

**ENVIRONMENTAL PROTECTION AGENCY****40 CFR Part 51**

[EPA-HQ-OAR-2008-0348; FRL-8784-5]

RIN 2060-AO58

**Methods for Measurement of Filterable PM<sub>10</sub> and PM<sub>2.5</sub> and Measurement of Condensable Particulate Matter Emissions From Stationary Sources****AGENCY:** Environmental Protection Agency (EPA).**ACTION:** Proposed rule.

**SUMMARY:** This action proposes amendments to Methods 201A and 202. The proposed amendments to Method 201A would add a particle-sizing device to allow for sampling of particulate matter (PM) with mean aerodynamic diameters less than or equal to 2.5 micrometers (µm) (PM<sub>2.5</sub> or fine PM). The proposed amendments to Method 202 would revise the sample collection and recovery procedures of the method to reduce the formation of reaction artifacts that could lead to inaccurate measurements of condensable particulate matter (CPM). Additionally, the proposed amendments to Method 202 would eliminate most of the hardware and analytical options in the existing method, thereby increasing the precision of the method and improving the consistency in the measurements obtained between source tests performed under different regulatory authorities. Finally, in this notice we are soliciting comments on whether to end the transition period for CPM in the New Source Review (NSR) program on a date earlier than the current end date of January 1, 2011. The proposed amendments would improve the measurement of fine particulates and would help State and local agencies in implementing CPM control measures to attain the PM<sub>2.5</sub> National Ambient Air Quality Standards (NAAQS) which were established to protect public health and welfare.

**DATES:** *Comments.* Comments must be received on or before May 26, 2009.

**ADDRESSES:** Submit your comments, identified by Docket ID Number EPA-HQ-OAR-2008-0348, by one of the following methods:

- *http://www.regulations.gov.* Follow the on-line instructions for submitting comments.
- *E-mail:* Send your comments via electronic mail to *a-and-r-docket@epa.gov*.
- *Fax:* (202) 566-9744.
- *Mail:* Methods for Measurement of Filterable PM<sub>10</sub> and PM<sub>2.5</sub> and

Measurement of Condensable Particulate Matter Emissions from Stationary Sources, Environmental Protection Agency, Mailcode 2822T, 1200 Pennsylvania Ave., NW., Washington, DC 20460. Please include a total of two copies.

- *Hand Delivery:* EPA Docket Center EPA Headquarter Library, Room 3334, EPA West Building, 1301 Constitution Ave., NW., Washington, DC, 20460. Such deliveries are accepted only during the Docket's normal hours of operation, and special arrangements should be made for deliveries of boxed information.

*Instructions:* Direct your comments to Docket ID No. EPA-HQ-OAR-2008-0348. EPA's policy is that all comments received will be included in the public docket without change and may be made available online at *http://www.regulations.gov*, including any personal information provided, unless the comment includes information claimed to be Confidential Business Information (CBI) or other information whose disclosure is restricted by statute. Do not submit information that you consider to be CBI or otherwise protected through *http://www.regulations.gov* or e-mail. The *http://www.regulation.gov* Web site is an "anonymous access" system, which means EPA will not know your identity or contact information unless you provide it in the body of your comment. If you send an e-mail comment directly to EPA without going through *http://www.regulations.gov*, your e-mail address will be automatically captured and included as part of the comment that is placed in the public docket and made available on the Internet. If you submit an electronic comment, EPA recommends that you include your name and other contact information in the body of your comment and with any disk or CD-ROM you submit. If EPA cannot read your comment due to technical difficulties and cannot contact you for clarification, EPA may not be able to consider your comment. Electronic files should avoid the use of special characters, any form of encryption, and be free of any defects or viruses. For additional information about EPA's public docket, visit the EPA Docket Center homepage at *http://www.epa.gov/epahome/dockets.htm*.

*Docket:* All documents in the docket are listed in the *http://www.regulations.gov* index. Although listed in the index, some information is not publicly available, e.g., CBI or other information whose disclosure is restricted by statute. Certain other material, such as copyrighted material, will be publicly available only in hard

copy. Publicly available docket materials are available either electronically in *http://www.regulations.gov* or in hard copy at the Methods for Measurement of Filterable PM<sub>10</sub> and PM<sub>2.5</sub> and Measurement of Condensable Particulate Matter Emissions from Stationary Sources Docket, EPA/DC, EPA West Building, Room 3334, 1301 Constitution Ave., NW., Washington, DC. The Public Reading Room/Docket Center is open from 8:30 a.m. to 4:30 p.m., Monday through Friday, excluding legal holidays. The telephone number for the Public Reading Room is (202) 566-1744, and the telephone number for the Air Docket Center is (202) 566-1742.

*Public Hearing:* If anyone contacts EPA requesting to speak at a public hearing concerning our proposal to revise the PM test methods by April 14, 2009, we will hold a public hearing on or about April 24, 2009. Persons interested in presenting oral testimony should contact Ms. Kristal Mozingo, Measurement Policy Group (D243-05), Sector Policies and Programs Division, EPA, Research Triangle Park, NC 27711, *telephone number:* (919) 541-9767, *e-mail address:* *mozingo.kristal@epa.gov*. Persons interested in attending the public hearing should also call Ms. Mozingo to verify the time, date, and location of the hearing. A public hearing will provide interested parties the opportunity to present data, views, or arguments concerning the proposed test method revisions.

If a public hearing is held, it will be held at 10 a.m. at the Conference Facilities at EPA's Main Campus, Research Triangle Park, NC, or an alternate site nearby.

**FOR FURTHER INFORMATION CONTACT:** For general information, contact Ms. Candace Sorrell, U.S. EPA, Office of Air Quality Planning and Standards, Air Quality Assessment Division, Measurement Technology Group (E143-02), Research Triangle Park, NC 27711; *telephone number:* (919) 541-1064; *fax number:* (919) 541-0516; *e-mail address:* *sorrell.candace@epa.gov*. For technical questions, contact Mr. Ron Myers, U.S. EPA, Office of Air Quality Planning and Standards, Sector Policies and Programs Division, Measurement Policy Group (D243-05), Research Triangle Park, NC 27711; *telephone number:* (919) 541-5407; *fax number:* (919) 541-1039; *e-mail address:* *myers.ron@epa.gov*.

**SUPPLEMENTARY INFORMATION:**

**I. General Information****A. Does This Action Apply to Me?**

This action would apply to you if you operate a stationary source that is subject to applicable requirements for total PM or total PM<sub>10</sub> where EPA Method 202 is incorporated as a component of the applicable compliance method.

In addition, this action would apply to you if Federal, State, or local agencies take certain additional independent actions. For example, this action would

apply to sources through actions by State and local agencies which implement CPM control measures to attain the PM<sub>2.5</sub> NAAQS and specify the use of this test method to demonstrate compliance with the control measure. Actions that State and local agencies would have to implement include: (1) Adopting this method in rules or permits (either by incorporation by reference or by duplicating the method in its entirety), and (2) promulgating an emissions limit requiring the use of this method (or an incorporated method

based upon this method). This action would also apply to stationary sources that are required to meet new applicable CPM requirements established through Federal or State permits or rules, such as New Source Performance Standards and New Source Review, which specify the use of this test method to demonstrate compliance with the control measure.

The source categories and entities potentially affected include, but are not limited to, the following:

Category	SIC <sup>1</sup> code	NAICS <sup>2</sup> code	Examples of potentially regulated entities
Industry .....	3569	332410	Fossil fuel steam generators.
	3569	332410	Industrial, commercial, institutional steam generating units.
	3569	332410	Electricity generating units.
	2911	324110	Petroleum refineries.
	4953	562213	Municipal waste combustors.
	2621	322110	Pulp and paper mills.
	2819	325188	Sulfuric acid plants.
	3241	327310	Portland Cement Plants.
	3274	327410	Lime Manufacturing Plants.
	1222	211111	Coal Preparation Plants.
	1231	212111	
		212112	
		212113	
	3334	331312	Primary and Secondary Aluminum Plants.
	3341	331314	
	3312	331111	Iron and Steel Plants.
	3325	331513	
	2493	321219	Plywood and Reconstituted Products Plants.
	2435	321211	
	2436	321212	

<sup>1</sup> Standard Industrial Classification.

<sup>2</sup> North American Industrial Classification System.

**B. What Should I Consider as I Prepare My Comments for EPA?**

Do not submit information containing CBI to EPA through <http://www.regulations.gov> or e-mail. Send or deliver information identified as CBI only to the following address: Roberto Morales, OAQPS Document Control Officer (C404-02), U.S. EPA, Office of Air Quality Planning and Standards, Research Triangle Park, NC 27711, Attention Docket ID No. EPA-HQ-OAR-2008-0348. Clearly mark the part or all of the information that you claim to be CBI. For CBI information on a disk or CD-ROM that you mail to EPA, mark the outside of the disk or CD-ROM as CBI, and then identify electronically within the disk or CD-ROM the specific information that is claimed as CBI. In addition to one complete version of the comment that includes information claimed as CBI, a copy of the comment that does not contain the information claimed as CBI must be submitted for inclusion in the public docket. Information so marked will not be

disclosed except in accordance with procedures set forth in 40 CFR part 2.

**C. Where Can I Obtain a Copy of This Action and Other Related Information?**

In addition to being available in the docket, an electronic copy of today's proposed amendments is also available on the Worldwide Web (<http://www.epa.gov/ttn/>) through the Technology Transfer Network (TTN). Following the Administrator's signature, a copy of the proposed amendment will be posted on the TTN's policy and guidance page for newly proposed or promulgated rules at <http://www.epa.gov/ttn/oarpg>. The TTN provides information and technology exchange in various areas of air pollution control.

**D. How Is This Document Organized?**

The information in this preamble is organized as follows:

**I. General Information****A. Does This Action Apply to Me?****B. What Should I Consider as I Prepare My Comments for EPA?****C. Where Can I Obtain a Copy of This Action and Other Related Information?****D. How Is This Document Organized?****II. Background****A. Why Is EPA Issuing This Proposed Rule?****B. Particulate Matter National Ambient Air Quality Standards****C. Measuring PM Emissions****1. Method 201A****2. Method 202****III. This Action****A. What Are the Proposed Amendments to Method 201A?****B. What Are the Proposed Amendments to Method 202?****C. How Will the Proposed Amendments to Methods 201A and 202 Affect Existing Emission Inventories, Emission Standards, and Permit Programs?****D. Request for Comments****1. Items Associated With Both Test Methods****2. Items Associated With Method 201A****2. Items Associated With Method 202****IV. Statutory and Executive Order Reviews****A. Executive Order 12866: Regulatory Planning and Review****B. Paperwork Reduction Act****C. Regulatory Flexibility Act****D. Unfunded Mandates Reform Act****E. Executive Order 13132: Federalism**

- F. Executive Order 13175: Consultation and Coordination With Indian Tribal Governments
- G. Executive Order 13045: Protection of Children From Environmental Health and Safety Risks
- H. Executive Order 13211: Actions Concerning Regulations That Significantly Affect Energy Supply, Distribution, or Use
- I. National Technology Transfer and Advancement Act
- J. Executive Order 12898: Federal Actions To Address Environmental Justice in Minority Populations and Low-Income Populations

## II. Background

### A. Why Is EPA Issuing This Proposed Rule?

On April 25, 2007 (70 FR 20586), we promulgated the Clean Air Fine Particle Implementation Rule regarding the Clean Air Act (CAA) requirements for State and Tribal plans to implement the 1997 PM<sub>2.5</sub> NAAQS. These rules require that each State having a PM<sub>2.5</sub> nonattainment area must submit, by April 5, 2008, an attainment demonstration and adopt regulations to ensure the area will attain the standards as expeditiously as practicable, but even those areas for which the Administrator determines an extension from the 2010 attainment date is appropriate may not receive an extension later than a 2015 attainment date. The emissions inventories and analyses used in the attainment demonstrations must consider filterable and condensable fractions of PM<sub>2.5</sub> emissions from stationary sources that are significant contributors of direct PM<sub>2.5</sub> emissions. Direct PM<sub>2.5</sub> emissions means the solid particles or liquid droplets emitted directly from an air emissions source or activity, or the gaseous emissions or liquid droplets from an air emissions source or activity that condense to form PM or liquid droplets at ambient temperatures.

The preamble to the April 25, 2007, rule acknowledged that there remain questions whether the available test methods provide the most accurate representation of primary PM emissions even though some States have established emissions limits for CPM. As a result, the final rule established a transitional period for developing emissions limits and regulations for condensable PM<sub>2.5</sub>. During this transitional period, EPA has committed to devote resources to assessing and improving the available test methods for CPM.

In response to this commitment and to address the need for improved measurement of fine PM, EPA is proposing amendments to the following

test methods in 40 CFR Part 51, Appendix M (Recommended Test Methods for State Implementation Plans (SIPs)):

- Method 201A—Determination of PM<sub>10</sub> Emissions (Constant Sampling Rate Procedure), and
- Method 202—Determination of Condensable Particulate Emissions from Stationary Sources.

These amendments to Method 201A add a particle-sizing device to allow for sampling of PM<sub>2.5</sub>, PM<sub>10</sub>, or both PM<sub>10</sub> and PM<sub>2.5</sub>. With regard to Method 202, we are aware that the method and the various hardware and analytic options described therein are sometimes applied inappropriately, which can lead to inaccurate and imprecise CPM measurements. We are also aware that Method 202 can produce inaccurate CPM measurements when sampling certain types of emissions sources, due to formation of reaction artifacts. The amendments to Method 202 revise the sample collection and recovery procedures of the method to provide for more accurate and precise measurement of CPM.

### B. Particulate Matter National Ambient Air Quality Standards

Section 108 and 109 of the CAA govern the establishment and revision of the NAAQS. Section 108 (42 U.S.C. 7408) directs the Administrator to identify and list “air pollutants” that “in his judgment, may reasonably be anticipated to endanger public health and welfare” and whose “presence \* \* \* in the ambient air results from numerous or diverse mobile or stationary sources” and to issue air quality criteria for those that are listed. Air quality criteria are intended to “accurately reflect the latest scientific knowledge useful in indicating the kind and extent of identifiable effects on public health or welfare which may be expected from the presence of [a] pollutant in ambient air\* \* \*.” Section 109 (42 U.S.C. 7409) directs the Administrator to propose and promulgate primary and secondary NAAQS for pollutants listed under section 108 to protect public health and welfare, respectively. Section 109 also requires review of the NAAQS at 5-year intervals and that an independent scientific review committee “shall complete a review of the criteria \* \* \* and the national primary and secondary ambient air quality standards \* \* \* and shall recommend to the Administrator any new \* \* \* standards and revisions of existing criteria and standards as may be appropriate \* \* \*.” Since the early 1980s, this independent review function has been performed by the Clean Air

Scientific Advisory Committee (CASAC).

Initially EPA established the NAAQS for PM on April 30, 1971 (36 FR 8186) based on the original criteria document (Department of Health, Education, and Welfare, 1969). The reference method specified for determining attainment of the original standards was the high-volume sampler, which collects PM up to a nominal size of 25 to 45 μm (referred to as total suspended particulates or TSP). On October 2, 1979 (44 FR 56730), EPA announced the first periodic review of the air quality criteria and NAAQS for PM, and significant revisions to the original standards were promulgated on July 1, 1987 (52 FR 24634). In that decision, EPA changed the indicator for particles from TSP to PM<sub>10</sub>. When that rule was challenged, the court upheld revised standards in all respects. *Natural Resources Defense Council v. Administrator*, 902 F. 2d 962 (D.C. Cir. 1990, *cert. denied*, 498 U.S. 1082 (1991)).

In April 1994, EPA announced its plans for the second periodic review of the air quality criteria and NAAQS for PM, and the Agency promulgated significant revisions to the NAAQS on July 18, 1997 (62 FR 38652). In that decision, EPA revised the PM NAAQS in several respects. While EPA determined that the PM NAAQS should continue to focus on particles less than or equal to 10 μm in diameter (PM<sub>10</sub>), EPA also determined that the fine and coarse fractions of PM<sub>10</sub> should be considered separately. The EPA added new standards, using PM<sub>2.5</sub> as the indicator for fine particles (with PM<sub>2.5</sub> referring to particles with a nominal mean aerodynamic diameter less than or equal to 2.5 μm), and using PM<sub>10</sub> as the indicator for purposes of regulating the coarse fraction of PM<sub>10</sub>.

Following promulgation of the 1997 PM NAAQS, petitions for review were filed by a large number of parties, addressing a broad range of issues. In May 1999, a three-judge panel of the U.S. Court of Appeals for the District of Columbia Circuit issued an initial decision that upheld EPA’s decision to establish fine particle standards. *American Trucking Associations v. EPA*, 175 F.3d 1027, 1055 (D.C. Cir. 1999), reversed in part on other grounds in *Whitman v. American Trucking Associations*, 531 U.S. 457 (2001). The Panel also found “ample support” for EPA’s decision to regulate coarse particle pollution but vacated the 1997 PM<sub>10</sub> standards, concluding that EPA had not provided a reasonable explanation justifying use of PM<sub>10</sub> as an indicator for coarse particles. *Id. at 1054–55*. Pursuant to the court’s

decision, EPA removed the vacated 1997 PM<sub>10</sub> standards but retained the pre-existing 1987 PM<sub>10</sub> standards (65 FR 80776, December 22, 2000).

On October 23, 1997, EPA published its plans for the third periodic review of the air quality criteria and NAAQS for PM (62 FR 55201), including the 1997 PM<sub>2.5</sub> standards and the 1987 PM<sub>10</sub> standards. On October 17, 2006, EPA issued its final decisions to revise the primary and secondary NAAQS for PM to provide increased protection of public health and welfare, respectively (71 FR 61144). With regard to the primary and secondary standards for fine particles, EPA revised the level of the 24-hour PM<sub>2.5</sub> standard to 35 µg per cubic meter (µg/m<sup>3</sup>), retained the level of the annual PM<sub>2.5</sub> annual standard at 15 µg/m<sup>3</sup>, and revised the form of the annual PM<sub>2.5</sub> standard by narrowing the constraints on the optional use of spatial averaging. With regard to the primary and secondary standards for PM<sub>10</sub>, EPA retained the 24-hour PM<sub>10</sub> standard (150 µg/m<sup>3</sup>) and revoked the annual standard because available evidence generally did not suggest a link between long-term exposure to current ambient levels of coarse particles and health or welfare effects.

### C. Measuring PM Emissions

Section 110 of the CAA, as amended (42 U.S.C. 7410), requires that State and local air pollution control agencies develop and submit plans for EPA approval that provide for the attainment, maintenance, and enforcement of the NAAQS in each air quality control region (or portion thereof) within such State. These plans are known as SIPs. 40 CFR part 51 (Requirements for Preparation, Adoption, and Submittal of Implementation Plans) specifies the requirements for SIPs. Appendix A to subpart A of 40 CFR part 51, defines primary PM<sub>10</sub> and PM<sub>2.5</sub> as including both the filterable and condensable fractions of PM. Filterable PM consists of those particles that are directly emitted by a source as a solid or liquid at the stack (or similar release conditions) and captured on the filter of a stack test train. Condensable PM is the material that is in vapor phase at stack conditions but which condenses and/or reacts upon cooling and dilution in the ambient air to form solid or liquid PM immediately after discharge from the stack.

Promulgation of the 1987 NAAQS created the need for methods to quantify PM<sub>10</sub> emissions from stationary sources. In response, EPA developed and promulgated the following test methods:

- Method 201A—Determination of PM<sub>10</sub> Emissions (Constant Sampling Rate Procedure), and
- Method 202—Determination of Condensable Particulate Emissions from Stationary Sources.

#### 1. Method 201A

On April 17, 1990 (56 FR 65433), EPA promulgated Method 201A in Appendix M of 40 CFR Part 51 to provide a test method for measuring filterable PM<sub>10</sub> emissions from stationary sources. In EPA Method 201A, a gas sample is extracted at a constant flow rate through an in-stack sizing device which directs particles with aerodynamic diameters less than or equal to 10 µm to a filter. The particulate mass collected on the filter is determined gravimetrically after removal of uncombined water. With the exception of the PM<sub>10</sub>-sizing device, the current Method 201A sampling train is the same as the sampling train used for EPA Method 17 of Appendix A-3 to 40 CFR Part 60.

Method 201A cannot be used to measure emissions from stacks that have entrained moisture droplets (e.g., from a wet scrubber stack) since these stacks may have water droplets that are larger than the cut size of the PM<sub>10</sub>-sizing device. The presence of moisture would prevent an accurate measurement of total PM<sub>10</sub> since any PM<sub>10</sub> dissolved in larger water droplets would not be collected by the sizing device and would consequently be excluded in determining the total PM<sub>10</sub> mass. To measure PM<sub>10</sub> in stacks where water droplets are known to exist, EPA's Technical Information Document (TID) 09 (Methods 201 and 201A in Presence of Water Droplets), recommends use of Method 5 of Appendix A-3 to 40 CFR Part 60 (or a comparable method) and consideration of the total particulate catch as PM<sub>10</sub> emissions.

Method 201A is also not applicable for stacks with small diameters (*i.e.*, 18 inches or less). The presence of the in-stack nozzle/cyclones and filter assembly in a small duct will cause significant cross-sectional area interference and blockage leading to incorrect flow calculation and particle size separation. Additionally, the type of metal used to construct the Method 201A cyclone may limit the applicability of the method when sampling at high stack temperatures (e.g., stainless steel cyclones are reported to gall and seize at temperatures greater than 260 °C).

#### 2. Method 202

On December 17, 1991 (56 FR 65433), EPA promulgated Method 202 in Appendix M of 40 CFR Part 51 to

provide a test method for measuring CPM from stationary sources. Method 202 uses water-filled impingers to cool, condense, and collect materials that are vaporous at stack conditions and become solid or liquid PM at ambient air temperatures. Method 202, as promulgated, contains several optional procedures that were intended to accommodate the various test methods used by State and local regulatory entities at the time Method 202 was being developed.

When conducted consistently and carefully, Method 202 provides acceptable precision for most emission sources, and the method has been used successfully in regulatory programs where the emission limits and compliance demonstrations are established based on a consistent application of Method 202 and its associated options. However, when the same emission source is tested using different combinations of the optional procedures, there may appear to be large variations in the measured CPM emissions. Additionally, during validation of the promulgated method, we determined that sulfur dioxide (SO<sub>2</sub>) gas (a typical component of emissions from several types of stationary sources) can be absorbed partially in the impinger solutions and can react chemically to form sulfuric acid. This sulfuric acid "artifact" is not related to the primary emission of CPM from the source but may be counted erroneously as CPM when using Method 202. As we have maintained consistently, the artifact formation can be reduced by at least 90 percent if a one-hour nitrogen purge of the impinger water is used to remove SO<sub>2</sub> before it can form sulfuric acid (this is our preferred application of the Method 202 optional procedures). Inappropriate use (or omission) of the preferred or optional procedures in Method 202 can increase the potential for artifact formation.

Considering the potential for variations in measured CPM emissions, we believe that further verification and refinement of Method 202 is appropriate to minimize the potential for artifact formation. We have performed several studies to assess artifact formation when using Method 202. The results of our 1998 laboratory study and field evaluation commissioned to evaluate the impinger approach can be found in "Laboratory and Field Evaluation of the EPA Method 5 Impinger Catch for Measuring Condensable Matter from Stationary Sources" at the following Internet address: <http://www.epa.gov/ttn/emc/methods/m202doc1.pdf>. Essentially, the 1998 study verified the need for a nitrogen purge when SO<sub>2</sub> is

present in stack gas and also provided guidance for analyzing the collected samples. In 2005, an EPA contractor conducted a second study ("Laboratory Evaluation of Method 202 to Determine Fate of SO<sub>2</sub> in Impinger Water") that replicated some of the earlier EPA work and addressed some additional issues. The report of that work is available at the following Internet address: <http://www.epa.gov/ttn/emc/methods/m202doc2.pdf>. This report also verified the need for a nitrogen purge and identified the primary factors that affect artifact formation.

Also in 2005, a private testing contractor presented a possible minor modification to Method 202 at the Air and Waste Management Association (AWMA) specialty conference. The proposed modification, described in their presentation titled "Optimized Method 202 Sampling Train to Minimize the Biases Associated with Method 202 Measurement of Condensable Particulate Matter Emissions," involved the elimination of water from the first impingers. The presentation (which is available at the following Internet address: <http://www.epa.gov/ttn/emc/methods/m202doc3.pdf>) concluded that modification of the promulgated method to use dry impingers resulted in a significant additional reduction in the sulfate artifact.

In 2006, we began to conduct laboratory studies, in collaboration with several stakeholders, to characterize the artifact formation and other uncertainties associated with conducting Method 202 and to identify procedures that would minimize uncertainties when using Method 202. Since August 2006, we have held two workshops in Research Triangle Park, North Carolina. These meetings were held to present and seek comments on our plan for evaluating potential modifications to Method 202 that would reduce artifact formation. Also, these meetings were held to discuss our progress in characterizing the performance of the modified method, issues that require additional investigation, the results of our laboratory studies, and our commitments to extend the investigation through stakeholders external to EPA. We held another meeting with experienced stack testers and vendors of emissions monitoring equipment to discuss hardware issues associated with modifications of the sampling equipment and the glassware for the proposed CPM test method. Summaries of the method evaluations, as well as meeting minutes from our workshops, can be found at the

following Internet address: <http://www.epa.gov/ttn/emc/methods/method202.html>.

The laboratory studies that were performed fulfill a commitment in the preamble to the Clean Air Fine Particle Implementation Rule (72 FR 20586, April 25, 2007) to examine the relationship between several critical CPM sampling and analysis parameters and, to the extent necessary, propose revisions to incorporate improvements in the method. While these improvements in the stationary source test method for CPM will provide for more accurate and precise measurement of all PM, the addition of PM<sub>2.5</sub> as an indicator of health and welfare effects by the 1997 NAAQS revisions generates the need to quantify PM<sub>2.5</sub> emissions from stationary sources. To respond to this need, we are proposing revisions to incorporate this capability into the test method for filterable PM<sub>10</sub>.

### III. This Action

This action proposes to provide the capability of measuring PM<sub>2.5</sub> using Method 201A and to provide for more accurate measurement of the filterable and condensable components of fine PM (particles with mean aerodynamic diameters less than or equal to 2.5 m) and coarse PM (particles with mean aerodynamic diameters less than or equal to 10 m) when using Method 202. Method 201A proposed amendments would add a particle-sizing cyclone to the sampling train. Method 202 proposed amendments would reduce the formation of sulfuric acid artifact by at least an additional 90 percent (compared to our recommended procedures for the existing Method 202), provide for greater consistency between testing contractors in method application, improve the precision of the method, and provide for more accurate quantification of direct (i.e., primary) PM emissions to the ambient air (the method will not measure secondarily-formed PM). The proposed amendments would also affect the measurement of total PM, PM<sub>10</sub>, and PM<sub>2.5</sub>. Additionally, we are proposing to revise the format of Methods 201A and 202 to be consistent with the format developed by EPA's Environmental Monitoring Management Council (EMMC). A guidance document describing the EMMC format can be found at the following Internet address: <http://www.epa.gov/ttn/emc/guidlnd/gd-045.pdf>.

#### A. What Are the Proposed Amendments to Method 201A?

On July 18, 1997 (62 FR 38652), we revised the NAAQS for PM to add new

standards for fine particles, using PM<sub>2.5</sub> as the indicator. This action will modify the current Method 201A sampling train configuration to allow for measurement of filterable PM<sub>10</sub>, filterable PM<sub>2.5</sub>, or both filterable PM<sub>10</sub> and filterable PM<sub>2.5</sub> from stationary sources. These amendments combine the existing method with the PM<sub>2.5</sub> cyclone to create a sampling train that includes a total of two cyclones (one cyclone to size particles with aerodynamic diameters greater than 10 m and one cyclone to size particles with aerodynamic diameters greater than 2.5 m) and a final filter to collect particles with aerodynamic diameters less than or equal to 2.5 m. The PM<sub>2.5</sub> cyclone would be inserted between the PM<sub>10</sub> cyclone and the filter of the Method 201A sampling train.

