

TABLE 1—FORMAT FOR SUBMITTING COMMENTS

|   |   |
|---|---|
| Name of regulation<br><br>Type of product or FDA Center regulating the product.<br>Citation to Code of Federal Regulations and statutory citation (as applicable).<br>Approved information collection and OMB Control Number (as applicable).<br>Brief description of concern .....<br>Available data on cost or economic impact .....<br><br>Proposed solution ..... | (For example, what innovation makes the regulation outdated? Why?)<br>(Quantified costs and/or cost savings. Qualitative description, if needed.)<br><br>(Include your solution. For example, how would you modify the regulation? Provide specific text if you are recommending a modification.) |
|---|---|

**III. References**

The following references are on display in the Dockets Management Staff office (see **ADDRESSES**) and are available for viewing by interested persons between 9 a.m. and 4 p.m., Monday through Friday; they are also available electronically at <https://www.regulations.gov>. FDA has verified the Web site addresses, as of the date this document publishes in the **Federal Register**, but Web sites are subject to change over time.

1. Executive Order 13771 (January 30, 2017); available at <https://www.federalregister.gov/documents/2017/02/03/2017-02451/reducing-regulation-and-controlling-regulatory-costs>.
2. Executive Order 13777 (February 24, 2017); available at <https://www.federalregister.gov/documents/2017/03/01/2017-04107/enforcing-the-regulatory-reform-agenda>.

Dated: August 30, 2017.

**Anna K. Abram,**

*Deputy Commissioner for Policy, Planning, Legislation, and Analysis.*

[FR Doc. 2017–19047 Filed 9–7–17; 8:45 am]

**BILLING CODE 4164–01–P**

**ENVIRONMENTAL PROTECTION AGENCY**

**40 CFR Part 51**

[EPA–HQ–OAR–2016–0456; FRL–9966–75–OAR]

**RIN 2060–AS91**

**Method 202—Dry Impinger Method for Determining Condensable Particulate Emissions From Stationary Sources**

**AGENCY:** Environmental Protection Agency (EPA).

**ACTION:** Proposed rule.

**SUMMARY:** In this action, the Environmental Protection Agency (EPA) proposes editorial and technical revisions to the EPA’s Method 202—Dry Impinger Method for Determining

Condensable Particulate Emissions from Stationary Sources to improve the consistency in results achieved across the testing community.

**DATES:**

*Comments.* Comments must be received on or before November 7, 2017.

*Public Hearing.* If a public hearing is requested by September 18, 2017, then we will hold a public hearing on October 10, 2017 at the location described in the **ADDRESSES** section. The last day to pre-register in advance to speak at the public hearing will be October 6, 2017.

**ADDRESSES:** Submit your comments, identified by Docket ID No. EPA–HQ–OAR–2016–0456, to the Federal eRulemaking Portal at <http://www.regulations.gov>. Follow the online instructions for submitting comments. Once submitted, comments cannot be edited or withdrawn. The EPA may publish any comment received to its public docket. Do not submit electronically any information you consider to be Confidential Business Information (CBI) or other information whose disclosure is restricted by statute. Multimedia submissions (audio, video, etc.) must be accompanied by a written comment. The written comment is considered the official comment and should include discussion of all points you wish to make. The EPA will generally not consider comments or comment contents located outside of the primary submission (*i.e.*, on the Web, Cloud, or other file sharing system). For additional submission methods, the full EPA public comment policy, information about CBI or multimedia submissions, and general guidance on making effective comments, please visit <http://www2.epa.gov/dockets/commenting-epa-dockets>.

*Public Hearing.* If a public hearing is requested, it will be held at EPA Headquarters, William Jefferson Clinton East Building, 1201 Constitution Avenue NW., Washington, DC 20004. If a public hearing is requested, then we will provide details about the public

hearing on our Web site at: <https://www.epa.gov/emc/emc-proposed-test-methods>. The EPA does not intend to publish another document in the **Federal Register** announcing any updates on the request for a public hearing. Please contact Mr. Ned Shappley at (919) 541–7903 or by email at [shappley.ned@epa.gov](mailto:shappley.ned@epa.gov) to request a public hearing, to register to speak at the public hearing, or to inquire as to whether a public hearing will be held.

