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US Army Contract DAAE30-02-C-1095
FY 2003 Tasks
WBS # 2.2.2

ASTM Method Development: Dilution Test Method for Determining PM2.5 and PM10 Mass in Stack Gases

Technikon # 1410-222

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(revised for public distribution)

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TECHNIKON
ENVIRONMENTAL DEVELOPMENT CENTER™

Measurement Technologies

Memorandum

December 16, 2004

To: File cc:

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

Subject: US Army Contract DAAE30-02-C-1095, FY2003 Tasks, WBS # 2.2.2

Technikon Measurement Technologies staff has participated as part of an American Society for Testing and Materials (ASTM) subcommittee that is developing a performance based method for the determination of particulate material 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.

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 emissions. PM10 included all particulate material with an apparent aerodynamic diameter of 10µm 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 its own, Technikon staff is co-chairing an ASTM subcommittee to write a method that will provide accurate and precise measurements in a variety of industries. The subcommittee is comprised of representatives from manufacturing, utilities, academia, research consortia, and state and federal environmental agency personnel. There have been two (2) all day meetings and three (3) multiple hour conference calls since the subcommittee's first meeting in March 2004. The sixth (6th) draft of the method has recently been completed. A copy of this current draft is attached.

Technikon's co-chair for this subcommittee's efforts will continue as work products in Subtask 2.2 of the FY2004 tasks under the subject contract. The subcommittee has committed to have a completed method to present to the full ASTM D22 committee at the April 2005 meeting. Following comment from D22 (the ASTM committee for this standard), the subcommittee will submit the method for formal vote at the ASTM October 2005 meeting.

NOTE: The attached document is the Draft Standard that is undergoing review by the ASTM subcommittee. The comments in the margins are comments from the co-chair subcommittee.

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Dilution Test Method for Determining PM2.5 and PM10 Mass in Stack Gases[1]

This standard is issued under the fixed designation X XXXX; 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 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 microns aerodynamic diameter (PM10) or the mass concentrations of particles with aerodynamic diameters between 2.5 and 10 microns. This method is not applicable for the determination of ultrafine particulate (<0.1µ) aerodynamic diameter.

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 of approximately 0.02 mg/dscm has been achieved while testing a natural gas-fired power plant with a 47 mm Teflon membrane filter and 27 dry standard cubic meters (dscm) sample volume and a dilution ratio of approximately 20:1 (20 degrees Celsius standard temperature, 760

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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

millimeters of mercury standard pressure). Including estimated measurement background in the dilution air, an in-stack minimum detection limit of 0.20 mg/dscm has been achieved under the same conditions above.

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 should 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.*

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[1]

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) [1]

D 3631 Test Methods for Measuring Surface Atmospheric Pressure[1]

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-224

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

E 1 Specification for ASTM Thermometers[2]

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 PRE 004 – 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 Analar 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 distribution from stationary combustion sources of coal, oil, and natural gas,: Chang, M.C, Yi, S., Hopke, P.K, England, G. C., Chow, J.C., Watson, J.G. Accepted for publication in Journal of Air and Waste Management Association (2004).

N13 “SCAQMD Method 100.1 – Instrumental Analyzer Procedures for Continuous Gaseous Emission Sampling” SCAQMD Source Test Manual, March 1989.

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

N15 Deleted.

N 16 “§ 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

N 17 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 Science and Technology. 10: pp. 193-204.

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 and then mixed with a stack gas sample in a dilution sampling system

3.2.4 dilution ratio – ratio of the dilution air flow rate through a dilution system to the stack gas sample flow rate through the system.

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 a stack plume.

3.2.6 stack gas sample – undiluted stack gas extracted from the gas 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 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

3.2.11a PM2.5 – particulate matter with an aerodynamic diameter less than 2.5 micrometers and captured on a Teflon filter maintained at a temperature of not greater than 47 ºC during sampling (N16) and weighed at temperature of 20 ºC and a relative humidity of 35 ± 5 percent.

3.2.11b PM10 – PM2.5 - particulate matter with an aerodynamic diameter between 2.5 and 10 micrometers and captured downstream of an in-stack cyclone with a 50 percent cutoff diameter of 10 micrometers and in an in-stack cyclone with a 50 percent cutoff diameter of 2.5 micrometers, and in the connections between the two cyclones (see N6).