We are not proposing any amendments to address the use of this method when the stack gas has entrained moisture or when the method is used for stack gases with high temperatures. In July 1979, we published a research document (EPA-600/7-79-166) to report the preliminary development of a method for measuring and characterizing the particles in the vent stream from a wet scrubber used to control sulfur oxide emissions. The method was based on the use of a heated, electrified wire placed in the vent stream. When a water droplet impacted the wire, the electric current flowing through the wire was attenuated in proportion to the size of the water droplet. We decided it was not appropriate to promulgate the preliminary method and, at this time, we are not aware of any commercially-available equipment that can determine the aerodynamic size of PM contained in, or dissolved in, liquid water droplets as they would exist in the ambient air following release and evaporation in the ambient air. While we are aware of several optical aerosol droplet spectrometers for measuring the size distribution of liquid droplets in exhaust gases, we are not aware of any commercial instruments that can measure size distributions of particles emitted from stationary sources. We also lack knowledge on the relative effects of solids concentration in the liquid droplets and the possible presence of dry particles in addition to the liquid droplets. Consequently, we recommend the use of EPA Method 5 (40 CFR Part 60, Appendix A-3—Determination of Particulate Matter Emissions from Stationary Sources) when measuring PM in stacks with saturated water vapors containing entrained water droplets. With this application of EPA Method 5,

all of the collected material would be considered PM<sub>2.5</sub>.

#### *B. What Are the Proposed Amendments to Method 202?*

This action proposes amendments incorporating modifications that would reduce the formation of artifacts at both low and high concentrations of SO<sub>2</sub> in the sample gas stream. The modifications were developed based on the method evaluations discussed in Section II.C.2 of this preamble.

Method 202, as promulgated in 1991, is a set of sampling procedures for collecting PM in water-filled impingers and a set of sample recovery procedures that are performed on the water following its collection. The water-filled impingers are nearly identical to the four chilled impingers used in standard stationary source sampling trains for PM (e.g., Method 5 and Method 17 of Appendix A-3 and A-6, 40 CFR Part 60). In principle, CPM is collected in the impinger portion of a Method 17-type sampling train. Our preferred operation of the promulgated method requires that the impinger contents be purged with nitrogen after the test run to remove dissolved SO<sub>2</sub> gas from the impinger contents. The impinger solution is then extracted with methylene chloride to separate the organic CPM from the inorganic CPM. The organic and aqueous fractions are then dried and the residues weighed. The sum of both fractions represents the total CPM.

These proposed amendments to Method 202 sampling train and sample recovery procedures would achieve at least an additional 90 percent reduction in sulfuric acid artifact formation compared to the current Method 202 using the nitrogen purge option, provide testing contractors with a more standardized application of the method, improve the precision of the method, and quantify more accurately direct PM emission to the ambient air.

The proposed changes to the sampling train of this method include:

- Installing a condenser between the filter in the front-half of the sample train and the first impinger to cool the sample gases to ambient temperature (less than 30 °C);
- Installing a recirculation pump in the ambient water bath to supply cooling water to the condenser;
- Changing the first two impingers from wet to dry, and placing these two dry impingers in a water bath at ambient temperature (less than 30 °C) (the first dry impinger will use a short-stem insert, and the second dry impinger will use a long-stem insert);
- Requiring the use of an out-of-stack, low-temperature filter (*i.e.*, the CPM

filter), as described in EPA Method 8, between the second and third impingers (a Teflon filter is used in place of the fiberglass filter described in EPA Method 8); and

- Requiring that the temperature of the sample gas drawn through the CPM filter be maintained at ambient temperature (less than 30 °C).
- It should be noted that under Method 202, the use of a CPM filter is an optional procedure that is used typically if the collection efficiency of the impinger is suspected to be low. These proposed amendments would make the use of a CPM filter a required procedure.

The proposed changes to Method 202 include:

- Extracting the CPM filter with water and organic solvent;
- Evaporating the liquid collected in the impingers in an oven or on a hot plate down to a minimum volume of 10 milliliters, instead of all the way to dryness;
- Evaporating the remaining liquid to dryness at ambient temperature prior to neutralization with ammonium hydroxide;
- Titrating the reconstituted residue with 0.1 normal ammonium hydroxide and a pH meter;
- Evaporating the neutralized liquid to a minimum volume of 10 milliliters in an oven or hot plate;
- Evaporating the final volume to dryness at ambient temperature; and
- Weighing the CPM sample residue to constant weight after allowing a minimum of 24 hours for equilibration in a desiccator.

Note that the requirements to evaporate liquids at ambient temperature and to titrate the reconstituted liquid exist already as options under this method. These optional steps are typically performed to retain CPM that might be lost at higher evaporation temperatures. Under these proposed amendments, these options would be required procedures.

#### *C. How Will the Proposed Amendments to Methods 201A and 202 Affect Existing Emission Inventories, Emission Standards, and Permit Programs?*

We anticipate that, over time, the changes in the test methods proposed in this action will result in, among other positive outcomes, more accurate emissions inventories of direct PM emissions and emissions standards that are more indicative of the actual impact of the source on the ambient air quality.

Accurate emission inventories are critical for regulatory agencies to develop the control strategies and demonstrations necessary to attain air

quality standards. If implemented, the proposed test method revisions would have the potential to improve our understanding of PM emissions due to the increased availability of more accurate emission tests and, eventually, through the incorporation of less biased test data into existing emissions factors. For CPM, the use of the proposed method would likely reveal a reduced level of CPM emissions from a source compared to the emissions that would have been measured using Method 202, as typically performed. However, there may be some cases where the proposed test method would reveal an increased level of CPM emissions from a source, depending on the relative emissions of filterable and CPM emissions from the source. For example, the existing Method 202 allows complete evaporation of the water containing inorganic PM at 105 °C (221 °F), where the proposed revision requires the last 10 ml of the water to be evaporated at room temperature (not to exceed 30 °C (85 °F)) thereby retaining the CPM that would evaporate at the increased temperature.

Prior to our adoption of the 1997 PM<sub>2.5</sub> NAAQS, several State and local air pollution control agencies had developed emission inventories that included CPM. Additionally, some agencies established enforceable CPM emissions limits or otherwise required that PM emissions testing include measurement of CPM. While this approach was viable in cases where the same test method was used to develop the CPM regulatory limits and to demonstrate facility compliance, there are substantial inconsistencies within and between States regarding the completeness and accuracy of CPM emission inventories and the test methods used to measure CPM emissions and to demonstrate facility compliance.

These amendments would serve to mitigate the potential difficulties that can arise when we and other regulatory entities attempt to use the test data from State and local agencies whose CPM test methods are inconsistent to develop emission factors, determine program applicability, or to establish emissions limits for CPM emission sources within a particular jurisdiction. For example, problems can arise when the test method used to develop a CPM emission limit is not the same as the test method specified in the rule for demonstrating compliance because the different test methods may quantify different components of PM (e.g., filterable versus condensable). Also, when emissions from State inventories are modeled to assess compliance with

the NAAQS, the determination of direct PM emissions may be biased high or low, depending on the test methods used to estimate PM emissions, and the atmospheric conversion of SO<sub>2</sub> to sulfates (or SO<sub>3</sub>) may be inaccurate or double-counted. Additionally, some State and local regulatory authorities have assumed that EPA Method 5 of Appendix A–3 to 40 CFR Part 60 (Determination of Particulate Matter Emissions from Stationary Sources) provides a reasonable estimate of PM<sub>10</sub> emissions. This assumption is incorrect because Method 5 does not provide particle sizing of the filterable component and does not quantify particulate caught in the impinger portion of the sampling train. Similar assumptions for measurements of PM<sub>2.5</sub> will result in greater inaccuracies.

With regard to State permitting programs, we recognize that, in some cases, existing Best Available Control Technology (BACT), Lowest Achievable Emission Rate (LAER), or Reasonably Available Control Technology (RACT) limits have been based on an identified control technology, and that the data used to determine the performance of that technology and establish the limits may have focused on filterable PM and thus did not completely characterize PM emissions to the ambient air. While the source test methods used by State programs that developed the applicable permit limit may not have fully characterized the PM emissions, we have no information that would indicate that the test methods are inappropriate indicators of the control technologies' performance for the portion of PM emissions that was addressed by the applicable requirement. As promulgated in the Clean Air Fine Particle Implementation Rule, after January 1, 2011, States are required to consider inclusion of CPM emissions in new or revised emissions limits which they establish. We will defer to the individual State's judgment as to whether, and at what time, it is appropriate to revise existing facility emission limits or operating permits to incorporate information from the revised CPM test method when it is promulgated.

With regard to operating permits, the Title V permit program does not generally impose new substantive air quality control requirements. In general, once emissions limits are established as CAA requirements under the SIP or a SIP-approved pre-construction review permit, they are included in the Title V permits. Obviously, Title V permits may have to be updated to reflect any revision of existing emission limits or new emission limits created in the

context of the underlying applicable requirements. Also, if a permit contains the previously promulgated test methods, it is not a given that the permit would always have to be revised should these test methods changes be finalized (e.g., where test methods are incorporated into existing permits through incorporation by reference, no permit terms or conditions would necessarily have to change to reflect changes to those test methods). In any event, the need for action in the permitting context due to these proposed changes to the test methods would be controlled by several factors, such as the exact wording of the existing operating permit, the requirements of the EPA-approved SIP, and any changes that may be made to pre-construction review permits with respect to a particular source test method that did not include CPM or on a set of procedures in Method 202 which underestimated emissions.

In recognition of these issues, the Clean Air Fine Particle Implementation Rule contains provisions establishing a transition period for developing emission limits for condensable direct PM<sub>2.5</sub> that are needed to demonstrate attainment of the PM<sub>2.5</sub> NAAQS. As discussed in the April 25, 2007, Clean Air Fine Particle Implementation Rule (72 FR 20586) and in the May 16, 2008, promulgation of the New Source Review Program Implementation for fine particulate matter (73 FR 28321), the transition period, which ends January 1, 2011, allows time to resolve and adopt appropriate testing procedures for CPM emissions and to collect total primary (filterable and condensable) PM<sub>2.5</sub> emissions data that are more representative of the emissions of each source in their areas. In the PM<sub>2.5</sub> NSR Implementation Rule, we stated that as part of this test methods rulemaking, we would "take comment on an earlier closing date for the transition period in the NSR program if we are on track to meet our expectation to complete the test method rule much earlier than January 1, 2011." See 73 FR at 28344. Accordingly, we are hereby soliciting comments on ending the NSR transition period for CPM on a date 60 to 90 days after the promulgation date of this test methods rulemaking.

During the transition period, we are available to provide technical support to States, as requested, in establishing emissions testing requirements. We will also solicit the involvement of interested stakeholders to collect new direct filterable and CPM emissions data using methodologies that provide more representative data of a source's direct PM<sub>2.5</sub> emissions. These data will be

used by us, States, and others to improve emissions factors and to help establish or revise source emissions limits in implementation plans. The transition period will also provide time for additional method evaluations. During the transition period, we expect that some States will continue to develop more complete inventories of direct PM<sub>2.5</sub> emissions, particularly for CPM. As needed to demonstrate attainment of the PM NAAQS, we also expect States to address the control of direct PM<sub>2.5</sub> emissions, including CPM, with any new actions taken after January 1, 2011 and to address CPM emissions in any direct PM<sub>2.5</sub> regulations or limits developed under any new PM NAAQS.

As with other methods, any new procedures approved by us will produce data that will be incorporated into the tools (e.g., emission factors, emission inventories, air quality modeling) used to assess the attainment of air quality standards. However, we do not believe that it is necessary to update continually the assessment tools or revise previous air quality analyses until evidence is presented that a mid-course corrective action is needed to achieve the air quality standards (a mid-course review is required by April 2011 for each area with an approved attainment date in 2014 or 2015). At that time, updated inventories and air quality models may be needed to identify and characterize the emission sources that are impeding adequate progress towards attaining the air quality standards. Additionally, the new test data could be used to improve the applicability and performance evaluations of various control technologies.

#### *D. Request for Comments*

We encourage stakeholders to continue to participate in the process to refine Methods 201A and 202. We are requesting public comments on all aspects of the proposed test methods. EPA has already engaged several stakeholder groups as described in Section II.C of this preamble. Stakeholders and other members of the public who have not yet participated are encouraged to submit comments. EPA is soliciting as many constructive comments as possible in order to make the most appropriate changes to the methods.

We are specifically interested in recommended alternatives to replace what we have proposed. When submitting comments on alternative approaches, please submit supporting information to substantiate the improvements that are achieved with your recommendation. For



recommended changes to the procedures, include supporting technical data and any associated cost information. For example, if you are proposing an alternative procedure, include data or information that would demonstrate how the alternative procedure would equal or improve the bias and precision of the proposed methods. In addition, provide data or cost information that would show the cost implications to testing companies and analytical laboratories of implementing the alternative procedure. Although our request for comments is not limited to these items, the following are examples of items for which we are specifically requesting comment.

#### 1. Items Associated With Both Test Methods

The proposed test methods are based upon EPA's assessment of comments made on the Clean Air Fine Particle Implementation Rule (April 25, 2007, 70 FR 20586). Commenters expressed that there is an overarching need for test methods that are unbiased with respect to primary particulate matter emissions to the atmosphere and that the test methods must provide a high degree of consistency (precision) in these measurements. As a result, we reduced the numerous options and alternative procedures in the existing methods to a single set of prescriptive procedures that already existed within the methods. In addition, we made a few minor changes to reduce further the bias caused by sulfate artifacts. We are requesting comments on the specific set of procedures we have proposed and any replacement procedures that would be less demanding but that would achieve or improve bias and precision. We are also requesting comments on our decision to eliminate options or alternatives within the existing methods that may not achieve comparable results. If we were to consider alternative procedures that may not achieve comparable results, then what level of difference would be acceptable?

#### 2. Items Associated With Method 201A

Regarding this proposed method, stakeholders have commented on the sample duration that would be required to collect a weighable mass. EPA is requesting comments on alternative methodologies or hardware that would reduce the sample duration in order to reach a reasonable detection limit or to demonstrate that emissions are below the regulatory limit. Commenters should provide information or data, including cost information, which supports their recommendation.

Stakeholders have expressed concern about the configuration and size of the proposed sampling train. Specifically, commenters have expressed concern that the size and length of the combined PM<sub>10</sub> cyclone and the PM<sub>2.5</sub> cyclone and filter require larger port opening(s) and a very large stack cross section to minimize blockage. In addition, stakeholders have stated that it is difficult to maintain stack temperature in the sampling train. Therefore, EPA requests comments on alternatives to the proposed procedures or hardware. EPA requests comments on alternative procedures or configurations that would reduce the blockage. EPA also requests comments on alternative configurations that would allow testers to maintain stack temperature in the sampling train, thus reducing or eliminating condensation in the primary or filterable particulate portions of the method. Recommendations to revise the sampling train size or configuration should include an assessment of the impacts of the recommended revisions on the sample size, required sample duration, and ability to collect a representative sample. Commenters should provide information or data, including cost information that supports their recommendation.

#### 3. Items Associated With Method 202

Stakeholders originally expressed concern about the formation of artifacts in Method 202 when sulfur dioxide was present in the stack gas. Based on laboratory experiments, the proposed revision to Method 202 eliminates at least an additional 90 percent of the artifact over the best practices procedures of the existing Method 202. In addition, the laboratory experiments show that the proposed revision to Method 202 reduces artifact at or below the detection limits of the method. EPA requests comments on any further concerns with the formation of artifacts in the proposed method.

Stakeholders have expressed concern about glassware cleaning. Specifically, stakeholders have questioned the requirement to bake glassware at 300 °C for 6 hours prior to use in order to reduce the background level of CPM. Stakeholders have stated that many stack testing firms and some analytical laboratories may not have ovens that can achieve this temperature. EPA requests information on the performance of a lower temperature oven in effectively reducing the blank level of CPM.

Another stakeholder concern is whether glassware needs to be completely cleaned between sampling runs. The proposed method requires

clean glassware at the start of each new source category test. EPA requests comments on alternatives that would minimize the cost of glassware preparation and reduce bias due to carryover from tests at the same source category and between source categories. Commenters should submit data or information to demonstrate that their alternative procedure would reduce or minimize the carryover or blank and would minimize the cost to prepare glassware.

Stakeholders expressed concern about the need for Method 202 following filtration at less than 30 °C (85 °F). EPA requests comments on how to clarify when Method 202 is or is not required.

Stakeholders have expressed concern about the appropriate type of CPM filter required by the proposed method. EPA requests comments on the construction material and porosity of the filter. Commenters should address the capture efficiency required by the method (i.e., the filter must have an efficiency of at least 99.95 percent (<0.05 percent penetration) on 0.3 micron particles). Commenters should include how their alternative would minimize the blank contribution from the filters.

Commenters have expressed concern about the additional analytical steps required to process the CPM filter. The proposed method requires extraction and combination of the filter extract with the appropriate impinger samples to accurately collect and measure sulfuric acid and other condensable material. Commenters should address alternative procedures for CPM filter analysis that would generate precise and unbiased analysis of CPM collected on the CPM filter.

Stakeholders have expressed concern about maintaining the stack gas flow through the Teflon® membrane filter. Stakeholders have commented on their need to use a supplementary support filter to maintain flow through the sample filter. EPA requests comments regarding the use of a support filter that would help maintain stack gas flow while minimizing or eliminating the support filter's contribution to the sample mass. EPA requests comments on the use of this alternative and its potential impact on bias and precision, as well as its potential impact on cost.

## IV. Statutory and Executive Order Reviews

### A. Executive Order 12866: Regulatory Planning and Review

Under Executive Order (EO) 12866 (58 FR 51735, October 4, 1993), this proposed action is a "significant regulatory action" since it raises novel



legal or policy issues arising out of legal mandates, the President's priorities, or the principles set forth in this Executive Order. Accordingly, EPA submitted this proposed action to the Office of Management and Budget (OMB) for review under Executive Order 12866 and any changes made in response to OMB recommendations have been documented in the docket for this action.

#### *B. Paperwork Reduction Act*

This proposed action does not impose an information collection burden under the provisions of the *Paperwork Reduction Act*, 44 U.S.C. 3501 *et seq.* Burden is defined at 5 CFR 1320.3(b). The proposed amendments do not contain any reporting or recordkeeping requirements. The proposed amendments revise two existing source test methods to allow one method to perform additional particle sizing at 2.5 micrometers and to improve the precision and accuracy of the other test method.

#### *C. Regulatory Flexibility Act*

The Regulatory Flexibility Act (RFA) generally requires an agency to prepare a regulatory flexibility analysis of any rule subject to notice and comment rulemaking requirements under the Administrative Procedure Act or any other statute unless the agency certifies that the rule will not have a significant economic impact on a substantial number of small entities. Small entities include small businesses, small organizations, and small governmental jurisdictions.

For purposes of assessing the impacts of this rule on small entities, small entity is defined as: (1) A small business as defined by the Small Business Administration's (SBA) regulations at 13 CFR 121.201; (2) a small governmental jurisdiction that is a government of a city, county, town, school district or special district with a population of less than 50,000; and (3) a small organization that is any not-for-profit enterprise which is independently owned and operated and is not dominant in its field.

After considering the economic impacts of this proposed rule on small entities, I certify that this action will not have a significant economic impact on a substantial number of small entities. We do not anticipate that the proposed changes to Methods 201A and 202 will result in a significant economic impact on small entities. Most of the emission sources that will be required by State regulatory agencies (and Federal regulators after 2011) to conduct tests using the revised methods are those that

have PM emissions of 100 tons per year or more. EPA expects that few, if any, of these emission sources will be small entities.

Although this proposed rule will not have a significant economic impact on a substantial number of small entities, EPA nonetheless has tried to reduce the impact of this rule on small entities. In this preamble, we explained that this rule does not require any entities to use these proposed test methods. Such a requirement would be mandated by a separate independent regulatory action. We indicated that upon promulgation of this rule, some entities may be required to use these test methods as a result of existing permits or regulations. Since the cost to use the proposed test methods is comparable to the cost of the methods they replace, little or no significant economic impact to small entities will accompany the increased precision and accuracy of the revised test methods which are proposed. We also indicated that after January 1, 2011, when the transition period established in the Clean Air Fine Particle Implementation Rule expires, States are required to consider inclusion of pollutants measured by these test methods in new or revised regulations. The economic impacts caused by any new or revised State regulations for fine PM would be associated with those State rules and not with this proposal to modify the existing test methods. Consequently, we believe that this rule imposes little if any adverse economic impact to small entities. However, we continue to be interested in the potential impacts of the proposed rule on small entities and welcome comments on issues related to such impacts.

#### *D. Unfunded Mandates Reform Act*

This rule does not contain a Federal mandate that may result in expenditures of \$100 million or more for State, local, and tribal governments, in the aggregate, or the private sector in any one year. The incremental costs associated with conducting the revised test methods (expected to be less than \$1,000 per test) do not impose a significant burden on sources. Thus, this rule is not subject to the requirements of sections 202 and 205 of the UMRA.

This rule is also not subject to the requirements of section 203 of UMRA because it contains no regulatory requirements that might significantly or uniquely affect small governments. The low incremental cost associated with the revised test methods mitigates any significant or unique effects on small governments.

#### *E. Executive Order 13132: Federalism*

Executive Order 13132, entitled "Federalism" (64 FR 43255, August 10, 1999), requires EPA to develop an accountable process to ensure "meaningful and timely input by State and local officials in the development of regulatory policies that have federalism implications." "Policies that have federalism implications" is defined in the Executive Order to include regulations that have "substantial direct effects on the States, on the relationship between the national government and the States, or on the distribution of power and responsibilities among the various levels of government."

This proposed rule does not have federalism implications. It will not have substantial direct effects on the States, on the relationship between the national government and the States, or on the distribution of power and responsibilities among the various levels of government, as specified in Executive Order 13132. In cases where a source of PM<sub>2.5</sub> emissions is owned by a State or local government, those governments may incur a minimal compliance costs associated with conducting tests to quantify PM<sub>2.5</sub> emissions using the revised methods when they are promulgated. However, such tests would be conducted at the discretion of the State or local government and the compliance costs are not expected to impose a significant burden on those governments. Thus, Executive Order 13132 does not apply to this rule.

In the spirit of Executive Order 13132, and consistent with EPA policy to promote communications between EPA and State and local governments, EPA specifically solicits comment on this proposed rule from State and local officials.

#### *F. Executive Order 13175: Consultation and Coordination With Indian Tribal Governments*

This action does not have tribal implications, as specified in Executive Order 13175 (65 FR 67249, November 9, 2000). In cases where a source of PM<sub>2.5</sub> emissions is owned by a tribal government, those governments may incur minimal compliance costs associated with conducting tests to quantify PM<sub>2.5</sub> emissions using the revised methods when they are promulgated. However, such tests would be conducted at the discretion of the tribal government and the compliance costs are not expected to impose a significant burden on those governments. Thus, Executive Order 13175 does not apply to this action.

EPA specifically solicits additional comment on this proposed rule from tribal officials.

*G. Executive Order 13045: Protection of Children From Environmental Health and Safety Risks*

EPA interprets EO 13045 (62 FR 19885, April 23, 1997) as applying only to those regulatory actions that concern health or safety risks, such that the analysis required under section 5–501 of the EO has the potential to influence the regulation. This action is not subject to EO 13045 because it does not establish an environmental standard intended to mitigate health or safety risks.

*H. Executive Order 13211: Actions Concerning Regulations That Significantly Affect Energy Supply, Distribution, or Use*

This action is not a “significant energy action” as defined in Executive Order 13211 (66 FR 28355 (May 22, 2001)), because it is not likely to have a significant adverse effect on the supply, distribution, or use of energy. This rule revises existing EPA test methods and does not affect energy supply, distribution, or use.

*I. National Technology Transfer and Advancement Act*

Section 12(d) of the National Technology Transfer and Advancement Act of 1995 (“NTTAA”), Public Law 104–113 (15 U.S.C. 272 note) directs EPA to use voluntary consensus standards (VCS) in its regulatory activities unless to do so would be inconsistent with applicable law or otherwise impractical. Voluntary consensus standards are technical standards (*e.g.*, materials specifications, test methods, sampling procedures, and business practices) that are developed or adopted by voluntary consensus standards bodies. NTTAA directs EPA to provide Congress, through OMB, explanations when the Agency decides not to use available and applicable voluntary consensus standards.

The rulemaking involves technical standards. Therefore, the Agency conducted a search to identify potentially applicable voluntary consensus standards. However, we identified no such standards, and none were brought to our attention in comments. Therefore, EPA has decided to amend portions of existing EPA test methods. While no comprehensive source test methods were identified, EPA identified two VCS which were applicable for use within the amended test methods. The first VCS cited in this proposal is American Society for Testing and Materials (ASTM) Method

D2986–95a (1999), “Standard Method for Evaluation of Air, Assay Media by the Monodisperse DOP (Diocetyl Phthalate) Smoke Test,” for its procedures to conduct filter efficiency tests. The second VCS cited in this proposed rule is ASTM D1193–06, “Standard Specification for Reagent Water,” for the proper selection of distilled ultra-filtered water. These VCS are available from the American Society for Testing and Materials, 100 Barr Harbor Drive, Post Office Box C700, West Conshohocken, PA 19428–2959.

EPA welcomes comments on this aspect of the proposed rulemaking and, specifically, invites the public to identify potentially applicable VCS and to explain why such standards should be used in this regulation.

*J. Executive Order 12898: Federal Actions To Address Environmental Justice in Minority Populations and Low-Income Populations*

Executive Order (EO) 12898 (59 FR 7629, February 16, 1994) establishes federal executive policy on environmental justice. Its main provision directs federal agencies, to the greatest extent practicable and permitted by law, to make environmental justice part of their mission by identifying and addressing, as appropriate, disproportionately high and adverse human health or environmental effects of their programs, policies, and activities on minority populations and low-income populations in the United States.

EPA has determined that this proposed rule will not have disproportionately high and adverse human health or environmental effects on minority or low-income populations because it does not affect the level of protection provided to human health or the environment. The proposed amendments revise existing test methods to improve the accuracies of the measurements which are expected to improve environmental quality and reduce health risks for areas that may be designated as nonattainment.

**List of Subjects in 40 CFR Part 51**

Administrative practice and procedure, Air pollution control, Carbon monoxide, Incorporation by reference, Intergovernmental relations, Lead, Nitrogen oxide, Ozone, Particulate matter, Reporting and recordkeeping requirements, Sulfur compounds, Volatile organic compounds.

Dated: March 16, 2009.

**Lisa P. Jackson,**  
*Administrator.*

For the reasons set out in the preamble, title 40, chapter I of the Code of Federal Regulations is proposed to be amended as follows:

**PART 51—[AMENDED]**

1. The authority citation for part 51 continues to read as follows:

**Authority:** 23 U.S.C. 101; 42 U.S.C 7401–7671q.

2. Amend Appendix M by revising Methods 201A and 202 to read as follows:

**Appendix M to Part 51—Recommended Test Methods for State Implementation Plans**

\* \* \* \* \*

**METHOD 201A—DETERMINATION OF PM<sub>10</sub> AND PM<sub>2.5</sub> EMISSIONS FROM STATIONARY SOURCES (Constant Sampling Rate Procedure)**

*1.0 Scope and Applicability*

1.1 Scope. The U.S. Environmental Protection Agency (U.S. EPA or “we”) developed this method to describe the procedures that the stack tester (“you”) must follow to measure particulate matter emissions equal to or less than a nominal aerodynamic diameter of 10 micrometer (PM<sub>10</sub>) and 2.5 micrometer (PM<sub>2.5</sub>). If the gas filtration temperature exceeds 30 °C (85 °F), this method includes procedures to measure only filterable particulate matter (material that does not pass through a filter or a cyclone/filter combination). If the gas filtration temperature exceeds 30 °C (85 °F), and you must measure total primary (direct) particulate matter emissions to the atmosphere, both the filterable and condensable (material that condenses after passing through a filter) components, then you must combine the procedures in this method with the procedures in Method 202 for measuring condensable particulate matter. However, if the gas filtration temperature never exceeds 30 °C (85 °F), then use of Method 202 is not required to measure total primary particulate matter.