*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 EPA Docket Center, EPA/DC, EPA WJC West Building, Room 3334, 1301 Constitution Avenue NW., Washington, DC. This Docket Facility 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 is (202) 566–1742.

**FOR FURTHER INFORMATION CONTACT:** Mr. Ned Shappley, Office of Air Quality Planning and Standards, Air Quality Assessment Division, Measurement Technology Group (E143–02), Environmental Protection Agency, Research Triangle Park, NC 27711; telephone number: (919) 541–5225; fax number: (919) 541–0516; email address: [shappley.ned@epa.gov](mailto:shappley.ned@epa.gov).

**SUPPLEMENTARY INFORMATION:** The following topics are discussed in this preamble.

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- H. Executive Order 13045: Protection of Children From Environmental Health Risks and Safety Risks
- I. Executive Order 13211: Actions That Significantly Affect Energy Supply, Distribution, or Use
- J. National Technology Transfer and Advancement Act (NTTAA)
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**I. General Information**

*A. Does this action apply to me?*

This action applies to you if you operate a stationary source that is subject to applicable requirements to control or measure condensable particulate matter (CPM) emissions where EPA Method 202 is incorporated as a component of the applicable test method. In addition, this action applies to you if federal, state, tribal, or local agencies take certain additional independent actions. For example, this

action applies to sources through actions by state and local agencies that implement CPM control measures to attain the National Ambient Air Quality Standards (NAAQS) for particles less than 2.5 micrometers in diameter (PM<sub>2.5</sub>) and specify the use of EPA Method 202 to demonstrate compliance with the control measures. State, tribal, and local agencies that specify the use of EPA Method 202 would have to implement the following requirements: (1) Adopt this method in rules or permits (either by incorporation by reference or by duplicating the method in its entirety) and (2) promulgate an emissions limit requiring the use of EPA Method 202 (or a method that incorporates EPA Method 202). This action also applies to stationary sources that are required to meet applicable CPM requirements established through federal, state, or tribal rules or permitting programs such as New Source Performance Standards and New Source Review (NSR), which specify the use of EPA Method 202 to demonstrate compliance with the control measures.

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

| Category       | NAICS <sup>a</sup> | Examples of regulated entities                                |
|----------------|--------------------|---|
| Industry ..... | 332410             | Fossil fuel steam generators.                                 |
|                | 332410             | Industrial, commercial, institutional steam generating units. |
|                | 332410             | Electricity generating units.                                 |
|                | 324110             | Petroleum refineries.   |
|                | 562213             | Municipal waste combustors.                                   |
|                | 322110             | Pulp and paper mills.   |
|                | 325188             | Sulfuric acid plants.   |
|                | 327310             | Portland cement plants.                                       |
|                | 327410             | Lime manufacturing plants.                                    |
|                | 211111             | Coal preparation plants.                                      |
|                | 212111             |   |
|                | 212112             |   |
|                | 212113             |   |
|                | 331312             | Primary and secondary aluminum plants.                        |
|                | 331314             |   |
|                | 331111             | Iron and steel plants.  |
|                | 331513             |   |
|                | 321219             | Plywood and reconstituted products plants.                    |
| 321211         |                    |   |
| 321212         |                    |   |

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

If you have any questions regarding the applicability of the proposed changes to Method 202, contact the person listed in the preceding **FOR FURTHER INFORMATION CONTACT** section.

*B. What should I consider as I prepare my comments?*

1. Submitting CBI

Clearly mark the part or all of the information that you claim to be CBI. For CBI information in a disk or CD-

ROM that you mail to the 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 marked as CBI will not be

disclosed except in accordance with procedures set forth in 40 Code of Federal Regulations (CFR) part 2.

Do not submit information that you consider to be CBI or otherwise protected through <http://www.regulations.gov> or email. Send or deliver information identified as CBI to only the following address: OAQPS Document Control Officer (Room C404-02), U.S. EPA, Research Triangle Park,

NC 27711, Attention Docket ID No. EPA-HQ-OAR-2016-0456.

