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US Army Contract DAAE30-02-C-1095
FY2004 Tasks
WBS # 2.2.1

Dilution Sampling Protocol: Draft of ASTM Test Method

Technikon # 1411-221

June 2005

Revised for public distribution

EPA United States Environmental Protection Agency
CISA
AFS
UNITED STATES COUNCIL FOR AUTOMOTIVE RESEARCH
USCAR
DAIMLERCHRYSLER Ford Motor Company General Motors

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ENVIRONMENTAL DEVELOPMENT CENTER™

Memorandum

Measurement Technologies

June 20, 2005

To: File cc:

From: C. R. Glowacki //Original Signed//

Subject: US Army Contract DAAE30-02-C-1095, FY2004, WBS # 2.2.1

Technikon's Measurement Technologies staff has continued to participate as part of an American Society for Testing and Materials (ASTM) work group under its sub committee D22.03. This subcommittee is developing a performance based method for the determination of particulate matter from stationary sources (stacks). The method will be specific for particulate matter having an apparent aerodynamic diameter of 10 micrometers (µm), 2.5µm, and for particulate formed by the condensation of vapors. Technikon staff has also participated in a second ASTM work group that is developing a Standard Practice for the determination of particulate matter from ambient stationary sources. This work group is in its infancy and just beginning to organize.

Current and recently promulgated, but not yet implemented, regulations designed to reduce particulate emissions from stationary sources are requiring manufacturing facilities, including metal casting operations, to determine their amount of PM10 and PM2.5 emissions. PM10 includes all particulate material with an apparent aerodynamic diameter of 10µm and smaller. PM2.5 includes all particulate material with an apparent aerodynamic diameter of 2.5 µm and smaller. Current methods for the determination of this material, especially the condensable particulate matter, significantly overestimate a facility's emissions resulting in apparent operating permit violations and possible plant closures.

Since the US EPA is required by executive order to adopt consensus body standards, if applicable, rather than develop their own, Technikon staff has continued to co-chair an ASTM subcommittee to write a method for measuring particulate matter that will provide accurate and precise measurements in a variety of industries. The committee is comprised of representatives from manufacturing, utilities, academia, research consortia, and state and federal environmental agency personnel. During FY2004 Tasks there have been three (3) all day meetings and six (6) multiple hour conference calls. On May 31, 2005 a draft method was submitted to ASTM for an acceptance vote by subcommittee D22.03. Results of this vote are expected by the end of July 2005. A copy of the submitted current draft is attached.

Participation of these two work groups will continue as work products in Subtask 2.2 under FY2005 Tasks when awarded. The Standard Method work group has committed to have a completed method to present to the full ASTM D22 committee for vote at its October 2005 meeting. The Standard Practice work group has the very ambitious goal of submitting a draft for subcommittee vote by October 2005.

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Dilution Test Method for Determining PM2.5 and PM10 Mass in Stack Gases[11]

1. Scope

1.1 This test method describes the sampling and conditioning of stack gas samples from stationary sources by dilution sampling that approximately simulates conditions experienced by stack emissions as they mix with the atmosphere. This method measures mass concentrations of particles smaller than 2.5 micrometers aerodynamic diameter (PM2.5). Optionally, the method also can be used to measure mass concentrations of particles with aerodynamic diameter smaller than 10 micrometers aerodynamic diameter (PM10) or the mass concentrations of particles with aerodynamic diameters between 2.5 and 10 micrometers. This method is not applicable for the determination of ultra fine particulate with an aerodynamic diameter of less than 0.1 micrometers. This method has been applied to stationary sources with low moisture and saturated moisture stack gases.

1.2 The in-stack minimum detection limit for PM2.5 or PM10 mass is dependent on the sample gas volume, dilution ratio (dilution air flow rate/stack gas flow rate), measurement background, and analytical method minimum detection limit. Ignoring measurement background, an in-stack minimum detection limit (three times standard deviation) of approximately 0.006 mg per dry standard cubic meter (dscm) has been achieved with six-hour tests on a natural gas-fired power plant with a 47 mm PTFE membrane filter and 27 dscm sample volume and a dilution ratio of approximately 20:1 (20 degrees Celsius standard temperature, 760 millimeters of mercury standard pressure). Including estimated measurement background in the dilution air, an in-stack minimum detection limit of 0.06 mg/dscm has been achieved under the same conditions. Estimated minimum detection limits for 1hour tests are 0.04 and 0.4 mg/dscm excluding and including dilution air background, respectively. See Section 16.3 for additional details.

1.3 A known limitation of this method concerns the presence of particles in the dilution air at very low concentrations, contributing to measurement background. This may be significant for certain very clean sources such as gas-fired power plants. Dilution air system blanks shall be obtained and reported with test results when sampling sources with anticipated PM2.5 or PM10 mass concentrations approximately equal to or less than 1.0 mg/dscm.

1.4 *This standard does not purport to address all of the safety problems associated with its use. It is the responsibility of the user of this standard to establish appropriate safety and health practices and to determine the applicability of regulatory limitations prior to use. For more specific safety precautionary information, see Section 19.*

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[1] This test method is under the jurisdiction of ASTM Committee D22 and is the direct responsibility of Subcommittee D22.03

Current edition approved XXX. XX, XXXX. Published XX XXXX

2. Referenced Documents

2.1 *ASTM Standards:*

D 1193 Specification for Reagent Water

D1356-00a Terminology Relating to Sampling and Analysis of Atmospheres

D 2986 Practice for Evaluation of Air Assay Media by the Monodisperse DOP (Dioctyl Phthalate) Smoke Test

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

D 3631 Test Methods for Measuring Surface Atmospheric Pressure

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

D 3670 Guide for Determination of Precision and Bias of Methods of Committee

D-22.04 D 3796 Practice for Calibration of Type S Pitot Tubes

D 6331-98 Test Method for Determination of Mass Concentration of Particulate Matter from Stationary Sources at Low Concentrations (Manual Gravimetric Method) 1

D 6831-02 Standard Test Method for Sampling and Determining Particulate Matter in Stack Gases Using an In-Stack, Inertial Microbalance

D 7036-04 Standard Practice for Competence of Air Emission Testing Bodies

E 1 Specification for ASTM Thermometers

1 Annual Book of ASTM Standards, Vol 11.03

2 Annual Book of ASTM Standards, Vol 14.03

2.2 *International Organization for Standardization (ISO) Standards*:

ISO 5725 Precision of test methods — Determination of repeatability and reproducibility by inter-laboratory tests

ISO 8178-1 Reciprocating internal combustion engines – Stack emission measurement – Part 1: Test-bed measurement of gaseous and particulate stack emissions.

ISO 9096 Stationary source emissions — Determination of concentration and mass flow rate of particulate material in gas-carrying ducts. Manual gravimetric method

ISO 10780 Stationary source emissions — Measurement of velocity and volume flow rate of gas stream in ducts

2.3 *Non-ASTM*

N1. "Particulate Matter (PM2.5) Speciation Document (Third Draft)" USEPA Monitoring and Quality Assurance Group. January 21, 1999.

N2. "EPA Method 19 – Determination of Sulfur Dioxide Removal Efficiency and Particulate Matter, Sulfur Dioxide, and Nitrogen Oxides Emission Rates" 40 CFR, Part 60, Appendix A.

N3. "Compendium of Methods for the Determination of Inorganic Compounds in Ambient Air" US EPA Office of Research and Development, EPA/625/R-96/010a, June 1999.

N4. "Compendium of Methods for the Determination of Toxic Organic Compounds in Ambient Air" US EPA Office of Research and Development, EPA/625/R-96/010b, January 1999.

N5 "Aerosol Technology: Properties, Behavior, and Measurement of Airborne Particles (Second Edition). Hinds, W. C., John Wiley & Sons (1999).

N6 "EPA Method CTM-040 – Method for the Determination of PM10 and PM2.5 Emissions (Constant Sampling Rate Procedure)," December 3, 2002.

N7 "DOE Standard – Specification for HEPA Filters Used by DOE Contractors." DOE_STD-3020-97, January 1997.

N8 "Reagent Chemicals, American Chemical Society Specifications," American Chemical Society, Washington, DC. For suggestions on the testing of reagents not listed by the American Chemical Society, see Analytical Standards for Laboratory Chemicals, BDH Ltd., Poole, Dorset, U.K., and the United States Pharmacopeia and National Formulary, U.S. Pharmacopeial Convention, Inc. (USPC), Rockville, MD.

N9 "Source Evaluation Society Safety Guidelines Book (First Edition). December 20, 2001.

N10 "EPA Method 2 – Determination of Stack Gas Velocity and Volumetric Flow Rate (Type S Pitot)" 40 CFR, Part 60, Appendix A.

N11 "EPA Method 1 – Sample and Velocity Traverses for Stationary Sources," 40 CFR, Part 60, Appendix A.

N12 "Measurement of Ultrafine Particle Size Distributions from Coal-, Oil-, and Gas-Fired Stationary Combustion Sources," Chang, M.C, Chow, J.C., Watson, J.G., Hopke, P.K, Yi, S., England, G. C. J. Air and Waste Manage. Assoc. 2004, 54:1494-1505.

N13 Chan, T and M. Lippmann. "Particle Collection Efficiencies of Air Sampling Cyclones: An Empirical Theory," Environ. Sci. Technol. 1977, 4:377-382.

N14 “Method 5 - Determination of Particulate Matter Emissions from Stationary Sources,” Appendix A, 40 CFR 60, U.S. EPA.

N15 McDow, S.R.; Huntzicker, J.J. "Vapor adsorption artifact in the sampling of organic aerosol: face velocity effects," Atmos. Environ. 1990, 24A:2563-2571.

N16 “§ 86.1310–2007 Stack gas sampling and analytical system for gaseous emissions from heavy-duty diesel-fueled engines and particulate emissions from all engines” 40 CFR 86 Subpart N Revisions

N17 Hildemann, L.M., G.R. Cass, and G.R. Markowski. 1989. A dilution stack sampler for organic aerosol emissions: Design, characterization, and field tests. Aerosol Sci. Technol. 10:193-204.

N18 England, G.C. 2004. “Development of Fine Particulate Emission Factors and Speciation Profiles for Oil and Gas-fired Combustion Systems,” Final Report, prepared for: U.S. Department of Energy, National Energy Technology Laboratory, Pittsburgh, PA; Gas Research Institute, Des Plaines, Illinois; and American Petroleum Institute, Washington, D.C. www.nyserda.org/programs/Environment/emepreports.asp accessed January, 2005.

N19 EPA Method CTM-039 - Measurement of PM2.5 and PM10 Emissions by Dilution Sampling (Constant Sampling Rate Procedures)

N20 “PM2.5 Guidance Document (Quality Assurance Guidance Document 2.12, Monitoring PM2.5 in Ambient Air Using Designated Reference or Class I Equivalent Methods,” USEPA Office of Research and Development, November 1998.

3. Terminology

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

3.2 *Definitions of Terms Specific to This Standard*

3.2.1 *ambient air sample*—QA sample collected by drawing unfiltered ambient air at the stationary source sampling location through the sampling media.

3.2.2 *diluted stack gas*—the combined stack gas sample and the dilution air, after mixing.

3.2.3 *dilution air*—ambient air that is filtered to remove solid particulate and organic compounds before mixing with a stack gas sample in a dilution sampling system.

3.2.4 *dilution ratio (DR)*—ratio of the dilution air flow rate through a dilution system to the undiluted stack gas sample flow rate through the system.

