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Standard Test Method for Sampling and Determining Particulate Matter in Stack Gases Using an In-Stack, Inertial Microbalance[1]

This standard is issued under the fixed designation D 6831; 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 is 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/m^3 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 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 *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]

D 1356 Terminology Relating to Atmospheric Sampling and Analysis

D 3154 Test Method Average Velocity in a Duct (Pitot Tube Method)

D 3685/D 3685M Test Methods for Sampling and Determination of Particulate Matter in Stack Gases

D 3796 Practice for Calibration of Type S Pitot Tubes

D 6331 Test Method for Determination of Mass Concentration of Particulate Matter from Stationary Sources at Low Concentrations (Manual Gravimetric Method)

3. Terminology

3.1 For definitions of terms used in this test method, refer to Terminology D 1356.

3.2 Definition of terms specific to this standard:

3.2.1 *particulate matter*—for solid particles of any shape, structure, or density 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

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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.

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 D 6331 or D 3685/D 3685M, 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 method 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 ±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^3 to 50 mg/m^3.

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.

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

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