If you have any questions about CBI or the procedures for claiming CBI, please consult the person identified in the **FOR FURTHER INFORMATION CONTACT** section.

## 2. Docket

The docket number for the Method 202 revisions is Docket ID No. EPA-HQ-OAR-2016-0456.

*C. Where can I get a copy of this document and other related information?*

*World Wide Web (WWW).* In addition to being available in the docket, an electronic copy of the proposed method revisions is available on the Air Emission Measurement Center (EMC) Web site at <https://www.epa.gov/emc/emc-proposed-test-methods>.

## II. Background

Section 110 of the Clean Air Act, as amended (42 U.S.C. 7410), requires state and local air pollution control agencies to develop, and submit for EPA approval, State Implementation Plans (SIPs) that provide for the attainment, maintenance, and enforcement of the NAAQS in each air quality control region (or portion thereof) within each state. The emissions inventory and analyses used in the state's attainment demonstrations must consider PM<sub>2.5</sub> and particles less than 10 micrometers in diameter (PM<sub>10</sub>) emissions from stationary sources that are significant contributors of primary PM<sub>10</sub> and PM<sub>2.5</sub> emissions. Primary or direct PM emissions are the solid particles or liquid droplets emitted directly from an air emissions source or activity and the gaseous emissions or liquid droplets from an air emissions source or activity that condense to form PM or liquid droplets at ambient temperatures.

Subpart A of 40 CFR part 51 (Requirements for Preparation, Adoption, and Submittal of Implementation Plans) defines primary PM<sub>2.5</sub> and PM<sub>10</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 sampling train. Condensable PM is the material that is in vapor phase at stack conditions but condenses and/or reacts upon cooling and dilution in the ambient air to form solid or liquid PM immediately after discharge from the stack. In response to the need to quantify primary PM<sub>10</sub> and PM<sub>2.5</sub> emissions from stationary sources, the

EPA previously developed and promulgated Method 202 (Determination of Condensable Particulate Emissions from Stationary Sources) in 40 CFR part 51, appendix M (Recommended Test Methods for State Implementation Plans).

Specifically, on December 17, 1991 (56 FR 65433), the EPA first promulgated Method 202 to provide a test method for measuring CPM from stationary sources. Method 202, as promulgated in 1991, used 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 in 1991, contains several optional procedures that were intended to accommodate the various test methods in use by state and local regulatory entities at the time Method 202 was being developed.

When conducted consistently and carefully, this version of the method provided improved precision for most emission sources, and has been successfully implemented 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 within the method, there were variations in the measured CPM emissions. Additionally, during validation of the 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. The EPA conducted additional studies to further examine the mechanism and the effects of sulfuric acid formation. The results of our 1989 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." The report of that work is available in the docket as EPA-HQ-OAR-2016-0456-0001. Essentially, the 1989 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 in the docket as EPA-HQ-OAR-2016-0456-0002. In 2009, an EPA contractor conducted a third study, "Evaluation and Improvement of Condensable Particulate Matter Measurement," that presents the results of a laboratory evaluation of a dry impinger modification to Method 202. The report of that work is available in the docket as EPA-HQ-OAR-2016-0456-0003.

In 2010, the EPA promulgated amendments to Method 202 (75 FR 80118) to improve the measurement of fine PM emissions. The final amendments revised the sample collection and recovery procedures of the method to: (1) Reduce the potential for CPM formation due to oxidation of dissolved SO<sub>2</sub> when using Method 202 (as promulgated in 1991) and (2) promote consistent application of the method by eliminating most of the hardware and analytical options in the existing method. The most significant procedural changes were the addition of a condenser prior to the first impinger, the removal of water from the two impingers between the condenser and the CPM filter, and the addition of the requirement for a post-test nitrogen purge. These revisions increased the precision of Method 202 and reduced potential positive and negative biases by removal of the myriad of options and elimination of water in the two impingers, which significantly improved the consistency in the measurements obtained between source tests performed under different regulatory authorities.