3.2.4.1 *dilution factor (DF)*—1+dilution ratio; the product of dilution factor and filter sample PM concentration is equal to the in-stack PM concentration

3.2.5 *dilution sampling system*—sampling equipment that dilutes stationary source stack gases with filtered ambient air to simulate the mixing and cooling processes in an stack plume.

3.2.6 *stack gas sample*—undiluted gas extracted from the duct or stack of a stationary source.

3.2.7 *field blank*—QA sample collected by assembling the dilution sampling equipment at the stationary source sampling location, performing a leak check, and then disassembling the equipment. No stack gas sample passes through the equipment.

3.2.8 *F-factor*—the ratio of combustion gas volume to heat input determined from fuel composition; determined by EPA Method 19 procedures.

3.2.9 *filtration temperature*—the temperature of the diluted sample gas immediately downstream of the filter. (The filter temp measurement device location shall be within 2.5 cm of the filter (N16)

3.2.10 *HEPA filter*—High Efficiency Particulate Arrest Filter

3.2.11 *PM2.5*—particulate matter with an aerodynamic diameter equal to and less than 2.5 micrometers and captured on a PTFE filter maintained at a temperature of not greater than 52 ºC during sampling (N16) and weighed at temperature of 23 ºC ± 2 ºC and a relative humidity of 35 ±5%.

3.2.12 *PM10*— particulate matter with an aerodynamic diameter equal to and less than 10 micrometers and captured on a PTFE filter maintained at a temperature of not greater than 52 ºC during sampling (N16) and weighed at temperature of 23 ºC ± 2 ºC and a relative humidity of 35 ±5%.

3.2.13 *ultrafine particulate matter*—Particulate matter with an aerodynamic diameter less than or equal to 0.1 micrometers.

3.2.14 *PM*—Particulate matter including PM2.5, PM10, and/or total suspended particulate matter.

3.2.15 *sampling point*—the specific position in a source vent at which a sample is extracted.

3.2.16 *dilution system blank*—QA sample collected by assembling the dilution sampling equipment at the stationary source sampling location and then drawing only dilution air through the dilution system and sampling media.

3.2.17 *weighing control procedures*—quality control procedures utilized for detecting/correcting apparent mass variations due to climatic or environmental changes between pre- and post-sampling weighing series.

3.2.18 *VOC*—volatile organic compounds, defined as organic compounds present with a vapor pressure below their saturated vapor pressure at ambient air pressure and temperature.

3.2.19 *Filter holder assembly*—filter holder or cassette that contains the filter media for collecting particulate samples.

3.2.20 *PTFE* – polytetrafluoroethylene

3.2.21 *Laboratory Blanks*—Laboratory blanks are conditioned, unsampled filters used to determine any weight change between pre- and postsampling weighings due to contamination in the microbalance environment.

3.2.22 *Standard temperature* (T_{std})— Absolute temperature at standard conditions, 298 K (25°C) (537°R).

3.2.23 *Standard pressure* (P_{std})— Absolute pressure at standard conditions, 101.3 kPa (29.9 in. Hg).

4. Summary of Test Method

4.1 A conceptual schematic of a dilution sampling system is shown in Figure 1. Other arrangements of components of the sampling system are acceptable as long as the Method criteria are met.

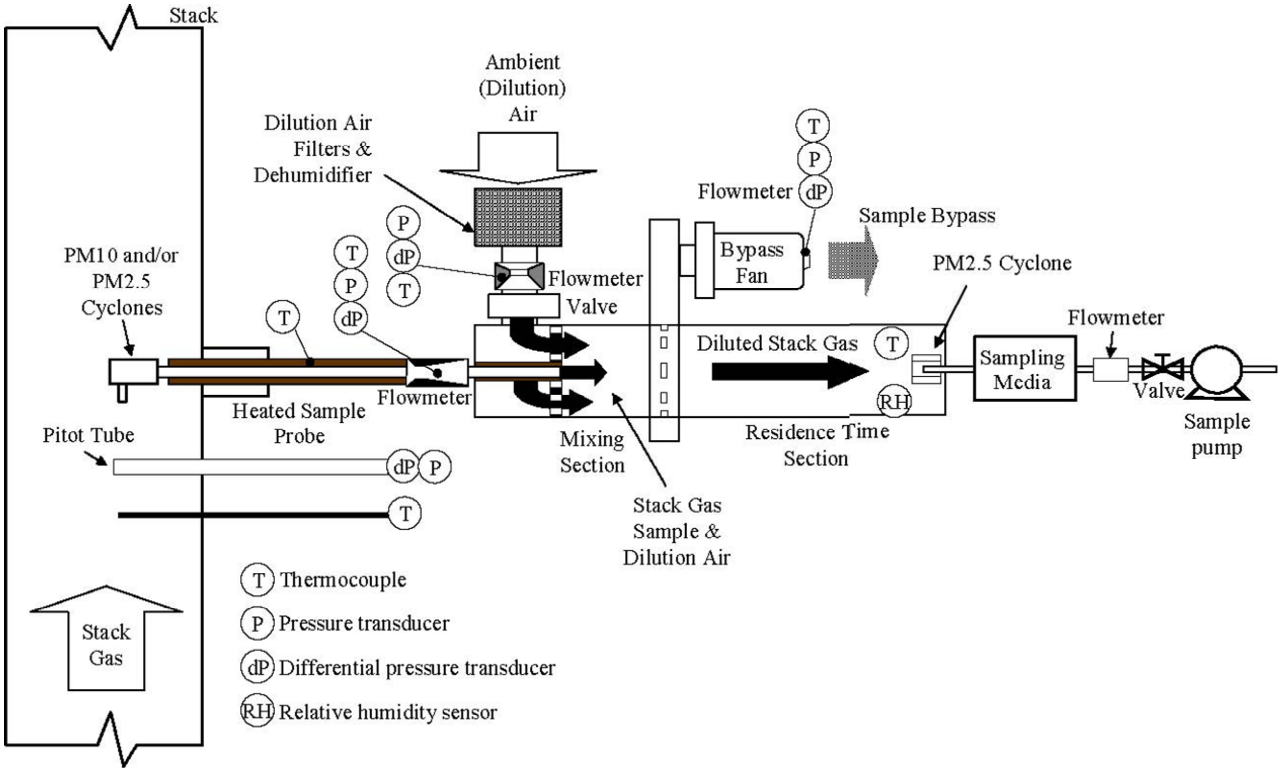

Figure 1. Dilution sampling system schematic.

4.2 A sample of stack gas from a stationary stack is drawn through a primary in-stack PM2.5 cyclone (and/or optionally an in-stack PM10 cyclone for simultaneous determination of total suspended particles, PM10, and PM2.5[GCE1]) and a heated probe to a dilution system where the sample gas is rapidly mixed with dilution air. When PM10 is being determined, the sample should be withdrawn from the stack gas isokinetically (N6). Isokinetic sampling is not required for PM2.5 sampling. The dilution ratio should be at least 20:1 unless higher dilution ratios are required to maintain the particle filter temperature at 52 ºC (N16) or lower. The lowest dilution ratio commensurate with achieving the filter temperature limitation should be used under these circumstances. When the stack gas temperature is very high, it may be necessary to cool the dilution air and/or filter holder assembly to achieve the filter temperature limitation. The dilution system approximately simulates the dilution and cooling processes that occur in the near field of an stack plume as it mixes with the atmosphere. These processes impact the homogeneous and heterogeneous nucleation of stack gas constituents. The diluted sample relative humidity should not exceed 70% because higher levels can significantly affect particle mass and size distribution within the dilution sampling system and thereby change the nucleation, condensation, and accumulation processes. A minimum post-mixing residence time of at least 10 seconds was shown to be necessary for gas-fired sources (N12). Shorter residence times may be acceptable for other source types. The diluted and aged gas is pulled through one or more secondary PM2.5 cyclones or PM10 cyclones if PM10 is being measured, to one or more sampling trains. The purpose of the secondary cyclone is to eliminate any particles that grow larger than 2.5 or 10 micrometers, respectively, during the dilution and aging processes such that the particles collected on the filter are more representative of particles in the stack gas upon cooling and dilution immediately after discharge from the stack. Particulate matter is collected on a single stage PTFE membrane filter or equivalent and subsequently measured gravimetrically or by inertial microbalance (D.6831-02). Stack gas flow rates required for determination of mass emissions are measured by a Pitot tube traverse of the stack, fuel flow rates and F-factors, or equivalent method.

5. Significance and Use

5.1 This test method is intended for PM2.5 and/or PM10 mass measurement. Test data collected using this method are applicable for developing PM2.5 or PM10 emission inventories used in State Implementation Plans for compliance with National Ambient Air Quality Standards for PM2.5 and PM10, visibility impact assessments, and source-receptor studies related to PM2.5 and PM10 emissions.

5.2 Optionally, with additional equipment and sampling/analytical procedures not described in this method, PM chemical speciation and physical characterization can be determined by applying ambient air sample collection and analysis methods (N1, N3, N4) to the diluted stack sample obtained using this dilution sampling method. Chemical speciation of PM is a key aspect of source apportionment analysis. Appropriate materials that do not contaminate the sample for the species of interest must be used for all sampler surfaces that contact the sample. Additional pre-test cleaning procedures must be applied to minimize contamination.

5.3 Optionally, with additional equipment and sampling/analytical procedures not described in this method, PM gaseous precursor emissions (e.g., SO_2, NO_X, ammonia, SO_3, HCl, VOC with carbon number greater than 7) can be determined by applying ambient air sample collection and analysis methods to the diluted stack sample obtained using this dilution sampling method (N3, N4).

6. Interferences

6.1 An important source of PM mass measurement bias is from the handling of the filters. Extreme care must be taken during the loading and unloading of filters to ensure the filters do not fray or break. It is highly recommended that the loading and unloading of the filter housings be performed in a laboratory, rather than at the testing location. Personnel loading and unloading filter housings shall wear gloves that are antistatic and powder-free when handling filters. A smooth, non-serrated forceps shall be used to handle filters. The forceps shall be used for this purpose only, cleaned with alcohol and lint-free wipes, and air-dried prior to use.

6.2 Particles may deposit on surfaces of the dilution sampling system and thus may not be collected on the sampling media. The dilution sampler shall be designed to minimize deposition of particles in the sampler prior to the sampling media. Losses in the dilution sampler may be caused by a combination of factors, e.g., thermophoretic losses in the sampling probe, and electrostatic, inertial, and diffusional losses in the sampler and tubing connecting sampling media to the sampler. See Section 12.4 for more details. Grounded electrically conductive surfaces shall be used to minimize electrostatic losses. The sample probe should be maintained at a temperature at or slightly above the stack gas temperature to minimize thermophoretic losses to the extent possible. Sharp changes in flow direction shall be avoided to minimize inertial losses.

6.2.1 *Particle losses from undiluted stack gas sample*—deposits of particulate matter on the surfaces of a dilution sampler in contact with the undiluted stack gas sample, such as the sample probe, were determined to be 18% of total PM2.5 and more than 43% of total PM10 (N17). Therefore, deposits on these sampler components must be recovered, analyzed, and included in the total PM2.5 and PM10 results.

