



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 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)

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)

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.

2.3 *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 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 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 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.
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 frequency is converted to a 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.

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

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

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.

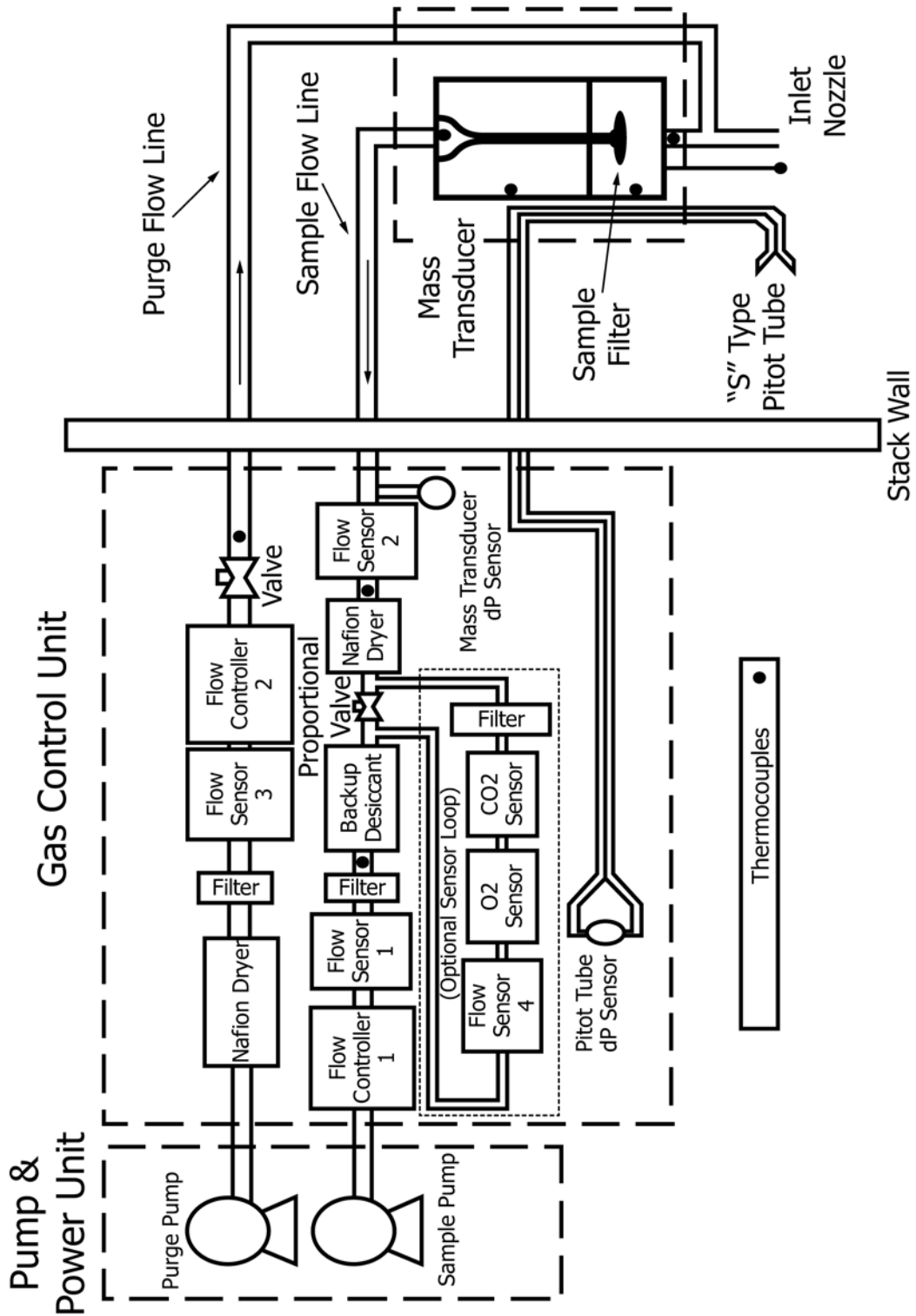


FIG. 1 Block Diagram of In-stack, Inertial Microbalance System

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 <math><30^\circ</math>, 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) 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 $\pm 1 \mu\text{g}$. 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 (K_0) is determined using a Mass Verification Filter. The calibration constant is deemed acceptable when the deviation from the mean of the individual K_0 measured for each filter is less than or equal to $\pm 1.5 \%$.

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 $\pm 0.025 \text{ mm}$ (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 $\pm 1 \%$.

7.3.3 Maximum allowable flow sensor calibration error is $\pm 2.0 \%$ 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 Q_1 and Q_2 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 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 $\pm 2.5 \text{ mm Hg}$ (0.1 in. Hg). The pressure sensor is accurate to within $\pm 2.5 \text{ mm Hg}$ (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.

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*: 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 ± 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 10.3.13.)