3.2.11 c ultrafine particulate matter- Particulate matter with an aerodynamic diameter less than or equal to 0.1 micrometers.

3.2.11d PM – Particulate matter including PM2.5 and/or PM10

3.2.12 sampling point – the specific position in a source vent at which a sample is extracted.

3.2.13 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.

3.2.14 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.

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

3.2.16. Filter holder assembly – filter holder or cassette that contains the filter media for collecting particulate samples.

4. Summary of Test Method

4.1 A schematic of a dilution sampling system is shown in Figure 1.

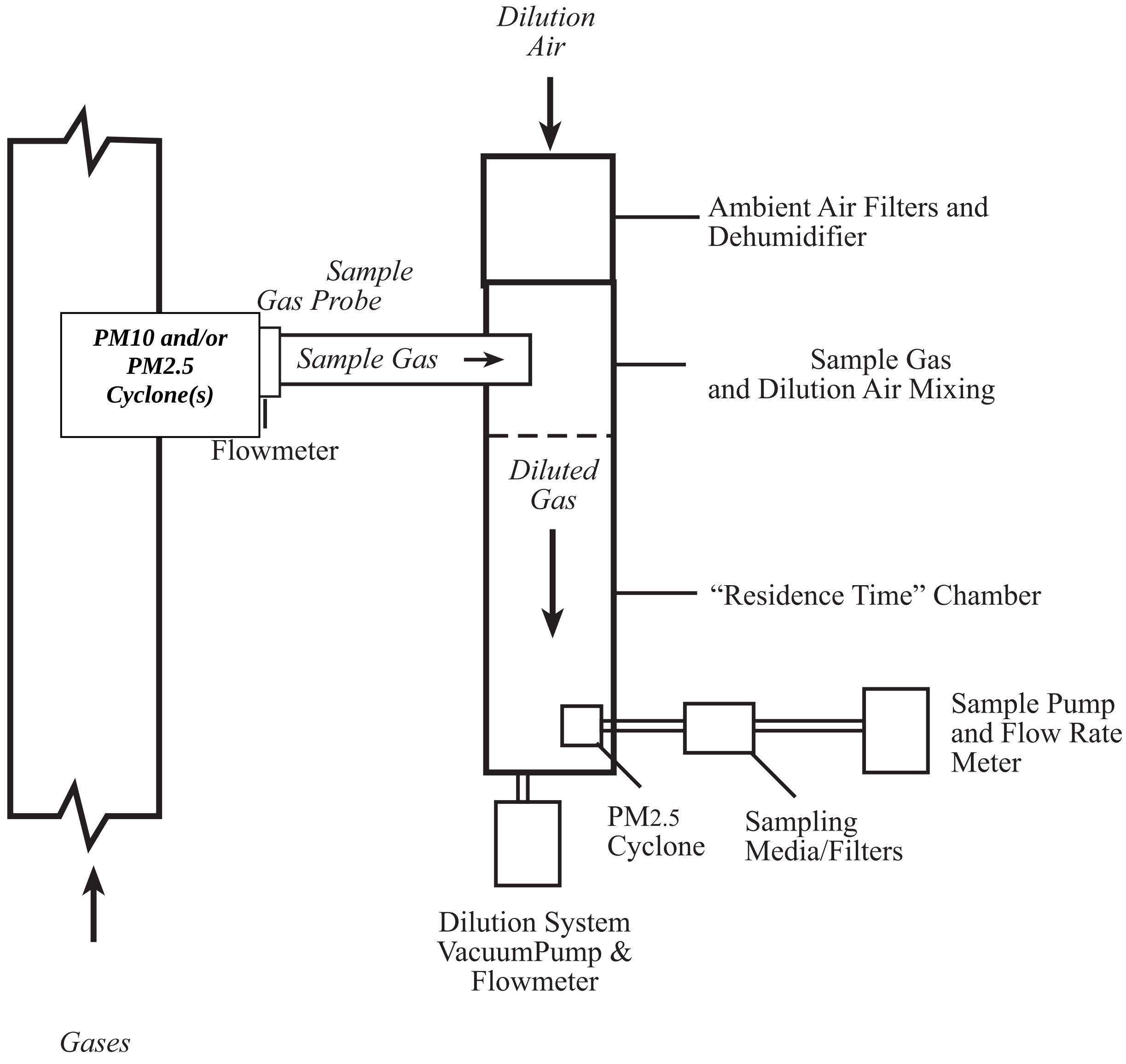


Figure 1. Dilution sampling system schematic

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) 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). The dilution ratio should be at least 20:1 unless higher dilution ratios are required to maintain the particle filter temperature at 47 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 percent 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 Teflon 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. Method performance has not been sufficiently established for demonstrating compliance with regulatory emission limits.