6.2.2 *Particle losses from diluted stack gas sample*—deposits of particulate matter on the surfaces of a dilution sampler in contact with the diluted stack gas sample, such as the mixing section and residence time section, were determined to be 3% or less of total PM2.5 and PM10 (N17). Therefore, dilution samplers designed to minimize particle deposits on these components should be capable of achieving equivalent performance. It is not necessary to recover deposits from these components of the sampler provided that the design is similar to that in N17 or that similar performance has been previously demonstrated.

6.3 Liquid water content can impact ambient aerosol size distribution. At ambient relative humidity greater than 70%, particle growth due to accumulation of moisture becomes significant. Therefore, the dilution air relative humidity must be controlled to prevent condensation. (N1, N5). Particle size growth from moisture condensation can bias PM measurements if agglomerated particles grow to a size greater than the target size (2.5 or 10 micrometer aerodynamic diameter) and are removed by the dilution sampling system prior to the sampling media. Therefore, the diluted sample humidity must be controlled below 70% to maintain particle growth conditions.

6.4 The sample may be altered during sample storage and shipping if appropriate conditions are not maintained as specified in Sections 9.8 and 9.9. For example, organic and nitrate compounds on filters may volatilize if the samples are stored at temperatures higher than the sample collection temperature contributing to negative bias in the filter mass.

7. Requirements for Sampling Plane and Sampling Points

7.1 Select a sampling site in accordance with the criteria of Test Method D 3154.

7.1.1 The velocity of the stack gas shall be in the range where a PM2.5 or PM10 cyclone can be used (N6). For PM2.5, the sample may be collected from a single point in the stack sufficiently far from the wall to eliminate wall flow disturbances. For PM10, the sample shall be withdrawn isokinetically at each traverse point.

7.1.2 Convenient access ports and a working platform are required for the testing. See Test Method D 3685/D 3685M for additional criteria.

7.2 *Sampling Plane:*

7.2.1 If stack gas velocity is to be measured using a pitot tube (D 3154, N10) or other in-stack method, the sampling plane shall be situated in a length of straight duct (preferably vertical) with a constant shape and constant cross-sectional area. The sampling shall be conducted as far as practical downstream and upstream from any obstruction that may cause a disturbance and produce a change in the direction of flow (Disturbances can be caused by bends, fans, or control equipment) (N11).

7.2.2 Stack gas velocity measurements at all the sampling points defined in 7.3 shall prove that the gas stream at the sampling plane meets the following requirements:

7.2.2.1 The angle of gas flow is less than 15° with regard to the duct axis (method for estimation is indicated in Annex C of ISO 10780);

7.2.2.2 No local negative flow is present;

7.2.2.3 The minimum velocity is measurable by the test method used (for example, using Test Method D 3154, a differential pressure larger than 5 Pa);

7.2.3 If the above requirements cannot be met, the sampling location will not be in compliance with this test method for measuring stack gas velocity with a pitot tube and alternative methods should be considered.

7.3 *Minimum Number and Location of Sampling Points for measuring stack gas velocities:*

7.3.1 See Test Method D 3154, Section 8, Figs. 7 and 8, and Tables 1 and 2.

7.4 *Access Ports:*

7.4.1 Provide sampling ports for access to the sampling points selected, in accordance with 7.3 and Test Method D 3154.

7.4.2 Ensure that the port dimensions offer ample space for the insertion and removal of the sampling equipment and associated devices.

8. Velocity and Gas Composition Measurement Apparatus

8.1 See Section 6, Test Method D 3154.

9. Sampling Apparatus

9.1 *Sampling Probe*—Stainless steel, glass, or other suitable material commensurate with the test objectives with a diameter between 10 mm and 13 mm equipped with an in-stack PM2.5 or PM10 cyclone (N6). The probe wall temperature shall be maintained at or above the source temperature and the water dew point, when practical.

9.2 *Sample Transfer Line*—(Optional, not recommended – only used if port logistics do not allow a direct connection of the probe and dilution system). Constructed of stainless steel and shall be as short as possible and of a diameter equal to or greater than the probe diameter, but no more than 13 mm. The sample transfer line shall be insulated and heated to a minimum wall temperature equal to the source temperature but no greater than 315ºC.

9.3 *Dilution Air High Efficiency Particulate Arresting (HEPA) filter*—designed for air flows to give dilution ratios of at least 20:1. HEPA filters shall have a minimum filtering efficiency of 99.97% when tested at an aerosol of 0.3 micrometers diameter (N7).

Note 1: Results of previous tests showed that significant background levels in the dilution air were found when testing gas-fired sources (N 18). The removal efficiency of ambient PM2.5 across the dilution air filtration system used in those tests, which included a HEPA filter, was estimated at approximately 80%. Depending on the test objectives, a filter system with higher filtration efficiency may be required.

9.4 *Dilution Air Activated carbon bed or filter*—Optional – Recommended if samples for analysis of organic compounds will be collected or if ambient air has high VOC concentrations. Design for air flows to give dilution ratios of at least 20:1.

9.5 *Dilution Air Dehumidifier (optional)* —designed for air flows to give dilution ratios of at least 20:1 and diluted gas humidity less than 70 % at temperatures equal to or less than 52ºC.

9.6 *Dilution Air Heater (optional)*—designed for air flows to give dilution ratios of at least 20:1 and diluted gas temperatures equal to or less than 52ºC.

9.7 *Dilution sampling system mixer/residence time chamber*—with following specifications:

9.7.1 The inlet will be the point of mixing between the dilution air and stack gas sample. Mixing must be rapid with completeness verified by measurement of the downstream CO_2 profile for combustion sources at a minimum of four points with a maximum difference of 10% between any two traverse points. Other verification procedures such as VOC profiling may be used for non-combustion sources. Mixer designs can include jet entrainment, mixing orifice, venturi, or equivalent.

9.7.2 The flow capacity of the dilution system shall be sufficient to provide a minimum 20:1 dilution ratio. Larger dilution ratios may be required to completely eliminate water condensation in dilution and sampling systems and maintain temperature of the diluted stack at or below 52 ºC immediately upstream of the residence time chamber’s cyclone(s).

9.7.3 The minimum residence time shall be ten (10) seconds after mixing is complete (N12).

9.7.4 The system shall be and designed and constructed of non-reactive materials to minimize deposition or alteration of the particulates and electrically earthed to prevent electrostatic effects.

9.7.5 *Temperature control system (Optional)*—Insulation, cooling, or heating may be required to maintain target temperature range.

9.7.6 *Dilution air vacuum pump*—sufficient capacity to maintain specified dilution air flows.

9.8 *Residence time chamber sampling system*

9.8.1 *Stainless steel cyclone(s)*—depending on the test objectives, one or more PM2.5 cyclones may be be used to withdraw the diluted and aged sample from the residence time chamber. The flow rate through the cyclone(s) shall be sufficient to provide flow to the sample filter and other sample media. The cyclone shall be constructed of stainless steel (316 or equivalent). The cyclone performance shall meet the size cutoff characteristics shown in Figure 2 (N13), and shall be operated to achieve a 50% cutoff diameter of 2.5 micrometers. A Unico 240 cyclone (available from Desert Research Institute, 2215 Raggio Parkway, Reno, NV 89512-1095) is an example of a cyclone with these characteristics.

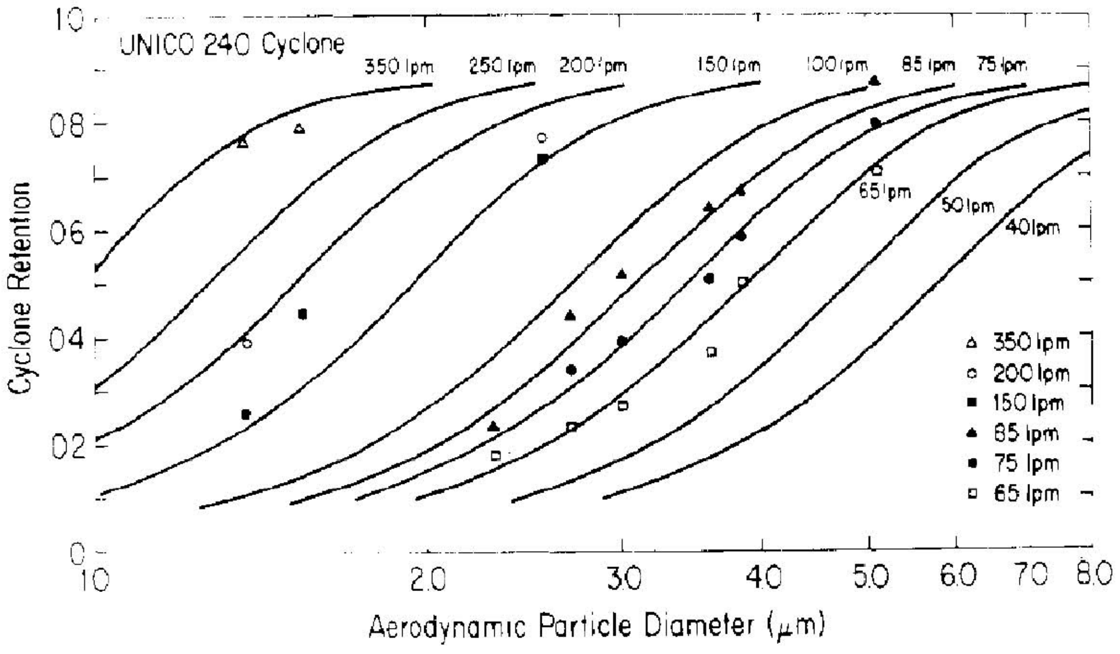

Figure 2. Residence time chamber PM2.5 cyclone performance specifications (N13).

9.8.2 *Transfer line*—The transfer line(s) from the cyclone(s) shall be designed to minimize PM deposition (gradual bends if any), constructed of electrically conductive material (stainless steel tube is recommended) that will not react with gas constituents, and electrically grounded. The transfer line may be heated to no more than 52 ºC before stack gas is introduced to the chamber containing the filter packs and may be insulated. The transfer line shall be as short as possible. A maximum length of 1.02 meters is recommended.

9.8.3 Filter cassette assembly (gravimetric analysis method)—constructed of 302, 303, or 304 stainless steel, polycarbonate or acrylonitrile/butadiene/styrene (ABS) resin with a minimum 47 mm stain area. A combination of these materials may also be used. Recommended loading of stain area: 0.5 mg on 47 mm filter; 1.3 mg on 70 mm filter; 2.3 mg on 90 mm filter; 3.6 mg on 100 mm filter. The filter packs may be heated or insulated to maintain a maximum filter temperature of 52 ºC.

9.8.3.1 The filter holder assembly design shall avoid sample losses that have a significant adverse affect on systematic and random test result variations.:

9.8.3.1.2 The entrance to the filter holder shall be designed using sound engineering judgment and leak free construction, to an inside diameter no smaller than 0.85 cm, maintaining the 12.5º angle from the inlet of the top filter holder to the area near the sealing surface of the top of the filter cartridge assembly. Figure 3 shows an acceptable design. Variations using sound engineering design are also acceptable provided that they provide even flow distribution across the filter media and a similar leak-free seal with the filter cartridge assembly.

9.8.3.1.3 A bevel introduced on the inside diameter of the entrance to the filter cartridge, is also acceptable (see Figure 4).