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 ± 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 m/s ((g/g-mol)/K) ^{1/2}
M_D	= dry molecular weight of stack gas, g-g/mol
M_S	= molecular weight of sample gas, g-g/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 ₂ O
P_2	= flow sensor 2 inlet pressure, inches H ₂ O
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 ₂ O
ΔP_2	= pressure drop across orifice of flow sensor 2, inches H ₂ O
Q_{IACT}	= 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, °C
T_2	= temperature of flow sensor 2, °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 f_1 and f_2 , where f_1 is the frequency measured without mass m , and f_2 is the frequency measured with mass m loaded onto the filter and oscillator. Using the relationship described by Eq 2, for each frequency measurement:

$$f_1^2 = K_0/m_1 \quad (4)$$

and

$$f_2^2 = K_0/m_2 \quad (5)$$

From these two equations, K_0 can be calculated for the particular hollow tube oscillator:

$$K_0 = (m_2 - m_1) \left(\frac{1}{f_2^2} - \frac{1}{f_1^2} \right) \quad (6)$$

The hollow tube oscillator system is constructed using a non-fatiguing inert material. Since the value of K_0 is a function of the (non-changing) physical characteristics of the hollow tube oscillator system, the hollow tube oscillator retains its calibration indefinitely. The starting frequency, f_1 , can be defined at any arbitrary time. Consequently the mass measurement does not depend on the knowledge of the previous filter loading. Furthermore, tracking frequency with time can yield the mass rate ($\mu\text{g}/\text{min}$).

12.2 *Dry Gas Volume*—The sample gas volume corrected to standard conditions is calculated using Eq 7.

$$V_{std} = V_a \left(\frac{T_{std}}{T_m} \right) \left(\frac{P_{bar}}{P_{std}} \right) \quad (7)$$

12.3 *Dry Molecular Weight*—The dry molecular weight of the sample gas is calculated using Eq 8.

$$M_D = 0.320 (\%O_2) + 0.440 (\%CO_2) + 0.280 (\%N_2) \quad (8)$$

where:

%CO₂ = percent carbon dioxide by volume, dry basis

- $\%O_2$ = percent oxygen by volume, dry basis
 N_2 = percent carbon dioxide by volume, dry basis
 0.440 = molecular weight of carbon dioxide, divided by 100
 0.320 = molecular weight of oxygen, divided by 100
 0.280 = molecular weight of nitrogen or carbon dioxide, divided by 100

12.4 *Molecular Weight of Stack Gas*—The molecular weight of the stack gas is calculated using Eq 9.

$$M_S = [(1 - BWS) * M_D] + 18 * BWS \quad (9)$$

12.5 *Source Gas Velocity*—Eq 10 is used for calculating the source gas velocity.

$$(V_s) = K_p C_p \left(\sqrt{\Delta P_{avg}} \right) \sqrt{\frac{(T_s)}{P_s M_s}} \quad (10)$$

where:

$$K_p = 129 \text{ m/s (g/g-mol-K)}^{1/2}$$

12.6 *Moisture Content*—Eq 11-13 are used for calculating moisture content of the source gas.

$$Q_{1STP} = Q_{1ACT} * \frac{T_{STD}}{(T_1 + 273.15)} * \frac{\frac{P_{BAR}}{51.75} + P_1}{P_{STD}} \quad (11)$$

$$Q_{2STP} = Q_{2ACT} * \frac{T_{STD}}{(T_1 + 273.15)} * \frac{\frac{P_{BAR}}{51.75} + P_2}{P_{STD}} \quad (12)$$

$$BWS = \frac{Q_{WV}}{Q_{WV} + Q_{2STP}} \quad (13)$$

12.7 *Mass Concentration of Particulate Matter*—Eq 14 is used to calculate the mass concentration of particulate matter in the source gas.

$$C_{PM} = \frac{TM}{V_{std}} \quad (14)$$

12.8 *Variation From Isokinetic*:

$$I = \left(\frac{Q_{MainSP} - Q_{STP}}{Q_{MainSP}} \right) * 100 \quad (15)$$

12.9 *Percent Variation from Isokinetic*:

$$\%I = 100 \% - I \quad (16)$$

13. Quality Control Procedures

13.1 Flow sensor calibration and audit—Calibrations of the sample flow sensors are performed by the factory prior to the initial shipment or whenever a flow sensor fails a flow audit.

13.1.1 Flow calibrations are performed in accordance with 7.3.

13.1.2 Flow audits are performed, at minimum, following each test either at the field site or in the laboratory.

13.1.3 Flow audits are performed using primary flow standards or NIST-traceable transfer standards. Flow audit devices must be calibrated to an error ± 2.0 % of the measuring range of the audit device.

13.2 Gas temperature sensor calibration and audit - Initial and periodic gas temperature sensor calibrations and audits are performed against a mercury-in-glass or equivalent thermometer or NIST traceable temperature transfer standard.