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 The most likely 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. 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. Teflon membrane filters are required because they are less prone to fraying than glass and quartz fiber filters. The Teflon membrane filters shall incorporate a outer diameter polycarbonate support ring. Liquid water can also impact the weights of collected particulate samples. To minimize this effect, the temperature and relative humidity of the filters' weighing chamber are specified to be 22 ºC ± 3 ºC and 45% ± 8%, (9.5 ºC dewpoint ± 1 ºC) respectively. Electrostatic charges can bias filter weights. To minimize static charge effects during filter weighings, static charge shall be neutralized in the weighing chamber prior to weighing by a polonium 210 neutralizer or device of similar effect. 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 condition that can contribute to significant positive bias in filter weight. The oxidation of acid gases (e.g. SO2, NO2) 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 Teflon membrane filters that are less prone to gas adsorption than quartz fiber filters.

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. Grounded electrically conductive surfaces shall be used to minimize electrostatic losses. The sample probe shall be maintained at a temperature at or slightly above the stack gas temperature to minimize thermophoretic losses. Sharp changes in flow direction shall be avoided to minimize inertial losses.

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. (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 micron aerodynamic diameter) and are removed by the dilution sampling system prior to the sampling media. Therefore, the diluted air humidity is controlled below 70%.

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

7. Requirements for Sampling Plane and Sampling Points

7.1 The dilution sampling system sample shall be collected at a location where the stack gases are not stratified (N13). 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 shall be collected from a single point in the stack sufficiently far from the wall to eliminate wall flow disturbances. For PM10, the sample will be withdrawn isokinetically at each traverse point.

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

7.1 2 If stack gas velocity is to be measured using pitot tube methods (D 3154), the sampling location should have a sufficiently homogeneous gas velocity such that the ratio of the highest to lowest local gas velocities is less than 3:1.

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 probe with diameter between 10 mm and 13 mm equipped with an in-stack PM2.5 or PM10 cyclone constructed per EPA Method PRE-004 requirements (N6). The probe wall temperature shall be maintained at or above the source temperature.

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 microns diameter (N7).

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 less than 47ºC ±5ºC.

9.6 Dilution Air Heater (optional) - designed for air flows to give dilution ratios of at least 20:1 and diluted gas temperatures less than 47ºC ± 5ºC.

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 CO2 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 minimum residence time shall be ten (10) seconds after mixing is complete (N12). 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 47 ºC ± 5 ºC immediately upstream of the residence time chamber's cyclone(s).

9.7.3 Constructed of electrically conductive, non-reactive materials such as stainless steel. The system shall be electrically earthed to prevent electrostatic effects and designed to minimize deposition or alteration of the particulates

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

9.7.5 Dilution air vacuum pump with sufficient capacity to maintain specified dilution air flows.

9.8 *Residence chamber sampling system*

9.8.1 Stainless steel cyclone(s) - constructed per EPA Method PRE-004 requirements (N6) and with sufficient flow rate for all sampling media.

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 47ºC 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 holder assembly – constructed of glass, Teflon, or other inert material with a minimum 47 mm stain area. Recommended loading of 465 ug/mm^2 (0.5 mg/1,075 mm2) 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 47 ºC.). The filter holder assembly design shall avoid sample losses that have a significant adverse affect on systematic and random test result variations. For example, the filter holder assembly may comply with the specifications set forth for ambient PM measurement in 40 CFR Part 50, Appendix L 7.3.5, Figures L–25 and L–26, with the following exceptions:

(A) The material shall be 302, 303, or 304 stainless steel instead of anodized aluminum.