9.8.4 *(deleted)*

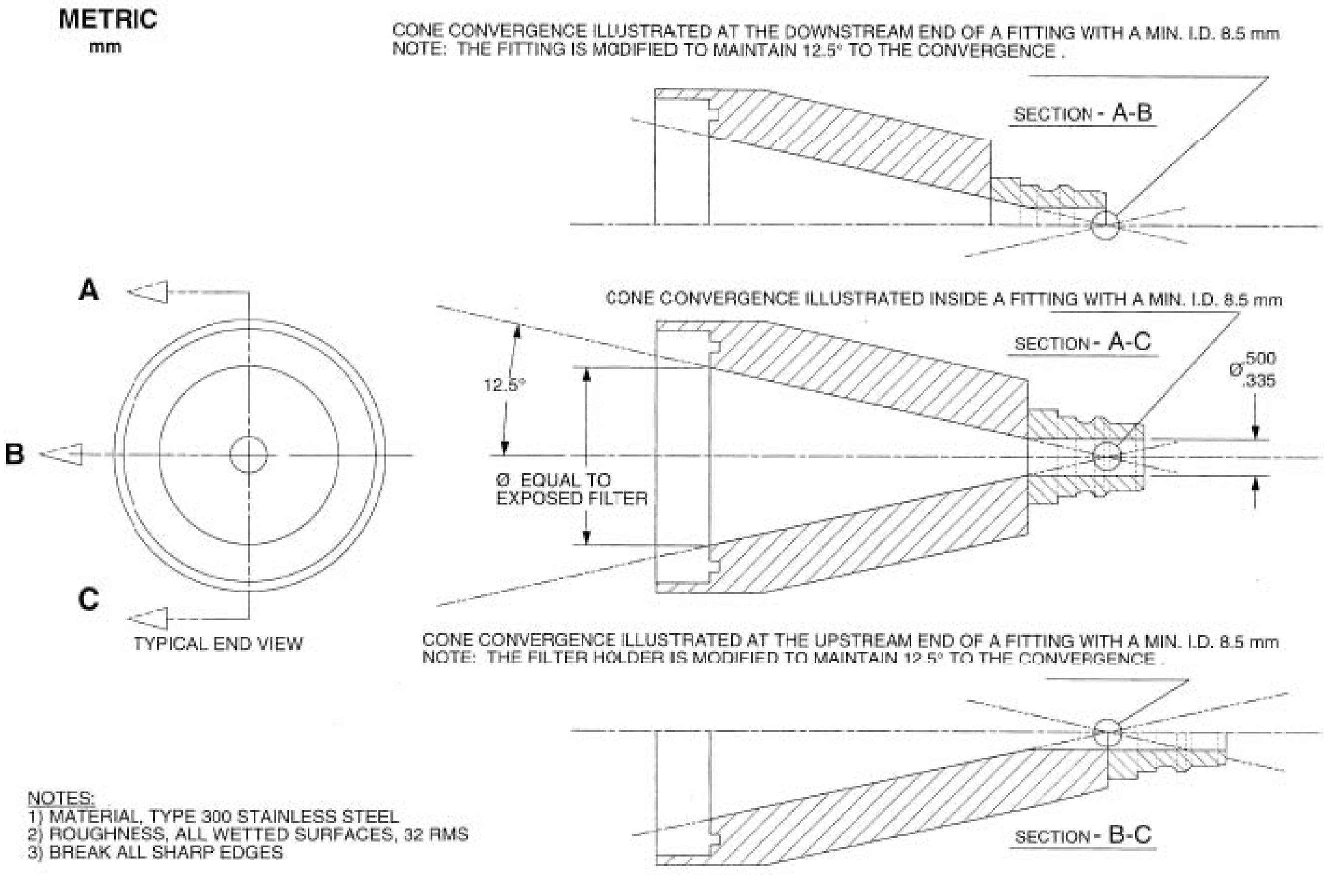

Figure 3. Filter holder geometry.

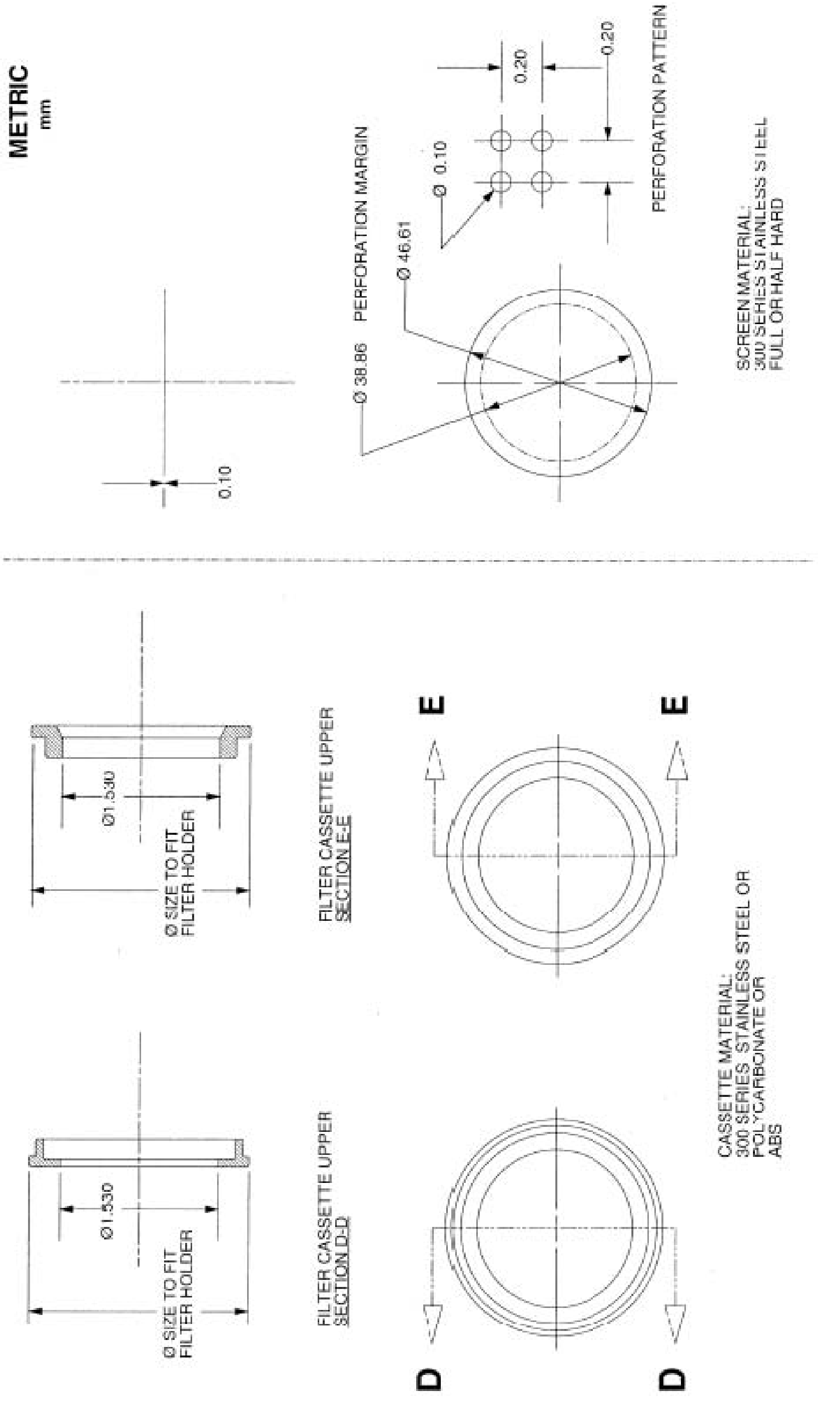

Figure 4. Filter Cassette and screen.

9.8.5 *Filter Cassette Containers*—Containers must protect the cassette and prevent it from moving within the container during transport. Containers should be fabricated from materials that have been demonstrated not to cause a static charge buildup. To avoid losses of particulate matter, do not allow the surface of the filter to touch any interior surfaces of the protective container. Containers should be marked for identification purposes.

9.8.6 *Insulated Shipping Container*—An insulated shipping container, capable of maintaining a temperature below 25 °C, is needed to transport the filter cassette containers between the field and laboratory after sampling. Coolers, such as those used for recreational purposes, filled with leak-proof ice substitutes have proven suitable for this use. The sample cassette temperature should be monitored from the time of collection until the sample is equilibrated (i.e., during shipping and handling). Monitoring can be done by placing an inexpensive electronic max-min thermometer in the shipping container for each leg of the trip to the laboratory.

9.8.7 *Sample Flow Measurements*—Sample gas flow shall be determined with flow measurement devices accurate to at least ± 4%

9.8.8 *Inertial microbalance design* (inertial mass measurement analysis method) - The construction and assembly of the inertial microbalance shall be in accordance with Sections 6 and 8.

9.9 *Particulate Filter Analysis System.*

9.9.1 Analytical balance

9.9.1.1 Precision—95% confidence uncertainty bounds: 5 µg for filter diameter less than or equal to 70 mm; 50 µg for filter diameter greater than 70 mm

9.9.1.2 Resolution: Filter size <70 mm: 1 ug; Filter size ≥70 mm 10 ug

9.9.2 The balance shall be located in the same climate-controlled environment in which the filters are conditioned. Temperature and humidity controls and meters shall be used to maintain and monitor the conditions within the filter conditioning and weighing room environment. The mean temperature of the weighing chamber should be maintained between 20 and 23 °C, with a variability of not more than ±2 °C over 24 hours and the mean relative humidity should be maintained between 30 and 40%, with a variability of not more than ±5% over 24 hours.

9.9.3 The analytical balance shall be electrically earthed to prevent electrostatic effects.

9.9.4 Static charge neutralizer –A polonium 210 neutralizer or device of similar effect shall be employed to eliminate or minimize static charge effects.

9.9.4.1 *Discussion:* It may be necessary to place a polonium 210 neutralizer or other similar device in the weighing chamber to prevent electrostatic charge buildup. It may also be necessary to pass each filter near, but not touching, an antistatic strip before it is weighed. Charge neutralization times may need to be longer than 60 sec for sampling situations in which (1) a high amount of charge has developed on collected particles due to their origin or (2) the particle loading on a filter is large. Electrostatic charge buildup becomes greater as the air becomes drier. A 60-sec charge neutralization may be sufficient in ambient indoor air conditioned to 37% RH and 23 °C but not in 20% RH and 23 °C in arid environments. This latter environment may require that the filter sit for more time on the antistatic strip. The longer neutralization period may have to be performed inside the weighing chamber or in a second small chamber used only for charge neutralization.

9.9.5 The analytical balance shall be calibrated at least annually using certified NIST-traceable mass reference standards. In addition, working standards shall be used to verify the calibration of the analytical balance before each use. Working standards shall be verified every 3 to 6 months against National Institute of Standards and Technology (NIST) traceable laboratory primary standards.

9.9.5.1 Select mass reference standards so that they are in the expected mass range for clean, unexposed filters and exposed filters. They should be certified as being traceable to NIST mass standards (see ASTM 1993b). Additionally, they should have an individual tolerance of no more than 0.025 mg.

9.9.6 Locate the microbalance on a sufficiently level, sturdy, vibration-free base (ideally, a level, stone weighing table), away from any equipment that produces vibrations, out of direct sunlight, and away from local heating or cooling sources such as open flames, hot plates, water baths, ventilation ducts, windows, and heat producing lamps.

10. Reagents and Materials

10.1 *Purity of Reagents*—Reagent grade chemicals shall be used in all tests. All reagents shall conform to the specifications of the committee on Analytical Reagents of the American Chemical Society. (N8)

10.2 *Purity Of Water*—Unless otherwise specified, water shall be Type III reagent water conforming to ASTM Specification D 1193.

