platform are required for the testing.

9.4 See Test Method D6331, Section 7 for specific requirements related to identification of appropriate sampling plane(s) and sample point locations.

10. Measurement Procedures

10.1 *Preparation of System:*

10.1.1 Install the monitor as described in Section 8. A suitable test location must be provided as follows:

- 10.1.1.1 A suitable flange, capable of supporting the probe and bearing assemblies.
- 10.1.1.2 Sufficient clearance to allow insertion of the probe into the bearing assembly.

10.2 *Configure Software/Firmware*

10.2.1 Configure system identification information and calibration factors, sample times, test parameters, and so forth, needed to provide the system software test instructions.

10.2.2 Transfer configuration data from a laptop computer to the control unit computer.

10.3 *Initiate Test*

10.3.1 Verify test setup configuration choices. Edit data fields to correct any errors in the test configuration.

10.3.2 Install new filter on the hollow tube oscillator.

10.3.3 Install inlet nozzle. If velocity traverse is not performed proceed to 10.3.5.

NOTE 5—Select the appropriate nozzle based on expected gas velocity. Final inlet selection will be made after velocity traverse.

10.3.4 Velocity traverse.

10.3.4.1 Prior to conducting a velocity traverse, perform leak check on Pitot tube assembly / pneumatic system.

10.3.4.2 The acceptable leak rate for the “impact” pitot is $\leq 2.0\%/15\text{ s}$ with the system pressure between 0.75 KPa and 1.12 KPa at initiation of the leak check. The acceptable leak rate for the draft pressure Pitot tubes is $\leq 2.0\%/15\text{ s}$ with the system pressure between -0.75 KPa and -1.12 KPa at initiation of the leak check. The acceptable leak rate for the “stack static” Pitot $\leq 2.0\%/15\text{ s}$ with either a positive or negative system pressure between ± 0.75 and 1.12 KPa at initiation of the leak check.

10.3.4.3 Determine velocity traverse point sampling locations in accordance with Test Method D3154.

10.3.4.4 Perform velocity measurements at each test point for a period of a least one minute.

10.3.5 Select and install the proper inlet based on the velocity traverse test results or historical gas velocity information. Install the appropriate nozzle.

10.3.6 Leak check sample and purge supply pneumatic lines.

10.3.6.1 Perform leak check on pneumatic lines (sample and purge supply).

10.3.6.2 The acceptable leak rate is less than 1.5 KPa/60 s with the system pressure equal to or below -40 KPa at initiation of the leak check.

10.3.7 Configure the system heater setpoints.

10.3.7.1 If the source gas being sampled is “dry” (not saturated with respect to moisture and below the boiling point of water), set the system heater set points 5°C or greater above the expected source gas temperature.

10.3.7.2 Alternatively, for “dry” source gas streams, set the heaters for automatic control based on maintaining the set points at the source gas temperature $+5^\circ\text{C}$.

10.3.7.3 If the source gas is “wet” (saturated with respect to moisture and below the boiling point of water), set the system heater set points at 125°C .

10.3.8 If onboard CO_2/O_2 sensors are active, perform a CO_2/O_2 system calibration check.

10.3.8.1 Connect gas cylinders certified in the appropriate concentration ranges (see 7.8.1) to the calibration gas input ports on the Control Unit. Set all calibration gas regulators to deliver calibration gas at a pressure consistent with manufacturer’s specifications.

10.3.8.2 Calibration check errors must be less than 2 % of the sensor range and bias check errors must be less than or equal to $\pm 5\%$ of the sensor range.

10.3.9 Insert probes to the first sample point. Rotate the mass transducer so that the inlet nozzle points directly into the gas stream.

10.3.10 Verify that the flow is isokinetic.

10.3.11 Run test.

10.3.11.1 Start the test procedure.

10.3.11.2 Verify that the mass transducer cap and case temperatures are being maintained to within $\pm 0.2^\circ\text{C}$ of the temperature set point, tare the total mass reading to zero.

10.3.11.3 Monitor total mass until it has stabilized to a constant value, then re-zero the total mass reading.

10.3.11.4 Begin sampling at the first traverse point. Before resuming sampling on each subsequent traverse line, verify that the mass transducer cap and case temperatures are being maintained to within $\pm 0.2^\circ\text{C}$ of their set points before resuming.