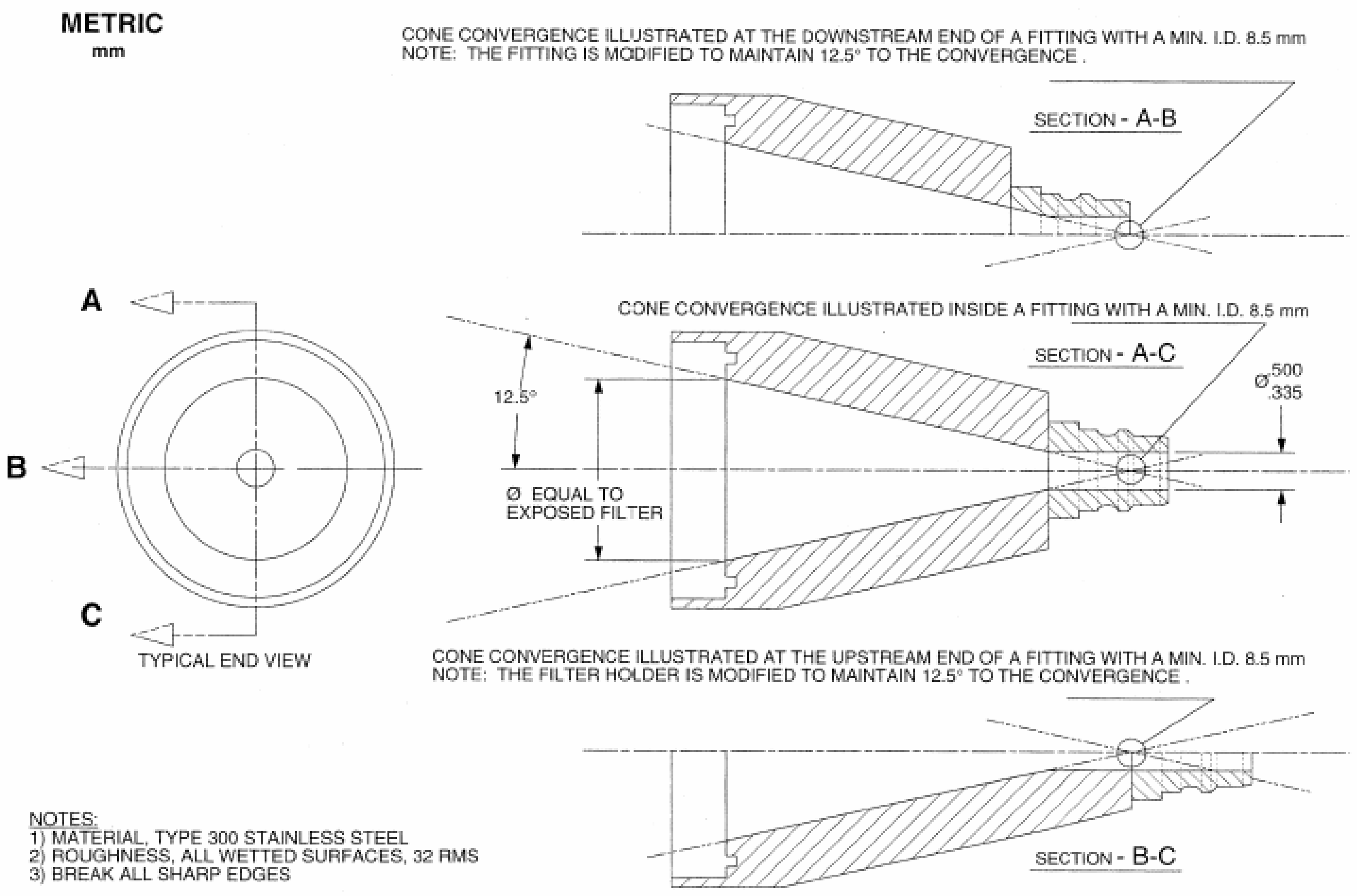
(B) The 2.84 cm diameter entrance to the filter holder may be adapted, using sound engineering judgment and leak free construction, to an inside diameter no smaller than 0.85 cm, maintaining the 12.5º angle from the inlet of the top filter holder to the area near the sealing surface of the top of the filter cartridge assembly. Figure N07–2 shows acceptable variation from the design in 40 CFR Part 50, Appendix L. Similar 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.