10.4 *Purity of Acetone*—Reagent ACS grade acetone with < 0.001 % residue in glass bottles. Acetone supplied in metal containers is unacceptable due to the prevalently high residue levels. Reject the acetone if blank residue mass (see 10.3) is >0.001 % of the total acetone mass.

10.4 *Soap*.

10.5 *Kimwipes*.

10.6 Towels.

10.7 *Brushes*—to clean sample probes and lines that conform the the specifications in ASTM Specification D-3685

10.8 *PTFE tape*.

10.9 *Wash bottles* – Polyethylene, PTFE, or equivalent

10.10 *Shipping containers and materials for filters*—see 9.8.6.

10.11 *Gloves*, insulated, heat-resistant.

10.12 *Filters*. If 47 mm filters are being used, the filters shall meet the following specifications:

10.12.1 *Size*—circular, 46.2-mm diameter ±0.25 mm (with support ring)

10.12.2 *Medium*—polytetrafluoroethylene (PTFE) with integral support ring

10.12.3 *Support ring*—polymethylpentene (PMP) or equivalent inert material, 0.38 ± 0.04 mm thickness, outer diameter 46.2 ± 0.25 mm, and width of 3.68 mm. The support ring diameter should also be checked for consistency. Variations in diameter will affect the filter exposure area, which, in turn, will affect any operations that involve use of a portion of the filter.

10.12.4 *Pore size*—2 µm

10.12.5 *Thickness*—30 to 50 µm.

10.12.6 *Maximum pressure drop (clean filter)*—30 cm H_2O column at 16.67 L/min clean air flow.

10.12.7 *Maximum moisture pickup*—No more than 10 µg weight increase after a 24-hour exposure to air at 40% RH, relative to the weight after a 24-hour exposure to air at 35% RH

10.12.8 *Collection efficiency*—greater than 99.7%, as measured by the dioctyl phthalate (DOP) test (ASTM 2986 or equivalent) with 0.3-µm particles at the sampler's operating face velocity. The manufacturer's quality control test data are sufficient for validation of efficiency.

10.12.9 *Filter weight stability (including test for loose, surface-particle contamination and test for temperature stability)*—filter weight loss <20 µg in either test.

10.12.10 *Alkalinity*—less than 25 microequivalents/g of filter.

10.12.11 Check the filters for irregularities, flaws, or pinholes by holding them up against a light source.

10.12.12 Although not required for determination of PM2.5 mass concentration under this standard, additional specifications for the filter should be developed by users who intend to subject archived PM2.5 filter samples to subsequent chemical analysis. These supplemental specifications include background chemical contamination of the filter and any other filter parameters that may be required by the chemical analysis method.

10.13 Personal protective equipment, as needed. See Section 19.

11. Pre-Sampling, Filter Conditioning and Weighing Procedures

11.1 General Aspects—Before carrying out any measurements, discuss the purpose of the sampling and the sampling procedures with the plant personnel concerned. The nature of the plant process, for example, steady state or cyclic, can affect the sampling program. If the process can be performed in a steady state, it is important that this state be maintained during sampling.

11.1.1 Agree upon, with the plant management, the dates, starting times, duration of survey, and sampling periods as well as plant operating conditions during these periods.

11.1.2 Make preliminary calculations on the basis of the expected particulate loading to determine the appropriate nozzle size, sample rate, and sampling conditions. Also determine whether the chosen nozzle size, sampling rate and sampling time will result in sufficient matter collected to obtain a sample filter mass at least five (5) times the dilution system blank filter mass. If possible, use system blank results from previous tests using the same dilution sampling system. If no previous data are available, then select run times based on minimum detection limits including background levels summarized in Section 16.3 (N18).

11.2 Collect a dilution system blank sample after each series of test runs on the same source following the procedure described in 13.8.2.

11.3 *Gravimetric Filter Weighing Procedures:*

11.3.1 *Presampling Filter Conditioning:*

11.3.1.1 Verify that the filter chamber mean relative humidity was maintained between 30 and 40%, with a variability of not more than ±5% over 24 hours. Verify that the mean temperature was between 20 and 23 °C, with a variability of not more than ±2 °C over 24 hours.

11.3.1.2 Record the equilibration chamber's relative humidity and temperature on the laboratory data sheet.

11.3.1.3 Condition the filters for a minimum period for a minimum of 24 hours and until weight changes between successive weighings are less than <15 µg.

11.3.2 Postsampling filter equilibration and storage:

11.3.2.1 Equilibrate filters for at least 24 hours.

11.3.2.2 Weighing should be completed within 240 hours (10 days) after the end of sampling, unless the filter is maintained at 4°C or below during the entire time between retrieval from the sampler and start of the conditioning, in which case the period shall not exceed 30 days.

11.3.2.3 If filters were to be maintained at reduced temperature (4°C or below) during the time between retrieval from the sampler and start of the conditioning, verify that the container was continuously maintained at 4°C or below using a min/max thermometer or other suitable device that was placed in the container at the time of shipment or by inspecting the cool packs to determine if they are still frozen or cold. If the temperature is between 4 and 25 ºC, but is below the maximum temperature that was recorded during the filter sampling period, assess the temperature and note other relevant information and evaluate the need to determine a shorter analysis deadline for the sample(s).

11.3.2.4 Verify that the filter chamber mean relative humidity was maintained between 30 and 40%, with a variability of not more than ±5% over 24 hours. Verify that the mean temperature was between 20 and 23 °C, with a variability of not more than ±2 °C over 24 hours. Record the equilibration chamber's relative humidity and temperature on the laboratory data sheet.

11.3.2.5 If mean relative humidity and/or temperature variation exceeds the specification in 11.3.2, repeat equilibration period.

11.3.3 Gravimetric Pre- and postsampling filter weighing:

11.3.3.1 Before each weighing series verify the balance calibration using standard weights. Weights of the mass standards should bracket the weights of the filters being measured.

11.3.3.2 Filter identification—suitably mark the filter container and laboratory data form to identify each filter.

11.3.3.3 Weigh the filter on a suitable balance (see section 9.9). Both the pre- and postsampling filter weighings should be performed on the same analytical balance. Smooth, non-serrated forceps shall be used to handle filters. The forceps shall be used for this purpose only, cleaned with alcohol and lint-free wipes, and air-dried prior to use.

11.3.3.3.1 Discussion: Because of the greater sensitivity needed for measuring microgram-range weights or weight differences, microbalances are vulnerable to relatively small changes in physical environmental conditions, such as vibration, electrostatic charge buildup, temperature, or relative humidity (RH). Features to offset the effect of these variables on the measurements may be integrated into the design of the microbalances, or they may be offered as options (see 9.9.4). The following general guidelines should be followed to control environmental factors that may affect microbalance performance:

11.3.3.3.1.1 Select a room that is not subject to large temperature or humidity variations and that has its heating and air conditioning maintained 24 hours a day, including weekends.

11.3.3.3.1.2 Locate the microbalance away from potential sources of drafts such as doors, windows,aisles with frequent traffic, ventilation ducts, and equipment with fans or moving parts.

11.3.3.3.1.3 If these arrangements are not possible, isolate the microbalance from such equipment by placing a stabilizing slab under the microbalance and/or by placing composite vibration damping pads at three points under the microbalance’s legs or under the stabilizing slab. Placing the pads at only three points eliminates any possible rocking associated with pads placed at four points.

11.3.3.3.1.4 Ensure that the microbalance’s base is sufficiently level to permit leveling of the microbalance according to the manufacturer’s instructions.• Filter weighing chamber shall be free of any ambient contaminants (such as dust) that could settle on filters during stabilization.

11.3.3.4 Observe all weighing procedures. Perform all quality control (QC) checks.

11.3.3.5 Passive deposition of dust on sample filters prior to and following sampling can bias PM measurements. This can be minimized by loading and unloading filter housings in the laboratory under controlled conditions and the use of the prescribed filter cassettes. Gases may be collected on sampling filters or on particles captured by the filter creating an artifact in the filter weight. Adsorption of organic gases on quartz filters and/or the sample is a known artifact that can contribute to significant positive bias in filter weight. The oxidation of acid gases (e.g. SO_2, NO_2) or retention of gaseous nitric acid on the surface of the filter also can contribute to positive bias in filter weight. These effects can be minimized by the use of PTFE membrane filters that are less prone to gas adsorption than quartz fiber filters. Measurement artifacts due to filter handling can be reduced or eliminated by using an inertial mass measurement technique because filters are not handled during the mass measurement process.

11.3.3.6 Repeat weighing.

11.3.3.7 At least two reference filters shall be weighed within four hours of sample filters weighings. If the average mass of the reference filters changes between sample filter weighings by more than 5% of the recommended minimum filter loading (refer to 9.9), then flag data and report deviation in reference filter weights.

11.4 Inertial Mass Measurement Procedures. If PM mass determination is made using an inertial microbalance, equipment configuration, operational procedures and QA/QC requirements shall performed in accordance with ASTM D6831-02 as follows:

11.4.1 Equipment configuration and setup—D6831-02, Section 6, except, depending on the configuration of the inertial microbalance sampling port, it may not be necessary to configure the system with integrated gas temperature and gas velocity measurement devices.

11.4.2 Operation procedures—D6831-02, Section 10, except that it may be acceptable to sample at non-isokinetic sampling conditions ($I \leq 90\%$ or $I \geq 110\%$) if the source sample is collected through a size selective inlet with a design cutpoint of 2.5µm or smaller. Also, the mass transducer temperature shall be maintained at or above the diluted sample gas temperature. Typically, the mass transducer temperature control is set to maintain the temperature at approximately 5°C above the sample gas temperature.

11.4.3 Calibration and QA/QC procedures—D6831-02, Sections 7 and 13. Field and laboratory blanks for the inertial microbalance filters are not required since handling of filter cartridges is not performed while sampling and PM mass determinations are performed. A sample filter is conditioned and tared after the cartridge has been installed and the final, collected PM mass determination is made after post-sampling filter conditioning has been completed but before the exposed filter cartridge is removed from the mass transducer.

12. Sampling

12.1 Preparation:

12.1.1 Clean (brush and rinse), calibrate, and check all the equipment before the first run of a new test. Exercise care not to reuse any part of a sampling train from a previous test program without a thorough cleaning.

12.1.2 Prepare the filters to be weighed for each sample run. This includes filters for the blank tests and additional filters to accommodate process and equipment malfunctions.

12.1.2.1 If using filters for the gravimetric measurement of particulate mass prepare them according to Section 11.3.

12.1.1.2 If using an inertial microbalance for the measurement of particulate mass refer to Section

11.4.

12.1.3 Perform the weighing procedures according to 11.3 and load the filters into the filter cassettes.

12.1.4 Protect all the weighed filters and the other parts or equipment that will come into contact with the sample from contamination during transportation and storage. Store and transport loaded filter cassettes in the filter cassette containers and insulated shipping containers described in Sections

9.8 and 9.9.

12.2 Pre-Sampling Measurements:

12.2.1 Determine the stack sampling location and the stack gas temperature, pressure, moisture content, velocity and molecular weight according to ASTM Test Method D-3154.

12.2.2 Determine the constant sample flowrate and nozzle size necessary to achieve the target in-stack cyclone particle size cutpoint(s) and a nozzle sample gas velocity that is within 20% of the average isokinetic rate based on the preliminary calculations (11.1.2), the measured stack gas velocity profile and other stack parameters (12.2.1). If measurements are intended for determination of PM2.5 only, isokinetic sampling is not required.