1.2 Applicability. You can use this method to measure filterable particulate matter from stationary sources only. Filterable particulate matter is collected in-stack with this method (*i.e.*, the method measures materials that are solid or liquid at stack conditions).

1.3 Responsibility. You are responsible for obtaining the equipment and supplies you will need to use this method. You must also develop your own procedures for following this method and any additional procedures to ensure accurate sampling and analytical measurements.

1.4 Results. To obtain results, you must have a thorough knowledge of the following test methods that are found in Appendices A–1 through A–3 of 40 CFR Part 60.

(a) Method 1—Sample and Velocity Traverses for Stationary Sources.

(b) Method 2—Determination of Stack Gas Velocity and Volumetric Flow Rate (Type S Pitot Tube).

(c) Method 3—Gas Analysis for the Determination of Dry Molecular Weight.

(d) Method 4—Determination of Moisture Content in Stack Gases.

(e) Method 5—Determination of Particulate Matter Emissions from Stationary Sources.

1.5 Additional Methods. We do not anticipate that you will need additional test methods to measure ambient contributions of particulate matter to source emissions because ambient contributions are insignificant for most of the sources that are expected to be measured using this test method. However, when an adjustment for the ambient air particulate matter is needed, use the ambient air reference methods to quantify the ambient air contribution. If the source gas filtration temperature never exceeds 30 °C (85 °F) and condensable particulate is not measured by Method 202, then the correction for ambient particulate matter must be adjusted for condensable material that vaporizes at the process temperature.

1.6 Limitations. You cannot use this method to measure emissions following a wet scrubber because this method is not applicable for in-stack gases containing water droplets. To measure PM<sub>10</sub> and PM<sub>2.5</sub> in emissions where water droplets are known to exist, we recommend that you use Method 5. This method may not be suitable for sources with stack gas temperatures exceeding 260 °C (500 °F). You may need to take extraordinary measures—including the use of specialty metals (e.g., Inconel) to achieve reliable particulate mass since the threads of the cyclones may gall or seize, thus preventing the recovery of the collected particulate matter and rendering the cyclone unusable for subsequent use.

1.7 Conditions. You can use this method to obtain both particle sizing and total filterable particulate if the isokinetics are within 90–110 percent, the number of sampling points is the same as Method 5 or 17, and the in-stack filter temperature is within the acceptable range. The acceptable range for the in-stack filter temperature is generally defined as the typical range of temperature for emission gases. The acceptable range varies depending on the source and control technology. To satisfy Method 5 criteria, you may need to remove the in-stack filter and use an out-of-stack filter and recover the PM in the probe between the PM<sub>2.5</sub> particle sizer and the filter. In addition, to satisfy Method 5 and Method 17 criteria, you may need to sample from more than 12 traverse points. Be aware that this method determines in-stack PM<sub>10</sub> and PM<sub>2.5</sub> filterable emissions by sampling from a recommended maximum of 12 sample points, at a constant flow rate through the train (the constant flow is necessary to maintain the size cuts of the cyclones), and with a filter that is at the stack temperature. In contrast, Method 5 or Method 17 trains are operated isokinetically with varying flow rates through the train. Method 5 and Method 17 require sampling from as many as 24 sample points. Method 5 uses an out-of-stack filter that is maintained at a constant

temperature of 120 °C (248 °F). Further, to use this method in place of Method 5 or Method 17, you must extend the sampling time so that you collect the minimum mass necessary for weighing on each portion of this sampling train. Also, if you are using this method as an alternative to a required performance test, then you must receive approval from the appropriate authorities prior to conducting the test.

## 2.0 Summary of Method

2.1 Summary. To measure PM<sub>10</sub> and PM<sub>2.5</sub>, extract a sample of gas at a predetermined constant flow rate through an in-stack sizing device. The sizing device separates particles with nominal aerodynamic diameters of 10 microns and 2.5 microns. To minimize variations in the isokinetic sampling conditions, you must establish well-defined limits. Once a sample is obtained, remove uncombined water from the particulate, then use gravimetric analysis to determine the particulate mass for each size fraction. Changes in the original Method 201A of Appendix M to 40 CFR part 51, supplement the filterable particulate procedures with the PM<sub>2.5</sub> cyclone from a conventional five-stage cascade cyclone train. The addition of a PM<sub>2.5</sub> cyclone between the PM<sub>10</sub> cyclone and the stack temperature filter in the sampling train supplements the measurement of PM<sub>10</sub> with the measurement of fine particulate matter. Without the addition of the PM<sub>2.5</sub> cyclone, the filterable particulate portion of the sampling train may be used to measure total and PM<sub>10</sub> emissions. Likewise, with the exclusion of the PM<sub>10</sub> cyclone, the filterable particulate portion of the sampling train may be used to measure total and PM<sub>2.5</sub> emissions. Figure 1 of Section 17 presents the schematic of the sampling train configured with these changes.

## 3.0 Definitions

[Reserved]

## 4.0 Interferences

You cannot use this method to measure emissions following a wet scrubber because this method is not applicable for in-stack gases containing water droplets. Stacks with entrained moisture droplets may have water droplets larger than the cut sizes for the cyclones. These water droplets normally contain particles and dissolved solids that become PM<sub>10</sub> and PM<sub>2.5</sub> following evaporation of the water.

## 5.0 Safety

Disclaimer: You may have to use hazardous materials, operations, and equipment while using this method. We do not provide information on appropriate safety and health practices. You are responsible for determining the applicability of regulatory limitations and establishing appropriate safety and health practices. Handle materials and equipment properly.

## 6.0 Equipment and Supplies

Figure 2 of Section 17 shows details of the combined cyclone heads used in this method. The sampling train is the same as Method 17 of Appendix A–6 to Part 60 with the exception of the PM<sub>10</sub> and PM<sub>2.5</sub> sizing devices. The following sections describe the

sampling train's primary design features in detail.

### 6.1 Filterable Particulate Sampling Train Components.

6.1.1 Nozzle. You must use stainless steel (316 or equivalent) or Teflon®-coated stainless steel nozzles with a sharp tapered leading edge. We recommend one of the 12 nozzles listed in Figure 3 of Section 17 because they meet design specifications when PM<sub>10</sub> cyclones are used as part of the sampling train. We also recommend that you have a large number of nozzles in small diameter increments available to increase the likelihood of using a single nozzle for the entire traverse. We recommend one of the nozzles listed in Figure 4A or 4B of Section 17 because they meet design specifications when PM<sub>2.5</sub> cyclones are used without PM<sub>10</sub> cyclones as part of the sampling train.

6.1.2 PM<sub>10</sub> and PM<sub>2.5</sub> Sizing Device. Use a stainless steel (316 or equivalent) PM<sub>10</sub> and PM<sub>2.5</sub> sizing devices. The sizing devices must be cyclones that meet the design specifications shown in Figures 3, 4, 5, and 6 of Section 17. Use a caliper to verify the dimensions of the PM<sub>10</sub> and PM<sub>2.5</sub> sizing devices to within ±0.02 cm of the design specifications. Example suppliers of PM<sub>10</sub> and PM<sub>2.5</sub> sizing devices include the following:

- (a) Environmental Supply Company, Inc., 2142 Geer Street, Durham, North Carolina 27704, (919) 956–9688 (phone), (919) 682–0333 (fax).
- (b) Apex Instruments, P.O. Box 727, 125 Quantum Street, Holly Springs, North Carolina 27540, (919) 557–7300 (phone), (919) 557–7110 (fax).
- (c) Andersen Instruments Inc., 500 Technology Court, Smyrna, Georgia 30082, (770) 319–9999 (phone), (770) 319–0336 (fax).

You may use alternative particle sizing devices if they meet the requirements in Development and Laboratory Evaluation of a Five-Stage Cyclone System, EPA–600/7–78–008 (incorporated by reference) and are approved by the Administrator. The Director of the Federal Register approves this incorporation by reference in accordance with 5 U.S.C. 552(a) and 1 CFR part 51. You may obtain a copy from National Technical Information Service, <http://www.ntis.gov> or (800) 553–6847. You may inspect a copy at the Office of Federal Register, 800 North Capitol Street, NW., Suite 700, Washington, DC.

6.1.3 Filter Holder. Use a filter holder that is either stainless steel (316 or equivalent) or Teflon®-coated stainless steel. A heated glass filter holder may be substituted for the steel filter holder when filtration is performed out-of-stack. Commercial size filter holders are available depending upon project requirements, including commercial filter holders to support 25-, 47-, and 63-mm diameter filters. Commercial size filter holders contain a Teflon® O-ring, a stainless steel screen that supports the filter, and a final Teflon® O-ring. Screw the assembly together and attach to the outlet of cyclone IV.

6.1.4 Pitot Tube. You must use a pitot tube made of heat resistant tubing. Attach the pitot tube to the probe with stainless steel

fittings. Follow the specifications for the pitot tube and its orientation to the inlet nozzle given in Section 6.1.1.3 of Method 5.

6.1.5 Probe Liner. The probe extension must be glass-lined or Teflon®. Follow the specifications in Section 6.1.1.2 of Method 5.

6.1.6 Differential Pressure Gauge, Condensers, Metering Systems, Barometer, and Gas Density Determination Equipment. Follow the requirements in Sections 6.1.1.4 through 6.1.3 of Method 5, as applicable.

## 6.2 Sample Recovery Equipment.

6.2.1 Filterable Particulate Recovery. Use the following equipment to quantitatively determine the amount of filterable particulate matter recovered from the sampling train. Follow the requirements specified in Sections 6.2.1 through 6.2.8 of Method 5, respectively.

- (a) Filter holder brushes
- (b) Wash bottles
- (c) Glass sample storage containers
- (d) Petri dishes
- (e) Graduated cylinders and balance
- (f) Plastic storage containers
- (g) Funnel
- (h) Rubber policeman

## 7.0 Reagents, Standards, and Sampling Media

7.1 Sample Collection. To collect a sample, you will need a filter and silica gel. You must also have water and crushed ice. Additional information on these items is in the following paragraphs.

7.1.1 Filter. Use a glass fiber, quartz, or Teflon® filter that does not have an organic binder. The filter must also have an efficiency of at least 99.95 percent (<0.05 percent penetration) on 0.3 micron dioctyl phthalate smoke particles. Conduct the filter efficiency test in accordance with ASTM Method D2986-95a—Standard Method for Evaluation of Air, Assay Media by the Monodisperse DOP (Dioctyl Phthalate) Smoke Test (incorporated by reference). The Director of the Federal Register approves this incorporation by reference in accordance with 5 U.S.C. 552(a) and 1 CFR part 51. You may obtain a copy from American Society for Testing and Materials (ASTM), 100 Barr Harbor Drive, Post Office Box C700, West Conshohocken, PA 19428-2959. You may inspect a copy at the Office of Federal Register, 800 North Capitol Street, NW., Suite 700, Washington, DC. Alternatively, you may use test data from the supplier's quality control program. If the source you are sampling has sulfur dioxide (SO<sub>2</sub>) or sulfite (SO<sub>3</sub>) emissions, you must use a filter that will not react with SO<sub>2</sub> or SO<sub>3</sub>. Depending on your application and project data quality objectives (DQOs), filters are commercially available in 25-, 47-, 83-, and 110-mm sizes.

7.1.2 Silica Gel. Use an indicating-type silica gel of 6 to 16 mesh. We must approve other types of desiccants (equivalent or better) before you use them. Allow the silica gel to dry for 2 hours at 175 °C (350 °F) if it is being reused. You do not have to dry new silica gel.

7.1.3 Crushed ice. Obtain from the best readily available source.

7.2 Sample Recovery and Analysis Reagents. You will need acetone and anhydrous sodium sulfate for the sample

analysis. Unless otherwise indicated, all reagents must conform to the specifications established by the Committee on Analytical Reagents of the American Chemical Society. If such specifications are not available, then use the best available grade. Additional information on each of these items is in the following paragraphs.

7.2.1 Acetone. Use acetone that is stored in a glass bottle. Do not use acetone from a metal container because it normally produces a high residue blank. You must use acetone with blank values <1 ppm, by weight residue. Analyze acetone blanks prior to field use to confirm low blank values. In no case shall a blank value of greater than 1E-06 of the weight of acetone used in sample recovery be subtracted from the sample weight (*i.e.*, the maximum blank correction is 0.079 mg per 100 mL of acetone used to recover samples).

7.2.2 Particulate Sample Desiccant. Use indicating-type anhydrous sodium sulfate to desiccate samples prior to weighing.

## 8.0 Sample collection, Preservation, Storage, and Transport

8.1 Qualifications. This is a complex test method. To obtain reliable results, you must be trained and experienced with in-stack filtration systems (such as cyclones, impactors, and thimbles) and their operations.

8.2 Preparations. Follow the pretest preparation instructions in Section 8.1 of Method 5.

8.3 Site Setup. You must complete the following to properly set up for this test:

- (a) Determine the sampling site location and traverse points.
- (b) Calculate probe/cyclone blockage.
- (c) Verify the absence of cyclonic flow.
- (d) Complete a preliminary velocity profile, and select a nozzle.

8.3.1 Sampling Site Location and Traverse Point Determination. Follow the standard procedures in Method 1 to select the appropriate sampling site. Then do all of the following:

- (a) Sampling site. Choose a location that maximizes the distance from upstream and downstream flow disturbances.
- (b) Traverse points. The recommended maximum number of total traverse points at any location is 12 as shown in Figure 7 of Section 17. Prevent the disturbance and capture of any solids accumulated on the inner wall surfaces by maintaining a 1-inch distance from the stack wall (½ inch for sampling locations less than 24 inches in diameter).

(c) Round or rectangular duct or stack. If a duct or stack is round with two ports located 90 degrees apart, use six sampling points on each diameter. Use a 3 x 4 sampling point layout for rectangular ducts or stacks. Consult with the Administrator to receive approval for other layouts before you use them.

(d) Sampling ports. To accommodate the in-stack cyclones for this method, you may need larger diameter sampling ports than those used by Method 5 or Method 17 for total filterable particulate sampling. When you must use nozzles smaller than 0.16 inch in diameter, the sampling port diameter must be 6 inches. Do not use the conventional 4-

inch diameter port because the combined dimension of the PM<sub>10</sub> cyclone and the nozzle extending from the cyclone exceeds the internal diameter of the port.

[**Note:** If the port nipple is short, you may be able to "hook" the sampling head through a smaller port into the duct or stack.]

8.3.2 Probe/Cyclone Blockage Calculations. Follow the procedures in the next two sections, as appropriate.

### 8.3.2.1 Ducts with diameters greater than 24 inches.

Minimize the blockage effects of the combination of the in-stack nozzle/cyclones and filter assembly for ducts with diameters greater than 24 inches by keeping the cross-sectional area of the assembly at 3 percent or less of the cross-sectional area of the duct.

8.3.2.2 Ducts with diameters between 18 and 24 inches. Ducts with diameters between 18 and 24 inches have blockage effects ranging from 3 to 6 percent, as illustrated in Figure 8 of Section 17. Therefore, when you conduct tests on these small ducts, you must adjust the observed velocity pressures for the estimated blockage factor whenever the combined sampling apparatus blocks more than 3 percent of the stack or duct (see Sections 8.7.2.2 and 8.7.2.3 on the probe blockage factor and the final adjusted velocity pressure, respectively).

8.3.3 Cyclonic Flow. Do not use the combined cyclone sampling head at sampling locations subject to cyclonic flow. Also, you must follow procedures in Method 1 to determine the presence or absence of cyclonic flow and then perform the following calculations.

(a) As per Section 11.4 of Method 1, find and record the angle that has a null velocity pressure for each traverse point using a S-type pitot tube.

(b) Average the absolute values of the angles that have a null velocity pressure. Do not use the sampling location if the average absolute value exceeds 20°.

[**Note:** You can minimize the effects of cyclonic flow conditions by moving the sampling location, placing gas flow straighteners upstream of the sampling location or applying a modified sampling approach as described in EPA Guideline Document 008. You may need to obtain an alternate method approval prior to using a modified sampling approach.]

8.3.4 Preliminary Velocity Profile. Conduct a preliminary velocity traverse by following Method 2 velocity traverse procedures. The purpose of the preliminary velocity profile is to determine all of the following:

(a) The gas sampling rate for the combined probe/cyclone sampling head in order to meet the required particle size cut.

(b) The appropriate nozzle to maintain the required gas sampling rate for the velocity pressure range and isokinetic range. If the isokinetic range cannot be met (*e.g.*, batch processes, extreme process flow or temperature variation), void the sample or use methods subject to the approval of the Administrator to correct the data.

(c) The necessary sampling duration to obtain sufficient particulate catch weights.

8.3.4.1 Preliminary traverse. You must use an S-type pitot tube with a conventional

thermocouple to conduct the traverse. Conduct the preliminary traverse as close as possible to the anticipated testing time on sources that are subject to hour-by-hour gas flow rate variations of approximately  $\pm 20$  percent and/or gas temperature variations of approximately  $\pm 10$  °C ( $\pm 50$  °F).

[**Note:** You should be aware that these variations can cause errors in the cyclone cut diameters and the isokinetic sampling velocities.]

8.3.4.2 *Velocity pressure range.* Insert the S-type pitot tube at each traverse point, and record the range of velocity pressures measured on data form in Method 2. You will use this later to select the appropriate nozzle.

8.3.4.3 *Initial gas stream viscosity and molecular weight.* Determine the average gas temperature, average gas oxygen content, average carbon dioxide content, and estimated moisture content. You will use this information to calculate the initial gas stream viscosity (Equation 3) and molecular weight (Equations 1 and 2).

[**Note:** You must follow the instructions outlined in Method 4 to estimate the moisture content. You may use a wet bulb-dry bulb measurement or hand-held hygrometer measurement to estimate the moisture content of sources with gas temperatures less than 71 °C (160 °F).]

8.3.4.4 *Particulate matter concentration in the gas stream.* Determine the particulate matter concentration for the PM<sub>2.5</sub> and the PM<sub>2.5</sub> to PM<sub>10</sub> components of the gas stream through qualitative measurements or estimates. Having an idea of the particulate concentration in the gas stream is not essential but will help you determine the appropriate sampling time to acquire sufficient particulate matter weight for better accuracy at the source emission level. The collectable particulate matter weight requirements depend primarily on the types of filter media and weighing capabilities that are available and needed to characterize the emissions. Estimate the collectable particulate matter concentrations in the >10 micrometer,  $\leq 10$  and >2.5 micrometers, and  $\leq 2.5$  micrometer size ranges. Typical particulate matter concentrations are listed in Table 1 of Section 17. Additionally, relevant sections of AP-42 may contain particle size distributions for processes characterized in those sections and Appendix B2 of AP-42 contains generalized particle size distributions for nine industrial process categories (e.g., stationary internal combustion engines firing gasoline or diesel fuel, calcining of aggregate or unprocessed ores). The generalized particle size distributions can be used if source-specific particle size distributions are unavailable. Appendix B2 also contains typical collection efficiencies of various particulate control devices and example calculations showing how to estimate uncontrolled total particulate emissions, uncontrolled size-specific emissions, and controlled size-specific particulate emissions.

8.4 Pre-test Calculations. You must perform pre-test calculations to help select the appropriate gas sampling rate through cyclone I (PM<sub>10</sub>) and cyclone IV (PM<sub>2.5</sub>). Choosing the appropriate sampling rate will

allow you to maintain the appropriate particle cut diameters based upon preliminary gas stream measurements, as specified in Table 2 of Section 17.

8.4.1 *Gas Sampling Rate.* The gas sampling rate is defined by the performance curves for both cyclones, as illustrated in Figure 9 of Section 17. You must use the calculations in Section 8.5 to achieve the appropriate cut size specification for each cyclone. The optimum gas sampling rate is the overlap zone defined as the range below the cyclone IV 2.25 micrometer curve down to the cyclone I 11.0 micrometer curve (area between the two dark, solid lines in Figure 9 of Section 17).

8.4.2 *Choosing the Appropriate Sampling Rate.* You must select a gas sampling rate in the middle of the overlap zone (discussed in Section 8.4.1), as illustrated in Figure 9 of Section 17 to maximize the acceptable tolerance for slight variations in flow characteristics at the sampling location. The overlap zone is also a weak function of the gas composition.

[**Note:** The acceptable range is limited, especially for gas streams with temperatures less than approximately 100 °F. At lower temperatures, it may be necessary to perform the PM<sub>10</sub> and PM<sub>2.5</sub> separately in order to meet the necessary particle size criteria shown in Table 2 of Section 17.0.]

8.5 Test Calculations. You must perform all of the calculations in Table 3 of Section 17 and the calculations described in Sections 8.5.1 through 8.5.5.

8.5.1 *The Assumed Reynolds Number.* Verify the assumed Reynolds number ( $N_{re}$ ) by substituting the sampling rate ( $Q_s$ ) calculated in Equation 7 into Equation 8. Then use Table 5 of Section 17 to determine if the  $N_{re}$  used in Equation 5 was correct.

8.5.2 *Final Sampling Rate.* Recalculate the final sampling rate ( $Q_s$ ) if the assumed Reynolds number used in your initial calculation is not correct. Use Equation 7 to recalculate the optimum sampling rate ( $Q_s$ ).

8.5.3 *Meter Box  $\Delta H$ .* Use Equation 9 to calculate the meter box  $\Delta H$  after you calculate the optimum sampling rate and confirm the Reynolds number.

[**Note:** The stack gas temperature may vary during the test, which could affect the sampling rate. If the stack gas temperature varies, you must make slight adjustments in the meter box  $\Delta H$  to maintain the correct constant cut diameters. Therefore, use Equation 9 to recalculate the  $\Delta H$  values for 50 °F above and below the stack temperature measured during the preliminary traverse (see Section 8.3.4.1), and document this information in Table 4 of Section 17.]

8.5.4 *Choosing a Sampling Nozzle.* Select one or more nozzle sizes to provide for near isokinetic sampling rate (that is, 80 percent to 120 percent). This will also minimize an isokinetic sampling error for the particles at each point. First calculate the mean stack gas velocity,  $v_s$ , using Equation 11. See Section 8.7.2 for information on correcting for blockage and use of different pitot tube coefficients. Then use Equation 12 to calculate the diameter of a nozzle that provides for isokinetic sampling at the mean stack gas velocity at flow  $Q_s$ . From the

available nozzles just smaller and just larger of this diameter,  $D$ , select the most promising nozzle. Perform the following steps for the selected nozzle.

8.5.4.1 *Minimum/maximum nozzle/stack velocity ratio.* Use Equation 14 to calculate the minimum nozzle/stack velocity ratio,  $R_{min}$ . Use Equation 15 to calculate the maximum nozzle/stack velocity ratio,  $R_{max}$ .

8.5.4.2 *Minimum gas velocity.* Use Equation 16 to calculate the minimum gas velocity ( $v_{min}$ ) if  $R_{min}$  is an imaginary number (negative value under the square root function) or if  $R_{min}$  is less than 0.5. Use Equation 17 to calculate  $v_{min}$  if  $R_{min}$  is greater than or equal to 0.5.

8.5.4.3 *Maximum stack velocity.* Use Equation 18 to calculate the maximum stack velocity ( $v_{max}$ ) if  $R_{max}$  is less than 1.5. Use Equation 19 to calculate the stack velocity if  $R_{max}$  is greater than or equal to 1.5.

8.5.4.4 *Conversion of gas velocities to velocity pressure.* Use Equation 20 to convert  $v_{min}$  to minimum velocity pressure,  $\Delta p_{min}$ . Use Equation 21 to convert  $v_{max}$  to maximum velocity pressure,  $\Delta p_{max}$ .

8.5.4.5 Compare minimum and maximum velocity pressures with the observed velocity pressures at all traverse points during the preliminary test (see Section 8.3.4.2).

8.5.5 *Optimum sampling nozzle.* The nozzle you selected is appropriate if all the observed velocity pressures during the preliminary test fall within the range of the  $\Delta p_{min}$  and  $\Delta p_{max}$ . Make sure the following requirements are met. Then follow the procedures in Sections 8.5.5.1 and 8.5.5.2.

(a) Choose an optimum nozzle that provides for isokinetic sampling conditions as close to 100 percent as possible. This is prudent because even if there are slight variations in the gas flow rate, gas temperature, or gas composition during the actual test, you have the maximum assurance of satisfying the isokinetic criteria. Generally, one of the two candidate nozzles selected will be closer to optimum (see Section 8.5.4).

(b) When testing is for PM<sub>2.5</sub> only, you may have only two traverse points out of 12 that are outside the range of the  $\Delta p_{min}$  and  $\Delta p_{max}$  (i.e., 16 percent failure rate rounded to the nearest whole number). If the coarse fraction for PM<sub>10</sub> determination is included, only one traverse point out of 12 can fall outside the minimum-maximum velocity pressure range (i.e., 8 percent failure rate rounded to the nearest whole number).

8.5.5.1 *Precheck.* Visually check the selected nozzle for dents before use.

8.5.5.2 *Attach the pre-selected nozzle.* Screw the pre-selected nozzle onto the main body of cyclone I using Teflon® tape. Use a union and cascade adaptor to connect the cyclone IV inlet to the outlet of cyclone I (see Figure 2 of Section 17).

8.6 *Sampling Train Preparation.* A schematic of the sampling train used in this method is shown in Figure 1 of Section 17. First, assemble the train and complete the leak check on the combined cyclone sampling head and pitot tube. Use the following procedures to prepare the sampling train.

[**Note:** Do not contaminate the sampling train during preparation and assembly. Keep all openings where contamination can occur

covered until just prior to assembly or until sampling is about to begin.]

**8.6.1 Sampling Head and Pitot Tube.** Assemble the combined cyclone train. The O-rings used in the train have a temperature limit of approximately 205 °C (400 °F). Use cyclones with stainless steel sealing rings when stack temperatures exceed 205 °C (400 °F). This method may not be suitable for sources with stack gas temperatures exceeding 260 °C (500 °F). You may need to take extraordinary measures including the use of specialty metals (e.g., Inconel) to achieve reliable particulate mass since the threads of the cyclones may gall or seize, thus preventing the recovery of the collected particulate matter and rendering the cyclone unusable for subsequent use. You must also keep the nozzle covered to protect it from nicks and scratches.

**8.6.2 Filterable Particulate Filter Holder and Pitot Tube.** Attach the pre-selected filter holder to the end of the combined cyclone sampling head (see Figure 2 of Section 17). Attach the S-type pitot tube to the combined cyclones after the sampling head is fully attached to the end of the probe.

[**Note:** The pitot tube tip must be mounted: slightly beyond the combined head cyclone sampling assembly; and at least one inch off the gas flow path into the cyclone nozzle. This is similar to the pitot tube placement in Method 17.]

Weld the sensing lines to the outside of the probe to ensure proper alignment of the pitot tube. Provide unions on the sensing lines so that you can connect and disconnect the S-type pitot tube tips from the combined cyclone sampling head before and after each run.

[**Note:** Calibrate the pitot tube on the sampling head because the cyclone body is a potential source flow disturbance.]

**8.6.3 Filter.** You must number and tare the filters before use. To tare the filters, desiccate each filter at  $20 \pm 5.6$  °C (68  $\pm$  10 °F) and ambient pressure for at least 24 hours and weigh at intervals of at least 6 hours to a constant weight, i.e., <0.5 mg change from previous weighing; record results to the nearest 0.1 mg. During each weighing, the filter must not be exposed to the laboratory atmosphere for longer than 2 minutes and a relative humidity above 50 percent. Alternatively, the filters may be oven-dried at 104 °C (220 °F) for 2 to 3 hours, desiccated for 2 hours, and weighed. Use tweezers or clean disposable surgical gloves to place a labeled (identified) and pre-weighed filter in both filterable and condensable particulate filter holders. You must center the filter and properly place the gasket so that the sample gas stream will not circumvent the filter. Check the filter for tears after the assembly is completed. Then screw the filter housing together to prevent the seal from leaking.