(C) If additional or multiple filter cartridges are stored in a particulate sampler as part of an automatic sequential sampling capability, all such filter cartridges, unless they are installed in the sample flow (with or without flow established) shall be covered or sealed to prevent communication of semi-volatile matter from filter to filter; contamination of the filters before and after sampling; or loss of volatile or semi-volatile particulate matter after sampling.

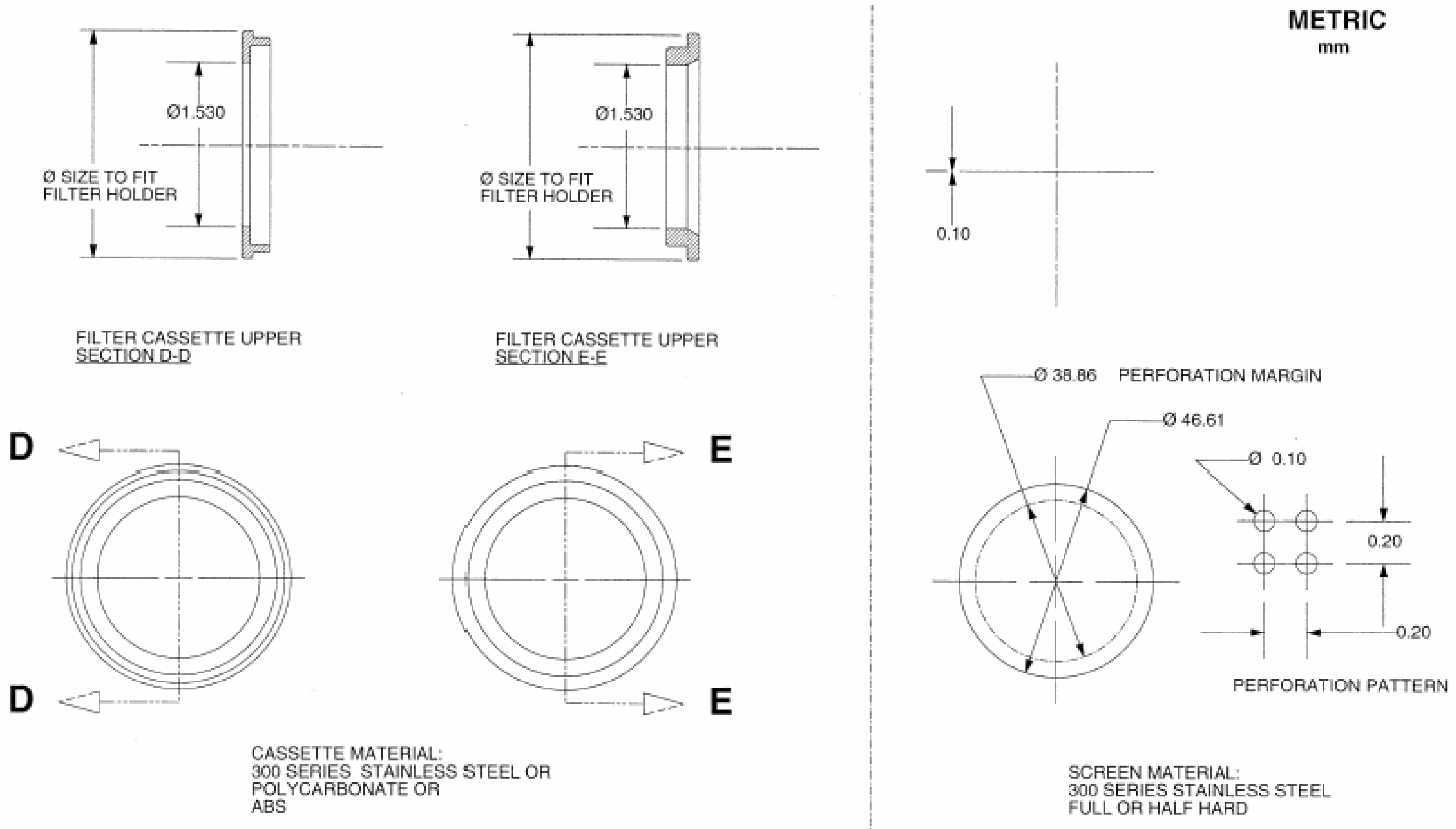
9.8.4 *Filter cartridge assembly.* The filter cartridge assembly shall comply with the specifications set forth for ambient PM measurement in 40 CFR Part 50, Appendix L 7.3.5, figures L–27,L–28, and L–29, with the following exceptions:

(A) In addition to the specified material, 302, 303, or 304 stainless steel, polycarbonate or acrylonitrile/butadiene/styrene (ABS) resin, or a combination of these materials may also be used.