12.3 Sampling:

12.3.1 Assemble the dilution sampling system in an area free of potential contaminants. Immediately before and after each test run, check for leaks in the in-stack cyclone/sample probe/sample flowmeter assembly and in the entire dilution sampler assembly. If using tracer gas procedures to determine the dilution ration then only that portion of the sampling system up stream of the first tracer senser location must be leak checked.

12.3.1.1 Pre-Test Leak Check (for components that normally operate under negative pressure)— It is important to avoid any leakage of ambient air or other sources of contamination into the sampling system. Most systems will withdraw stack gas sample by inducing flow with a pump, in which case the stack gas sample in the components upstream of the pump is under negative pressure. All components that normally operate under negative pressure must be leak checked. For systems in which the entire dilution system operates under negative pressure, this leak check must be performed in two steps: first, the probe and components that are in contact with the undiluted sample alone, and second the entire system. For system that operate with some or all components under positive pressure and which determine dilution ratio using tracer measurements, no leak check is necessary.

12.3.1.2 Pre-Test Leak Check –Undiluted Sample Components. Temporarily seal the in-stack cyclone sample nozzle and draw a vacuum on the cyclone/sample probe/sample flowmeter assembly equal to the maximum vacuum experienced during a run and measure the sample flow (leak) rate for a period of approximately 5 minutes. If the pre-run leak rate is greater than 2% of the target sample flow rate determined in Section 12.2.2, identify the source of the leak and eliminate or reduce it until the leak rate is equal to or less than 2% of the target sample flow rate. If the leak rate after the test run is greater than 2% of the actual average sample flow rate during the run, the results of that run may not be valid and must be flagged in the report.

12.3.1.2 Pre-Test Leak Check – Entire System. Attach the in-stack cyclone/sample probe/sample flowmeter assembly to the dilution sampler. Temporarily seal the cyclone sample nozzle, dilution air inlet, and diluted sample bypass. Then draw a vacuum on the entire assembly equal to the maximum vacuum experienced during a run and measure the sample flow (leak) rate for a period of approximately 5 minutes. If the leak rate before the test run is greater than 2% of the total flow through the dilution chamber during a run, then the source(s) of the leak(s) must be identified and eliminated until the leak rate is equal to or less than 2% of the total flow through the dilution chamber. If the leak rate after the test run is greater than 2% of the total flow through the dilution chamber during the run, the results of that run may not be valid and must be flagged in the report.

12.3.2 Conduct a stack gas Pitot tube traverse per ASTM D 3154 for determination of stack gas velocity and flow rate.

12.3.2.1 If sampling is being performed at a single point for PM2.5 in the stack gas, conduct Pitot tube traverses immediately before and after the test run and measure the velocity head with the Pitot tube at the single sampling point continually during the test run. Calculate the ratio of the average velocity over the full traverse to the velocity at the single sampling point based on the pre- and post-run Pitot tube traverses. Calculate the average stack gas velocity at the single sampling point during the test run based on the velocity head measurements during sampling, and multiply this by the average-to-single point velocity ratio determined from the pre-and post-run traverses. Use the resulting corrected velocity to calculate average stack gas flow rate during the test run.

12.3.2.2 If sampling is being performed at multiple traverse points for PM10 in the stack gas, conduct the Pitot tube traverse during sampling.

12.3.2.3 Alternatively, for combustion sources, stack gas flow rate may be measured by stack gas oxygen, fuel flow and fuel composition measurement according to ISO 8178-1 Annex A, or other equivalent method.

12.3.3 Place the sample probe nozzle at the first sampling point. Avoid contacting the nozzle with any deposits on surfaces of the access port or stack walls. Seal the opening of the access port to minimize air in-leakage or exposure of operators to toxic gases.

12.3.4 Turn the pump on to start the dilution air flow

12.3.5 Preheat the stack gas sample probe to the specified temperature (9.1). After the minimum probe temperature has been achieved, start the stack gas sample flow (11.1.2). Adjust the dilution air flow rate to achieve a dilution ratio of at least 20:1. Higher dilution ratios may be used if required to completely eliminate water condensation or to lower the temperature of the diluted stack gas to less than or equal to 52 ºC at the cyclone(s) upstream of the filter holders (see also Section 9.7). If dilution ratios greater than 30:1 are required, the minimum dilution ratio necessary to achieve the maximum humidity and maximum temperature criteria should be used. Adjust the temperature of dilution air if needed.

Adjust the humidity of the dilution air, if necessary, to maintain relative humidity in the diluted sample gas equal to or less than 70%.

12.3.6 Verify the dilution ratio—Methods may include:

12.3.6.1 Direct measurement of flows (stack gas sample, dilution air, total diluted stack gas sample; at least 2 of the 3 needed); or

12.3.6.2 Measurement of CO_2, NO_X, VOC, or other appropriate tracer in the stack gas sample, dilution air, total diluted stack gas sample and calculation of dilution ratio by mass balance; or

12.3.6.3 From carbon balance by measuring CO_2, fuel composition, and flow according to ISO 8178-1, Annex A.

12.3.7 After the dilution tunnel temperature has stabilized to ±2 ºC for three (3) minutes, turn on the filter sample system pump and adjust the flow to the target rate. The filter face velocity should not exceed 100 cm/sec (N15) and the pressure differential across the filter should not exceed 75 cm of water column.

12.3.8 Continue to collect the sample for the target sampling duration. Target sampling durations will vary depending on process conditions, the concentration of particulate matter in the stack gas, and the test objectives for minimum detection and quantification limits. Sampling durations of thirty (30) to sixty (60) minutes may be sufficient for most continuous processes. Batch processes may require sampling for one (1) or more process cycles. Record dilution tunnel and sample system operating conditions (temperature, flow rates, humidity, dilution ratio, pressure differential across the sample filter) a minimum of twelve (12) times during a test run, or more often if needed, to capture expected process variations, The data collection should be conducted at equally spaced intervals during each test run.

12.3.9 At the end of the run, record the final dilution system and sample filter operating data. Turn off the sampling pump and record the final sample volumes. Shut down the dilution sampling system. Perform the post-run leak checks.

12.3.10 Recover the sample filter cassette with filter intact, place the filter cassette in the filter cassette holder and seal tightly, and affix a sample label with a unique number to each sample. Record sample information on a suitable sample tracking or chain of custody form. Pack the samples in the insulated shipping and storage container for shipment or transfer to the analytical laboratory. Store and transport samples in accordance with Section 9.8.6.

12.3.10.1 Inspect the sample lines for condensed material, record any observations.

12.4 Recovery of Deposits Upstream of the Filter:

12.4.1 Depending on the test objectives and whether both the PM10 and PM2.5 in-stack cyclones are used, recovery may involve the quantitative transfer of particles in the following size ranges: (1) larger than 10 micrometers, (2) smaller than or equal to 10 micrometers but larger than 2.5 micrometers, and (3) smaller than or equal to 2.5 micrometers. Use a Nylon brush and an ultrapure acetone rinse to recover particles from the cyclone sampling head, sample probe and sample gas flowmeter. Keep any dust found on the external surfaces out of the samples. Minimize the volume of acetone used for recovery to minimize the influence of acetone blanks.

12.4.1.1 Container #1—Quantitatively recover the (1) particulate matter from the PM10 cyclone cup and acetone rinses (and brush cleaning) of the cyclone cup, (2) internal surface of the nozzle, and (3) PM10 cyclone internal surfaces, including the outside surface of the internal downcomer tube. Seal the container and mark the liquid level on the outside of the container. This container holds particulate matter larger than 10 micrometers.

12.4.1.2 Container #2—Place the solids from the PM2.5 cyclone cup and the acetone (and brush cleaning) rinses of the PM2.5 cyclone turnaround cup (above inner downcomer tube), inside of the downcomer tube, and interior surfaces of the PM2.5 cyclone into the container. Seal the container and mark the liquid level on the outside. This container holds particulate matter smaller than 10 micrometers and larger than 2.5 micrometers.

12.4.1.3 Container #3—Retrieve the acetone rinses (and brush cleaning) of the exit tube of the PM2.5 cyclone, the sample probe, the sample gas flowmeter, and all surfaces downstream of the cyclones in contact with the undiluted sample gas into container #3. Seal the container and mark the liquid level on the outside of the container. This container holds particulate matter that is smaller than

2.5 micrometers.

12.4.1.4 Container #4—Depending on the design of the dilution sampler, it may be necessary to recover particle deposits found on the surfaces of the dilution sampler in contact with the diluted stack gas sample. If necessary, recover these deposits with acetone into sample container #4.

Note 2: Previous studies have shown that particle losses on dilution sampler surfaces in contact with the diluted stack gas sample can be reduced to 3% or lower by proper design (N17). Provided that similar designs are used or previous test results demonstrate that particle losses on the surfaces in contact with the diluted stack gas sample are less than or equal to 3%, recovery of particle deposits from surfaces in contact with the diluted stack gas sample is not required.

12.4.1.5 Container #5—Collect 250 mL of acetone from the wash bottle used to perform sample recovery rinses into sample container #5. This is the acetone field reagent blank.

12.5 Validation of Results:

12.5.1 Parameters Depending on the Stationary Source:

12.5.1.1 If testing was conducted at an unsuitable location or was carried out under fluctuating plant operating conditions, the representativeness of the sample(s) may be questioned and the uncertainty of the measurement results unknown. In such cases, the test report shall clearly indicate that the test was not conducted according to the specifications of this test method.

12.5.1.2 The test report must include details of the flow characteristics at the sampling location or on the variations of the flow rate in the stack while sampling, or both.

13. Calibration and QA/QC Activities:

13.1 Calibrate the velocity and gas composition measurement apparatus according to Test Method D 3154.

13.2 Gas flow rate meters—Calibrate stack gas flow rate meters at least every six (6) months or whenever a system repair or change is made that could influence calibration. Calibrate sampling system flow rate meters at least every three (3) months or whenever a system repair or change is made that could influence calibration. The gas flow rate meters shall be calibrated with a NIST-traceable standard or equivalent. The readings shall agree within ±4%.

13.3 Analytical balance—See Section 9.9.

13.3.1 The analytical balance calibration shall be verified at the beginning of each weighing session. Conduct calibration verifications using NIST-traceable mass standards or equivalent.

13.4 Temperature sensors—Verify the calibration thermocouples at least every three months or whenever a system repair or change is made that could influence calibration. Thermocouples should be calibrated with a NIST-traceable standard or equivalent and agreement should be within ±2 ºC.

13.5 Relative humidity meters—Verify the calibration of relative humidity meters at least every month or whenever a system repair or change is made that could influence calibration. Meters should be calibrated with a NIST traceable standard or equivalent and agreement should be within ±3% of reading.

13.6 Pressure sensors— Calibrate pressure sensors at least every 3 months or whenever a system repair or change is made that could influence calibration. The sensors should be calibrated with a NIST traceable standard or equivalent and agreement should be within ±2% of reading.

13.7 QA/QC Activities

13.7.1 Pre-test dilution sampling system cleaning (before each test condition) - Wash and rinse all components with acetone and allow to dry completely. Wipe down large surfaces.