**8.6.7 Moisture Trap.** If you are measuring only filterable particulate (or you are sure that the filtration temperature will be maintained below 30 °C (85 °F)), then an empty modified Greenburg Smith impinger followed by an impinger containing silica gel is required. Alternatives described in Method 5 may also be used to collect moisture that passes through the ambient filter. If you are

measuring condensable particulate matter in combination with this method, then follow the procedures in Method 202 for moisture collection.

**8.6.8 Leak Check.** Use the procedures outlined in Section 8.4 of Method 5 to leak check the entire sampling system. Specifically perform the following procedures:

**8.6.8.1 Sampling train.** You must pretest the entire sampling train for leaks. The pretest leak check must have a leak rate of not more than 0.02 ACFM or 4 percent of the average sample flow during the test run, whichever is less. Additionally, you must conduct the leak check at a vacuum equal to or greater than the vacuum anticipated during the test run. Enter the leak check results on the field test data sheet (see Section 11.1) for the specific test.

[**Note:** Do not conduct a leak check during port changes.]

**8.6.8.2 Pitot tube assembly.** After you leak check the sample train, perform a leak check of the pitot tube assembly. Follow the procedures outlined in Section 8.4.1 of Method 5.

**8.6.9 Sampling Head.** You must preheat the combined sampling head to the stack temperature of the gas stream at the test location ( $\pm 10$  °C,  $\pm 50$  °F). This will heat the sampling head and prevent moisture from condensing from the sample gas stream. Record the site barometric pressure and stack pressure on the field test data sheet.

**8.6.9.1 Unsaturated stacks.** You must complete a passive warmup (of 30–40 min) within the stack before the run begins to avoid internal condensation.

[**Note:** Unsaturated stacks do not have entrained droplets and operate at temperatures above the local dew point of the stack gas.]

**8.6.9.2 Shortened warm-up of unsaturated stacks.** You can shorten the warmup time by thermostated heating outside the stack (such as by a heat gun). Then place the heated sampling head inside the stack and allow the temperature to equilibrate.

**8.7 Sampling Train Operation.** Operate the sampling train the same as described in Section 4.1.5 of Method 5, except use the procedures in this section for isokinetic sampling and flow rate adjustment. Maintain the flow rate calculated in Section 8.4.1 throughout the run, provided the stack temperature is within 28 °C (50 °F) of the temperature used to calculate  $\Delta H$ . If stack temperatures vary by more than 28 °C (50 °F), use the appropriate  $\Delta H$  value calculated in Section 8.5.3. Determine the minimum number of traverse points as in Figure 7 of Section 17. Determine the minimum total projected sampling time ( $t_t$ ), based on achieving the data quality objectives or emission limit of the affected facility. We recommend you round the number of minutes sampled at each point to the nearest 15 seconds. Perform the following procedures:

**8.7.1 Sample Point Dwell Time.** You must calculate the dwell time (that is, sampling time) for each sampling point to ensure that the overall run provides a

velocity-weighted average that is representative of the entire gas stream. Vary the dwell time, or sampling time, at each traverse point proportionately with the point velocity.

**8.7.1.1 Dwell time at first sampling point.** Calculate the dwell time for the first point,  $t_1$ , using Equation 22. You must use the data from the preliminary traverse. Here,  $N_{tp}$  equals the total number of traverse points.

**8.7.1.2 Dwell time at remaining sampling points.** Calculate the dwell time at each of the remaining traverse points,  $t_n$ , using Equation 23. This time you must use the actual test run data.

[**Note:** Round the dwell times to the nearest 15 seconds.] Each traverse point must have a dwell time of at least 2 minutes.

**8.7.2 Adjusted Velocity Pressure.** When selecting your sampling points using your preliminary velocity traverse data, your preliminary velocity pressures must be adjusted to take into account the increase in velocity due to blockage. Also, you must adjust your preliminary velocity data for differences in pitot tube coefficients. Use the following instructions to adjust the preliminary velocity pressure.

**8.7.2.1 Different pitot tube coefficient.** You must use Equation 24 to correct the recorded preliminary velocity pressures if the pitot tube mounted on the combined cyclone sampling head has a different pitot tube coefficient than the pitot tube used during the preliminary velocity traverse (see Section 8.3.4).

**8.7.2.2 Probe blockage factor.** You must use Equation 25 to calculate an average probe blockage correction factor ( $b_f$ ) if the diameter of your stack or duct is between 18 and 24 inches. A probe blockage factor is calculated because of the flow blockage caused by the relatively large cross-sectional area of the combined cyclone sampling head, as discussed in Section 8.3.2.2 and illustrated in Figure 8 of Section 17.

[**Note:** The sampling head (including the PM<sub>10</sub> cyclone, PM<sub>2.5</sub> cyclone, pitot and filter holder) has a projected area of approximately 20.5 square inches when oriented into the gas stream. As the probe is moved from the most outer to the most inner point, the amount of blockage that actually occurs ranges from approximately 4 square inches to the full 20.5 inches. The average cross-sectional area blocked is 12 square inches.]

**8.7.2.3 Final adjusted velocity pressure.** Calculate the final adjusted velocity pressure ( $\Delta p_{s2}$ ) using Equation 26.

[**Note:** Figure 8 of Section 17 illustrates that the blockage effect of the large combined cyclone sampling head increases rapidly below diameters of 18 inches. Therefore, you must follow the procedures outlined in Method 1A to conduct tests in small stacks (< inches diameter). You must conduct the velocity traverse downstream of the sampling location or immediately before the test run.]

**8.7.3 Sample Collection.** Collect samples the same as described in Section 4.1.5 of Method 5, except use the procedures in this section for isokinetic sampling and flow rate adjustment. Maintain the flow rate calculated in Section 8.5 throughout the run, provided the stack temperature is within 28 °C (50 °F)

of the temperature used to calculate  $\Delta H$ . If stack temperatures vary by more than 28 °C (50 °F), use the appropriate  $\Delta H$  value calculated in Section 8.5.3. Calculate the dwell time at each traverse point as in Equations 22 and 23. In addition to these procedures, you must also use running starts and stops if the static pressure at the sampling location is more negative than 5 in. water column. This prevents back pressure from rupturing the sample filter. If you use a running start, adjust the flow rate to the calculated value after you perform the leak check (see Section 8.4).

#### 8.7.3.1 Level and zero manometers.

Periodically check the level and zero point of the manometers during the traverse. Vibrations and temperature changes may cause them to drift.

8.7.3.2 *Portholes*. Clean the portholes prior to the test run. This will minimize the chance of collecting deposited material in the nozzle.

8.7.3.3 *Sampling procedures*. Verify that the combined cyclone sampling head temperature is at stack temperature ( $\pm 10$  °C,  $\pm 50$  °F).

[**Note:** For many stacks, portions of the cyclones and filter will be external to the stack during part of the sampling traverse. Therefore, you must heat or insulate portions of the cyclones and filter that are not within the stack in order to maintain the sampling head temperature at the stack temperature. Maintaining the temperature will insure proper particle sizing and prevent condensation on the walls of the cyclones.]

Remove the protective cover from the nozzle. To begin sampling, immediately start the pump and adjust the flow to calculated isokinetic conditions. Position the probe at the first sampling point with the nozzle pointing directly into the gas stream. Ensure the probe/pitot tube assembly is leveled.

[**Note:** When the probe is in position, block off the openings around the probe and porthole to prevent unrepresentative dilution of the gas stream.]

(a) Traverse the stack cross-section, as required by Method 1 with the exception that you are only required to perform a 12-point traverse. Do not bump the cyclone nozzle into the stack walls when sampling near the walls or when removing or inserting the probe through the portholes. This will minimize the chance of extracting deposited materials.

(b) Record the data required on the field test data sheet for each run. Record the initial dry gas meter reading. Then take dry gas meter readings at the following times: the beginning and end of each sample time increment; when changes in flow rates are made; and when sampling is halted. Compare the velocity pressure measurements (Equations 20 and 21) with the velocity pressure measured during the preliminary traverse. Keep the meter box  $\Delta H$  at the value calculated in Section 8.5.3 for the stack temperature that is observed during the test. Record all the point-by-point data and other source test parameters on the field test data sheet. Do not leak check the sampling system during port changes.

(c) Maintain the flow through the sampling system at the last sampling point. Remove

the sampling train from the stack while it is still operating (running stop). Then stop the pump, and record the final dry gas meter reading and other test parameters on the field test data sheet.

8.7.4 *Process Data*. You must document data and information on the process unit tested, the particulate control system used to control emissions, any non-particulate control system that may affect particulate emissions, the sampling train conditions, and weather conditions. Discontinue the test if the operating conditions may cause non-representative particulate emissions.

8.7.4.1 *Particulate control system data*. Use the process and control system data to determine if representative operating conditions were maintained throughout the testing period.

8.7.4.2 *Sampling train data*. Use the sampling train data to confirm that the measured particulate emissions are accurate and complete.

8.7.5 *Sample Recovery*. First remove the sample head (combined cyclone/filter assembly) from the stack. After the sample head is removed, perform a post-test leak check of the probe and sample train. Then recover the components from the cyclone/filter. Refer to the following sections for more detailed information.

8.7.5.1 *Remove sampling head*. At the conclusion of the test, document final test conditions and remove the pitot tube and combined cyclone sampling head from the source. Make sure that you do not scrape the pitot tube or the combined cyclone sampling head against the port or stack walls.

[**Note:** After you stop the gas flow, make sure you keep the combined cyclone head level to avoid tipping dust from the cyclone cups into the filter and/or down-comer lines.]

After cooling and when the probe can be safely handled, wipe off all external surfaces near the cyclone nozzle, and cap the inlet to cyclone I. Remove the combined cyclone/filter sampling head from the probe. Cap the outlet of the filter housing to prevent particulate matter from entering the assembly.

8.7.5.2 *Leak check probe/sample train assembly (post-test)*. Leak check the remainder of the probe and sample train assembly (including meter box) after removing the combined cyclone head/filter. You must conduct the leak rate at a vacuum equal to or greater than the maximum vacuum achieved during the test run. Enter the results of the leak check onto the field test data sheet. If the leak rate of the sampling train (without the combined cyclone sampling head) exceeds 0.02 ACFM or 4 percent of the average sampling rate during the test run (whichever is less), the run is invalid, and you must repeat it.

8.7.5.3 *Weigh or measure the volume of the liquid collected in the water collection impingers and silica trap*. Measure the liquid in the first impingers to within 1 ml using a clean graduated cylinder or by weighing it to within 0.5 g using a balance. Record the volume of the liquid or weight of the liquid present to be used to calculate the moisture content of the effluent gas.

8.7.5.4 *If a balance is available in the field, weigh the silica impinger to within 0.5*

g. Note the color of the indicating silica gel in the last impinger to determine whether it has been completely spent, and make a notation of its condition. If you are measuring condensable particulate matter in combination with this method, then leave the silica in the impinger for recovery after the post-test nitrogen purge is complete.

8.7.5.5 *Recovery of particulate matter*. Recovery involves the quantitative transfer of particles in the following size range: > 10 micrometers;  $\leq 10$  micrometers but > 2.5 micrometers; and  $\leq 2.5$  micrometers. You must use a Nylon or Teflon brush and an acetone rinse to recover particles from the combined cyclone/filter sampling head. Use the following procedures for each container.

(a) *Container #1,  $\leq PM_{2.5}$  micrometer filterable particulate*—Use tweezers and/or clean disposable surgical gloves to remove the filter from the filter holder. Place the filter in the petri dish that you identified as Container #1. Using a dry Nylon bristle brush and/or a sharp-edged blade, carefully transfer any particulate matter and/or filter fibers that adhere to the filter holder gasket or filter support screen to the petri dish. Seal the container. This container holds particles  $\leq 2.5$  micrometers that are caught on the in-stack filter.

(b) *Container #2, >  $PM_{10}$  micrometer filterable particulate*—Quantitatively recover the particulate matter from the cyclone I cup and acetone rinses (and brush cleaning) of the cyclone cup, internal surface of the nozzle, and cyclone I internal surfaces, including the outside surface of the downcomer line. Seal the container and mark the liquid level on the outside of the container. You must keep any dust found on the outside of cyclone I and cyclone nozzle external surfaces out of the sample. This container holds particulate matter > 10 micrometers.

(c) *Container #3, Filterable particulate  $\leq 10$  micrometer and > 2.5 micrometers*—Place the solids from cyclone cup IV and the acetone (and brush cleaning) rinses of the cyclone I turnaround cup (above inner downcomer line), inside of the downcomer line, and interior surfaces of cyclone IV into Container #3. Seal the container and mark the liquid level on the outside. This container holds particulate matter  $\leq 10$  micrometers but > 2.5 micrometers.

(d) *Container #4,  $\leq PM_{2.5}$  micrometers acetone rinses of the exit tube of cyclone IV and front half of the filter holder*—Retrieve the acetone rinses (and brush cleaning) of the exit tube of cyclone IV and the front half of the filter holder in container #4. Seal the container and mark the liquid level on the outside of the container. This container holds particulate matter that is  $\leq 2.5$  micrometers.

(e) *Container #5, Cold impinger water*—If the water from the cold impinger used for moisture collection has been weighed in the field, it can be discarded. Otherwise quantitatively transfer liquid from the cold impinger that follows the ambient filter into a clean sample bottle (glass or plastic). Mark the liquid level on the bottle. This container holds the remainder of the liquid water from the emission gases.

(f) *Container #6, Silica Gel Absorbent*—Transfer the silica gel to its original container



and seal. A funnel may make it easier to pour the silica gel without spilling. A rubber policeman may be used as an aid in removing the silica gel from the impinger. It is not necessary to remove the small amount of silica gel dust particles that may adhere to the impinger wall and are difficult to remove. Since the gain in weight is to be used for moisture calculations, do not use any water or other liquids to transfer the silica gel. If the silica gel has been weighed in the field to measure water content, it can be discarded. Otherwise the contents of Container #6 are weighed during sample analysis.

(g) *Container #7, Acetone Rinse Blank*—Take 100 ml of the acetone directly from the wash bottle you used, and place it in Container #7 labeled Acetone Rinse Blank.

8.7.6 *Transport Procedures*. Containers must remain in an upright position at all times during shipping. You do not have to ship the containers under dry or blue ice.

## 9.0 Quality Control

9.1 *Daily Quality Checks*. You must perform daily quality checks using data quality indicators that require review of recording and transfer of raw data, calculations, and documentation of testing procedures.

9.2 *Calculation Verification*. Verify the calculations by independent, manual checks. You must flag any suspect data and identify the nature of the problem and potential effect on data quality. After you complete the test, prepare a data summary, and compile all the calculations and raw data sheets.

9.3 *Conditions*. You must document data and information on the process unit tested, the particulate control system used to control emissions, any non-particulate control system that may affect particulate emissions, the sampling train conditions, and weather conditions. Discontinue the test if the operating conditions may cause non-representative particulate emissions.

9.4 *Health and Safety Plan*. Develop a health and safety plan to ensure the safety of your employees who are on site conducting the particulate emission test. Your plan must conform to all applicable OSHA, MSHA, and DOT regulatory requirements. The procedures must also conform to the plant health and safety requirements.

9.5 *Calibration Checks*. Perform calibration check procedures on analytical balances each time they are used.

9.6 *Glassware*. Use class A volumetric glassware for titrations, or calibrate your equipment against NIST traceable glassware.

## 10.0 Calibration and Standardization

[**Note:** Maintain a laboratory log of all calibrations.]

10.1 *Gas Flow Velocities*. Measure the gas flow velocities at the sampling locations using Method 2. You must use an S-type pitot tube that meets the required EPA specifications (EPA Publication 600/4-77-0217b) during these velocity measurements. You must also complete the following:

(a) Visually inspect the S-type pitot tube before sampling.

(b) Leak check both legs of the pitot tube before and after sampling.

(c) Maintain proper orientation of the S-type pitot tube while making measurements.

10.1.1 *S-type pitot tube orientation*. The S-type pitot tube is oriented properly when the yaw and the pitch axis are 90 degrees to the air flow.

10.1.2 *Average velocity pressure record*. Instead of recording either high or low values, record the average velocity pressure at each point during flow measurements.

10.1.3 *Pitot tube coefficient*. Determine the pitot tube coefficient based on physical measurement techniques described in Method 2.

[**Note:** You must calibrate the pitot tube on the sampling head because of potential interferences from the cyclone body. Refer to Section 8.7.2 for additional information.]

10.2 *Thermocouple Calibration*. Calibrate the thermocouples using the procedures described in Section 10.1.4.1.2 of Method 2 to calibrate the thermocouples. Calibrate each temperature sensor at a minimum of three points over the anticipated range of use against an NIST-traceable mercury-in-glass thermometer.

10.3 *Nozzles*. You may use stainless steel (316 or equivalent) or Teflon®-coated nozzles for isokinetic sampling. Make sure that all nozzles are thoroughly cleaned, visually inspected, and calibrated according to the procedure outlined in Section 10.1 of Method 5.

10.4 *Dry Gas Meter Calibration*. Calibrate your dry gas meter following the calibration procedures in Section 16.1 of Method 5. Also, make sure you fully calibrate the dry gas meter to determine the volume correction factor prior to field use. Post-test calibration checks must be performed as soon as possible after the equipment has been returned to the shop. Your pretest and post-test calibrations must agree within  $\pm 5$  percent.

## 11.0 Analytical Procedures

11.1 *Analytical Data Sheet*. Record all data on the analytical data sheet. Obtain the data sheet from Figure 5–6 of Method 5. Alternatively, data may be recorded electronically using software applications such as the Electronic Reporting Tool (ERT) located at the following internet address: ([http://www.epa.gov/ttn/chief/ert/ert\\_tool.html](http://www.epa.gov/ttn/chief/ert/ert_tool.html)).

11.2 *Dry Weight of Particulate Matter*. Determine the dry weight of particulate following procedures outlined in this section.

11.2.1 *Container #1,  $\leq PM_{2.5}$  micrometer filterable particulate*. Transfer the filter and any loose particulate from the sample container to a tared glass weighing dish. Desiccate for 24 hours in a desiccator containing anhydrous calcium sulfate or indicating silica gel. Weigh to a constant weight, and report the results to the nearest 0.1 mg. For the purposes of this section, the term “constant weight” means a difference of no more than 0.5 mg or 1 percent of total weight less tare weight, whichever is greater, between two consecutive weighings, with no less than 6 hours of desiccation time between weighings.

11.2.2 *Container #2,  $> PM_{10}$  micrometer filterable particulate acetone rinse*. Separately treat this container like Container #1.

11.2.3 *Container #3, Filterable particulate  $\leq 10$  micrometer and  $\geq 2.5$  micrometers acetone rinse*. Separately treat this container like Container #1.

11.2.4 *Container #4,  $\leq PM_{2.5}$  micrometers acetone rinse of the exit tube of cyclone IV and front half of the filter holder*. Note the level of liquid in the container, and confirm on the analysis sheet whether leakage occurred during transport. If a noticeable amount of leakage has occurred, either void the sample or use methods, subject to the approval of the Administrator, to correct the final results. Quantitatively transfer the contents to a tared 250 ml beaker, and evaporate to dryness at ambient temperature and pressure. Desiccate for 24 hours, and weigh to a constant weight. Report the results to the nearest 0.1 g.

11.2.5 *Container #5, Cold impinging water*. If the amount of water has not been determined in the field, note the level of liquid in the container, and confirm on the analysis sheet whether leakage occurred during transport. If a noticeable amount of leakage has occurred, either void the sample or use methods, subject to the approval of the Administrator, to correct the final results. Measure the liquid in this container either volumetrically to  $\pm 1$  ml or gravimetrically to  $\pm 0.5$  g.

11.2.6 *Container #6, Silica gel absorbent*. Weigh the spent silica gel (or silica gel plus impinger) to the nearest 0.5 g using a balance. This step may be conducted in the field.

11.2.7 *Container #7, Acetone rinse blank*. Use 100 ml of acetone from the blank container for this analysis. If insufficient liquid is available or if the acetone has been lost due to container breakage, either void the sample or use methods, subject to the approval of the Administrator, to correct the final results. Transfer 100 ml of the acetone to a clean 250 ml beaker. Evaporate the acetone at room temperature and pressure in a laboratory hood to approximately 10 ml. Quantitatively transfer the beaker contents to a 50 ml preweighed tin, and evaporate to dryness at room temperature and pressure in a laboratory hood. Following evaporation, desiccate the residue for 24 hours in a desiccator containing anhydrous calcium sulfate. Weigh and report the results to the nearest 0.1 mg.

## 12.0 Calculations and Data Analysis

12.1 *Nomenclature*. Report results in International System of Units (SI units) unless the regulatory authority for compliance testing specifies English units. The following nomenclature is used.

A = Area of stack or duct at sampling location, square inches.

A<sub>n</sub> = Area of nozzle, square feet.

b<sub>f</sub> = Average blockage factor calculated in Equation 25, dimensionless.

B<sub>ws</sub> = Moisture content of gas stream, fraction e.g., 10% H<sub>2</sub>O is B<sub>ws</sub> = 0.10).

C = Cunningham correction factor for particle diameter, D<sub>p</sub>, and calculated using the actual stack gas temperature, dimensionless.

%CO<sub>2</sub> = Carbon Dioxide content of gas stream, % by volume.

C<sub>a</sub> = Acetone blank concentration, mg/mg.

C<sub>fPM10</sub> = Conc. of filterable PM<sub>10</sub> particulate matter, gr/DSCF.

$C_{\text{IPM2.5}}$  = Conc. of filterable  $\text{PM}_{2.5}$  particulate matter, gr/DSCF.

$C_p$  = Pitot coefficient for the combined cyclone pitot, dimensionless.

$C_p'$  = Coefficient for the pitot used in the preliminary traverse, dimensionless.

$C_r$  = Re-estimated Cunningham correction factor for particle diameter equivalent to the actual cut size diameter and calculated using the actual stack gas temperature, dimensionless.

$C_{\text{if}}$  = Conc. of total filterable particulate matter, gr/DSCF.

$C_1 = -150.3162$  (micropoise)

$C_2 = 18.0614$  (micropoise/ $K^{0.5}$ ) = 13.4622 (micropoise/ $R^{0.5}$ )

$C_3 = 1.19183 \times 10^6$  (micropoise/ $K^2$ ) =  $3.86153 \times 10^6$  (micropoise/ $R^2$ )

$C_4 = 0.591123$  (micropoise)

$C_5 = 91.9723$  (micropoise)

$C_6 = 4.91705 \times 10^{-5}$  (micropoise/ $K^2$ ) =  $1.51761 \times 10^{-5}$  (micropoise/ $R^2$ )

$D$  = Inner diameter of sampling nozzle mounted on Cyclone I, in.

$D_p$  = Physical particle size, micrometers.

$D_{50}$  = Particle cut diameter, micrometers.

$D_{50-1}$  = Re-calculated particle cut diameters based on re-estimated  $C_r$ , micrometers.

$D_{50LL}$  = Cut diameter for cyclone I corresponding to the 2.25 micrometer cut diameter for cyclone IV, micrometers.

$D_{50N}$  =  $D_{50}$  value for cyclone IV calculated during the Nth iterative step, micrometers.

$D_{50(N+1)}$  =  $D_{50}$  value for cyclone IV calculated during the N+1 iterative step, micrometers.

$D_{50T}$  = Cyclone I cut diameter corresponding to the middle of the overlap zone shown in Figure 9 of Section 17, micrometers.

$I$  = Percent isokinetic sampling, dimensionless.

in. = Inches

$K_p = 85.49$ , [(ft/sec)/(pounds/mole - °R)].

$m_a$  = Mass of residue of acetone after evaporation, mg.

$M_d$  = Molecular weight of dry gas, pounds/pound mole.

$M_w$  = Molecular weight of wet gas, pounds/pound mole.

$M_1$  = Milligrams of particulate matter collected on the filter,  $\leq 2.5$  micrometers.

$M_2$  = Milligrams of particulate matter recovered from Container #2 (acetone blank corrected),  $>10$  micrometers.

$M_3$  = Milligrams of particulate matter recovered from Container #3 (acetone blank corrected),  $\leq 10$  and  $>2.5$  micrometers.

$M_4$  = Milligrams of particulate matter recovered from Container #4 (acetone blank corrected),  $\leq 2.5$  micrometers.

$N_{\text{ip}}$  = Number of iterative steps or total traverse points.

$N_{\text{re}}$  = Reynolds number, dimensionless.

$\%O_{2,\text{wet}}$  = Oxygen content of gas stream, % by volume of wet gas.

[Note: The oxygen percentage used in Equation 3 is on a wet gas basis. That means that since oxygen is typically measured on a dry gas basis, the measured  $\%O_2$  must be multiplied by the quantity  $(1 - B_{ws})$  to convert to the actual volume fraction. Therefore,  $\%O_{2,\text{wet}} = (1 - B_{ws}) * \%O_{2,\text{dry}}$ ]

$P_{\text{bar}}$  = Barometric pressure, in. Hg.

$P_s$  = Absolute stack gas pressure, in. Hg.

$Q_s$  = Sampling rate for cyclone I to achieve specified  $D_{50}$ , ACFM.

$Q_{sT}$  = Dry gas sampling rate through the sampling assembly, DSCFM.

$Q_I$  = Sampling rate for cyclone I to achieve specified  $D_{50}$ , ACFM.

$Q_{IV}$  = Sampling rate for cyclone IV to achieve specified  $D_{50}$ , ACFM.

$R_{\text{max}}$  = Nozzle/stack velocity ratio parameter, dimensionless.

$R_{\text{min}}$  = Nozzle/stack velocity ratio parameter, dimensionless.

$T_m$  = Meter box and orifice gas temperature, °R.

$t_n$  = Sampling time at point n, min.

$t_r$  = Total projected run time, min.

$T_s$  = Absolute stack gas temperature, °R.

$t_1$  = Sampling time at point 1, min.

$v_{\text{max}}$  = Maximum gas velocity calculated from Equations 18 or 19, ft/sec.

$v_{\text{min}}$  = Minimum gas velocity calculated from Equations 16 or 17, ft/sec.

$v_n$  = Sample gas velocity in the nozzle, ft/sec.

$v_s$  = Velocity of stack gas, ft/sec.

$V_a$  = Volume of acetone blank, ml.

$V_{aw}$  = Volume of acetone used in blank wash, ml.

$V_c$  = Quantity of water captured in impingers and silica gel, ml.

$V_m$  = Dry gas meter volume sampled, ACF.

$V_{ms}$  = Dry gas meter volume sampled, corrected to standard conditions, DSCF.

$V_{ws}$  = Volume of water vapor, SCF.

$V_b$  = Volume of aliquot taken for IC analysis, ml.

$V_{ic}$  = Volume of impinger contents sample, ml.

$W_a$  = Weight of residue in acetone blank wash, mg.

$Z$  = Ratio between estimated cyclone IV  $D_{50}$  values, dimensionless.

$\Delta H$  = Meter box orifice pressure drop, in. W.C.

$\Delta H_{\text{or}}$  = Pressure drop across orifice at flow rate of 0.75 SCFM at standard conditions, in. W.C.

[Note: specific to each orifice and meter box.]

$[(\Delta p)^{0.5}]_{\text{avg}}$  = Average of square roots of the velocity pressures measured during the preliminary traverse, in. W.C.

$\Delta p_m$  = Observed velocity pressure using S-type pitot tube in preliminary traverse, in. W.C.

$\Delta p_{\text{max}}$  = Maximum velocity pressure, in. W.C.

$\Delta p_{\text{min}}$  = Minimum velocity pressure, in. W.C.