13.2.1 Sensor calibrations and audits shall be performed at two different temperatures to check the accuracy and linearity of the gas temperature sensor.

13.2.2 The calibration tolerance for acceptable performance is $\pm 4^\circ\text{C}$.

13.3 Gas flow rate sensor (Pitot tube) calibration and audit - Initial and periodic Pitot tube calibrations shall be performed against a calibrated standard Pitot tube under dynamic conditions in a wind tunnel and in accordance with 7.5. Field verification (audit) of the Pitot tube assembly is made by checking the geometry of the Pitot tubes' openings and orientation with precision calipers.

13.3.1 The dimensions of the Pitot tube assembly shall conform to dimensional specifications supplied by the manufacturer. Repair or return any assembly that does not conform to the manufacturer supplied specifications.

13.4 Inlet Nozzle Dimensional Audits

13.4.1 Prior to each use, the sample inlet nozzle should be visually inspected for damage to the nozzle tip such as dents, nicks, or corrosion. If damage is suspected, either reject the nozzle, reshape or return to the supplier. Measurement of the nozzle's inside diameter shall be made using either precision measuring pins or a micrometer. The specification for the inside diameter is ≤ 0.125 mm.

13.5 *CO₂ and O₂ Gas Sensor Calibration and Audit*.

13.5.1 This section is only applicable if isokinetic sampling conditions are determined using the monitor's onboard molecular weight measurement system.

13.5.2 Sensor calibrations may be performed in the laboratory prior to mobilization of the instrumentation for use at a field test site or may be performed at the test site immediately prior to running a test. In general, the calibration, audit and bias checks, follow U.S. EPA Method 3A (40CFR60, Appendix A).

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

13.5.4 The cylinder gas mixtures should be Protocol gases and have the following concentrations: Zero: Nitrogen - 100 %. Residual CO₂ and O₂ must be less than 0.25 % of the CO₂ and O₂ sensor span. High: CO₂ and O₂ in N₂ balance - 80 to 100 % of CO₂ sensor span, 80 to 100 % of O₂ sensor span, remainder N₂. Mid: CO₂ and O₂ in N₂ balance - 40 to 60 % of CO₂ sensor span, 40 to 60 % of O₂ sensor span, remainder N₂.

13.5.5 Connect the cylinders to the appropriate ports on the Control Unit.

13.5.6 Set each cylinder regulator for delivery pressure of 10 psi.

13.6 Start the calibration check or audit. A successful calibration is achieved when the error at any test point is less than 2 % of the sensor span. If the sensor calibration check or audit does not pass, check to make sure all gas cylinders are set to deliver the proper pressure consistent with manufacturer's specifications, there are no leaks in the system, gas cylinder concentrations are entered correctly into the firmware, and then retest. If results are still outside of specifications, recalibrate the sensor electronic hardware.

13.7 *Hollow tube oscillator K_o Verification*.

13.7.1 Mass verification filters must be stored in a clean, dry container between uses.

13.7.2 Mass verification filters are accurate to within $\pm 1\mu\text{g}$.

13.7.3 Conduct the K_0 audit only after the system is allowed to warm up and stabilize at operating temperature.

13.7.4 If the K_0 audit result is not within $\pm 5\%$, change the mass verification filter and repeat the audit. Replace any mass transducer hollow tube oscillator that doesn't meet the audit specification.

13.7.5 *Discussion*—While the potential for corrosion or other changes in the physical characteristics of a hollow tube oscillator exists in some source gas chemistries, which may change the mass/frequency response of hollow tube oscillators, extended use of the hollow tube oscillator in a wide variety of source gas types has not been found to cause a measured change in the K_0 value.

14. Precision and Bias

14.1 This test method has been subjected to replicate testing in accordance with U.S. EPA Reference Method 301 (40CFR63, Subpart A) using quadruplet sampling trains to compare the in-situ, inertial microbalance method to EPA Test Methods 5 and 17. Additionally, methods comparisons were performed using two probe installation configurations to assess the influence of probe inlet nozzle placement on the compatibility of measurements.

14.1.1 Probe configuration “A” shown in Fig. 2 and Fig. 3 was used to represent the typical installation in which all four sampling trains are inserted into the stack using sample ports at the same vertical elevation. This configuration results in the inertial microbalance probe nozzles being introduced into the