On April 8, 2014, the EPA issued interim guidance on the treatment of CPM results in the Prevention of Significant Deterioration (PSD) and Nonattainment NSR Permitting Programs. The purpose of this guidance was to address concerns that CPM test results obtained with the method could include a positive bias that results in the overestimation of emissions due to the potential for blank contamination associated with the implementation of Method 202. In this interim guidance, we recommend to air agencies and permit applicants that it is appropriate on an interim basis to allow major source permit applicants to depart from one aspect of Method 202, specifically the current upper limit of 2.0 milligrams (mg) for the field train recovery blank. Consistent with this guidance, during the prescribed interim period, air agencies may allow permit applicants to use field train proof blanks, in lieu of the field train recovery blanks, and blank values as high as 5.1 mg can then

be used in the calculation of CPM emissions. As part of this guidance, the EPA announced plans to issue guidance on best practices for Method 202 implementation and to revise Method 202 as necessary. In addition, this guidance stated that the interim guidance period will end on the effective date of any revision that the EPA may make for Method 202 regarding the use of blanks in the field train on individual test results. We intend that the interim guidance will no longer apply as of the effective date of the final rule resulting from this proposal. A copy of the interim guidance is available in the docket (EPA-HQ-OAR-2016-0456-003) and on the EMC Web site at <https://www3.epa.gov/ttn/emc/methods/psdnsrinterimcmapmemo4814.pdf>.

On March 10, 2016, the EPA released the EPA Method 202 Best Practices Handbook. This handbook provides quality control procedures for evaluating the cause of blank contamination and practices to reduce contamination, so that testers may achieve the expected results when using Method 202. A copy of this handbook is available in the docket as EPA-HQ-OAR-2016-0456-004 and on the EMC Web site at <https://www3.epa.gov/ttn/emc/methods/m202-best-practices-handbook.pdf>.

### III. Summary of Proposed Revisions

In this action, we are proposing technical revisions and editorial changes to clarify and update the requirements and procedures specified in Method 202. Proposed editorial changes include correcting inconsistent terminology, improving readability, and simplifying text to aid in consistent implementation of the method. Proposed technical revisions are discussed below.

#### A. Blank Correction

In this action, we propose to replace the field train recovery blank requirement used to determine the blank correction (up to 2.0 mg) with a field train proof blank requirement. In the current version of Method 202, the result of the field train recovery blank is used as the basis for the blank correction (up to 2.0 mg). Specifically, we propose to revise section 8.5.4.10 (and renumber as section 8.5.5.8) to require conducting a field train proof blank to demonstrate the cleanliness of the sampling train. We propose to revise sections 9.9, 12.1, and 12.2.2, and Figures 4, 5, and 6 to replace the field train recovery blank with the field train proof blank. We also propose to remove the field train recovery blank

requirement and the associated text in section 9.10 from the method.

The EPA received technical information and recommendations from the National Council on Air and Stream Improvement (NCASI) supporting the use of a field train proof blank to evaluate method blank correction. The EPA believes the updated field train proof blank is a better indicator of the total systematic blank error for Method 202 sample runs. Under the proposed amendments, a clean and prepared sampling train is transported and fully assembled at the sampling location, leak checked, left in place without collecting a sample, purged with nitrogen, and recovered in the same manner as a sample collection train. All components of the Method 202 sampling train must be included in the field train proof blank to properly quantify the blank value. The field train proof blank represents the systematic bias associated with all of the uncertainty from the reagents, sampling media, glassware preparation, recovery and analysis procedures, environmental contamination, leak checks, and test crew sample handling.

#### B. Procedures for the Field Train Proof Blank

In the current version of Method 202, the setup and recovery procedures for the field train proof blank are incomplete. We are proposing the following revisions for the field train proof blank setup and recovery procedures specified in sections 8.5.5.8, 8.5.5.8.1, 8.5.5.8.2, and 9.9:

- Adding a full sampling train setup including the front half of the train for collecting filterable PM, probe extension and/or transfer line, condenser, impingers, and filter used to collect the CPM.
- Requiring that the entire filterable PM and CPM sampling train is transported to and assembled at the sampling location.
- Adding pre- and post-test leak checks.
- Exposing the assembled field train proof blank sampling train to the sampling environment for the same duration as the test runs to be conducted.
- Performing a post-test nitrogen purge of the field train proof blank.
- Requiring recovery of the sampling train components identical to how field samples are recovered.