$\Delta p_n$  = Velocity pressure measured at point n during the test run, in. W.C.

$\Delta p_s$  = Velocity pressure calculated in Equation 24, in. W.C.

$\Delta p_{s1}$  = Velocity pressure adjusted for combined cyclone pitot tube, in. W.C.

$\Delta p_{s2}$  = Velocity pressure corrected for blockage, in. W.C.

$\Delta p_1$  = Velocity pressure measured at point 1, in. W.C.

$\gamma$  = Dry gas meter gamma value, dimensionless.

$\mu$  = Gas viscosity, micropoise.

$\theta$  = Total run time, minutes.

$\rho_a$  = Density of acetone, mg/ml (see label on bottle).

12.0 = Constant calculated as 60 percent of 20.5 square inch cross-sectional area of combined cyclone head, square inches.

12.2 Calculations. Perform all of the calculations found in Table 6 of Section 17.

Table 6 of Section 17 also provides instructions and references for the calculations.

12.3 Analyses. Analyze  $D_{50}$  of cyclone IV and the concentrations of the particulate matter in the various size ranges.

12.3.1  $D_{50}$  of cyclone IV. To determine the actual  $D_{50}$  for cyclone IV, recalculate the Cunningham correction factor and the Reynolds number for the best estimate of cyclone IV  $D_{50}$ . The following sections describe additional information on how to recalculate the Cunningham correction factor and determine which Reynolds number to use.

12.3.1.1 *Cunningham correction factor.* Recalculate the initial estimate of the Cunningham correction factor using the actual test data. Insert the actual test run data and  $D_{50}$  of 2.5 micrometers into Equation 4. This will give you a new Cunningham correction factor that is based on actual data.

12.3.1.2 *Initial  $D_{50}$  for cyclone IV.* Determine the initial estimate for cyclone IV  $D_{50}$  using the test condition Reynolds number calculated with Equation 8 as indicated in Table 3 of Section 17. Refer to the following instructions.

(a) If the Reynolds number is less than 3,162, calculate the  $D_{50}$  for cyclone IV with Equation 33, using actual test data.

(b) If the Reynolds number is equal to or greater than 3,162, calculate the  $D_{50}$  for cyclone IV with Equation 34, using actual test data.

(c) Insert the "new"  $D_{50}$  value calculated by either Equation 33 or 34 into Equation 35 to re-establish the Cunningham Correction Factor ( $C_r$ ).

[Note: Use the test condition calculated Reynolds number to determine the most appropriate equation (Equation 33 or 34).]

12.3.1.3 *Re-establish cyclone IV  $D_{50}$ .* Use the re-established Cunningham correction factor (calculated in the previous step) and the calculated Reynolds number to determine  $D_{50-1}$ .

(a) Use Equation 36 to calculate the re-established cyclone IV  $D_{50-1}$  if the Reynolds number is less than 3,162.

(b) Use Equation 37 to calculate the re-established cyclone IV  $D_{50-1}$  if the Reynolds number is equal to or greater than 3,162.

12.3.1.4 *Establishing "Z" values.* The "Z" value is the result of an analysis that you must perform to determine if the Cunningham correction factor is acceptable. Compare the calculated cyclone IV  $D_{50}$  (either Equation 33 or 34) to the re-established cyclone IV  $D_{50-1}$  (either Equation 36 or 37) values based upon the test condition calculated Reynolds number (Equation 38). Follow these procedures.

(a) Use Equation 38 to calculate the "Z". If the "Z" value is between 0.99 and 1.01, the  $D_{50-1}$  value is the best estimate of the cyclone IV  $D_{50}$  cut diameter for your test run.

(b) If the "Z" value is greater than 1.01 or less than 0.99, re-establish a Cunningham correction factor based on the  $D_{50-1}$  value determined in either Equations 36 or 37, depending upon the test condition Reynolds number.

(c) Use the second revised Cunningham correction to re-calculate the cyclone IV  $D_{50}$ .

(d) Repeat this iterative process as many times as necessary using the prescribed

equations until you achieve the criteria documented in Equation 39.

12.3.2 Particulate concentration. Use the particulate catch weights in the combined cyclone sampling train to calculate the concentration of particulate matter in the various size ranges. You must correct the concentrations for the acetone blank.

12.3.2.1 *Acetone blank concentration.* Use Equation 41 to calculate the acetone blank concentration ( $C_a$ ).

12.3.2.2 *Acetone blank weight.* Use Equation 42 to calculate the acetone blank weight ( $W_a$ ).

[**Note:** Correct each of the particulate matter weights per size fraction by subtracting the acetone blank weight (that is,  $M_{2,3,4}-W_a$ )].

12.3.2.3 *Particulate weight catch per size fraction.* Subtract the weight of the acetone blank from the particulate weight catch in each size fraction.

[**Note:** Do not subtract a blank value of greater than 0.001 percent of the weight of the acetone used from the sample weight. Use the following procedures.]

(a) Use Equation 43 to calculate the particulate matter recovered from Containers #1, #2, #3, and #4. This is the total collectable particulate matter ( $C_{if}$ ).

(b) Use Equation 44 to determine the quantitative recovery of  $PM_{10}$  particulate matter ( $C_{IPM10}$ ) from Containers #1, #3, and #4.

(c) Use Equation 45 to determine the quantitative recovery of  $PM_{2.5}$  particulate matter ( $C_{IPM2.5}$ ) recovered from Containers #1 and #4.

12.4 Reporting. You must include the following list of conventional elements in the emissions test report.

(a) Emission test description including any deviations from this protocol.

(b) Summary data tables on a run-by-run basis.

(c) Flowchart of the process or processes tested.

(d) Sketch of the sampling location.

(e) Preliminary traverse data sheets including cyclonic flow checks.

(f) Raw field data sheets.

(g) Laboratory analytical sheets and case narratives.

(h) Sample calculations.

(i) Pretest and post-test calibration data.

(j) Chain of custody forms.

(k) Documentation of process and air pollution control system data.

12.5 Equations. Use the following equations to complete the calculations required in this test method.

*Molecular Weight of Dry Gas.* Calculate the molecular weight of the dry gas using Equation 1.

$$M_d = 0.44 (\% CO_2) + 0.32 (\% O_2) + 0.28 (100 - \% O_2 - \% CO_2) \quad \text{Eq. 1}$$

*Molecular Weight of Wet Gas.* Calculate the molecular weight of the stack gas on a wet basis using Equation 2.

$$M_w = M_d (1 - B_{ws}) + 18 (B_{ws}) \quad \text{Eq. 2}$$

*Gas Viscosity.* Calculate the gas viscosity using Equation 3. This equation uses constants for gas temperatures in °R.

$$\mu = C_1 + C_2 \sqrt{T_s} + C_3 T_s^{-2} + C_4 (\% O_{2,wet}) - C_5 B_{ws} + C_6 B_{ws} T_s^2 \quad \text{Eq. 3}$$

*Cunningham Correction Factor.* The Cunningham correction factor is calculated for a 2.25 micrometer diameter particle.

$$C = 1 + 0.0057193 \left[ \frac{\mu}{P_s D_p} \right] \left[ \frac{T_s}{M_w} \right]^{0.5} \quad \text{Eq. 4}$$

*Lower Limit Cut Diameter for Cyclone I for  $N_{re} < 3,162$ .* The Cunningham correction

factor is for a 2.25 micrometer diameter particle.

$$D_{50LL} = 9.507 C^{0.3007} \left[ \frac{M_w P_s}{T_s} \right]^{0.1993} \quad \text{Eq. 5} \quad (N_{re} < 3,162)$$

*Cut Diameter for Cyclone I for the Middle of the Overlap Zone.*

$$D_{50T} = \left( \frac{11 + D_{50LL}}{2} \right) \quad \text{Eq. 6}$$

*Sampling Rate.*

$$Q_s = Q_l = 0.07296 (\mu) \left[ \frac{T_s}{M_w P_s} \right]^{0.2949} \left[ \frac{1}{D_{50 T}} \right]^{1.4102} \quad \text{Eq. 7}$$

Reynolds Number.

$$N_{re} = 8.64 \times 10^5 \left[ \frac{P_s M_w}{T_s} \right] \left[ \frac{Q_s}{\mu} \right] \quad \text{Eq. 8}$$

Meter Box Orifice Pressure Drop.

$$\Delta H = \left[ \frac{Q_s (1 - B_{ws}) P_s}{T_s} \right]^2 \left[ \frac{1.083 T_m M_d \Delta H_{@}}{P_{bar}} \right] \quad \text{Eq. 9}$$

Lower Limit Cut Diameter for Cyclone I for  $N_{re} \geq 3,162$ . The Cunningham correction

factor is for a 2.25 micrometer diameter particle.

$$D_{50 LL} = 10.0959 C^{0.4400} \left[ \frac{M_w P_s}{T_s} \right]^{0.0600} \quad \text{Equation 10} \\ (N_{re} < 3162)$$

Velocity of Stack Gas. Correct the mean preliminary velocity pressure for  $C_p$  and blockage using Equations 23, 24, and 25.

$$v_s = K_p C_p \left( \sqrt{(\Delta p)} \right)_{avg} \left[ \sqrt{\frac{T_s}{P_s M_w}} \right] \quad \text{Eq. 11}$$

Calculated Nozzle Diameter for Acceptable Sampling Rate.

Velocity of Gas in Nozzle.

Minimum Nozzle/Stack Velocity Ratio Parameter.

$$D = \left[ \frac{3.056 Q_s}{v_s} \right]^{0.5} \quad \text{Eq. 12}$$

$$V_n = \frac{\left( \frac{Q_s}{60} \right)}{A_n} \quad \text{Eq. 13}$$

$$R_{min} = \left[ 0.2457 + \left( 0.3072 - \frac{0.2603 (\mu) (Q_s)^{0.5}}{v_n^{1.5}} \right)^{0.5} \right] \quad \text{Eq. 14}$$

Maximum Nozzle/Stack Velocity Ratio Parameter.

$$R_{max} = \left[ 0.4457 + \left( 0.5690 + \frac{0.2603 (\mu) (Q_s)^{0.5}}{v_n^{1.5}} \right)^{0.5} \right] \quad \text{Eq. 15}$$

Minimum Gas Velocity for  $R_{min} \leq 0.5$ .

$$v_{min} = v_n (0.5) \quad \text{Eq. 16}$$

Minimum Gas Velocity for  $R_{min} \geq 0.5$ .

$$v_{min} = v_n R_{min} \quad \text{Eq. 17}$$

Maximum Gas Velocity for  $R_{max} < 1.5$ .

$$v_{max} = v_n R_{max} \quad \text{Eq. 18}$$

Maximum Gas Velocity for  $R_{max} \geq 1.5$ .

$$v_{\max} = v_n (1.5) \quad \text{Eq. 19}$$

*Minimum Velocity Pressure.*

$$\Delta p_{\min} = 1.3686 \times 10^{-4} \left[ \frac{P_s M_w}{T_s} \right] \left[ \frac{v_{\min}}{C_p} \right]^2 \quad \text{Eq. 20}$$

*Maximum Velocity Pressure.*

$$\Delta p_{\max} = 1.3686 \times 10^{-4} \left[ \frac{P_s M_w}{T_s} \right] \left[ \frac{v_{\max}}{C_p} \right]^2 \quad \text{Eq. 21}$$

*Sampling Time at Point 1.  $N_{tp}$  is the total number of traverse points. You must use the preliminary velocity traverse data.*

$$t_1 = \left[ \frac{\sqrt{\Delta p_1}}{(\sqrt{\Delta p})_{\text{avg}}} \right] \left[ \frac{t_r}{N_{tp}} \right] \quad \text{Eq. 22}$$

*Sampling Time at Point n. You must use the actual test run data at each point, n, and test run point 1.*

$$t_n = t_1 \frac{\sqrt{\Delta p_n}}{\sqrt{\Delta p_1}} \quad \text{Eq. 23}$$

*Adjusted Velocity Pressure.*

$$\Delta p_s = \Delta p_m \left[ \frac{C_p}{C_p} \right]^2 \quad \text{Eq. 24}$$

*Average Probe Blockage Factor.*

$$b_f = \frac{12.0}{A} \quad \text{Eq. 25}$$

*Velocity Pressure.*

$$\Delta p_s 2 = \Delta p_s 1 \left[ \frac{1}{(1 - b_f)} \right]^2 \quad \text{Eq. 26}$$

*Dry Gas Volume Sampled at Standard Conditions.*

$$v_{ms} = \left[ \frac{528}{29.92} \right] [\gamma V_m] \left[ \frac{\left( P_{\text{bar}} + \frac{\Delta H}{13.6} \right)}{T_m} \right] \quad \text{Eq. 27}$$

*Sample Flow Rate at Standard Conditions.*

$$Q_{sST} = \frac{V_{ms}}{\theta} \quad \text{Eq. 28}$$

*Volume of Water Vapor.*

$$V_{ws} = 0.04707 V_c \quad \text{Eq. 29}$$

*Moisture Content of Gas Stream.*

$$B_{ws} = \left[ \frac{V_{ws}}{V_{ms} + V_{ws}} \right] \quad \text{Eq. 30}$$

*Sampling Rate.*

$$Q_s = \frac{29.92}{528} Q_{sST} \left[ \frac{1}{(1 - B_{ws})} \right] \left[ \frac{T_s}{P_s} \right] \quad \text{Eq. 31}$$

[Note: The viscosity and Reynolds Number must be recalculated using the actual stack temperature, moisture, and oxygen content.

*Actual Particle Cut Diameter for Cyclone I.*  
This is based on actual temperatures and pressures measured during the test run.

$$D_{50} = 0.15625 \left[ \frac{T_s}{M_w P_s} \right]^{0.2091} \left[ \frac{\mu}{Q_s} \right]^{0.7091} \quad \text{Eq. 32}$$

*Particle Cut Diameter for  $N_{re} < 3,162$  for Cyclone IV.* C must be recalculated using the actual test run data and a  $D_{50}$  ( $D_p$ ) of 2.5.

$$D_{50} = 0.0024302 \left[ \frac{\mu}{Q_s} \right]^{1.1791} \left[ \frac{1}{C} \right]^{0.5} \left[ \frac{T_s}{P_s M_w} \right]^{0.6790} \quad \text{Equation 33} \\ (N_{re} < 3162)$$

*Particle Cut Diameter for  $N_{re} \geq 3,162$  for Cyclone IV.* C must be recalculated using the actual test run data and a  $D_{50}$  ( $D_p$ ) of 2.5.

$$D_{50} = 0.019723 \left[ \frac{\mu}{Q_s} \right]^{0.8058} \left[ \frac{1}{C} \right]^{0.5} \left[ \frac{T_s}{P_s M_w} \right]^{0.3058} \quad \text{Equation 34} \\ (N_{re} < 3162)$$

*Re-estimated Cunningham Correction Factor.* You must use the actual test run Reynolds Number ( $N_{re}$ ) value and select the

appropriate  $D_{50}$  from Equation 32 or 33 (or Equation 36 or 37 if reiterating).

$$C_r = 1 + 0.0057193 \left[ \frac{\mu}{P_s D_{50}} \right] \left[ \frac{T_s}{M_w} \right]^{0.5} \quad \text{Eq. 35}$$

*Re-calculated Particle Cut Diameter for  $N_{re} < 3,162$ .*

$$D_{50-1} = 0.0024302 \left[ \frac{\mu}{Q_s} \right]^{1.1791} \left[ \frac{1}{C_r} \right]^{0.5} \left[ \frac{T_s}{P_s M_w} \right]^{0.6790} \quad \text{Equation 36} \\ (N_{re} < 3162)$$

*Re-calculated Particle Cut Diameter for  $N \geq 3,162$ .*

$$D_{50-1} = 0.019723 \left[ \frac{\mu}{Q_s} \right]^{0.8058} \left[ \frac{1}{C_r} \right]^{0.5} \left[ \frac{T_s}{P_s M_w} \right]^{0.3058} \quad \text{Equation 37} \\ (N_{re} < 3162)$$

*Ratio (Z) Between  $D_{50}$  and  $D_{50-1}$  Values.*

$$Z = \frac{D_{50-1}}{D_{50}} \quad \text{Eq. 38}$$

*Acceptance Criteria for Z Values.* The number of iterative steps is represented by N.

$$0.99 \leq \left[ Z = \left( \frac{D_{50N}}{D_{50N+1}} \right) \right] \leq 1.01 \quad \text{Eq. 39}$$

Percent Isokinetic Sampling.

$$I = \left( \frac{100 T_s V_{ms} 29.92}{60 v_s A_n P_s (1 - B_{ws}) 528} \right) \quad \text{Eq. 40}$$

Acetone Blank Concentration.

$$C_a = \frac{m_a}{V_a \rho_a} \quad \text{Eq. 41}$$

Acetone Blank Weight.

$$W_a = C_a V_{aw} \rho_a \quad \text{Eq. 42}$$

Concentration of Total Filterable Particulate Matter.

$$C_{tf} = \left( \frac{7000}{453,592} \right) \left[ \frac{M_1 + M_2 + M_3 + M_4}{V_{ms}} \right] \quad \text{Eq. 43}$$

Concentration of Filterable PM<sub>10</sub> Particulate Matter.

$$C_{fPM10} = \left( \frac{7000}{453,592} \right) \left[ \frac{M_1 + M_3 + M_4}{V_{ms}} \right] \quad \text{Eq. 44}$$

Concentration of Filterable PM<sub>2.5</sub> Particulate Matter.

$$C_{fPM2.5} = \left( \frac{7000}{453,592} \right) \left[ \frac{M_1 + M_4}{V_{ms}} \right] \quad \text{Eq. 45}$$

### 13.0 Method Performance

(a) Field evaluation of PM<sub>10</sub> and total particulate matter showed that the precision of constant sampling rate method was the same magnitude as Method 17 (approximately 5 percent). Precision in PM<sub>10</sub> and PM<sub>10</sub> fraction between multiple trains showed standard deviations of 2 to 4 percent and total mass compared to 4.7 percent observed for Method 17 in simultaneous test runs at a Portland cement clinker cooler exhaust. The accuracy of the constant sampling rate PM<sub>10</sub> method for total mass, referenced to Method 17, was  $-2 \pm 4.4$  percent. A small bias was found between Method 201A and Method 17 total particulate matter (10%) (Farthing, 1988).

(b) Laboratory evaluation and guidance for PM<sub>10</sub> cyclones were designed to limit error due to spatial variations to 10 percent. The maximum allowable error due to anisokinetic sampling was limited to  $\pm 20$  percent for 10

µm particles in laboratory tests (Farthing, 1988b).

### 14.0 Pollution Prevention

[Reserved]

### 15.0 Waste Management

[Reserved]

### 16.0 References

We used the following references to develop this test method:

1. Dawes, S.S., and W.E. Farthing. "Application Guide for Measurement of PM<sub>2.5</sub> at Stationary Sources," U.S. Environmental Protection Agency, Atmospheric Research and Exposure Assessment Laboratory, Research Triangle Park, NC 27511, EPA-600/3-90/057 (NTIS No.: PB 90-247198), November 1990.

2. U.S. Environmental Protection Agency, Federal Reference Methods 1 through 5 and Method 17, 40 CFR 60, Appendix A.

3. Richards, J.R. "Test protocol: PCA PM<sub>10</sub>/PM<sub>2.5</sub> Emission Factor Chemical Characterization Testing," PCA R&D Serial No. 2081, Portland Cement Association, 1996.

4. Farthing and Co-workers, 1988a "PM<sub>10</sub> Source Measurement Methodology: Field Studies," EPA 600/3-88/055, NTIS PB89-194287/AS, U.S. Environmental Protection Agency, Research Triangle Park, NC 27711.

5. Farthing and Dawes, 1988b "Application Guide for Source PM<sub>10</sub> Measurement with Constant Sampling Rate," EPA/600/3-88-057, U.S. Environmental Protection Agency, Research Triangle Park, NC 27711.

### 17.0 Tables, Diagrams, Flowcharts, and Validation Data

You must use the following tables, diagrams, flowcharts, and data to complete this test method successfully.

TABLE 1—TYPICAL PARTICULATE MATTER CONCENTRATIONS

Particle size range	Concentration and % by weight
Total collectable particulate	0.015 gr/DSCF.
≤ 10 and > 2.5 micrometers	40% of total collectable particulate matter.
≤ 2.5 micrometers	20% of total collectable particulate matter.



TABLE 2—REQUIRED CYCLONE CUT DIAMETERS ( $D_{50}$ )

Cyclone	Min. cut diameter (Micrometer)	Max. cut diameter (Micrometer)
PM <sub>10</sub> Cyclone (Cyclone I from five stage cyclone) .....	9	11
PM <sub>2.5</sub> Cyclone (Cyclone IV from five stage cyclone) .....	2.25	2.75

TABLE 3—PRETEST CALCULATIONS

If you are using . . .	To calculate . . .	Then use . . .
Preliminary data .....	dry gas molecular weight, $M_d$ .....	Equation 1.
Dry gas molecular weight ( $M_d$ ) and preliminary moisture content of the gas stream.	wet gas molecular weight, $M_w$ .....	Equation 2 <sup>a</sup> .
Stack gas temperature, and oxygen and moisture content of the gas stream.	gas viscosity, $\mu$ .....	Equation 3.
Gas viscosity, $\mu$ .....	Cunningham correction factor <sup>b</sup> , $C$ .....	Equation 4.
Reynolds Number <sup>c</sup> ( $N_{re}$ ) .....	preliminary lower limit cut diameter for cyclone I, $D_{50LL}$ .....	Equation 5.
$N_{re} < 3,162$ .....		
$D_{50LL}$ from Equation 5 .....	cut diameter for cyclone I for middle of the overlap zone, $D_{50T}$ .	Equation 6.
$D_{50T}$ from Equation 6 .....	final sampling rate for cyclone I, $Q_I(Q_s)$ .....	Equation 7.
$Q_I(Q_s)$ from Equation 7 .....	(verify) the assumed Reynolds number .....	Equation 8.

<sup>a</sup> Use Method 4 to determine the moisture content of the stack gas. Use a wet bulb-dry bulb measurement device or hand-held hygrometer to estimate moisture content of sources with gas temperature less than 160 °F.

<sup>b</sup> For the lower cut diameter of cyclone IV, 2.25 micrometer.

<sup>c</sup> Verify the assumed Reynolds number using the procedure in Section 8.5.1, before proceeding to Equation 9.

TABLE 4— $\Delta H$  VALUES BASED ON PRELIMINARY TRAVERSE DATA

Stack temperature (°R)	$T_s - 50^\circ$	$T_s$	$T_s + 50^\circ$
$\Delta H$ , (in. W.C.) .....	—	—	—

TABLE 5—VERIFICATION OF THE ASSUMED REYNOLDS NUMBER

If the $N_{re}$ is . . .	Then . . .	And . . .
$< 3,162$ .....	Calculate $\Delta H$ for the meter box.	
$\geq 3,162$ .....	Recalculate $D_{50LL}$ using Equation 10 .....	Substitute the “new” $D_{50LL}$ into Equation 6 to recalculate $D_{50T}$ .

TABLE 6—CALCULATIONS FOR RECOVERY OF PM<sub>10</sub> AND PM<sub>2.5</sub>

Calculations	Instructions and references
Average dry gas meter temperature .....	See field test data sheet.
Average orifice pressure drop .....	See field test data sheet.
Dry gas volume ( $V_{ms}$ ) .....	Use Equation 27 to correct the sample volume measured by the dry gas meter to standard conditions (20 °C, 760 mm Hg or 68 °F, 29.92 in. Hg).
Dry gas sampling rate ( $Q_{sST}$ ) .....	Must be calculated using Equation 28.
Volume of water condensed ( $V_{ws}$ ) .....	Use Equation 29 to determine the water condensed in the impingers and silica gel combination. Determine the total moisture catch by measuring the change in volume or weight in the impingers and weighing the silica gel.
Moisture content of gas stream ( $B_{ws}$ ) .....	Calculate this with Equation 30.
Sampling rate ( $Q_s$ ) .....	Calculate this with Equation 31.
Test condition Reynolds number <sup>a</sup> .....	Use Equation 8 to calculate the actual Reynolds number during test conditions.
Actual $D_{50}$ of Cyclone I .....	Calculate this with Equation 32. This calculation is based on the average temperatures and pressures measured during the test run.
Stack gas velocity ( $v_s$ ) .....	Calculate this with Equation 11.
Percent isokinetic rate (%) .....	Calculate this with Equation 40.

<sup>a</sup> Calculate the Reynolds number at the cyclone IV inlet during the test based on: (1) The sampling rate for the combined cyclone head, (2) the actual gas viscosity for the test, and (3) the dry and wet gas stream molecular weights.