Top and Side View

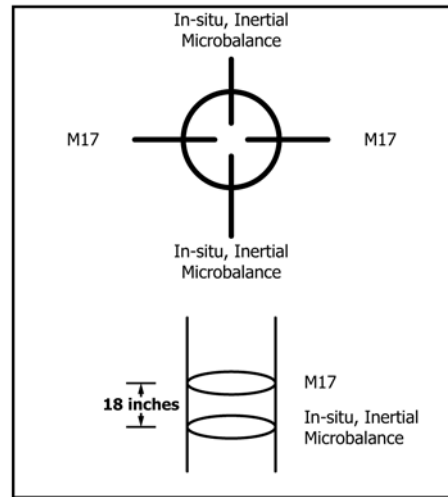


FIG. 3 Method 301 Test Probe Configuration A

**Probe Configuration B
Top View**

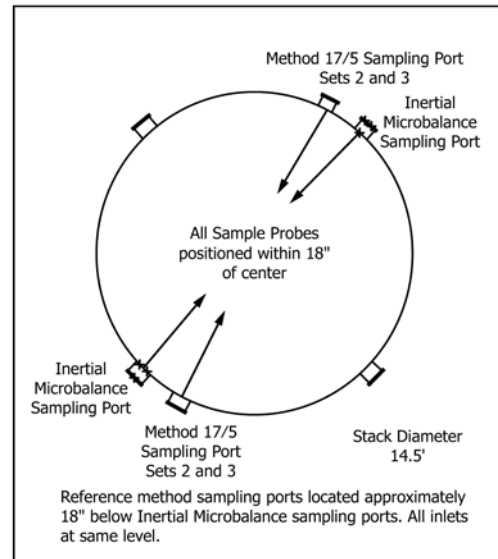


FIG. 4 Method 301 Test Probe Configuration B

Top View

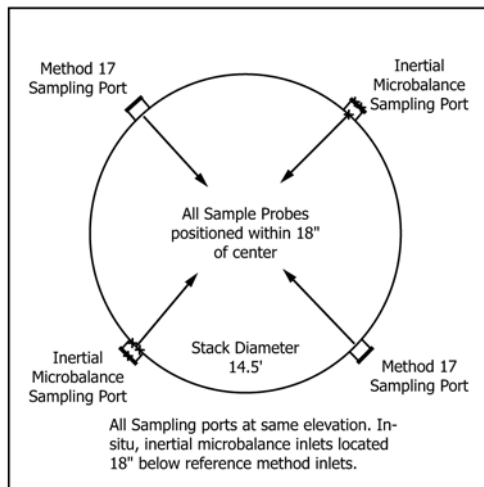


FIG. 2 Method 301 Test Probe Configuration A

gas stream approximately 18 inches upstream of the reference method sample inlets.

14.1.2 Probe configuration “B” allowed the inlet nozzles for the paired reference method sampling trains to be presented into the gas stream at the same vertical elevation as the inertial microbalance sample nozzles. Figures Fig. 4 and Fig. 5 graphically describe this configuration. Results of replicate

**Configuration B
Top and Side View**

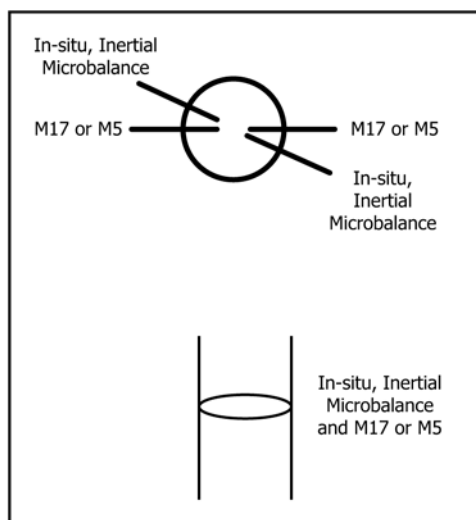


FIG. 5 Method 301 Test Probe Configuration B

testing with comparison to the U.S. EPA Reference Methods 17 and 5 (40CFR60, Appendix A) are shown in the table below.

In-situ, Inertial Microbalance vs. Method 17–Probe Configuration A				
Test No.	Method 17 Results Train 1 (mg/m ³)	Method 17 Results Train 2 (mg/m ³)	Inertial Microbalance Results System 1 (mg/m ³)	Inertial Microbalance Results System 2 (mg/m ³)
1	38.46	36.21	37.23	33.30
2	1.74	1.61	2.25	2.20
3	2.64	3.77	4.18	3.92
4	12.45	14.45	15.14	15.79
Bias	-0.336	EPA Test Method Variance (S _v ²)		1.292
t-statistic	0.389	Inertial Microbalance Method Variance (S _p ²)		1.992
Critical t value	1.638	Experimental F Value		1.542
RSD _p (%)	13.98 %	Critical F Value		3.787

In-situ, Inertial Microbalance vs. Method 17–Probe Configuration B				
Test No.	Method 17 Results Train 1 (mg/m ³)	Method 17 Results Train 2 (mg/m ³)	Inertial Microbalance Results System 1 (mg/m ³)	Inertial Microbalance Results System 2 (mg/m ³)
1	2.98	4.03	3.63	3.61

2	3.93	1.71	2.94	3.24
3	8.76	5.93	6.21	6.20
4	4.95	5.26	6.06	5.90
Bias	-0.029	EPA Test Method Variance (S _v ²)		1.763
t-statistic	0.068	Inertial Microbalance Method Variance (S _p ²)		0.015
Critical t value	1.638	Experimental F Value		0.008
RSD _p (%)	0.31 %	Critical F Value		3.787