(B) A bevel introduced on the inside diameter of the entrance to the filter cartridge, as used by some commercially available automated sequential particulate filter cartridge changers, is also acceptable (see Figure N07–3).



N07-2 FILTER HOLDER GEOMETRY



N07-3 FILTER CASSETTE AND SCREEN

9.8.5 Sample Flow Measurements – Sample gas flow shall be determined with flow measurement devices accurate to at least ± 4%

9.9 Particulate Filter Analysis System.

9.9.1 Analytical balance

9.9.1.1 Precision: Filter size <70 mm: 1 ug; Filter size ≥70 mm 10 ug

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

Note: The balance should be in climate controlled area with temperature and humidity controls and meters.

9.9.2 System shall be electrically earthed to prevent electrostatic effects.

9.9.3 Humidity meter.

9.9.4 Static charge neutralizer – polonium neutralizer or device of similar effect.

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)

10.1.1 Acetone—Reagent ACS grade acetone in glass bottles with < 0.001 % residue. (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.

10.1.2 Water—Unless otherwise specified, water shall be Type III reagent water conforming to ASTM Specification D 1193.

10.2 Soap

10.3 Kimwipes

10.4 Towels

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

10.6 Teflon tape

10.7 Petri dishes for filters

10.8 Wash bottles – Polyethylene, PTFE, or equivalent

10.9 Shipping containers and materials for filters/petri dishes (Action item, language for filter storage & shipping containers – EB)

10.10 Teflon membrane filter. The filters shall exhibit at least 99.95 % collection efficiency of a 0.3-mm dioctyl phthalate smoke particle, in accordance with Practice D 2986. The manufacturer's quality control test data are sufficient for validation of efficiency.

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

10.11 Personal protective equipment, as needed. See Section 19

11. **Weighing and Pre-Sampling Procedure**

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[2].

11.2 Collect a dilution system blank sample after each measurement series following the procedure described in 13.8.2. Calculate, using the blank sample data, the blank average and repeatability resulting from dilution air background, contamination of filters and rinsing solutions ,and transport, storage and handling in the laboratory. This information will be included in the test report.

11.3 Weighing Procedures: (Action item, Add reference for 1.)gravimetric measurement and 2.) inertial microbalance D.6831-02 – GE&EB)

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 Depending on the measurement program, 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 the ASTM D-6331

12.1.1.2 If using an inertial microbalance for the measurement of particulate mass refer to ASTM D-6831

12.1.3 Perform the weighing procedures according to 11.3.

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.

12.2 Pre-Sampling Measurements:

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

12.2.2 Select an appropriate nozzle for in-stack 10 µm cyclone sampling that provides the correct flow for the cyclone and is within 20% of the Isokinetic rate calculated from the preliminary calculations (11.1.2), the measured stack velocities and other stack parameters (12.2.1). If only a 2.5 µm cyclone is used in-stack, 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 by drawing a vacuum

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[2] Historical data since DSB not known before test. Run and analyze DSB before test if no historical data exists.

on the sample probe and in the dilution sampler equal to the maximum vacuum experienced during a run, and measuring the sample flow (leak) rate for a period of approximately 5 minutes. If the leak rate before the test run is greater than 2 percent of the total flow through the dilution chamber during a test, then the source(s) of the leak(s) must be identified and eliminated until the leak rate is less than the value above. If the leak rate after the test run is greater than 2 percent 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 Collect volumetric flow measurement data either before and after, or during sampling, whichever is appropriate for method used. Stack flow may be measured by pitot tube traverse per ASTM D 3154; stack oxygen, fuel flow and composition measurement, carbon balance method (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 within the stack. 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 the target dilution ratio (Section 9.3). The dilution ratio shall be a minimum of 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 . 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 temperature of dilution air as needed. Adjust the humidity of dilution air to produce a diluted sample gas relative humidity of 70% or less.