10.3.11.5 When all traverses have been completed, stop sampling and begin the post sampling filter desiccation process. The filter desiccation process is deemed complete when the total mass (TM) reading is stable (less than $\pm 2\ \mu\text{g}$ change in TM averaged over a 180 s period).

10.3.11.6 *Discussion*—The determination of stable mass when desiccating the filter should be assessed as a function of source particulate matter mass concentration. For source concentrations less than $1\ \text{mg}/\text{m}^3$, initial and final stabilization criteria should be based on the anticipated mass concentration. At very low concentrations, the measurement uncertainty decreases as the total mass drift rate decreases.

10.3.12 If required, capture any particulate matter that has collected on the inside walls of the inlet nozzle by performing the following inlet recovery procedure. (See *Discussion* in ~~10.3.11.6~~ in 10.3.13.)

10.3.12.1 Remove the probe from the sample port and allow the mass transducer temperature to restabilize. The total mass reading must be stable (less than $\pm 2\ \mu\text{g}$ change in TM averaged over a 180 s period) before the inlet recovery is performed.

10.3.12.2 When the temperature is stable, briefly turn off the purge air supply (being used to desiccate the filter and keep stack gas from entering the nozzle).

10.3.12.3 With the purge air turned off (and the sample vacuum still on), use a clean, properly sized, soft bristle brush to gently dislodge any particles that are trapped on the inner walls of the inlet nozzle. Carefully insert the brush in the inlet. Gently push the brush into the inlet taking care not to contact the filter with the brush. Remove the brush and repeat the brushdown two more times. As the particles are loosened, the sample vacuum draws the dislodged particles onto the sample filter where its mass can be determined and combined with the mass reading obtained during sampling.

10.3.12.4 Allow the total mass to stabilize (less than $\pm 2\ \mu\text{g}$ change in TM averaged over a 30 s period) before ending the inlet recovery sequence.

10.3.13 *Discussion*—The amount of mass retained in the inlet during sampling may or may not be significant depending on the physical/chemical characteristics of the particulate matter and the concentration of particulate matter in the source being tested. Generally, it has been found that as the concentration of particulate matter decreases, the relative importance of the inlet catch on the total mass captured during the test increases. However, because particulate matter characteristics and concentrations can vary considerably during a test, it is difficult to determine a precise concentration at which the inlet recovery procedure must be used to obtain the precision and accuracy stated in this test method. The precision and bias assessment reported in Section 14 included inlet recovery.

10.3.14 Conduct post-test leak checks

10.3.14.1 Perform a leak check on the sample and purge pneumatic lines.

10.3.14.2 Perform a leak check on the Pitot tube pneumatic lines.

10.3.14.3 If any leak check does not pass (less than 0.2 KPa/s at a starting pressure of 140 KPa or above), troubleshoot to determine and correct the cause of the leak before proceeding with any additional test runs.

10.3.15 Conduct a flow audit if required by the test protocol.

10.3.16 Conduct a gas sensor post-test bias check.

10.3.16.1 Before starting gas sensor bias checks, verify that the calibration gas regulators are set to deliver 68.9 kPa [~~10 psi~~].

10.3.16.2 The acceptance criterion for a gas sensor drift check is $\pm 3\%$ of sensor full scale.

11. Data Records

11.1 Raw Data Files

11.1.1 Store all sample collection and analysis data used for the determination of the measured mass concentration in ASCII, tab-delimited, electronic data files that can be processed by the system's associated data reporting software or can be read into standard spreadsheet software for manual processing or review.

11.1.2 Generated one data file for each test. Record data at any interval in multiples of the system clock interval (approximately three seconds). Include in each data record: the date, time, system operating status, raw frequency, sample flow percent of isokinetic flowrate, mass transducer case temperature, total mass, mass concentration, sample volume, flue gas temperature and flue gas velocity.

11.1.3 Store additional, user-defined, data parameters in each record for quality control or other purposes.

11.1.4 Retain raw data files in the control unit's hard drive. Transfer data files to a laptop for subsequent processing, if desired.