In-situ, Inertial Microbalance vs. Method 5–Probe Configuration B				
Test No.	Method 17 Results Train 1 (mg/m ³)	Method 17 Results Train 2 (mg/m ³)	Inertial Microbalance Results System 1 (mg/m ³)	Inertial Microbalance Results System 2 (mg/m ³)
1	5.11	5.78	4.40	4.19
2	1.57	1.43	2.12	1.59
3	1.93	2.12	1.44	1.65
4	1.64	1.34	1.09	1.28
Bias	0.396	EPA Test Method Variance (S _v ²)		0.074
t-statistic	1.280	Inertial Microbalance Method Variance (S _p ²)		0.051
Critical t value	1.638	Experimental F Value		0.682
RSD _p (%)	2.28 %	Critical F Value		3.787

14.2 The precision of this test method is potentially influenced by some of the same factors inherent in the manual test methods for particulate matter, such as Test Methods **D3685/D3685M**, or U.S. EPA Methods 5 and 17 (40CFR60, Appendix A). Adherence to the measurement and calibration procedures described in this method is necessary to reduce the effect of the various factors. However, this instrumental method can standardize the sample collection and analysis process through the use of integrated, standardized, menu-driven software. An onboard computer can perform automatically all data calculations for isokinetic flow control and mass concentration determination. Also, this instrumental method completely eliminates operator/laboratory handling of the sample media during the collection and analysis process. As a result, the influence on the field operator or the laboratory on the result is significantly reduced.

15. Keywords

15.1 hollow tube oscillator; in-situ, inertial microbalance; low concentration particulate matter measurement; mass concentration; particulate matter; real-time source particulate matter measurement

APPENDIXES
(Nonmandatory Information)
X1. GUIDELINES FOR ASSESSING SPATIAL VARIATION IN PARTICULATE MATTER CONCENTRATION

X1.1 Discussion—Assessment of the spatial variation in particulate matter (PM) concentration within the cross section of a stack or duct test location may be useful to determine whether a particular sampling point or limited number of sampling points can be used to acquire representative PM samples. The results may be used to evaluate a particular sampling point for a permanently installed particulate matter continuous monitoring system (PM CEMS) or to reduce the number of sampling points and duration of PM test runs necessary to characterize PM concentrations during short term tests. This can provide advantages for tests conducted to track temporal variations in PM concentrations to assess the effects of changing conditions (including changing fuel, feed, process operation, and air pollution control device operation) or when test data are correlated with PM CEMS data. Well controlled sources with sampling locations in long straight ducts or stacks generally have only small particles and fully developed turbulent flow, which tends toward exhibiting little or no spatial variation (“stratification”) in PM concentration, even though gas velocity and PM emission rates will vary across the duct. Where PM concentration stratification does exist due to flow disturbances and inertial effects on particles, or other causes, it may not be possible to characterize fully the PM stratification or account for factors that would change the PM concentration profile over time. The procedures in this Appendix are intended to be used primarily for demonstrating that a particular sampling point, or region within the cross section, provides a homogeneous PM concentration for PM measurements consistent with the objectives of the measurement program.

X1.2 Approach—The inertial microbalance method is well suited to providing sequential PM concentration measurements at various points on a diameter for circular ducts or straight line for rectangular ducts by repositioning the mass transducer at each measurement point. Additional time and effort are required to move and reinstall the inertial microbalance equipment on another sample port and more time is required for stabilization of the equipment when changing ports. In some cases, measurements along one line may be sufficient for the test purposes. For example, if there is an upstream bend, then a traverse across the expected potential stratification gradient (traverse along a line parallel to plane defined by duct and upstream bend) is the “worst case” investigation. If this traverse reveals no spatial variation in PM concentration, then a second traverse on a perpendicular diameter is not necessary. Similarly, if there is no apparent reason to expect stratification in a particular duct, and none is found on a traverse along one diameter, then it is unlikely that stratification would be present on a second diameter. In cases where measurements from multiple sample ports are necessary, see **X1.6**.

X1.3 Preliminaries—An indication of the PM concentration variability over time is needed to determine the appropriate procedures for assessment of spatial variation. The temporal variability of the PM concentration can be evaluated based on the sequential measurements provided by a PM CEMS, by a continuous opacity monitoring system (COMS), or by preliminary testing using the inertial microbalance.

X1.3.1 Traverse Point Sample Duration—Determine the sampling interval to be used for each traverse point based on PM concentration variability. At many sources, sampling for a period of 5 to 10 minutes is sufficient to acquire an accurate and representative inertial microbalance PM measurement result. However, if the short-term measurement data are “noisy,” or if intermittent spikes are present, a longer averaging period may be needed. If regular periodic spikes occur, such as when an off-line cleaning fabric filter is returned to service, it may be necessary to avoid sampling during these periods, or discard data where the effects are apparent.