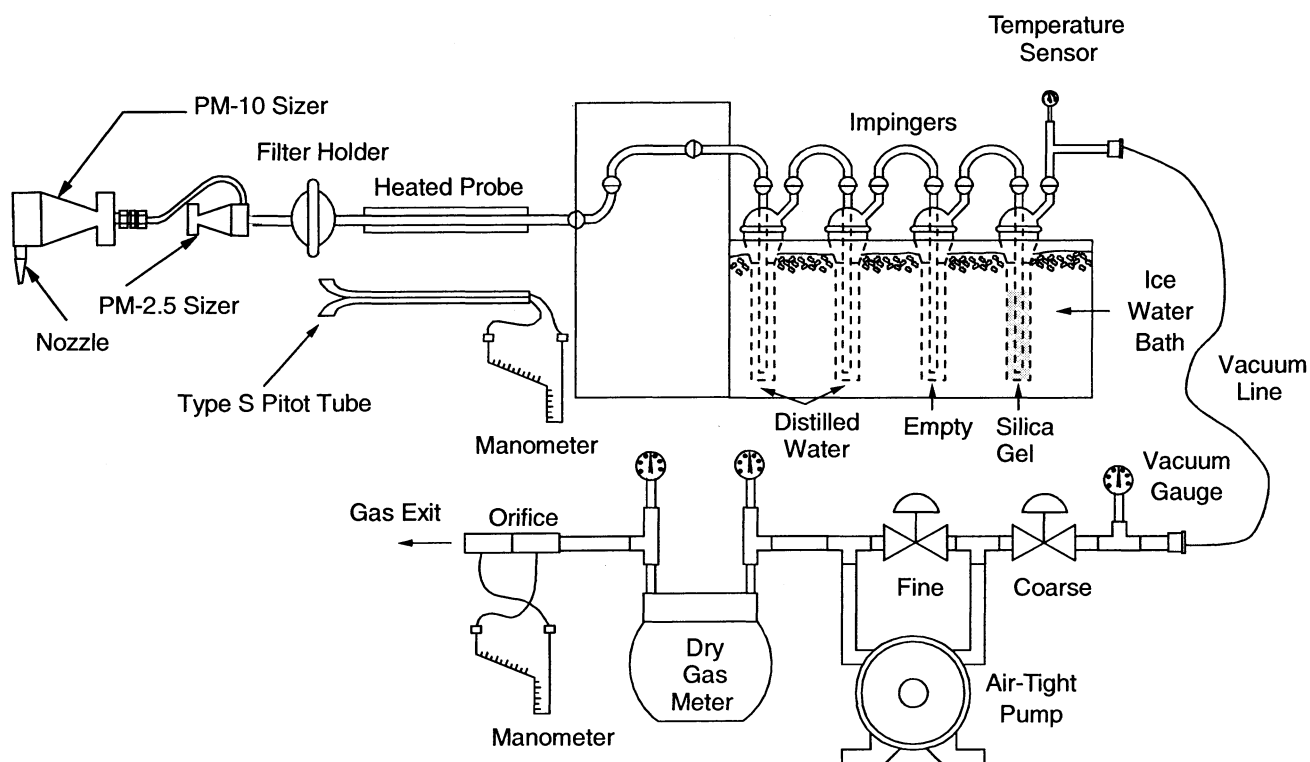


Figure 1. In-stack  $PM_{10}$  and  $PM_{2.5}$  Sampling Train

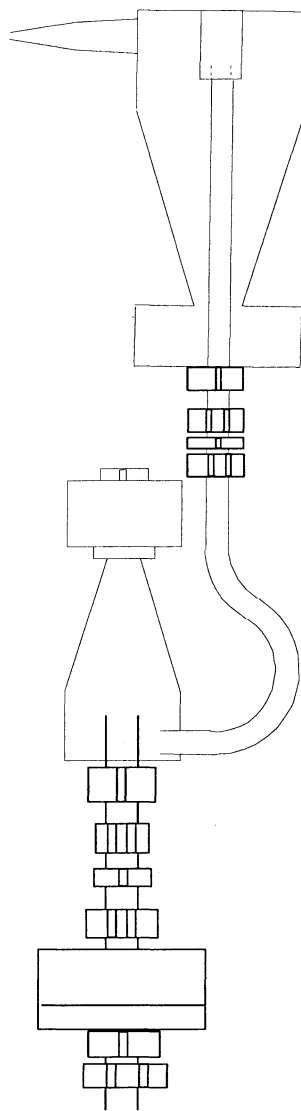
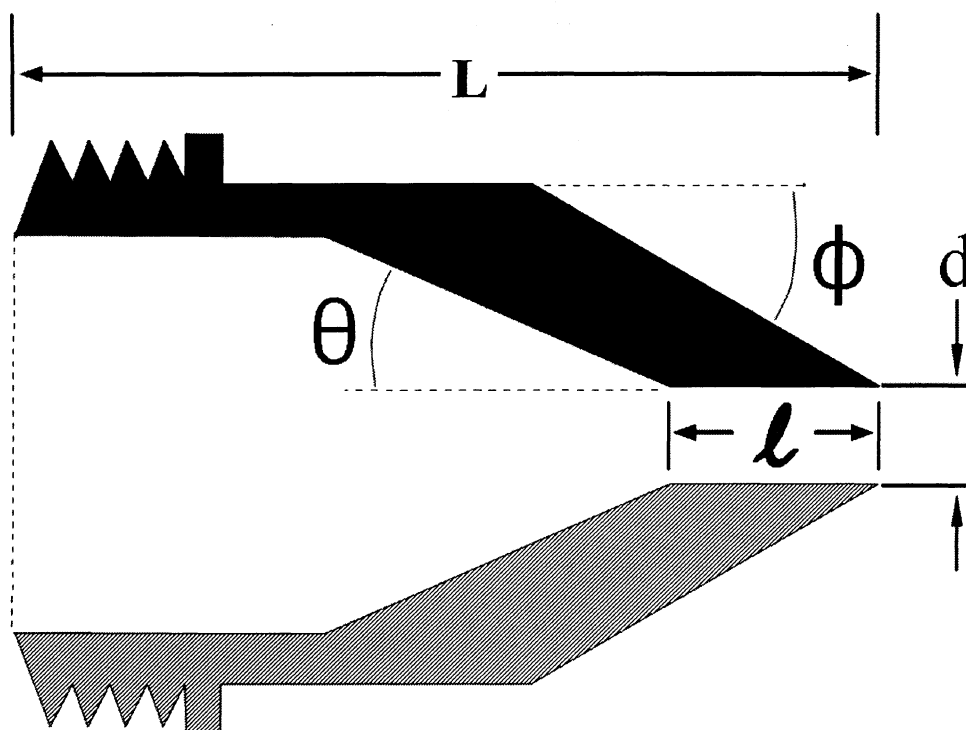
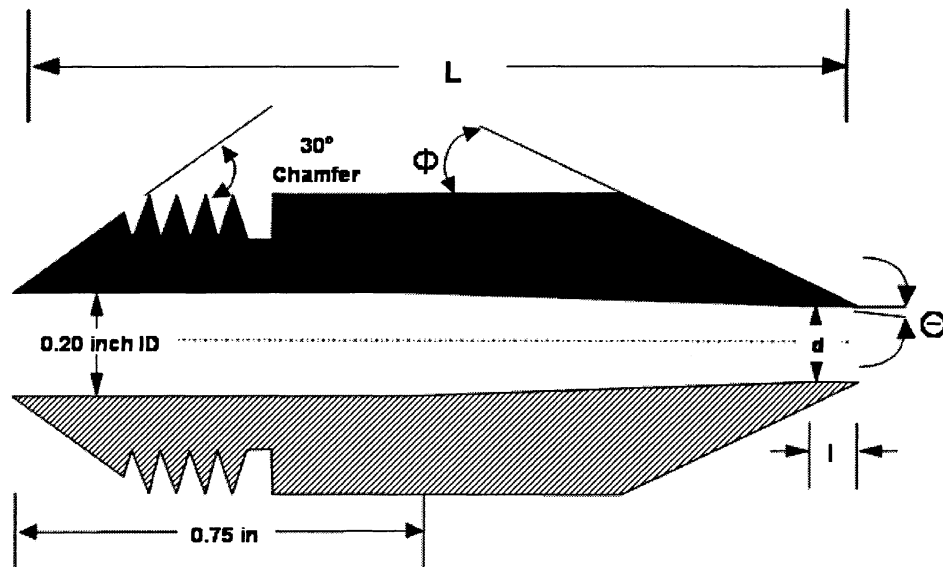


Figure 2. Combined Cyclone Sampling Head



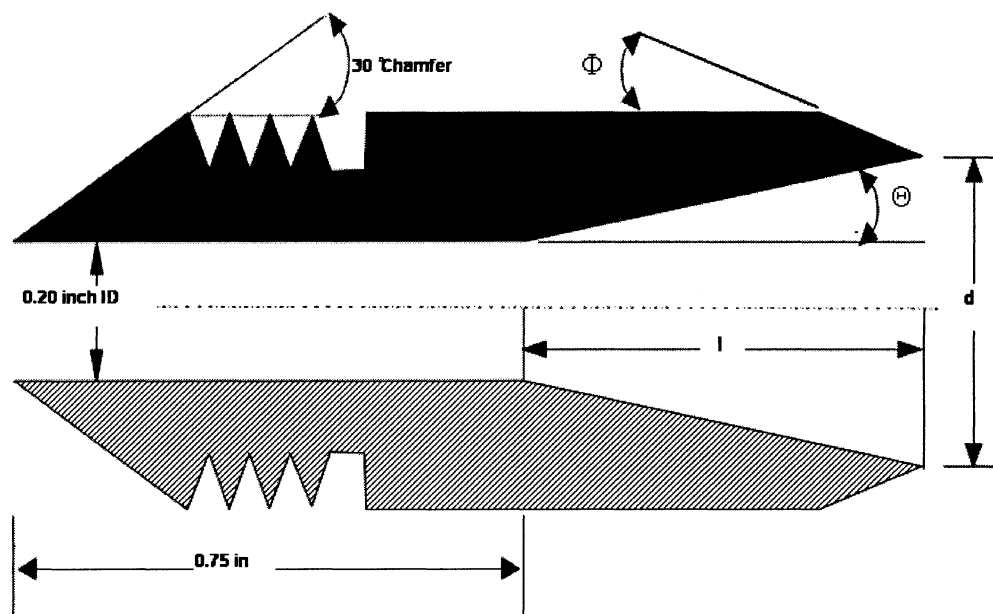
Nozzle Diameter, $d$ (inches)	Cone Angle, $\theta$ (degrees)	Outside Taper, $\phi$ (degrees)	Straight Inlet Length, $l$ (inches)	Total Length, $L$ (inches)
0.125	4	15	<0.05	$2.710 \pm 0.05$
0.136	4	15	<0.05	$2.653 \pm 0.05$
0.150	4	15	<0.05	$2.553 \pm 0.05$
0.164	5	15	<0.05	$1.970 \pm 0.05$
0.180	6	15	<0.05	$1.572 \pm 0.05$
0.197	6	15	<0.05	$1.491 \pm 0.05$
0.215	6	15	<0.05	$1.450 \pm 0.05$
0.233	6	15	<0.05	$1.450 \pm 0.05$
0.264	5	15	<0.05	$1.450 \pm 0.05$
0.300	4	15	<0.05	$1.480 \pm 0.05$
0.342	4	15	<0.05	$1.450 \pm 0.05$
0.390	3	15	<0.05	$1.450 \pm 0.05$

Figure 3. Nozzle Design Specifications for PM<sub>10</sub> Cyclone



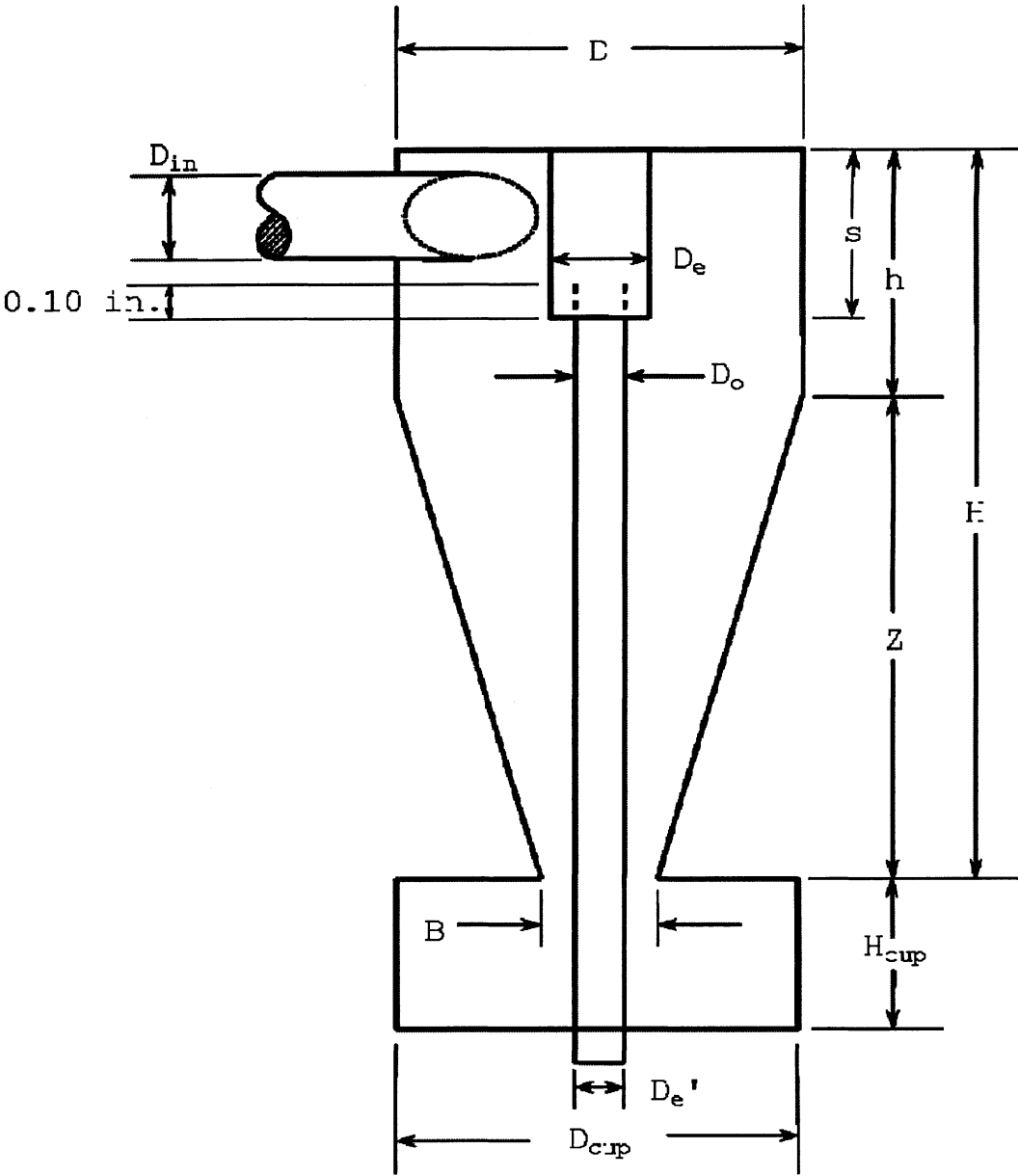
Nozzle Diameter, d (in.)	Internal Cone Angle, $\theta$ (degrees)	Outside Taper, $\phi$ (degrees)	Straight Inlet Length, l (in.)	Total Length, L (in.)
0.125	3	15	$\leq 0.05$	$1.45 \pm 0.05$
0.138	2	15	$\leq 0.05$	$1.45 \pm 0.05$
0.156	1	15	$\leq 0.05$	$1.45 \pm 0.05$
0.172	1	15	$\leq 0.05$	$1.45 \pm 0.05$
0.188	1	15	$\leq 0.05$	$1.45 \pm 0.05$
0.200	0	15	$\leq 0.05$	$1.45 \pm 0.05$

Figure 4A. Nozzle Design for PM<sub>2.5</sub> Cyclone (Higher Stack Flow)



Nozzle diameter, d (inches)	Cone Angle, $\theta$ (degrees)	Outside taper, $\phi$ (degrees)	Internal Taper length, l (inches)
0.216	5	15	0.093
0.234	5	15	0.194
0.253	5	15	0.304
0.274	5	15	0.422
0.296	5	15	0.549
0.320	5	15	0.688

**Figure 4B. Nozzle Design for PM<sub>2.5</sub> Cyclone (Lower Stack Flow)**

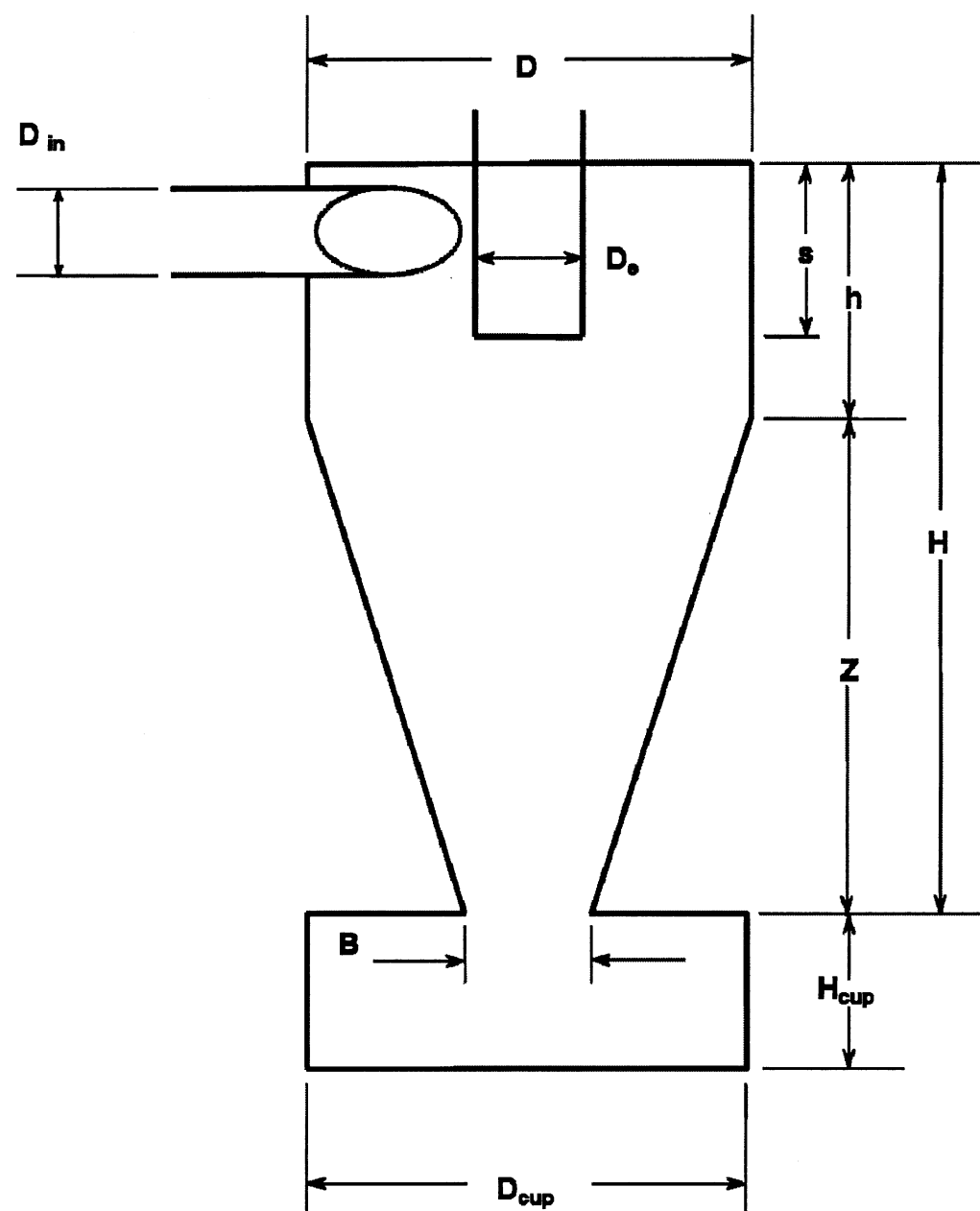


Cyclone I (10 Micrometer)	Cyclone Interior Dimensions (cm ± 0.02 cm)											
	$D_{in}$	$D$	$D_e$	$B$	$H$	$h$	$Z$	$S$	$H_{cup}$	$D_{cup}$	$D_e'$	$D_o$
	1.27	4.47	1.50	1.88	6.95	2.24	4.71	1.57	2.25	4.45	1.02	1.24

Figure 5. Design Specifications for Cyclone I  
(10 Micrometer)



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Cyclone IV (2.5 Micrometer)	Cyclone Interior Dimensions (cm $\pm$ 0.02 cm)									
	$D_{in}$	$D$	$D_e$	$B$	$H$	$h$	$Z$	$S$	$H_{cup}$	$D_{cup}$
	0.51	2.54	0.59	1.09	2.68	1.03	1.65	0.58	2.22	2.62

Figure 6. Design Specifications for Cyclone IV (2.5 Micrometer) Sizing Device

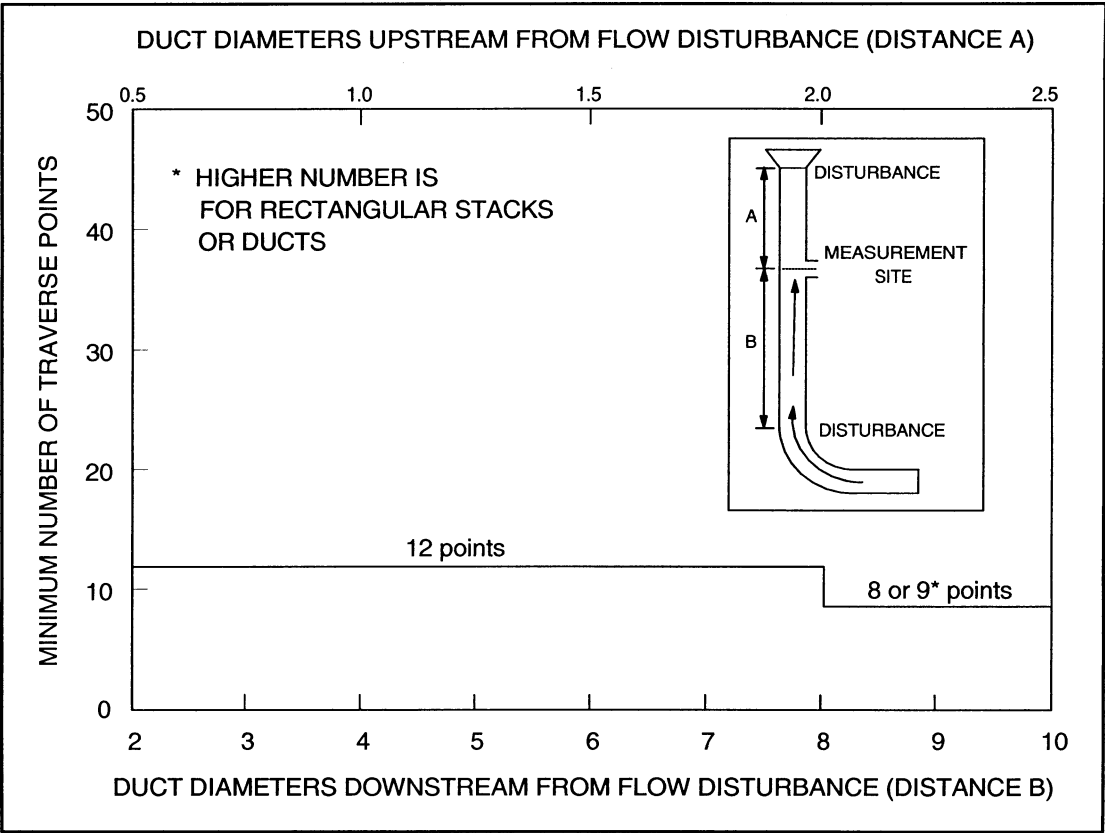


Figure 7. Minimum Number of Traverse Points  
for Preliminary Method 4 Traverse

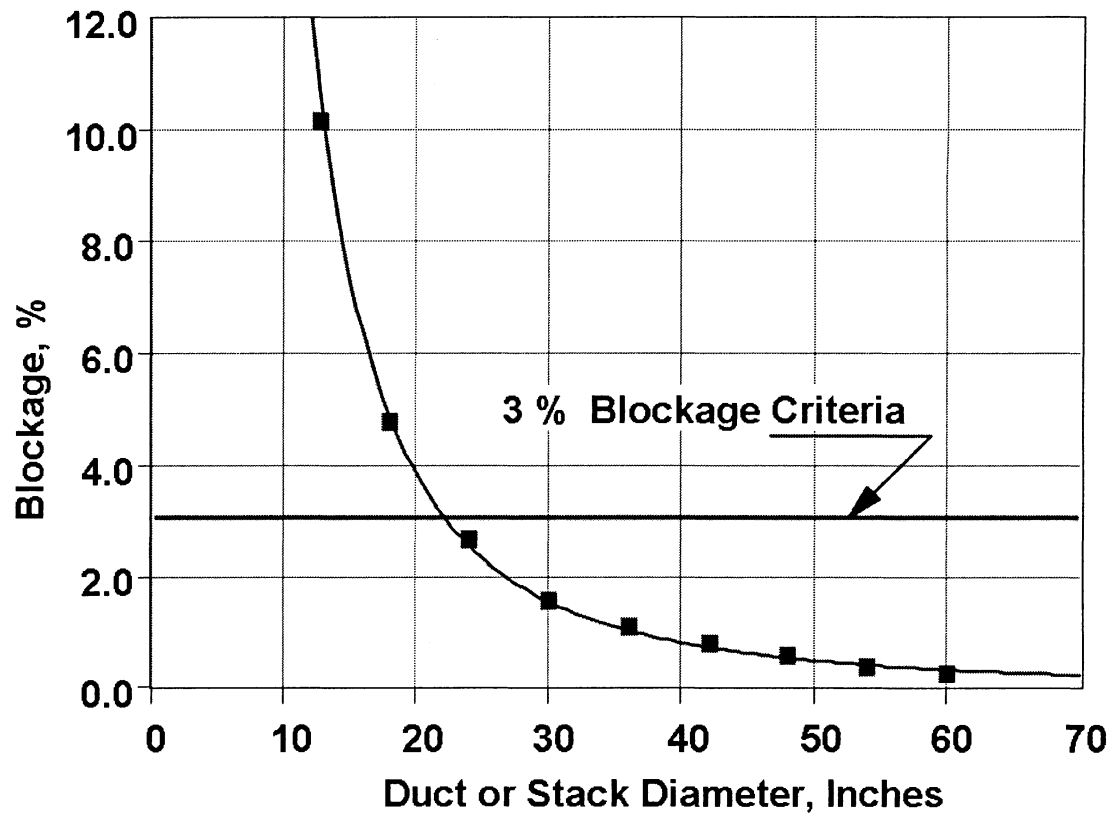


Figure 8. Gas Flow Blockage by the Combined Cyclone Sampling Head

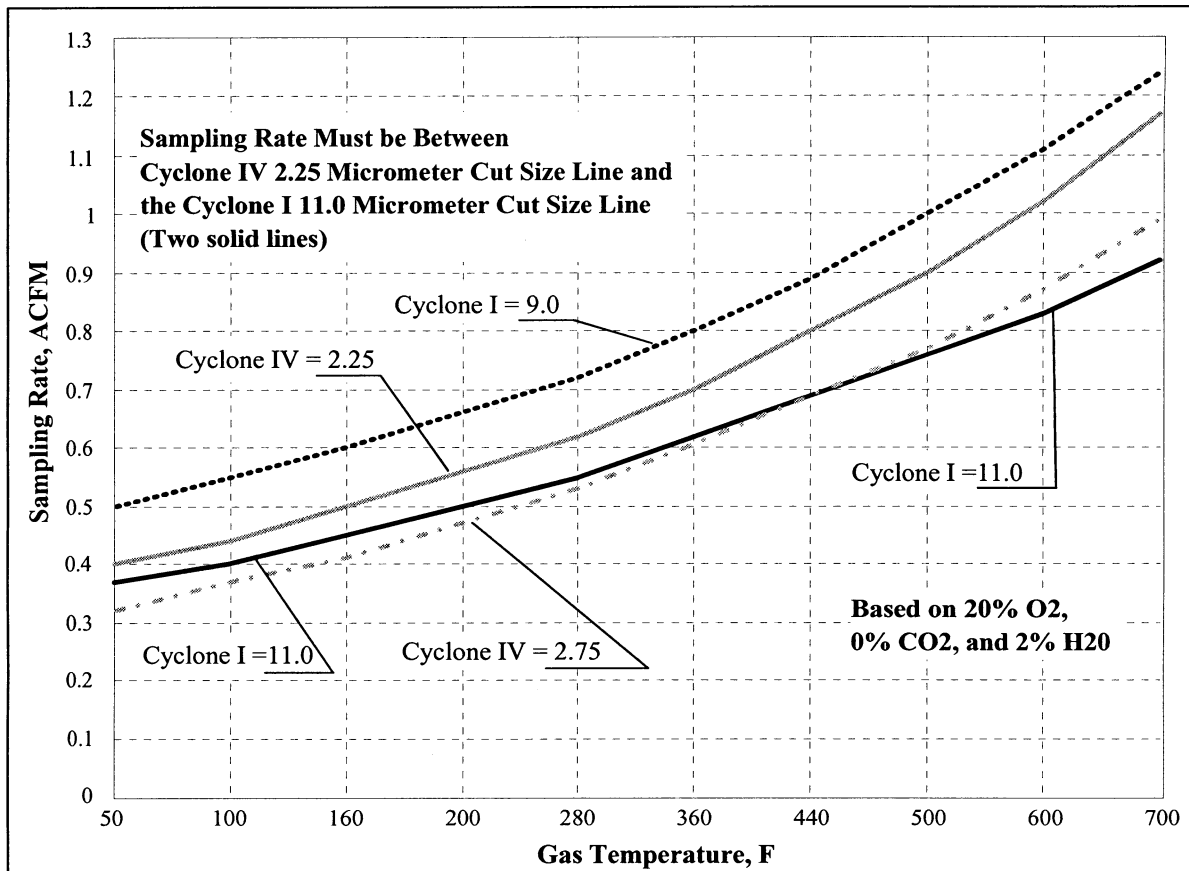


Figure 9. Acceptable Sampling Rate for Combined Cyclone Heads

## METHOD 202—DRY IMPINGER METHOD FOR DETERMINING CONDENSABLE PARTICULATE EMISSIONS FROM STATIONARY SOURCES

### 1.0 Scope and Applicability

1.1 Scope. The U.S. Environmental Protection Agency (U.S. EPA or “we”) developed this method to describe the procedures that the stack tester (“you”) must follow to measure condensable particulate matter (CPM) emissions from stationary sources. This method includes procedures for measuring both organic and inorganic CPM.