13.7.2 Pre-test dilution system blank—At least one per test and sampling location. For each test program, collect at least one dilution system blank as an indication of total measurement background due to dilution air background and contamination of filters and rinsing solutions during sample handling, transport and analysis. Calculate the equivalent in-stack concentration using mean values of dilution ratio and sample volume for the stack samples. The level of significance will vary depending on test objectives, but 20% of the average stack sample results may be used as a general guide. Report these results along with the stack sample results..

13.7.3 Ambient air sample (optional but recommended)—One per test. Collect the ambient air sample at the sampling location by directly pulling unfiltered ambient air through a PM10 or PM2.5 cyclone head (or equivalent) and through the sampling media only. Conduct sampling for the same duration as stack samples, or longer depending on test objectives. These results may be used in conjunction with the dilution system blank to evaluate the dilution air filtration system performance.

13.7.4 Field blank – Collected by assembling and disassembling sampling equipment without drawing any gas through the media. These results may be used to assess cumulative contamination in sampling media preparation, handling, storage, shipping and analysis.

13.7.5 Pre-run and Post-run leak checks per 12.3.1

13.7.6 Dilution ratio check– If dilution ratio is determined by tracer methods, this check is not required. If dilution ratio is determined by direct flow rate measurements, this check is recommended to verify the calibration and proper operation of the system. Perform a dilution ratio check before the start of each run using an appropriate method, such as by measuring CO_2, NO_X or other tracer concentrations in the raw stack gas sample and diluted stack gas sample (ISO 8178-1). The measured dilution ratio by the two methods should agree within 10% relative percent difference. If the pre-test dilution ratio check is not within 10% of the of the measured value, correct the error before starting the test. Include the results of the dilution ratio checks in the final report.

13.7.7 Verify that the gases in the dilution sampling system are uniformly mixed under the test conditions. Mixing may be verified by measurement of CO_2 or other tracer gas profile upstream of the residence time section or any sample collection media at a minimum of four points across the dilution sampler cross-sectional diameter. Concentration at each individual point shall be within 10% of the mean of the concentrations at all points. Alternately, the degree of mixing may be warranted by the equipment manufacturer/vendor using these procedures.

13.7.8 Gravimetric Weighing QA/QC—After approximately every 10th filter, rezero the microbalance and reweigh at least one working standard. Weigh approximately 10% laboratory blanks per weighing session. Reweigh one replicate filter at the end of the weighing session. Weigh approximately 10% field blanks.

13.7.8.1 The working standard measurements should agree within 3 µg of the verified values. If the working standard measurements do not agree within this limit, recalibrate the balance, or use an alternate calibrated balance, and reweigh all filters since the last successful working standard check.

13.7.8.2 Laboratory blank and replicate measurements should agree within 15 µg. If laboratory blanks or replicate measurements do not agree within this limit, determine and correct the source of the discrepancy and reweigh all filters since the last successful laboratory blank and replicate check.

13.7.9 Gravimetric Filter Handling—Careful handling of the filter during sampling, conditioning, and weighing is necessary to avoid measurement errors due to damaged filters or a gain or loss of collected particles on the filters. Whenever filters are handled, the analyst should wear antistatic, powder-free gloves; these gloves act as an effective contamination barrier. Gloves that are packed in a box can carry an electrostatic charge. It is a good practice to discharge them by touching a good electrical ground after putting them on. The filters should be handled carefully by the support ring, rather than by the filter material, with smooth, non-serrated forceps that are used only for that purpose. Mark these forceps to distinguish them from the forceps used to handle mass reference standards. Forceps should be cleaned with alcohol and lint-free wipes before handling filters and then allowed to air-dry. These precautions reduce the potential effect from body moisture or oils contacting the filters and subsequently affecting the measured weights. If filter loadings will be speciated chemically, the possibility of contamination from gloves should be considered. Some gloves have been found to be contaminated with sulfate. If antistatic gloves are used, ensure that ammonium chloride is not a component of the antistatic reagent. Powder-free gloves that are certified to be free of chloride, nitrate, and sulfate are available. In the laboratory, each filter should be transferred from its sealed manufacturer's packaging to a clean filter-handling container, such as a glass or plastic petri dish or petri slide, to reduce the risk of contamination. The filter should remain in this container, except for weighing, until it is loaded into a filter cassette prior to sampling. Each filter should have a unique identification number. If such a number is not provided by the filter manufacturer, a label that lists the filter number can be attached to the filter-handling container. It is recommended that each microbalance be assigned a block of filter numbers to be processed and used sequentially. Take care to avoid mistakenly assigning the same number twice or omitting a number. Color-coded labels may be helpful for separating groups of filters when more than one microbalance is being used. If sufficient resources are available, bar coding for filters can be introduced. Bar code readers and printers for microbalances are already generally available and can significantly improve the efficiency of filter inventory tracking and processing.

13.7.10 Laboratory, Field and System Blanks

13.7.10.1 Laboratory Blanks—Laboratory blanks should be kept inside the conditioning chamber except during weighing sessions. Weigh enough laboratory blanks during a presampling weighing session to provide at least one single-use laboratory blank during each subsequent postsampling weighing session. The pre- and postsampling weights should be recorded in the QC notebook and the laboratory data form or an equivalent database. If the weight change exceeds 15 µg, contamination in the conditioning chamber may be occurring. Take appropriate troubleshooting and corrective actions.

13.7.10.2 Field blanks should be transported to the sampling site, momentarily installed in the sampler, removed, and stored in their protective containers, inside the sampler's case at the sampling site until the exposed filters are retrieved for postsampling weighing. Field blanks should be collected at the start of the test series. At least one field blank should be collected for each test series.

13.7.10.3 Dilution system blanks—at least one dilution system blank should be collected during each test series. It may be collected either at the beginning or at the end of the test series. Dilution system blanks should be collected by collecting a sample with all openings except for the dilution air inlet plugged so that only dilution air is drawn through the dilution sampler. Collect and analyze the sample in the same manner as the stack gas samples.

13.7.11 Inertial Microbalance QA/QC—Activities and procedures should be performed in accordance with D6831-02, Section 7 and 13.

13.8 Personnel Qualifications--Persons using this test method shall have adequate training and experience in preparing, maintaining and operating the sampling equipment, conducting the test procedures and performing tests at similar sampling locations. It is strongly recommended, but not required, that the testing organization meet the criteria specified in D-7036.

14. Additional Aspects Discussion

14.1 Sampling Duration and Detection Limit:

14.1.1 In-stack minimum detection limits are dependent upon the greater of analytical uncertainty or dilution system blank levels and upon dilution ratio, sampling duration, sample flow rate, sample filter flow rate, filter face velocity, filter pressure drop, and potentially other factors. See Section 16 for additional discussion of analytical uncertainty and dilution system blanks. Based on the pre-test estimated analytical uncertainty and dilution system blank results, select the sample flow rate, dilution ratio, sample filter flow rate, and sampling duration to achieve in-stack minimum detection limits commensurate with the test objectives.

14.2 Particulate Deposits Upstream of the Filter:

14.2.1 Errors can be introduced into the measurements if particulate deposits on the surfaces of the sampling system in contact with the sample upstream of the filter are not recovered quantitatively. Such errors are more significant in the portions of the dilution sampler in contact with the undiluted stack gas sample.

14.2.2 Particulate deposits are dependent upon the design of the sampling equipment and the characteristics of the particulate matter to be sampled (see also Note in Section 12.4.1.4). Therefore, inspect all parts of the sampler in contact with the undiluted and diluted sample for the deposition of particulate matter and, if present, collect the particulate matter by using the rinsing procedures in 12.4.

14.2.3 The residue contained in reagent grade acetone (typically less than 1 part per million by weight) may be significant for some sources with very low particulate matter concentration in the stack gas, such as some natural gas-fired combustion equipment (see Section 16 for additional discussion). The analytical bias introduced by the acetone residue has been shown to be much greater than the analytical uncertainty associated with analyzing the filters by gravimetric methods (N-18). For such sources, it is preferable to neglect deposits upstream of the filter, rather than inflict a relatively large positive bias associated with acetone rinses on the final result, provided the dilution sampler is designed to minimize such losses. If the test objectives dictate the need to recover such deposits, rinsing with acetone following the procedures in Section 12.4 is not recommended. Other procedures with background levels commensurate with the test objectives must be used.

14.2.4 Tests on a No. 6 oil-fired research boiler simulator showed deposits in the undiluted sample components (sample probe, sample flow meter, other passages) averaged 3.1±3.4% (mean ±2 standard deviations) at a PM2.5 concentration of approximately 80 mg/dscm for one dilution sampler designed to minimize such deposits (N 18). Tests using the same dilution sampler on a Diesel engine equipped with a Diesel particulate filter showed such deposits were 8.0±1.8% at a PM2.5 concentration of approximately 4 mg/dscm. Tests using the same dilution sampler at two natural gas-fired sources showed that such deposits were indistinguishable at the 95% confidence level from acetone field reagent blanks taken over several different tests, at PM2.5 concentrations less than approximately 0.2 mg/dscm (filter only).

14.3 Improvement of the Weighing Procedure: see Section 14.3 of ASTM D-6331.

14.4 Gravimetric Filter Handling: Careful handing of the filter during sampling, conditioning, and weighing is necessary to avoid measurement errors due to damaged filters or a gain or loss of collected particles on the filters. Whenever filter are handled, the analyst should wear antistatic, powdered-free gloves; these gloves act as an effective contamination barrier. Gloves that are packed in a box can carry an electrostatic charge. It is a good practice to discharge them by touching a good clean electrical ground after putting them on. The filters should be handled carefully by the support ring, rather than by the filter material, with smooth, nonserrated forceps that are used only for that purpose. Mark these forceps to distinguish them from the forceps used to handle mass reference standards. Forceps should be cleaned with alcohol and lint-free wipes before handling filters and then allowed to air-dry. These precaution reduce the potential effect from body moisture or oils contacting the filters and subsequently affecting the measured weights. If filter loadings will be speciated chemically, the possibility of contamination from gloves should be considered. Some gloves have been found to be contaminated with sulfate. If antistatic gloves are used, ensure that ammonium chloride is not a component of the antistatic reagent. Powder free gloves that are certified to be free of chloride, nitrate, and sulfate are available. In the laboratory, each filter should be transferred from its sealed manufacturer's packaging to a clean filter-handling container, such as a glass or plastic petri dish or petri slides, to reduce the risk of contamination. The filter should remain in the container, except for weighing, until it is loaded into a filter cassette prior to sampling. Each filter should have a unique identification number. If such a number is not provided by the filter manufacture, a label that lists the identification number can be attached to the filter-handling container. It is recommended that each microbalance be assigned a bloc of filter numbers to be processed and used sequentially. Take care to avoid mistakenly assigning the same number twice or omitting a number. Color-coded labels may be helpful for separating groups of filters when more than one microbalance is being used. If sufficient resources are available, barcoding of filters can be introduced. Barcode readers and printers for microbalances are already generally available and can significantly improve the efficiency of filer inventory tracking and processing.