X1.3.2 Number and Location of Sample Points—Determine the number of sample points and their location on each traverse line in accordance with EPA Method 1 (40CFR60, Appendix A). However, do not sample in the flow boundary layer. If the application of Method 1 results in sample points closer than 15 cm (6 in.) to the stack wall, either (a) relocate the sample point to 15 cm from the stack wall (but do not sample the same point twice), or (b) increase the number of sample points on the traverse, and exclude the point(s) closest to the wall.

X1.3.2.1 For a traverse across a circular duct diameter, use at least six sample points, representing equal areas of the duct cross section.

X1.3.2.2 For a traverse across a circular duct radius use at least four sample points, representing equal areas of the duct cross section.

X1.3.2.3 For a traverse across a rectangular duct use at least six sample points, representing equal areas of the duct cross section.

X1.3.2.4 Additional points of interest, such as the candidate sampling point for a PM CEMS, may be added to any traverse.

X1.3.3 Temporal Controls—If PM concentrations at the test location are constant over the time required to perform the traverse(s), temporal control measurements and data adjustments are not needed for assessment of the spatial variation. Follow Procedures in **X1.4**. If PM concentrations at the test location are not constant over the time required to perform the traverse(s), an independent monitor or second microbalance measurement system may be needed. Follow procedures in **X1.5**.

X1.4 Traverse Without Temporal Controls—Use this procedure where the PM concentration is, or can be maintained, at

reasonably constant levels for the duration of the traverse. If a temporal change in the PM concentration occurs during the traverse, a non-stratified sampling location may appear to be stratified. This would be a false positive result indicating stratification. However, it is highly unlikely that a temporal change in the PM concentration would offset a coincidental spatial variation, resulting in a stratified location appearing to be non-stratified. Hence, false negative stratification results are unlikely.

X1.4.1 Prepare the inertial microbalance measurement system for use in isokinetic sample mode. Set the mass transducer temperatures to automatically maintain 5°C above effluent temperature. Configure automatic data recording/report generation software to determine sequential PM concentrations for the sampling intervals determined in X1.3.1.

NOTE X1.1—Because the purpose of this test is to determine the relative PM concentration at each sample point, the post-test desiccation of the filter cartridge and nozzle recovery steps are omitted. The determination is based on the “wet PM” (weight of PM collected before desiccation) per unit of dry gas volume sampled. Some systems report this as “Method A” results.

X1.4.2 Insert the inertial microbalance system into the duct and orient the sample nozzle into the sample flow. Position the system at the first sampling point. Perform the stabilization procedure.

X1.4.3 Start the test and sample at each point for the same duration. Determine the PM concentration reported by the report generation software for each point. Alternatively, calculate the PM concentration from objective analysis of the electronic data files using the same procedures for each sample point.

X1.4.4 Determine the average PM concentration for all points sampled on the traverse. Compare the measured PM concentrations for each sample point to the average.

X1.4.5 If the result for each sample point is within ±5 % of the average, or within ±0.5 mg/m³, whichever is least restrictive, the sampling location does not exhibit any spatial variation. For subsequent measurements, sampling at any point on the traverse (more than 15 cm from the duct wall) is expected to provide a representative sample for PM concentration.

X1.4.6 If the result for each sample point is within ±10 % of the average, or within ±1.0 mg/m³, whichever is least restrictive, the sampling location exhibits minimal spatial variations. For subsequent measurements, selection of a sampling point, or set of points, on the traverse (more than 15 cm from the duct wall) with observed PM concentration(s) either (a) greater than or equal to the average, but not more than +5 % above the average, or (b) greater than or equal to the average but not more than +0.5 mg/m³ above the average, whichever is least restrictive, is expected to provide a representative sample for PM concentration.

X1.5 *Traverse With Temporal Controls*—Use this procedure where the PM concentration cannot be assumed to be reasonably constant for the duration of the traverse. A second measurement system must be used to determine relative PM

concentrations at a stationary measurement location during the traverse with the inertial microbalance. This measurement system may be a second inertial microbalance, a PM CEMS or other device with a response that is, or can be corrected to be, linearly proportional to PM concentration.

X1.5.1 The stationary measurement system used to adjust for varying PM concentrations over time is designated “temporal reference” (TR). The TR may acquire measurements proportional to PM concentration at any point, region or path which is judged to have the same bulk stream PM concentration as the cross section where the spatial traverse is performed.

X1.5.2 The inertial microbalance measurement system shall be used to quantify the relative PM concentration at each traverse sample point (S₁, S₂ ... S_N) in accordance with X1.4.

X1.5.3 TR measurement data recorded concurrent with the sampling intervals for each inertial microbalance traverse sample point shall be recorded. For example TR₁, corresponds to S₁, TR₂ corresponds to S₂, and so forth.