1.2 Applicability. You can use this method to measure CPM from stationary source emissions after filterable particulate matter has been removed. CPM is measured in the emissions after removal from the stack and after passing through a filter. You can use Method 17 to collect condensable and filterable particulate material from sources operating at stack temperatures and/or samples collected below 30 °C (85 °F) if the filter is treated as described in Sections 8.5.4.4 and 11.2.1 of this method. You may use this method only for stationary source emission measurements.

1.3 Responsibility. You are responsible for obtaining the equipment and supplies you will need to use this method. You must also develop your own procedures for following this method and any additional procedures to ensure accurate sampling and analytical measurements.

1.4 Results. To obtain reliable results, you must have a thorough knowledge of the following test methods that are found in Appendices A-1 through A-3 and A-6 to Part 60, and in Appendix M to Part 51:

(a) Method 1—Sample and Velocity Traverses for Stationary Sources.

(b) Method 2—Determination of Stack Gas Velocity and Volumetric Flow Rate (Type S Pitot Tube).

(c) Method 3—Gas Analysis for the Determination of Dry Molecular Weight.

(d) Method 4—Determination of Moisture Content in Stack Gases.

(e) Method 5—Determination of Particulate Matter Emissions from Stationary Sources.

(f) Method 17—Determination of Particulate Matter Emissions from Stationary Sources (in-stack filtration method).

(g) Method 201A—Determination of PM<sub>10</sub> and PM<sub>2.5</sub> Emissions from Stationary Sources (Constant Sampling Rate Procedure)

1.5 Additional Methods. You will need additional test methods to measure filterable particulate matter. You may use this method to collect CPM in conjunction with Method 5 or 17 of Appendices A-1 through A-3 and A-6 to Part 60 or, Method 201A of Appendix M to Part 51. The sample train operation and front end recovery and analysis are conducted according to the filterable particulate method you choose. This method addresses the equipment, preparation, and analysis necessary to measure only CPM.

1.6 Limitations. You can use this method to measure emissions following a wet scrubber only when this method is combined with a filterable particulate method that operates at high enough temperatures to cause water droplets sampled through the probe to become gaseous.

1.7 Conditions. You must maintain isokinetic sampling conditions to meet the requirements of the filterable particulate method used in conjunction with this method. You must sample at the required number of sampling points specified in Method 5, 17, or 201A. Also, if you are using this method as an alternative to a required performance test method, you must receive approval from the appropriate authorities prior to conducting the test.

### 2.0 Summary of Method

2.1 Summary. The CPM is collected in dry impingers after filterable particulate material has been collected on filters maintained above 30 °C (85 °F) using Method 5, 17, or 201A. The organic and aqueous fractions of the impingers and an out-of-stack CPM filter are then taken to dryness and weighed. The total of all fractions represents the CPM. Compared to the December 17, 1991 promulgated Method 202, this method removes water from the impingers and includes the addition of a condenser followed by a water dropout impinger immediately after the final in-stack or heated filter. This method also includes the addition of one modified Greenburg Smith impinger and a CPM filter following the water dropout impinger. Figure 1 of Section 18 presents the schematic of the sampling train configured with these changes.

2.1.1 Condensable Particulate Matter. CPM is collected in the water dropout impinger, the modified Greenburg Smith impinger, and the CPM filter of the sampling train as described in this method. The impinger contents are purged with nitrogen (N<sub>2</sub>) immediately after sample collection to remove dissolved sulfur dioxide (SO<sub>2</sub>) gases from the impinger. The CPM filter is extracted with water and methylene chloride. The impinger solution is then extracted with methylene chloride (MeCl<sub>2</sub>). The organic and aqueous fractions are dried and the residues are weighed. The total of the aqueous and organic fractions represents the CPM.

2.1.2 Dry Impinger and Additional Filter. The potential artifacts from SO<sub>2</sub> are reduced using a condenser and dropout impinger to separate CPM from reactive gases. No water is added to the impingers prior to the start of sampling. To improve the collection efficiency of CPM, an additional filter (the CPM filter) is placed between the second and third impingers.

### 3.0 Definitions

3.1 Primary PM. Primary PM (also known as direct PM) means particles that enter the atmosphere as a direct emission from a stack or an open source. Primary PM comprises two components: filterable PM and condensable PM. These two PM components have no upper particle size limit.

3.2 Filterable PM. Filterable PM means particles that are emitted directly by a source as a solid or liquid at stack or release conditions and captured on the filter of a stack test train.

3.3 Primary PM<sub>10</sub>. Primary PM<sub>10</sub> (also known as direct PM<sub>10</sub>, total PM<sub>10</sub>, PM<sub>10</sub> or filterable PM<sub>10</sub>, and condensable PM, individually) means particulate matter with an aerodynamic diameter equal to or less than 10 micrometers.

3.4 Primary PM<sub>2.5</sub>. Primary PM<sub>2.5</sub> (also known as direct PM<sub>2.5</sub>, total PM<sub>2.5</sub>, PM<sub>2.5</sub>, or filterable PM<sub>2.5</sub>, and condensable PM, individually) means solid particles emitted directly from an air emissions source or activity, or gaseous emissions or liquid droplets from an air emissions source or activity that condense to form particulate matter at ambient temperatures. Direct PM<sub>2.5</sub> emissions include elemental carbon, directly emitted organic carbon, directly emitted sulfate, directly emitted nitrate, and other inorganic particles (including but not limited to crustal material, metals, and sea salt).

3.5 Condensable PM (CPM). Condensable PM means material that is vapor phase at stack conditions, but which condenses and/or reacts upon cooling and dilution in the ambient air to form solid or liquid PM immediately after discharge from the stack. Note that all condensable PM is assumed to be in the PM<sub>2.5</sub> size fraction (Reference: Part 51, Subpart Z (51.1000)).

### 4.0 Interferences [Reserved]

### 5.0 Safety

*Disclaimer:* You may have to use hazardous materials, operations, and equipment while performing this method. We do not provide information on appropriate safety and health practices. You are responsible for determining the applicability of regulatory limitations and establishing appropriate safety and health practices. Handle materials and equipment properly.

### 6.0 Equipment and Supplies

The equipment used in the filterable particulate portion of the sampling train is described in Methods 5 and 17 of Appendix A-1 through A-3 and A-6 to Part 60 and Method 201A in Appendix M to Part 51. The equipment used in the CPM portion of the train is described in this section.

6.1 Condensable Particulate Sampling Train Components. The sampling train for this method is consistent with the sampling train for collecting filterable particulate using Method 5, 17, or 201A with the following exceptions or additions:

6.1.1 Condenser and Impingers. You must add the following components to the filterable particulate sampling train: A Method 23 type condenser as described in Section 2.1.2 of Method 23 of Appendix A-8 to Part 60, followed by a dropout impinger or flask, followed by a modified Greenburg-Smith impinger with an open tube tip as described in Section 6.1.1.8 of Method 5.

6.1.2 CPM Filter Holder. The modified Greenburg-Smith impinger is followed by a filter holder that is either glass, stainless steel (316 or equivalent), or Teflon®-coated stainless steel. Commercial size filter holders are available depending on project requirements. Use a commercial filter holder capable of supporting 47 mm or greater diameter filters. Commercial size filter holders contain a Teflon® O-ring, stainless steel, ceramic or Teflon® filter support and a final Teflon® O-ring. At the exit of the CPM filter, install a Teflon®-coated or stainless steel encased thermocouple that is in contact with the gas stream.

6.1.3 Long Stem Impinger Insert. You will need a long stem modified Greenburg Smith

impinger insert for the dropout impinger to perform the nitrogen purge of the sampling train.

## 6.2 Sample Recovery Equipment.

### 6.2.1 Condensable Particulate Matter Recovery.

6.2.1.1 Nitrogen Purge Line. You must use inert tubing and fittings capable of delivering at least 20 liters/min of nitrogen gas to the impinger train from a standard gas cylinder (see Figure 2 of Section 18). You may use standard 0.6 cm (1/4-in.) tubing and compression fittings in conjunction with an adjustable pressure regulator and needle valve.

6.2.1.2 Rotameter. You must use a rotameter capable of measuring gas flow up to 20 L/min. The rotameter must be accurate to 5 percent of full scale.

6.2.1.3 Ultra-high Purity (UHP) Nitrogen Gas. Compressed ultra-pure nitrogen, regulator, and filter must be capable of providing at least 20 L/min purge gas for 1 hour through the sampling train.

6.3 Analysis. The following equipment is necessary for CPM sample recovery and analysis:

6.3.1 Separatory Funnel. Glass, 1 liter.

6.3.2 Weighing Tins. 50 mL.

6.3.3 Glass Beakers. 300 to 500 mL.

6.3.4 Drying Equipment. Hot plate or oven with temperature control.

6.3.5 Pipets. 5 mL.

6.3.6 Burette. Glass, 0 to 100 mL in 0.1 mL graduations.

6.3.7 Analytical Balance. Analytical balance capable of weighing 0.0001 g (0.1 milligram). For extremely low emission sources, a balance capable of weighing 0.00001 g (0.01 milligram) may be required.

6.3.8 pH Meter. A meter capable of determining the acidity of liquid within 0.1 pH units.

## 7.0 Reagents and Standards

7.1 Sample Collection. To collect a sample, you will need a Teflon® filter, crushed ice, and silica gel. You must also have water and nitrogen gas to purge the sampling train. You will find additional information on each of these items in the following summaries.

7.1.1 Filter. You must use a Teflon® membrane filter that does not have an organic binder. The filter must also have an efficiency of at least 99.95 percent (<0.05 percent penetration) on 0.3 micron particles. You may use test data from the supplier's quality control program to document filter efficiency. If the source you are sampling has SO<sub>2</sub> or sulfur trioxide (SO<sub>3</sub>) emissions, then you must use a filter that will not react with SO<sub>2</sub> or SO<sub>3</sub>. Depending on your application and project data quality objectives (DQOs), filters are commercially available in 47 mm and larger sizes.

7.1.2 Silica Gel. Use an indicating-type silica gel of 6 to 16 mesh. We must approve other types of desiccants (equivalent or better) before you use them. Allow the silica gel to dry for 2 hours at 175 °C (350 °F) if it is being reused. You do not have to dry new silica gel.

7.1.3 Water. Use deionized distilled ultra-filtered water (to conform to ASTM D1193–06, Type 1 water or equivalent) (incorporated

by reference) to recover material caught in the impinger, if required. The Director of the Federal Register approves this incorporation by reference in accordance with 5 U.S.C. 552(a) and 1 CFR part 51. You may obtain a copy from American Society for Testing and Materials (ASTM), 100 Barr Harbor Drive, Post Office Box C700, West Conshohocken, PA 19428–2959. You may inspect a copy at the Office of **Federal Register**, 800 North Capitol Street, NW., Suite 700, Washington, DC.

7.1.4 Crushed Ice. Obtain from the best readily available source.

7.1.5 Nitrogen Gas. Use Ultra-High Purity (UHP) compressed nitrogen or equivalent to purge the sampling train. The compressed nitrogen you use to purge the sampling train must contain no more than 1 ppm oxygen, 1 ppm total hydrocarbons as carbon, and 2 ppm moisture.

7.2 Sample Recovery and Analytical Reagents. You will need acetone, MeCl<sub>2</sub>, anhydrous sodium sulfate, ammonia hydroxide (NH<sub>4</sub>OH), and deionized water for the sample recovery and analysis. Unless otherwise indicated, all reagents must conform to the specifications established by the Committee on Analytical Reagents of the American Chemical Society. If such specifications are not available, then use the best available grade. Find additional information on each of these items in the following paragraphs:

7.2.1 Acetone. Use acetone that is stored in a glass bottle. Do not use acetone from a metal container because it normally produces a high residue blank. You must use acetone with blank values <1 ppm, by weight, residue.

7.2.2 Methylene Chloride, American Chemical Society (ACS) grade. You must use methylene chloride with a blank value <1.5 ppm, by weight, residue.

7.2.3 Water. Use deionized distilled ultra-filtered water (to conform to ASTM D1193–06, Type 1 or equivalent) (incorporated by reference) to recover material caught in the impinger.

7.2.4 Condensable Particulate Sample Desiccant. Use indicating-type anhydrous sodium sulfate to desiccate water and organic extract residue samples.

7.2.5 Ammonium Hydroxide. Use NIST traceable or equivalent (0.1 N) NH<sub>4</sub>OH.

7.2.6 Standard Buffer Solutions. Use one buffer with a neutral pH and a second buffer solution with an acid pH.

## 8.0 Sample Collection, Preservation, Storage, and Transport

8.1 Qualifications. This is a complex test method. To obtain reliable results, you must be trained and experienced with in-stack filtration systems (such as, cyclones, impactors, and thimbles) and impinger and moisture train systems.

8.2 Preparations. You must clean glassware prior to field tests as described in Section 8.4, including baking glassware at 300 °C for 6 hours prior to use. Cleaned, baked glassware is used at the start of each new source category tested. Analyze reagent blanks (water, acetone, and methylene chloride) before field tests to verify low blank concentrations. Follow the pretest

preparation instructions in Section 8.1 of Method 5.

8.3 Site Setup. You must follow the procedures required by filterable particulate sampling method setup run in conjunction with this method including:

(a) Determining the sampling site location and traverse points.

(b) Calculating probe/cyclone blockage.

(c) Verifying the absence of cyclonic flow.

(d) Completing a preliminary velocity profile, and selecting a nozzle(s).

8.3.1 Sampling Site Location and Traverse Point. Determination. Follow the standard procedures in Method 1 of Appendix A–1 to Part 60 to select the appropriate sampling site. Then you must do all of the following:

8.3.1.1 Sampling site. Choose a location that maximizes the distance from upstream and downstream flow disturbances.

8.3.1.2 Traverse points. Use the recommended maximum number of traverse points at any location, as found in Methods 5, 17, or 201A, whichever is applicable to your test requirements. You must prevent the disturbance and capture of any solids accumulated on the inner wall surfaces by maintaining a 1-inch distance from the stack wall (½ inch for sampling locations less than 24 inches in diameter).

8.4 Sampling Train Preparation. A schematic of the sampling train used in this method is shown in Figure 1 of Section 18. All sampling train glassware must be cleaned prior to the test with soap and water, and rinsed using tap water, deionized water, acetone, and finally, MeCl<sub>2</sub>. It is important to completely remove all silicone grease from areas that will be exposed to the MeCl<sub>2</sub> rinse during sample recovery. After cleaning, you must bake glassware at 300 °C for 6 hours prior to each source type sampled. Prior to each sampling run, the train glassware used to collect condensable particulate matter must be rinsed thoroughly with deionized, distilled ultra-filtered water that conforms to ASTM D1193–06, Type 1 or equivalent (incorporated by reference).

8.4.1 Condenser and Dropout Impinger. Add a Method 23 type condenser and a condensate dropout impinger without bubbler tube after the final in-stack or out-of-stack hot filter assembly. The Method 23 type stack gas condenser is described in Section 2.1.2 of Method 23. It must be capable of cooling the stack gas to less than 30 °C (85 °F).

8.4.2 Backup Impinger. The dropout impinger is followed by a modified Greenburg Smith impinger with no taper (see Figure 1 of Section 18). Place the dropout and other impingers in an insulated box with water at ≤ 30 °C (≤ 85 °F). At the start of the tests, the water dropout and backup impinger must be clean, without any water or reagent added.

8.4.3 CPM Filter. Place a filter holder with a filter meeting the requirements in Section 6.1.2 following the modified Greenburg-Smith impinger. The connection between the CPM filter and the moisture trap impinger includes a thermocouple fitting that provides a leak-free seal between the thermocouple and the stack gas.

[**Note:** A thermocouple well is not sufficient for this purpose because the

Teflon® or steel encased thermocouple must be in contact with the sample gas).]

**8.4.4 Moisture Traps.** You must use a modified Greenburg-Smith impinger containing 100 mL of water or the alternative described in Method 5 followed by an impinger containing silica gel to collect moisture that passes through the CPM filter. You must maintain the gas temperature below 20°C (68 °F) at the exit of the moisture traps.

**8.4.5 Silica Gel Trap.** Place 200 to 300 g of silica gel in each of several air-tight containers. Weigh each container, including silica gel, to the nearest 0.5 g, and record this weight on the filterable particulate data sheet. As an alternative, the silica gel need not be preweighed, but may be weighed directly in its impinger or sampling holder just prior to train assembly.

**8.4.6 Leak-Check (Pretest).** Use the procedures outlined in Method 5, 17, or 201A as appropriate to leak check the entire sampling system. Specifically, perform the following procedures:

**8.4.6.1 Sampling Train.** You must pretest the entire sampling train for leaks. The pretest leak-check must have a leak rate of not more than 0.02 actual cubic feet per minute (ACFM) or 4 percent of the average sample flow during the test run, whichever is less. Additionally, you must conduct the leak-check at a vacuum equal to or greater than the vacuum anticipated during the test run. Enter the leak-check results on the field test data sheet for the filterable particulate method.

(**Note:** Conduct leak-checks during port changes only as allowed by the filterable particulate method used with this method).

**8.4.6.2 Pitot Tube Assembly.** After you leak-check the sample train, perform a leak-check of the pitot tube assembly. Follow the procedures outlined in Section 8.4.1 of Method 5.

**8.5 Sampling Train Operation.** Operate the sampling train as described in the filterable particulate sampling method (*i.e.*, Method 5, 17, or 201A) with the following additions or exceptions:

**8.5.1 CPM Filter Assembly.** On the field data sheet for the filterable particulate method, record the CPM filter temperature readings at the beginning of each sample time increment and when sampling is halted. Maintain the CPM filter  $\leq 30^{\circ}\text{C}$  ( $\leq 85^{\circ}\text{F}$ ) during sample collection.

**8.5.2 Leak-Check Probe/Sample Train Assembly (Post-Test).** Conduct the leak rate check according to the filterable particulate sampling method used during sampling. If required, conduct the leak-check at a vacuum equal to or greater than the maximum vacuum achieved during the test run. If the leak rate of the sampling train exceeds 0.02 ACFM or 4 percent of the average sampling rate during the test run (whichever is less), then the run is invalid and you must repeat it.

**8.5.3 Post-Test Nitrogen Purge.** As soon as possible after the post-test leak-check, detach the probe, any cyclones, and in-stack or hot filters from the condenser and impinger train. Leave the ice in the second impinger box to prevent removal of moisture during the purge. If necessary, add more ice

during the purge to maintain the gas temperature measured at the exit of the silica gel impinger below 20 °C (68 °F).

**8.5.3.1** If no water was collected before the CPM filter, then you may skip the remaining purge steps and proceed with sample recovery (see Section 8.5.4).

**8.5.3.2** Replace the short stem impinger insert with a modified Greenberg Smith impinger insert. The impinger tip length must extend below the water level in the impinger catch. If insufficient water was collected, you must add a measured amount of degassed deionized, distilled ultra-filtered ASTM D1193–06, Type 1 or equivalent) (incorporated by reference) water until the impinger tip is at least 1 cm below the surface of the water. You must record the amount of water added to the dropout impinger (see Figure 4 of Section 18) to correct the moisture content of the effluent gas.

(**Note:** Prior to use, water must be degassed using a nitrogen purge bubbled through the water for at least 15 minutes to remove dissolved oxygen).

**8.5.3.3** With no flow of gas through the clean purge line and fittings, attach the line to a purged inline filter. Connect the filter outlet to the input of the impinger train (see Figure 2 of Section 18). To avoid over- or under-pressurizing the impinger array, slowly commence the nitrogen gas flow through the line while simultaneously opening the meter box pump valve(s). Adjust the pump bypass and nitrogen delivery rates to obtain the following conditions: (1) 20 liters/min or  $\Delta H@$ , and (2) a positive overflow rate through the rotameter of less than 2 liters/min. Condition (2) guarantees that the nitrogen delivery system is operating at greater than ambient pressure and prevents the possibility of passing ambient air (rather than nitrogen) through the impingers. During the purge, continue operation of the condenser recirculation pump, and heat or cool the water surrounding the first two impingers to maintain the gas temperature measured at the exit of the CPM filter below 30 °C (85 °F). Continue the purge under these conditions for 1 hour, checking the rotameter and  $\Delta H$  value(s) periodically. After 1 hour, simultaneously turn off the delivery and pumping systems.

**8.5.3.4** Weigh the liquid, or measure the volume of the liquid collected in the dropout, impingers, and silica trap. Measure the liquid in the first impinger to within 1 mL using a clean graduated cylinder or by weighing it to within 0.5 g using a balance. Record the volume or weight of liquid present to be used to calculate the moisture content of the effluent gas in the field log notebook.

**8.5.3.5** If a balance is available in the field, weigh the silica impinger to within 0.5 g. Note the color of the indicating silica gel in the last impinger to determine whether it has been completely spent, and make a notation of its condition in the field log book.

**8.5.4 Sample Recovery.**

**8.5.4.1** Recovery of Filterable Particulate Matter. Recovery of filterable particulate matter involves the quantitative transfer of particles according to the filterable particulate sampling method (*i.e.*, Method 5, 17 or 201A).

**8.5.4.2 CPM Container #1, Aqueous Liquid Impinger Contents.** Quantitatively transfer liquid from the dropout and the impinger prior to the CPM filter into a clean sample bottle (glass or plastic). Rinse the probe extension, condenser, each impinger and the connecting glassware, and the front half of the CPM filter housing twice with water. Recover the rinse water, and add it to the same sample bottle. Mark the liquid level on the bottle. CPM Container #1 holds the water soluble CPM captured in the impingers.

**8.5.4.3 CPM Container #2, Organic Rinses.** Follow the water rinses of the probe extension, condenser, each impinger and all of the connecting glassware and front half of the CPM filter with an acetone rinse. Then repeat the entire procedure with two rinses of  $\text{MeCl}_2$ , and save both solvents in a separate glass container identified as CPM Container #2. Mark the liquid level on the jar.

**8.5.4.4 CPM Container #3, CPM filter Sample.** Use tweezers and/or clean disposable surgical gloves to remove the filter from the CPM filter holder. Place the filter in the petri dish identified as CPM Container #3.

**8.5.4.5 CPM Container #4, Cold Impinger Water.** You must weigh or measure the volume of the contents of CPM Container #4 either in the field or during sample analysis (see Section 11.2.3). If the water from the cold impinger has been weighed in the field, it can be discarded. Otherwise, quantitatively transfer liquid from the cold impinger that follows the CPM filter into a clean sample bottle (glass or plastic). Mark the liquid level on the bottle. This container holds the remainder of the liquid water from the emission gases.

**8.5.4.6 CPM Container #5, Silica Gel Absorbent.** You must weigh the contents of CPM Container #5 in the field or during sample analysis (see Section 11.2.4). If the silica gel has been weighed in the field to measure water content, then it can be discarded. Otherwise, transfer the silica gel to its original container and seal. A funnel may make it easier to pour the silica gel without spilling. A rubber policeman may be used as an aid in removing the silica gel from the impinger. It is not necessary to remove the small amount of silica gel dust particles that may adhere to the impinger wall and are difficult to remove. Since the gain in weight is to be used for moisture calculations, do not use any water or other liquids to transfer the silica gel.

**8.5.4.7 CPM Container #6, Acetone Rinse Blank.** Take 150 mL of the acetone directly from the wash bottle you used, and place it in CPM Container #6, labeled Acetone Rinse Blank (see Section 11.2.5 for analysis). Mark the liquid level on the bottle.

**8.5.4.8 CPM Container #7, Water Rinse Blank.** Take 150 mL of the water directly from the wash bottle you used, and place it in CPM Container #7, labeled Water Rinse Blank (see Section 11.2.6 for analysis). Mark the liquid level on the bottle.

**8.5.4.9 CPM Container #8, Methylene Chloride Rinse Blank.** Take 150 mL of the  $\text{MeCl}_2$  directly from the wash bottle you used, and place it in CPM Container #8, labeled Methylene Chloride Rinse Blank (see



Section 11.2.7 for analysis). Mark the liquid level on the bottle.

8.5.5 Transport procedures. Containers must remain in an upright position at all times during shipping. You do not have to ship the containers under dry or blue ice. However, samples must be maintained at or below 30 °C (85 °F) during shipping.

#### 9.0 Quality Control

9.1 Daily Quality Checks. You must perform daily quality checks of field log books and data entries and calculations using data quality indicators from this method and your site-specific test plan. You must review and evaluate recorded and transferred raw data, calculations, and documentation of testing procedures. You must initial or sign log book pages and data entry forms that were reviewed.

9.2 Calculation Verification. Verify the calculations by independent, manual checks. You must flag any suspect data and identify the nature of the problem and potential effect on data quality. After you complete the test, prepare a data summary and compile all the calculations and raw data sheets.

9.3 Conditions. You must document data and information on the process unit tested, the particulate control system used to control emissions, any non-particulate control system that may affect particulate emissions, the sampling train conditions, and weather conditions. Discontinue the test if the operating conditions may cause non-representative particulate emissions.

9.4 Health and Safety Plan. Develop a health and safety plan to ensure the safety of your employees who are on-site conducting the particulate emission test. Your plan must conform with all applicable Occupational Safety and Health Administration (OSHA), Mine Safety and Health Administration (MSHA), and Department of Transportation (DOT) regulatory requirements. The procedures must also conform to the plant health and safety requirements.

9.5 Calibration Checks. Perform calibration check procedures on analytical balances each time they are used.

9.6 Glassware. Use class A volumetric glassware for titrations, or calibrate your equipment against National Institute of Standards and Technology (NIST) traceable glassware.

9.7 Analytical Balance. Check the calibration of your analytical balance each day you weigh CPM samples. You must use NIST Class S weights at a mass approximately equal to the weight of the sample plus container you will weigh.

9.8 Reagent Blanks. You must run blanks of water, acetone, and methylene chloride used for field recovery and sample analysis. Analyze at least one sample (100 mL minimum) of each reagent that you plan to use for sample recovery and analysis before you begin testing. Running blanks before field use will verify low blank concentrations, thereby reducing the potential for a high field blank on test samples.

9.9 Field Reagent Blanks. You must run at least one field blank of water, acetone, and methylene chloride you use for field recovery. Running independent reagent field

blanks will verify that low blank concentrations were maintained during field solvent use and demonstrate that reagents have not been contaminated during field tests.

9.10 Field Train Blank. You must recover a minimum of one field train blank for each set of compliance tests at the facility. You must assemble the sampling train as it will be used for testing. Prior to the purge, you must add 100 mL of water to the first impinger and record this data on Figure 3. You must purge the assembled train as described in Sections 8.5.3.2. and 8.5.3.3. You must recover field train blank samples as described in Section 8.5.4. From the field sample weight, you will subtract the condensable particulate mass you determine with this blank train or 0.002 g (2.0 mg), whichever is less.

9.11 Audit Procedure. Concurrent with compliance sample analysis, and if available, analyze audit material to evaluate the technique of the analyst and the standards preparation. Use the same staff, analytical reagents, and analytical system for both compliance samples and the EPA audit sample. If this condition is met, auditing of subsequent compliance analyses for the same enforcement agency within 30 days is not required. An audit sample set may not be used to validate different sets of compliance samples under the jurisdiction of different enforcement agencies, unless prior arrangements are made with both enforcement agencies.

9.12 Audit Samples. As of the publication date of this test method, audit materials are not available. If audit materials become available, audit samples will be supplied only to enforcement agencies for compliance tests. Audit samples can be requested by a State agency. Audit materials are requested online by authorized regulatory authorities at the following internet address: <http://www.sscap.net/>. Authorization can be obtained by contacting an EPA Emission Measurement Center QA Team Member listed on the EPA TTN Web site at the following internet address: <http://www.epa.gov/ttn/emc/email.html#qaqc>. The request for the audit sample must be made at least 30 days prior to the scheduled compliance sample analysis.

9.13 Audit Results. Calculate the audit sample concentration according to the calculation procedure described in the audit instructions included with the audit sample. Fill in the audit sample concentration and the analyst's name on the audit response form included with the audit instructions. Send one copy to the EPA Regional Office or the appropriate enforcement agency.