12.3.6 Verify the dilution ratio – Methods include: 1.) Direct measurement of flows (sample stack gas, dilution air, total diluted air - at least 2 of the 3 needed); 2.) Measurement of CO2, NOx, VOCs, or other parameter in the sample stack gas, dilution air, total diluted air, and calculation of dilution ratio via mass balance.

12.3.7 After the dilution tunnel temperature has stabilized to ± 2 ºC for three (3) minutes, turn on PM filter sample system pump and adjust the flow to the target rate. The filter face velocity should be less than 100cm/sec (Action item, references for filter pack face velocities – GE)

12.3.8 Collect PM on the filter for the target sampling time. Target sampling times will vary based on process conditions and the level of particulate in the stack. Sampling times of thirty (30) to sixty (60) minutes may be sufficient for continuous processes. Batch processes may require sampling one (1) or more process cycles. Record dilution tunnel and sample system operating conditions (temperature, flow rates, humidity, dilution ratio, filter change in pressure) at a minimum of twelve (12) equally spaced intervals over the sampling period.

12.3.9 After PM sampling is completed, turn off sampling pump and record final sample volumes and/or flow rates. Shut down dilution sampling system flows after recording final operating conditions. Perform post-run leak check.

12.3.10 Recover sample filter holders with filters intact after the test run, seal containers tightly, and affix sample label with unique identifier to each sample. Record sample information on a suitable sample tracking or chain of custody form. Pack the samples for shipment or transfer to the laboratory. The container material shall be sufficiently electrically conductive to avoid dust losses from the filter due to electrostatic charge effects. (Action item, reference ambient methods for sample storage and shipment – GE)

12.3.10.1 Inspect sample lines for condensed material, record observations.

12.4 Recovery of Deposits Upstream of the Filter:

12.4.1 A clean environment is recommended for the following procedure. Exercise special care to avoid contamination of the sample if the procedure is performed on-site.

12.4.2 Rinse the sampling assembly components that are in contact with the undiluted sample gas with water to recover the deposits. Collect the rinse into a storage bottle. Rinse these same surfaces with acetone and collect the acetone into the same bottle.

12.4. 3 Do not mechanically clean surfaces to recover deposits upstream of the filter after sampling. However, it is necessary to mechanically clean and rinse the equipment before the next measuring series is initiated.

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 will 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 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 Temperatures - Calibrate thermocouples at least every three months or whenever a system repair or change is made that could influence calibration. Thermocouple should be calibrated against a NIST traceable standard and agreement should be within ±2 K.

13. 4 Relative humidity meters – Calibrate relative humidity meters at least every month or whenever a system repair or change is made that could influence calibration. Meter should be calibrated against a NIST traceable standard and agreement should be within ±3% of reading

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13.8 QA/QC Activities

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

13.8.2 Pre-test dilution sampling system blank – One per test condition. Filtered dilution air is drawn through the dilution sampling system without stack gas.

13.8.3 Ambient Air Sample (optional but recommended) – One per sampling location. Collected at the sampling location by directly pulling unfiltered ambient air samples through the sampling media only.

13.8.4 Field blank – Collected by assembling and disassembling sampling equipment without drawing any gas through the media.

13.8.5 Leak checks per 12.6.2.

13.8.6 Check dilution ratio. The measured dilution ratio in the diluted stack gas shall be within 10% of the dilution ratio calculated from CO2, NOx, or other tracer gas concentration measured in the stack gas and in the dilution sampling system stack.

13.8.7 Verify that the gases in the dilution sampling system are uniformly mixed under the test conditions. Mixing may be verified by measurement of the CO2 or other tracer gas profile upstream of the aging tunnel or any sample collection media at a minimum of four (4) points across the stack diameter. All measurements shall be within ± 10% of the mean concentration in the stack Alternately, the degree of mixing may be warranted by the equipment manufacturer/vendor as described above.