X1.5.4 The inertial microbalance measurement results shall be adjusted for temporal variations indicated by TR measurements:

$$SA_n = S_n * \left(\frac{TR_1}{TR_n} \right) \quad (X1.1)$$

where:

SA_n = Adjusted relative PM concentration at each traverse sample point (SA₁, SA₂ ... SA_N)

S_n = Relative PM concentration at each traverse sample point (S₁, S₂ ... S_N)

TR₁ = Temporal reference measurement concurrent with PM concentration measurement at S₁

TR_n = Temporal reference measurement concurrent with at each traverse sample point (S₁, S₂ ... S_N)

X1.5.5 If the adjusted result for each sample point is within ±5 % of the average, or within ±0.5 mg/m³, whichever is least restrictive, the sampling location does not exhibit any spatial variations. For subsequent measurements, sampling at any point on the traverse (more than 15 cm from the duct wall) is expected to provide a representative sample for PM concentration.

X1.5.6 If the adjusted result for each sample point is within ±10 % of the average, or within ±1.0 mg/m³, whichever is least restrictive, the sampling location exhibits minimal spatial variations. For subsequent measurements, selection of a sampling point, or set of points, on the traverse (more than 15 cm from the duct wall) with observed PM concentration(s) either (a) greater than or equal to the average but not more than +5 % above the average, or (b) greater than or equal to the average but not more than +0.5 mg/m³ above the average, whichever is least restrictive, is expected to provide a representative sample for PM concentration.

X1.6 *Traverses in Multiple Sample Ports*—The procedures in X1.4 and X1.5 are used to assess spatial variations between sampling points on a traverse within a single sample port. This section addresses cases where multiple traverses in multiple

sample ports are performed due to large duct diameters, probe length limitations, physical constraints, or other reasons.

X1.6.1 If separate traverses are performed on the radius of a circular duct from opposite sample ports on the same diameter, the absence of spatial variations is demonstrated for the entire diameter if both traverses demonstrate the absence of spatial variations. Direct comparison of the measurement data for the two traverses is not necessary.

X1.6.2 Traverses of rectangular ducts should be performed across any expected PM concentration gradient. If PM measurements at sample points on traverses in multiple ports are required, the procedures in X1.5 must be used, and all traverse data must be corrected for temporal variations. The time required for port changes may adversely affect the ability to demonstrate the absence of spatial variations for the cross-sectional area.

X2. GUIDELINES FOR EXPEDITED PM CEMS CORRELATIONS

X2.1 *Discussion* —The inertial microbalance method is well suited to providing sequential PM concentration measurements. At many sources, sampling for a period of 5 to 10 minutes is sufficient to acquire an accurate and representative inertial microbalance PM measurement result at a single measurement point. (The automatic data recording/report generation software can be configured to determine sequential PM concentrations for such sampling intervals of 5 to 10 minutes, or longer periods.) Where “PM stratification tests” are performed (see for example Appendix X1) and results demonstrate that representative PM concentration measurements can be acquired at a single point, the inertial microbalance system can be used to acquire multiple PM measurement data during a single test run. This can expedite PM CEMS calibrations. Minimizing human interaction between sequential measurements and reducing steps between sample runs may reduce the uncertainty of the measurement process and allow higher correlation coefficients to be achieved for PM CEMS correlations. Also, the availability of PM concentration test results when the test run is completed may be used to determine the need for additional testing before leaving the plant site. Expediting the PM CEMS calibration may reduce operational costs for the source operator and may reduce air pollution emissions to the atmosphere because less plant operating time is required for testing at high PM concentrations to satisfy regulatory requirements such as those found in EPA Performance Specification 11 (40CFR60, Appendix B).

X2.2 *Applicability* —The approach described in this Appendix is applicable to establishing PM CEMS calibrations when: (a) correlations are determined based on co-located (“side-by-side”) inertial microbalance and PM CEMS measurements, and (b) other tests have demonstrated the absence of significant PM spatial variations and single-point inertial microbalance measurements can be compared with PM CEMS measurements; both measurements need not necessarily be at the same point in the duct.

X2.3 *Approach*—The standard inertial microbalance test method includes: (1) preparation steps including time required for pre-test desiccation of the filter cartridge and stabilization of the measurement system, (2) sampling for a period of time, typically 30 to 90 minutes at a series of sample points, (3) post-test desiccation of the filter cartridge and stabilization, (4) nozzle recovery and stabilization and final weighing. The results of these steps are often reported as the “Method B” results and are expressed as “dry particulate mass” per unit of

dry gas sampled. The Method B results have been successfully validated in accordance with EPA Method 301 (40CFR63, Appendix A) relative to EPA Method 5 and Method 17 (40CFR60, Appendix A).³ The inertial microbalance system also provides PM concentration measurements at each sequential point sampled during the test run (typically 6 to 12 points per run), but these results are based on the “wet PM” (weight of PM collected before desiccation) per unit of dry gas volume sampled, because desiccation is only performed after the last sample point. Some systems report this as “Method A” results. In some cases the difference between the average of the Method A result for all data points and the Method B result for the entire test run, as well as the contribution from the nozzle recovery procedure, are sometimes small or insignificant. When this is demonstrated for a particular test run, an adjustment can be applied to the Method A test results, to provide many discrete valid PM concentration measurement results (again, typically 6 to 12) for correlation with concurrent PM CEMS measurements, rather than a single result.