#### 10.0 Calibration and Standardization

Maintain a log of all condensable particulate sampling and analysis calibrations. Include copies of the relevant portions of the calibration and field logs in the final test report.

10.1 Thermocouple Calibration. You must calibrate the thermocouples using the procedures described in Section 10.1.4.1.2 of Method 2 of Appendix A-1 to Part 60. Calibrate each temperature sensor at a minimum of three points over the anticipated

range of use against an NIST-traceable mercury-in-glass thermometer.

10.2 Ammonium Hydroxide. The 0.1 N  $\text{NH}_4\text{OH}$  used for titrations in this method is made as follows: Add 7 mL of concentrated (14.8 M)  $\text{NH}_4\text{OH}$  to 1 liter of water. Standardize against standardized 0.1 N  $\text{H}_2\text{SO}_4$ , and calculate the exact normality using a procedure parallel to that described in Section 5.5 of Method 6 of Appendix A-4 to 40 CFR part 60. Alternatively, purchase 0.1 N  $\text{NH}_4\text{OH}$  that has been standardized against a NIST reference material. Record the normality on the Condensable Particulate Matter Work Table (see Figure 5 of Section 18).

#### 11.0 Analytical Procedures

11.1 Analytical Data Sheets. (a) Record the filterable particulate field data on the appropriate (*i.e.*, Method 5, 17, or 201A) analytical data sheets. Alternatively, data may be recorded electronically using software applications such as the Electronic Reporting Tool (ERT), available at the following internet address: [http://www.epa.gov/ttn/chief/ert/ert\\_tool.html](http://www.epa.gov/ttn/chief/ert/ert_tool.html). Record the condensable particulate data on the Condensable Particulate Matter Work Table (see Figure 5 of Section 18).

(b) Measure the liquid in all containers either volumetrically to  $\pm 1$  mL or gravimetrically to  $\pm 0.5$  g. Confirm on the filterable particulate analytical data sheet whether leakage occurred during transport. If a noticeable amount of leakage has occurred, either void the sample or use methods, subject to the approval of the Administrator, to correct the final results.

11.2 Condensable Particulate Matter Analysis. See the flow chart in Figure 6 of Section 18 for the steps to process and combine fractions from the CPM train.

11.2.1 Container #3, CPM Filter Sample. Extract the filter recovered from the low temperature portion of the train, and combine the extracts with the organic and inorganic fractions resulting from the aqueous impinger sample recovery. If the sample was collected by Method 17 because the stack temperature was below 30 °C (85 °F), process the filter extracts as described in this section without combination with any other portion from the train.

11.2.1.1 Extract the water soluble (aqueous or inorganic) CPM from the CPM filter as described in this section. Fold the CPM filter in quarters, and place it into a 50 mL extraction tube. Add sufficient deionized ultra-filtered water to cover the filter (*e.g.*, 10 mL of water). Place the extractor tube into a sonication bath and extract the water soluble material for a minimum of 2 minutes. Combine the aqueous extract with the contents of Container #1. Repeat this extraction step twice for a total of three extractions.

11.2.1.2 Extract the organic soluble CPM from the CPM filter as described in this section. Add sufficient methylene chloride to cover the filter (*e.g.*, 10 mL of water). Place the extractor tube into a sonication bath and extract the organic soluble material for a minimum of 2 minutes. Combine the organic extract with the contents of Container #2. Repeat this extraction step twice for a total of three extractions.

11.2.2 CPM Container #1, Aqueous Liquid Impinger Contents. Analyze the water soluble CPM in Container 1 as described in this section. Place the contents of Container #1 into a separatory funnel. Add approximately 30 mL of  $\text{MeCl}_2$  to the funnel, mix well, and drain off the lower organic phase. Repeat this procedure twice with 30 mL of  $\text{MeCl}_2$  each time combining the organic phase from each extraction. Each time, leave a small amount of the organic/ $\text{MeCl}_2$  phase in the separatory funnel, ensuring that no water is collected in the organic phase. This extraction should yield about 90 mL of organic extract.

11.2.2.1 CPM Container #2. Combine the organic extract from Container #1 with the organic train rinse in Container 2.

11.2.2.2 Organic Fraction Weight Determination. Place the organic phase in a clean glass beaker. Evaporate the organic extract at room temperature (not to exceed 30 °C (85 °F)) and pressure in a laboratory hood to not less than 10 mL. Quantitatively transfer the beaker contents to a 50-mL preweighed tin, and evaporate to dryness at room temperature (not to exceed 30 °C (85 °F)) and pressure in a laboratory hood. Following evaporation, desiccate the organic fraction for 24 hours in a desiccator containing anhydrous calcium sulfate. Weigh at intervals of at least 6 hours to a constant weight (*i.e.*,  $\leq 0.5$  mg change from previous weighing), and report results to the nearest 0.1 mg on the Condensable Particulate Matter Work Table (see Figure 5 of Section 18).

11.2.2.3 Inorganic Fraction Weight Determination. Transfer the aqueous fraction from the extraction to a clean 500-mL or smaller beaker. Evaporate to not less than 10 mL liquid on a hot plate or in the oven at 105 °C, and allow to dry at room temperature (not to exceed 30 °C (85 °F)). You must ensure that water and volatile acids have completely evaporated before neutralizing nonvolatile acids in the sample. Redissolve the residue in 100 mL of deionized distilled ultra-filtered water (ASTM D1193-06, Type 1 water or equivalent) (incorporated by reference).

11.2.2.4 Use titration to neutralize acid in the sample and remove water of hydration. Calibrate the pH meter with the neutral and acid buffer solutions; then titrate the sample with 0.1N  $\text{NH}_4\text{OH}$  to a pH of 7.0, as indicated by the pH meter. Record the volume of titrant used on the Condensable Particulate Matter Work Table (see Figure 5 of Section 18).

11.2.2.5 Using a hot plate or an oven at 105 °C, evaporate the aqueous phase to approximately 10 mL. Quantitatively transfer the beaker contents to a 50-mL preweighed tin, and evaporate to dryness at room temperature (not to exceed 30 °C (85 °F)) and pressure in a laboratory hood. Following evaporation, desiccate the residue for 24 hours in a desiccator containing anhydrous calcium sulfate. Weigh at intervals of at least 6 hours to a constant weight (*i.e.*,  $\leq 0.5$  mg change from previous weighing), and report results to the nearest 0.1 mg on the Condensable Particulate Matter Work Table (see Figure 5 of Section 18).

11.2.2.6 Calculate the correction factor to subtract the  $\text{NH}_4^+$  retained in the sample using Equation 1 in Section 12.

11.2.3 CPM Container #4, Cold Impinger Water. If the amount of water has not been

determined in the field, note the level of liquid in the container, and confirm on the filterable particulate analytical data sheet whether leakage occurred during transport. If a noticeable amount of leakage has occurred, either void the sample or use methods, subject to the approval of the Administrator, to correct the final results. Measure the liquid in Container #4 either volumetrically to  $\pm 1$  mL or gravimetrically to  $\pm 0.5$  g, and record the volume or weight on the filterable particulate analytical data sheet of the filterable particulate matter test method.

11.2.4 CPM Container #5, Silica Gel Absorbent. Weigh the spent silica gel (or silica gel plus impinger) to the nearest 0.5 g using a balance. This step may be conducted in the field. Record the weight on the filterable particulate analytical data sheet of the filterable particulate matter test method.

11.2.5 Container #6, Acetone Field Rinse Blank. Use 100 mL of acetone from the blank container for this analysis. If insufficient liquid is available or if the acetone has been lost due to container breakage, either void the sample, or use methods, subject to the approval of the Administrator, to correct the final results. Transfer 100 mL of the acetone to a clean 250-mL beaker. Evaporate the acetone at room temperature (not to exceed 30 °C (85 °F)) and pressure in a laboratory hood to approximately 10 mL. Quantitatively transfer the beaker contents to a 50-mL preweighed tin, and evaporate to dryness at room temperature (not to exceed 30 °C (85 °F)) and pressure in a laboratory hood. Following evaporation, desiccate the residue for 24 hours in a desiccator containing anhydrous calcium sulfate. Weigh at intervals of at least 6 hours to a constant weight (*i.e.*,  $\leq 0.5$  mg change from previous weighing), and report results to the nearest 0.1 mg on Figure 3.

11.2.6 Water Rinse Field Blank, Container #7. Use 100 mL of the water from the blank container for this analysis. If insufficient liquid is available, or if the water has been lost due to container breakage, either void the sample, or use methods, subject to the approval of the Administrator, to correct the final results. Transfer the water to a clean 250-mL beaker, and evaporate to approximately 10 mL liquid in the oven at 105 °C. Quantitatively transfer the beaker contents to a clean preweighed 50-mL tin, and evaporate to dryness at room temperature (not to exceed 30 °C (85 °F)) and pressure in a laboratory hood. Following evaporation, desiccate the residue for 24 hours in a desiccator containing anhydrous calcium sulfate. Weigh at intervals of at least 6 hours to a constant weight (*i.e.*,  $\leq 0.5$  mg change from previous weighing) and report results to the nearest 0.1 mg on Figure 3.

11.2.7 Methylene Chloride Field Reagent Blank, Container #8. Use 100 mL of  $\text{MeCl}_2$  from the blank container for this analysis. Transfer 100 mL of the  $\text{MeCl}_2$  to a clean 250-mL beaker. Evaporate the methylene chloride at room temperature (not to exceed 30 °C (85 °F)) and pressure in a laboratory hood to approximately 10 mL. Quantitatively transfer the beaker contents to a 50-mL preweighed tin, and evaporate to dryness at room temperature (not to exceed 30 °C (85 °F)) and pressure in a laboratory hood. Following

evaporation, desiccate the residue for 24 hours in a desiccator containing anhydrous calcium sulfate. Weigh at intervals of at least 6 hours to a constant weight (*i.e.*,  $\leq 0.5$  mg change from previous weighing), and report results to the nearest 0.1 mg on Figure 3.

## 12.0 Calculations and Data Analysis

12.1 Nomenclature. Report results in International System of Units (SI units) unless the regulatory authority for compliance testing specifies English units. The following nomenclature is used.

$\Delta H_{\text{or}}$  = Pressure drop across orifice at flow rate of 0.75 SCFM at standard conditions, in. W.C.

[**Note:** specific to each orifice and meter box.]

17.03 = mg/milliequivalents for ammonium ion.

ACFM = Actual cubic feet per minute.

$C_{\text{cpm}}$  = Concentration of the condensable particulate matter in the stack gas, dry basis, corrected to standard conditions, milligrams/dry standard cubic foot.

$m_c$  = Mass of the  $\text{NH}_4^+$  added to sample to form ammonium sulfate, mg.

$m_{\text{cpm}}$  = Mass of the total condensable particulate matter, mg.

$m_{\text{fb}}$  = Mass of field train total CPM blank, mg.

$m_i$  = Mass of inorganic CPM matter, mg.

$m_{\text{ib}}$  = Mass of field train inorganic CPM blank, mg.

$m_o$  = Mass of organic CPM, mg.

$m_{\text{ob}}$  = Mass of organic field train blank, mg.

$m_r$  = Mass of dried sample from inorganic fraction, mg.

$N$  = Normality of ammonium hydroxide titrant.

$V_{\text{m(std)}}$  = Volume of gas sample measured by the dry gas meter, corrected to standard conditions, dry standard cubic meter (dscm) or dry standard cubic foot (dscf) as defined in Equation 5-1 of Method 5.

$V_t$  = Volume of  $\text{NH}_4\text{OH}$  titrant, mL.

$V_p$  = Volume of water added during train purge.

12.2 Calculations. Use the following equations to complete the calculations required in this test method. Enter the appropriate results from these calculations on the Condensable Particulate Matter Work Table (see Figure 5 of Section 18).

12.2.1 Mass of ammonia correction. Correction for ammonia added during titration of 100 mL aqueous CPM sample. This calculation assumes no waters of hydration.

$$m_c = 17.03 \times v_t \times N \quad \text{Eq. 1}$$

12.2.2 Mass of the Field Blank (mg). Per Section 9.9, the mass of the field blank,  $m_{\text{fb}}$ , shall not exceed 2.0 mg.

$$m_{\text{fb}} = m_{\text{ib}} + m_{\text{ob}} \quad \text{Eq. 2}$$

12.2.3 Mass of Inorganic CPM (mg).

$$m_i = m_r - m_c \quad \text{Eq. 3}$$

12.2.4 Total Mass of CPM (mg).

$$m_{\text{cpm}} = m_i + m_o - m_{\text{fb}} \quad \text{Eq. 4}$$

12.2.5 Concentration of CPM (mg/dscf).

$$C_{\text{cpm}} = \frac{m_{\text{cpm}}}{V_{m(\text{std})}} \quad \text{Eq. 5}$$

12.3 Emissions Test Report. Include the following list of conventional elements in the emissions test report.

(a) Emission test description including any deviations from this protocol.

(b) Summary data tables on a run-by-run basis that include the condensable particulate mass.

(c) Flowchart of the process or processes tested.

(d) Sketch of the sampling location.

(e) Preliminary traverse data sheets including cyclonic flow checks.

(f) Raw field data sheets and copies of field log pages.

(g) Laboratory analytical sheets and case narratives.

(h) Pretest and post test reagent blank results.

(i) Sample calculations.

(j) Pretest and post-test calibration data.

(k) Chain of custody forms.

(l) Documentation of process and air pollution control system data.

#### 13.0 Method Performance [Reserved]

#### 14.0 Pollution Prevention [Reserved]

#### 15.0 Waste Management

Solvent and water are evaporated in a laboratory hood during analysis. No liquid waste is generated in the performance of this method. Organic solvents used to clean

sampling equipment should be managed as RCRA organic waste.

#### 16.0 Alternative Procedures [Reserved]

#### 17.0 References

1. U.S. Environmental Protection Agency, Federal Reference Methods 1 through 5 and Method 17, 40 CFR 60, Appendix A-1 through A-3 and A-6.

2. Richards, J., T. Holder, and D. Goshaw. "Optimized Method 202 Sampling Train to Minimize the Biases Associated with Method 202 Measurement of Condensable Particulate Matter Emissions." Paper presented at Air & Waste Management Association Hazardous Waste Combustion Specialty Conference. St. Louis, Missouri. November 2-3, 2005.

3. DeWees, W.D., S.C. Steinsberger, G.M. Plummer, L.T. Lay, G.D. McAlister, and R.T. Shigehara. "Laboratory and Field Evaluation of the EPA Method 5 Impinger Catch for Measuring Condensable Matter from Stationary Sources." Paper presented at the 1989 EPA/AWMA International Symposium on Measurement of Toxic and Related Air Pollutants. Raleigh, North Carolina. May 1-5, 1989.

4. DeWees, W.D. and K.C. Steinsberger. "Method Development and Evaluation of Draft Protocol for Measurement of Condensable Particulate Emissions." Draft Report. November 17, 1989.

5. Texas Air Control Board, Laboratory Division. "Determination of Particulate in Stack Gases Containing Sulfuric Acid and/or Sulfur Dioxide." Laboratory Methods for Determination of Air Pollutants. Modified December 3, 1976.

6. Nothstein, Greg. Masters Thesis.

University of Washington. Department of Environmental Health. Seattle, Washington.

7. "Particulate Source Test Procedures Adopted by Puget Sound Air Pollution Control Agency Board of Directors." Puget Sound Air Pollution Control Agency, Engineering Division. Seattle, Washington. August 11, 1983.

8. Commonwealth of Pennsylvania, Department of Environmental Resources. Chapter 139, Sampling and Testing (Title 25, Rules and Regulations, Part I, Department of Environmental Resources, Subpart C, Protection of Natural Resources, Article III, Air Resources). January 8, 1960.

9. Wisconsin Department of Natural Resources. Air Management Operations Handbook, Revision 3. January 11, 1988.

10. U.S. Environmental Protection Agency, "Laboratory Evaluation of Method 202 to Determine Fate of SO<sub>2</sub> in Impinger Water," EPA Contract No. 68-D-02-061, Work Assignment 3-14, September 30, 2005.

11. U.S. Environmental Protection Agency, "Evaluation and Improvement of Condensable Particulate Matter Measurement," EPA Contract No. EP-D-07-097, Work Assignment 2-03, October 2008.

12. Electric Power Research Institute (EPRI), "Laboratory Comparison of Methods to Sample and Analyze Condensable Particulate Matter," EPRI Agreement EP-P24373/C11811 Condensable Particulate Methods: EPRI Collaboration with EPA, October 2008.

## 18.0 Tables, Diagrams, Flowcharts, and Validation Data

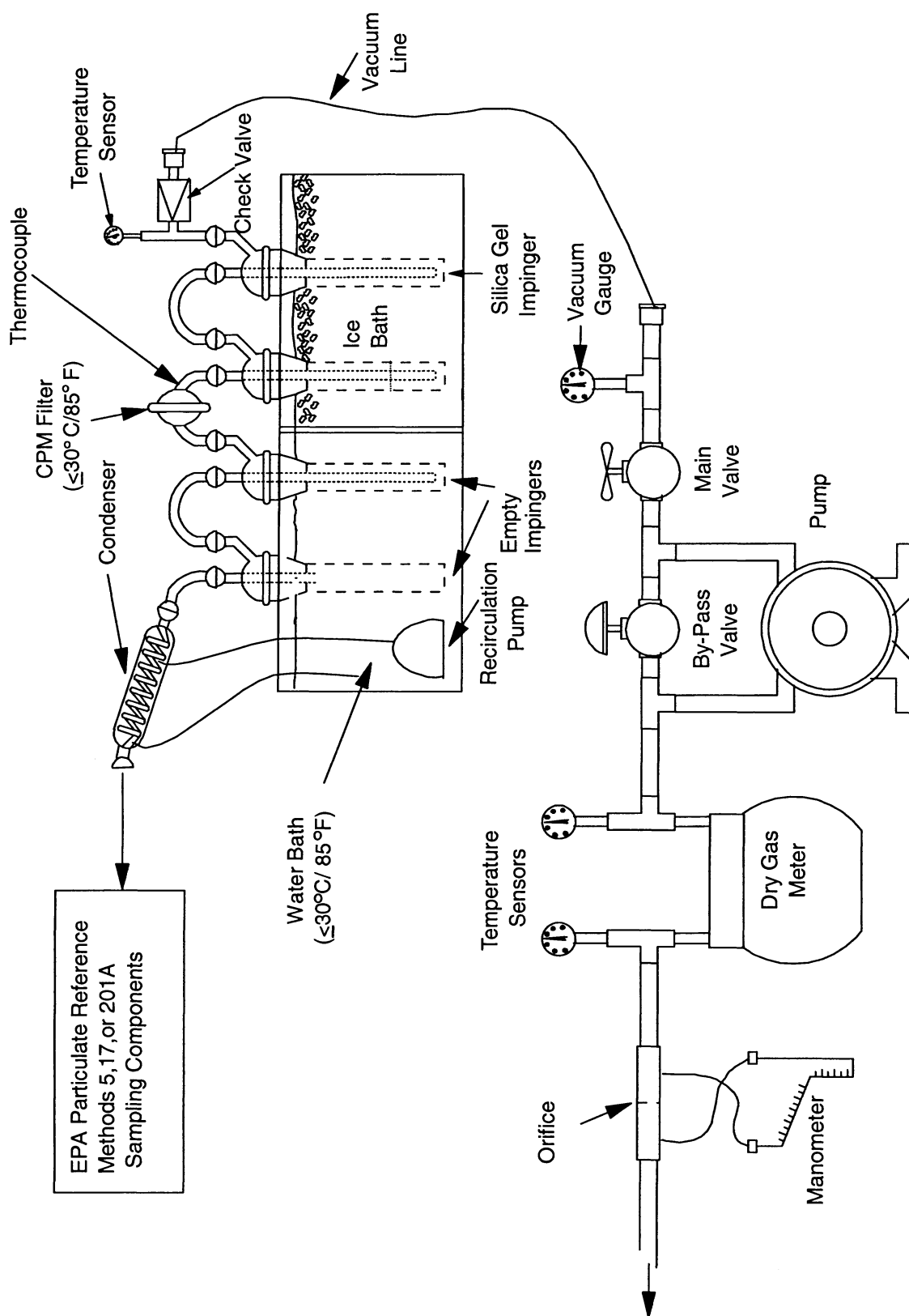


Figure 1. Schematic of Condensable Particulate Sampling Train

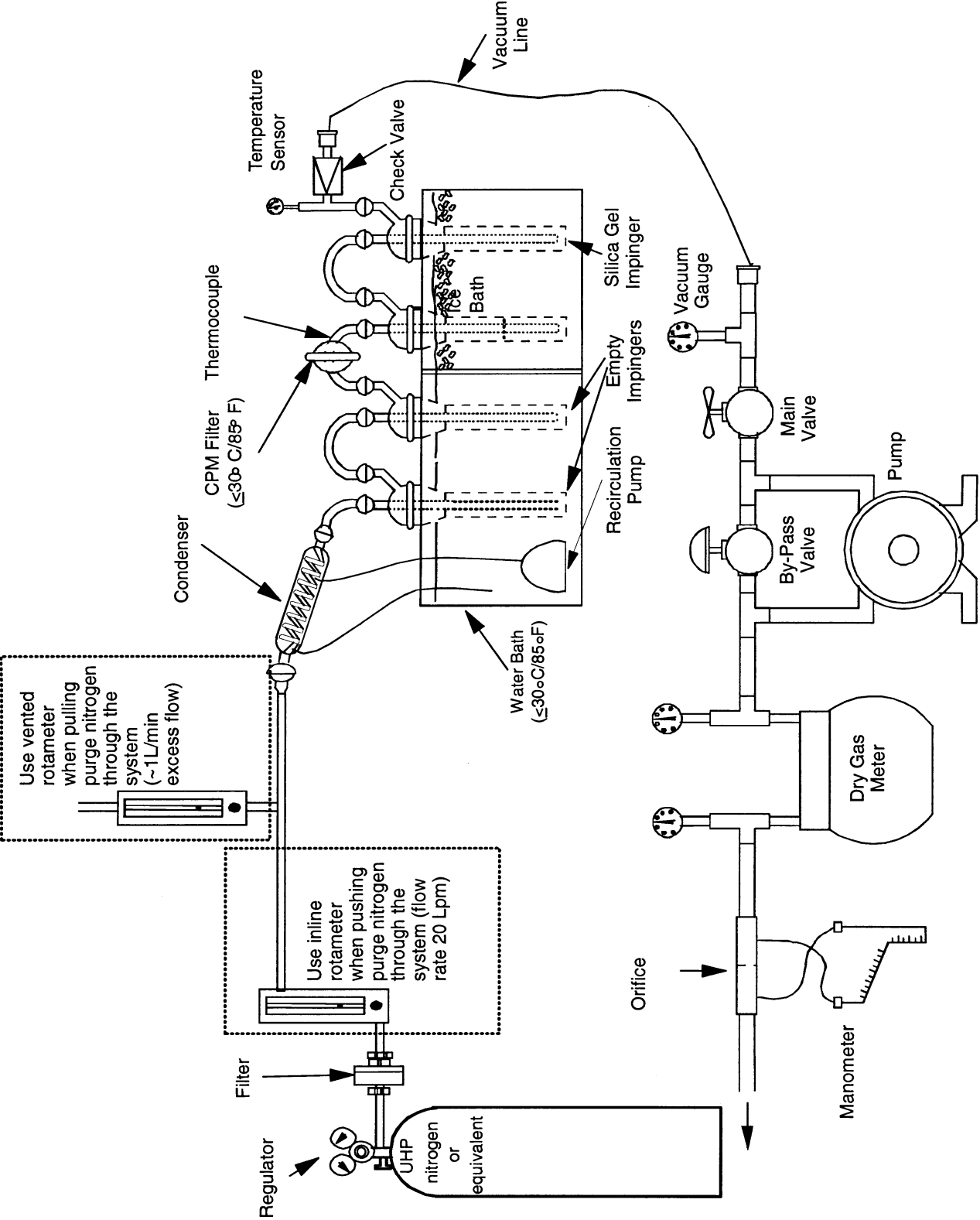
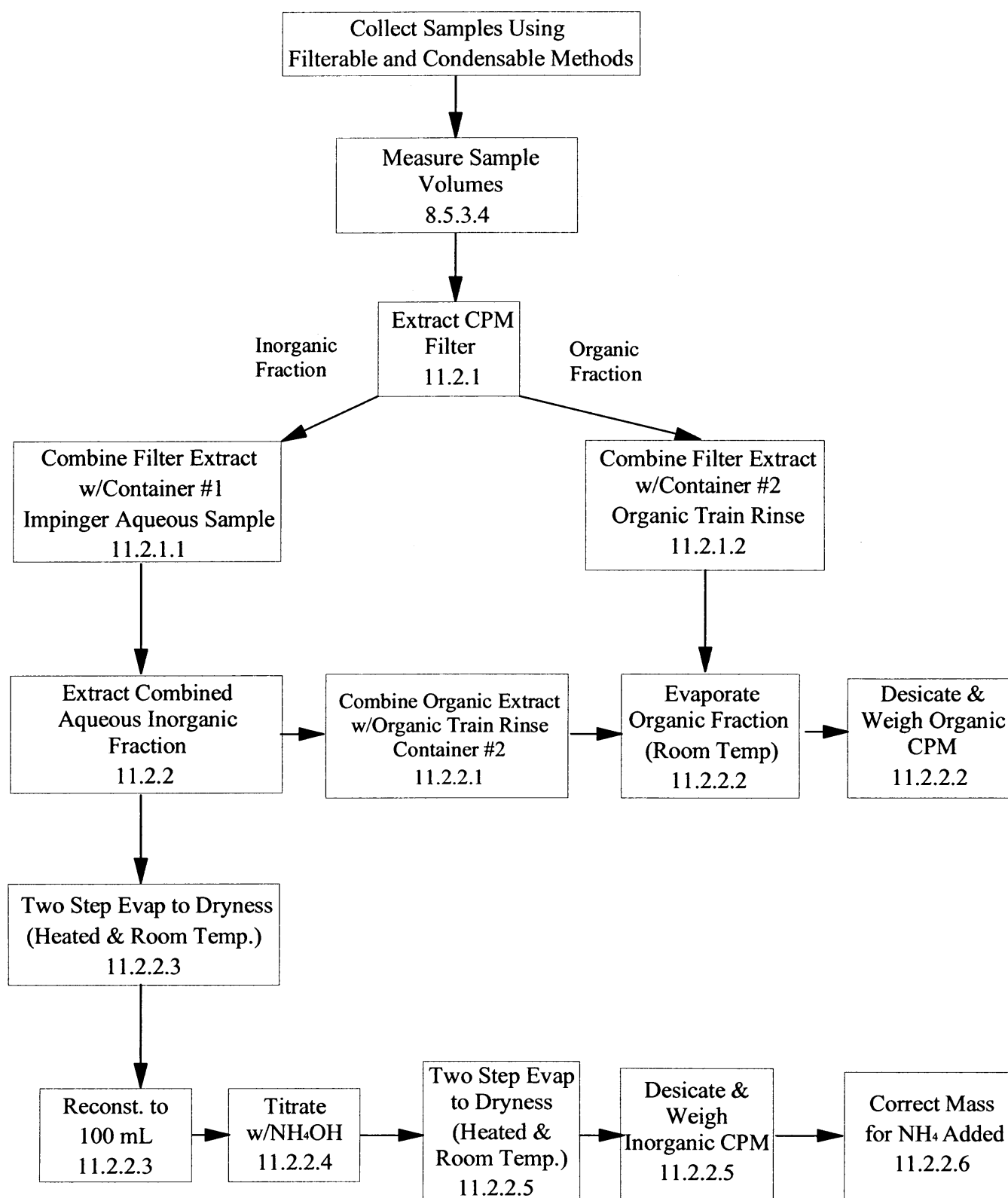


Figure 2. Nitrogen Purge Train Configuration





**Figure 6. CPM Sample Processing Flow Chart**