11.2 *Processed Data Files*—Process copies of the raw data files to compile a test report.

11.2.1 Test reports should include information pertaining to the source owner, test provider, site and sample location

information, system identification and quality control information and test data results in tabular and graphical formats.

11.2.2 Do not modify or change the raw data file during data processing and report generation.

12. Calculation

A_N	= cross-sectional area of the inlet nozzle, cm^2
f	= oscillating frequency of the hollow tube oscillator, Hz
BWS	= stack gas moisture content
CO_2	= concentration of CO_2 in stack gas, %, dry
C_P	= Pitot tube coefficient, dimensionless
C_{PM}	= concentration of particulate matter in stack gas, mg/m_3 , dry, standard conditions
I	= variation from Isokinetic
$\%I$	= percent variation from isokinetic
K_0	= calibration constant of the hollow tube oscillator
K_P	= Pitot tube constant, $128.9 \text{ m/s} \left[\frac{(\text{g/g-mol})/\text{K}}{((\text{g/g-mol})/\text{K})} \right]^{1/2}$
M_D	= dry molecular weight of stack gas, $\text{g-g}/\text{mol}$
M_S	= molecular weight of sample gas, $\text{g-g}/\text{mol}$
M_1	= slope of calibration curve for flow sensor 1
M_2	= slope of calibration curve for flow sensor 2
M_3	= slope of calibration curve for flow sensor 2 referenced to flow sensor 1
O_2	= concentration of O_2 in stack gas, %, dry
P_1	= flow sensor 1 inlet pressure, inches H_2O
P_2	= flow sensor 2 inlet pressure, inches H_2O
P_{BAR}	= barometric pressure at sampling location, mm Hg
P_S	= absolute stack gas pressure, kPa
P_{STATIC}	= stack gas static pressure kPa
P_{STD}	= standard ambient atmospheric pressure, kPa
ΔP	= velocity head of stack gas kPa
ΔP_1	= pressure drop across orifice of flow sensor 1, inches H_2O
ΔP_2	= pressure drop across orifice of flow sensor 2, inches H_2O
Q_{1ACT}	= volumetric flow rate of sample gas through flow sensor 1, wet actual conditions, lpm
Q_{2ACT}	= volumetric flow rate of sample gas through flow sensor 2, wet actual conditions, lpm
Q_{1STP}	= volumetric flow rate of sample gas through flow sensor 1, lpm, dry, corrected to standard conditions
Q_{2STP}	= volumetric flow rate of sample gas through flow sensor 2, lpm, dry, corrected to standard conditions
Q_{WV}	= volumetric flow rate of moisture in sample gas at standard conditions, lpm
TM	= mass of particulate matter collected on filter, mg, dry
T_1	= temperature of flow sensor 1, $^{\circ}\text{C}$
T_2	= temperature of flow sensor 2, $^{\circ}\text{C}$
T_S	= stack temperature, K
T_{STD}	= standard absolute temperature, 298.15K
V_S	= stack gas velocity, m/s
Y_1	= y intercept of calibration curve for flow sensor 1
Y_2	= y intercept of calibration curve for flow sensor 2
Y_3	= y intercept of calibration curve for flow sensor 2 referenced to flow sensor 1

12.1 Total mass—Determination of the mass from measured frequency is as follows:

$$f^2 = K_0 / M \quad (2)$$

where:

f = frequency

K_0 = calibration constant for the hollow tube oscillator, and

M = mass of the filter cartridge and any accumulated particulate matter

As sampled particulate matter begins to accumulate on the filter element, the change in mass (Δm) of the filter over a given time period can be calculated by measuring the frequencies f_1 and f_2 where f_1 is the frequency at time a, and f_2 is the frequency at time b.

$$\Delta m = K_0 \left(\frac{1}{f_2^2} - \frac{1}{f_1^2} \right) \quad (3)$$

The measured mass corresponding to each frequency sample is combined with the monitored sample gas volumetric flow rate data for the same sample period to calculate the mass concentration. The calibration of the hollow tube oscillator can be established as follows. A known mass, m_1 is determined gravimetrically and placed on the filter. K_0 can be determined by measuring frequency