X2.4 Procedure:

X2.4.1 Prepare the inertial microbalance measurement system for use in isokinetic sample mode. Set the mass transducer temperatures to automatically maintain 5°C above the effluent temperature. Configure automatic data recording/report generation software to determine sequential PM concentrations for sampling point intervals, typically 5 to 10 minutes per point.

X2.4.2 Insert the inertial microbalance system into the duct and orient the sample nozzle into the sample flow. Position the system at the selected sampling point. Perform stabilization procedure. Start the test and sample for the same duration at each “sample point.” Do not move the probe during the test.

X2.4.3 Perform the post-test stabilization, filter cartridge desiccation, and nozzle recovery steps in the standard method.

X2.4.4 Determine the “Method B” PM concentration reported by the report generation software for the test run, both with and without nozzle recovery contribution.

X2.4.5 Determine the average of the “Method A” PM concentration results run (that is, the average of A_1, A_2, \dots, A_n) reported by the report generation software for all sample intervals during the test. Alternatively, calculate the PM concentration from objective analysis of the electronic data files using the same procedures for each sample interval.

X2.4.6 *Data Analysis*—All Method A results are validated for test runs where the difference between the average of the

Method A result for all data points and the Method B result for the entire test run excluding nozzle recovery contribution, is less than 5 % of the Method B measurement result, or less than 0.5 mg/m³, whichever is least restrictive.

X2.4.7 If the Method A measurement results meet the validation criteria, apply a uniform correction factor to all Method A results so that the average of the Method A adjusted results is equal to the average of the Method B result including the nozzle recovery contribution:

$$\text{Method A Adjusted} = AA_i = A_i * \frac{\text{Method B with nozzle recovery}}{\text{Method A average}} \quad (\text{X2.1})$$

X2.4.8 Use adjusted Method A adjusted results (that is, AA_i) for direct comparison with concurrent PM CEMS measurement results to construct the correlation. If a particular Method A sample interval includes a non-representative spike or transient or other anomaly, it can be rejected as non-representative or as an outlier. Other Method A results in the same test run are valid.

X2.4.9 When applying this approach it is important to consider the response time of the PM CEMS and to check the timing of measurements to ensure comparison of concurrent measurements because the sample intervals are shorter than used for traditional manual tests.

Particulate - Data Sheet

Plant Name:	Example	Stack
Test Company:	Emission Monitoring Inc.	Control Unit SN: 7000A201200212
Test Operator:	LLK & JWP	Start Time: 9:40:36
Test Date:	Sometime	Stop Time: 11:31:17

Port Description	Point No.	Sampling Duration (s)	Distance from Near Wall (m)	Average Filter Temp(C)	Total Mass (ug)	Volume (m ³)	Flue Gas Velocity (m/s)	Method A Mass Conc. (mg/m ³)	Method A Adjusted Mass Conc. (mg/m ³)
Port 1	Water	90.00		170.00	5.22	0.0000			
Port 1	1	600.00	1.00	170.00	201.73	0.0148	11.67	13.66	14.14
Port 1	2	600.00	1.00	170.00	212.80	0.0148	11.47	14.43	14.94
Port 1	3	600.00	1.00	170.00	181.76	0.0151	11.63	12.05	12.47
Port 1	4	600.00	1.00	170.00	200.24	0.0150	11.60	13.38	13.85
Port 1	5	600.00	1.00	170.00	187.16	0.0150	11.74	12.45	12.89
Port 1	6	600.00	1.00	170.00	216.27	0.0147	11.85	14.75	15.27
Port 1	7	600.00	1.00	170.00	226.91	0.0145	12.02	15.62	16.17
Port 1	8	600.00	1.00	170.00	207.63	0.0147	12.13	14.14	14.63
Port 1	9	600.00	1.00	170.00	191.63	0.0150	12.27	12.75	13.20
Port 1	Desiccation	222.00		170.00	-11.93				
							Averages	13.69	14.17

FIG. X2.1 Example Data Sheet

Method A: Mass Concentration without nozzle recovery: 13.69 mg/m³
 Method B: Mass Concentration without nozzle recovery: 13.89 mg/m³
 Absolute Difference 0.20 mg/m³ Acceptable
 Percentage Difference 1.4% Acceptable

Method B: Mass Concentration with nozzle recovery: 14.17 mg/m³

Adjustment Factor 1.035 (Method B: Mass Concentration with nozzle recovery/Method A: Mass Concentration without nozzle recovery)
 Multiply each Method A result by 1.035 to determine the corresponding Method A Adjusted result

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