15. Calculations

15.1 Stack gas flow rate: For each run calculate the stack gas flow rate, QS, using the stack cross-sectional area, AS, and average stack gas velocity, VS (see Section 12.3.2), standard m^3/min:

$$Q_S = A_S V_S (T_{STD}/T_S)(P_S/P_{STD}) \quad (1)$$

Where,

A_S = the stack area in the same cross-sectional plane as the stack gas sample measurements, m^2

V_S = the average stack gas velocity in the same cross-sectional plane as the stack gas sample measurements, m/min;

T_S = average stack gas temperature, K;

P_S = average stack gas pressure, K;

T_{STD} is the standard temperature, K;

P_{STD} is the standard pressure, inches of mercury;

15.2 Dilution Ratio--For each run, calculate the dilution ratio, DR:

15.2.1 Calculated from tracer gas measurements (ISO 8178-1); or

15.2.2 Calculated from direct flow measurements:

$$DR = Q_{DA}/Q_{UDS} \quad (2)$$

Where,

Q_{DA} is the flow rate of the dilution air at standard temperature and pressure,

$$Q_{DA}= Q_{MDA}(T_{STD}/T_{MDA})(P_{MDA}/P_{STD}) \quad (3)$$

where:

Q_{MDA} is the measured dilution air flow rate at actual temperature and pressure, m_3/min;

T_{MDA} is the measured dilution air temperature at the flow meter, K;

P_{MDA} is the measured dilution air pressure at the flow meter, inches of mercury;

Q_{UDS} is the flow rate of the undiluted stack gas sample at the flow meter, at standard pressure and temperature, standard m^3/min,

$$Q_{UDS}= Q_{MUDS}(T_{STD}/T_{MUDS})(P_{MUDS}/P_{STD}) \quad (4)$$

where:

Q_{MUDS} is the measured undiluted stack gas sample flow rate at the flow meter, at actual temperature and pressure, m^3/min;

T_{MUDS} is the measured undiluted stack gas sample temperature at the flow meter, K;

P_{MUDS} is the measured undiluted stack gas sample pressure at the flow meter, inches of mercury.

15.3 PM2.5 and/or PM10 concentration--For each run, calculate the PM2.5 and/or the PM10 concentration(s) in the undiluted stack gas sample at standard temperature and pressure, mg/m^3:

$$C_{PM} = (1+DR)(M_{FP} - M_{FT})/V_{FSTD} \quad (5)$$

where,

M_{FP} is the mass of the particulate laden filter(s), mg;

M_{FT} is the tare mass of the filter(s), mg;

V_{FSTD} is the volume of the diluted stack gas, m^3, which passed through the filter corrected to standard temperature and pressure:

$$V_{FSTD} = V_F (T_{STD}/T_{MF})(P_{MF}/P_{STD}) \quad (6)$$

where,

V_F is the measured volume of diluted stack gas that passed thorough the filter, m^3;

T_{MF}= the measured temperature of the diluted stack gas sample at the filter flow meter, m^3.

15.4 For each run, calculate the PM2.5 and/or the PM10 mass emission rate (wet basis), M_{PM}, kg/hr:

$$M_{PM} = C_{PM}\, Qs\, 60\, 10^{-6} \tag{7}$$

16. Precision and Bias

16.1 General:

16.1.1 Because the stack gas composition may vary in the time, it is not possible to determine the repeatability and reproducibility of the test method according to Guide D 3670.

16.1.2 However, if one team performs successive parallel sampling tests with two identical sampling systems, such a procedure allows a statistical comparison between paired values x1 and x2 to be calculated.

16.1.3 The standard deviation of the differences can be calculated as follows:

$$S = (\Sigma(x_1 - x_2)^2/2n)^{0.5} \quad (8)$$

where n is the number of sample pairs.

16.1.4 The repeatability, r, (according to ISO 5725), that is the maximum difference between two measurements by the same team, for a 95 % confidence level is:

$$r = t\, S\, 2^{0.5} \quad (9)$$

where t is the Student factor for a 95% confidence and the degrees of freedom n-1.

16.1.5 When data are provided by several independent teams operating together, similar calculations can be conducted and provided as an estimation of:

16.1.5.1 The external uncertainty linked to an individual measurement carried out by any team fulfilling the requirements of the test method; and

16.1.5.2 The reproducibility (according to ISO 5725), that is the maximum difference, R, that can be expected at a 95 % confidence between two measurements by different teams working according to the test method at the process conditions.

16.1.6 When performing measurements at low level concentrations, the detection limit may be estimated:

16.1.6.1 By parallel measurements and calculation of the uncertainty; and

16.1.6.2 By successive measurements at near zero concentration. The detection limit is assumed to be three times the standard deviation.

16.2 Experimental Data for Diesel Engine Tests and 120 Minute Sampling Duration:

16.2.1 Comparison tests were conducted on a Diesel engine equipped with a catalytic particulate filter with two dilution samplers employing similar design principles but different configurations. Sampler A is a very similar derivative of previously validated reference design (N 17). Sampler B is a compact design (N 18). The measured PM2.5 concentration is approximately 4 mg per standard m3.

16.2.2 Accuracy—Results for 6 co-located samples for two different engine loads showed an average relative difference in PM2.5 concentration of 2.9% for Sampler B relative to Sampler A. The difference is not significant at the 95% confidence level based on a paired t-test.

16.2.3 Precision—The average relative standard deviation of 3 replicate samples at each of two different engine loads was 2 and 9% for Sampler A and Sampler B, respectively. The difference in precision for Sampler A and Sampler B is not significant at the 95% confidence level based on F-tests.

16.2.4 Confidence Interval—The average 95% confidence interval (two standard deviations) was 3 to 5% for Sampler A and 14 to 24% for Sampler B for three replicate samples at each of two different engine loads.

16.2.5 Uncertainty— The uncertainty of average emission rates in pounds per million British thermal units (lb/MMBtu), calculated including estimates of error in all underlying measurements according to ASME Performance Test Code 19.1, was approximately 15 to 16%, at the 95% confidence level.

16.3 Experimental Data for Natural Gas-Fired Sources and 360-Minute Sampling Duration:

16.3.1 Comparison tests with Sampler A and Sampler B were conducted on a natural gas-fired combined cycle power plant (with duct burners, CO oxidation catalyst and selective catalytic NOx reduction emission controls). Tests with Sampler B were conducted at a gas-fired cogeneration plant (with duct burners, CO oxidation catalyst and selective catalytic NOx reduction emission controls). The average PM2.5 concentration was approximately 0.1 to 0.2 mg per dscm. Dilution ratio was approximately 20, 47 mm PTFE filters were used, and the total sample volume through each filter was approximately 27 m^3. Note, acetone rinses were excluded from these results because they could not be distinguished from blanks at the 95% confidence level. (see N 18).

16.3.2 Relative Accuracy—Results for seven co-located samples under two loads at the power plant showed an average relative difference of approximately 6% for Sampler B relative to Sampler A. The difference is not significant at the 95% confidence level.

16.3.3 Bias and Minimum Detection Limit—Background PM2.5 concentration in the filtered dilution air during the power plant site tests was estimated to be approximately 0.04 to 0.09 mg per dscm (10th to 90th percentile), corrected to in-stack concentration at a dilution ratio of approximately

20. This is significant relative to the measured stack sample results. Based on bias due to background levels in the dilution air, the minimum in-stack detection limit (three times standard deviation of system blanks) in these tests is estimated to be 0.06 mg per dscm (mg/dscm) for a six hour test.

Extrapolating this result, the minimum in-stack detection limit for a 1-hour test is estimated to be 0.4 mg/dscm. If background PM2.5 in the dilution air is neglected (i.e., if additional measures are taken to reduce background to negligible levels) and only the analytical detection limit is used, the minimum in-stack detection limit for a 1-hour test is estimated to be 0.04 mg/dscm.

16.3.4 Precision—The PM2.5 relative standard deviation at the power plant for three to four replicate samples at two different process loads was 15 to 29% for Sampler A and 30 to 46% for Sampler B. The difference in precision for Sampler A and Sampler B is not significant at the 95% confidence level based on F-tests. The relative standard deviation of three replicate test runs at a gas-fired cogeneration plant was 23% at an average PM2.5 concentration of approximately 0.2 mg per dry standard m3.

16.3.5 Confidence Interval—The average 95% confidence interval (2 standard deviations) for a four run test at the power plant was approximately ±0.05 mg/dscm (or ±52% of 0.1 mg/dscm). It should be noted that these values represent conservative estimates of measurement precision since they include the combined variability in the measurements, the process, and external factors. Measurement variability is believed to be the dominant source of variation.

16.3.6 Uncertainty—The uncertainty of average emission rates in pounds per million British thermal units (lb/MMBtu), calculated including estimates of error in all underlying measurements according to ASME Performance Test Code 19.1, ranged from 37 to 41% at the power plant and 60% at the cogeneration plant, at the 95% confidence level.

17. Test Report

17.1 The test report shall refer to this test method and shall include the following information:

17.1.1 Description of the purpose of the test, identification of the site, date of sampling, and participants in the test program.

17.1.2 Description of the operating conditions of the plant process and any variation in operating conditions that may have occurred during the test program.

17.1.3 Identification of the sampling location(s) and gas parameters in the stack.

17.1.3.1 Stack dimensions, number and position of velocity measurement points, and the sampling points;

17.1.3.2 Velocity and temperature profiles;

17.1.3.3 O_2 and CO_2 (if relevant), humidity concentrations, gas volumes;

17.1.3.4 Compliance with the requirements of 7.2.

17.1.4 Measurement Procedures:

17.1.4.1 Velocity measurements, if taken, or other stack gas flow rate determinations (calibration of S Pitot tubes, and so forth);

17.1.4.2 Characteristics of sampling equipment: (a) Make (type, manufacturer) and detailed description of the sampling system; (b) Nozzle diameter, characteristic of filter (material, size, type); (c) Calibration of flow rate measurement devices; (d) Filtration temperature; (e) dilution ratio, and (f) response time.

17.1.4.3 Weighing procedures: Conditioning temperature and humidity;.

17.1.5 Test Results:

17.1.5.1 Number, date, duration of runs for each test run: (a) Mass of PM collected; (b) Temperature and relative humidity of the fully diluted stack gas sample in the dilution sampling system; (c) Dilution air temperature, relative humidity and dilution ratio; (d) Sampling duration; (e) Undiluted and diluted stack gas sample volume and flow rate; (f) Initial and final pressure drop across the filter pack; (g) filter face velocity; (h) Any special circumstances or incidents; (i) All intermediate and final calculation results; and (j) Raw results and corrected results (standard conditions).

17.1.6 Quality Assurance:

17.1.6.1 Leak tests results;

17.1.6.2 Blank values;

17.1.6.3 Calibration documentation

17.1.6.4 Dilution ratio verification.

17.1.7 Comments:

17.1.7.1 Record in the test report, the value of the ratio of the measured values to the blanks values.

17.1.7.2 Record in the test report any special circumstances that may have influenced the results and any information concerning the uncertainty of the results.

17.1.73 If it was necessary to modify or deviate from the test method for any reason, report this modification or deviation.

18. Keywords

18.1 PM; PM10; total suspended particulate matter; condensable particulate matter; PM2.5; dilution system sampling; stack sampling.

19. Safety Precautions

19.1 Collection of emissions samples may require working on stack platforms and other elevated surfaces. General safety precautions for working at elevation are specified in the Source Evaluation Society Safety Guidelines Book (N 9).

19.2 Protective clothing (heavy long sleeves and pants, hard hats, and eye protection) should be worn at all times while working in the vicinity of hot surfaces. Insulated gloves should be worn when handling hot probes or accessing stack sampling locations with hot surfaces. Hearing protection should be worn if needed.

19.3 Equipment cleaning requires the use of acetone. This work should be performed in a well-ventilated area to minimize fire and worker exposure hazards.