13.8.8 The test team shall meet the criteria specified in D-7036

14. Additional Aspects Discussion

14.1 *Sampling Duration and Detection Limit:*

14.1.1 Instack detection limits will vary with the sampling time, filter face velocity, and the objectives of the test.

14.2 *Particulate Deposits Upstream of the Filter:*

14.2.1 Errors can be introduced into the measurements if particulate deposits upstream of the filter are not recovered carefully.

14.2.1.1 Deposits in the undiluted portion of the sampling system shall be collected by the rinsing procedures in 12.4.

14.2.2 Particulate deposits are dependent upon the design of the sampling equipment and the characteristics of the particulate matter to be sampled. Therefore, check all non-weighed parts for the deposition of particulate matter. If there is observable deposition, collect the particulate matter by the rinsing procedures in 12.4.

14.3 Improvement of the Weighing Procedure:

14.3.1 Experience has shown that weighing uncertainties are not only related to the balance performance but to the whole procedure employed. Therefore, before performing any measurement, the user shall establish and validate a procedure, taking into account the sampling equipment and filters to be used.

14.3.2 Repeated weighing of the same filters, spread over several weeks in various conditions, that is, external temperature, humidity, and so forth, will provide an estimation of the actual precision of weighing, including the uncertainties related to the manipulation of the filters, equilibrium time, and so forth.

14.3.3 Use the results as a first estimate of the overall blank value, and provide a means of calculation of the gas volume to be sampled in order to obtain significant data, taking into account the anticipated range of particulate concentrations.

15. Calculations

15.1 Qs = stack gas flow rate, scm/min.

Where: scm/min = standard cubic meters per minute. .

15.2 DR = dilution ratio

15.2.1 Calculated from tracer gas measurements (TBD)

15.2.2 Calculated from direct flow measurements. DR = $Q_{DEX}/Q_{EX} = (Q_{DA} + Q_{EX})/Q_{EX}$, where:

- Q_{DEX} is the flow rate of the diluted stack gas at standard conditions, scm/min = $Q_{MDEX} * (T_{STD}/T_M)*(P_M/P_{STD})$, where:
- Q_{MDEX} is the measured diluted stack gas flow rate, cm/min;
- T_{STD} is the standard conditions temperature, K;
- T_M is the measured diluted stack gas temperature, K;
- P_{STD} is the standard conditions pressure, in Hg;
- P_M is the measured diluted stack gas pressure, in Hg;
- cm/min = cubic meters/min.
- Q_{DA} is the flow rate of the dilution air at standard conditions, scm/min = $Q_{MDA} * (T_{STD}/T_M)*(P_M/P_{STD})$, where:
- Q_{MDA} is the measured dilution air flow rate, cm/min;
- Q_{EX} is the flow rate of the stack gas sample at standard conditions, scm/min = $Q_{MEX} * (T_{STD}/T_M)*(P_M/P_{STD})$, where:
- Q_{MEX} is the measured stack gas sample flow rate, cm/min;

15.3 C_{PM} = PM Concentration in Stack Gas, mg/scm = DR * (M_{FP} – M_{FT})/ V_{FSTD}, 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 that passed through the filter corrected to standard conditions = V_F * (T_{STD}/T_M)*(P_M/P_{STD}), scm, where:
- V_F is the measured volume of diluted stack gas that passed thorough the filter, cm;
- cm = cubic meters.

15.4 M_{PM} = PM mass emission rate = C_{PM} * Qs * 60 * 10^{-6}, kg/hr.

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 = (sum of ((x1 – x2)^2)/2n)^0.5; where:

n, the number of sample pairs, may be used for the calculation of the repeatability confidence interval r = t*(2S)^0.5, where t is the Student factor for a 95 % confidence and the degrees of freedom n-1.

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:

$$r = t * s * 2^{0.5}$$

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.

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.

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.

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 point;

17.1.3.2 Velocity and temperature profiles;

17.1.3.3 O2 and CO2 (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) 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: (a) Mass of PM collected; (b) Temperature of the fully diluted stack gas sample in the dilution sampling system; (c) Dilution air temperature, dilution ratio, and gas composition (including moisture content); (d) Sampling time; (e) Sampling flow rate and sampled volume; (f) Initial and final change in pressure 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.

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

18. Keywords

18.1 PM; 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 (N9).

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. 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 hazards..