In this action, we are also proposing to add section 8.5.5.8.3 to include procedures for handling the CPM filter from the field train proof blank. We believe that the proposed revisions will generate blank samples that duplicate

sources of possible contamination experienced by the field samples.

#### C. Configuration of the Vertical Condenser

Currently, Method 202 does not specify the orientation of the moisture condenser located before the first impinger of the sampling train. Although the sampling trains depicted in Figures 1 through 3 show the placement of the condenser, the incline of the condenser in the figures is not specified.

When the condenser is installed horizontally or at an angle, condensed moisture may pool in the condenser coils, increasing the potential for SO<sub>2</sub> to dissolve into that water and slowly oxidize to form CPM that is not related to the primary emission of CPM from the source. We believe that requiring the condenser to be installed vertically will minimize pooling of condensed moisture in the condenser coils, thereby reducing the potential for this bias and promoting consistency in CPM measurement.

In this action, we propose revisions to sections 2.1.2, 6.1.2, and 8.4.1 to require that the moisture condenser be installed in a vertical orientation. We propose to revise Figures 1 through 3 to depict the condenser in the vertical position consistent with the changes to the method text. We also propose to revise section 6.1.4 (and renumber as section 6.1.3) to allow other equipment options to purge the water in the dropout impinger.

#### D. Use of Graduated Cylinders

Currently, Method 202 allows the use of a graduated cylinder to measure the volume of moisture collected in the impingers and the silica gel trap for the purpose of calculating the moisture content of the effluent gas. We believe that using a graduated cylinder to measure the accumulated water is not sensitive enough to measure the moisture and potentially adds an unnecessary additional source for potential loss of condensable particulate residual mass in samples measured by Method 202. Therefore, we propose to revise section 8.5.3.4 (and renumber as section 8.5.3) to remove the option to use graduated cylinders and to require use of a balance to determine the mass of each impinger for the purpose of measuring the moisture collected during sampling. Instructions to weigh each impinger before testing, which is a necessary step for determining the amount of moisture collected when using a balance, are proposed for relocation to section 8.4.5. We also propose to make accompanying

revisions in sections 8.5.1.1, 8.5.1.2, and 11.1(b) to clarify the procedures for weighing the impingers and captured moisture. Sections related to transferring the moisture-trap impinger and silica gel impinger contents in sample containers for measurement using graduated cylinders are proposed to be removed.

#### E. Limitations of Method 202

High moisture in the sampled gas stream can result in the accumulation of SO<sub>2</sub> in the collected moisture resulting in a positive bias for CPM measurements. As the moisture accumulates in the sample impingers, the method performs similarly to the original version of Method 202 where SO<sub>2</sub> in the effluent could react in the condensed moisture and form sulfuric acid that may be counted erroneously as CPM. In addition, longer sampling times coupled with high moisture can (in the water-contained impingers) allow more SO<sub>2</sub> conversion to CPM since the conversion of SO<sub>2</sub> to CPM has a relatively slow reaction rate.

Section 8.5.1.1 of Method 202 recommends removing moisture from the sampling train during the test run when the amount of moisture collected is greater than half the capacity of the water dropout impinger or the moisture level of the back-up impinger is above the impinger tip.

Longer sampling run times also delay the start of the post-test nitrogen purge. The post-test nitrogen purge is designed to remove dissolved gasses from the accumulated moisture and thus reduce the potential chemical reactions. In this action, we propose to amend Method 202 by adding a recommendation in section 1.5 to limit the sampling time to 2 hours for Method 202 testing when excessive moisture collection is expected. We also propose revisions to section 8.5.1.1 to specify that if accumulated water exceeds half of the capacity of the water dropout impinger, or if water accumulates in the back-up impinger sufficient to cover the impinger tip, the impinger(s) must be removed and replaced with new pre-weighed impingers and all resulting impingers must be weighed, purged and recovered following the procedures of the method.

The current version of Method 202 also prohibits the use of certain filterable particulate test methods in conjunction with Method 202. In this action, we propose revisions to section 1.4 to state only the acceptable filterable particulate test methods and to include a note that you must maintain the gas filtration temperature as specified in the filterable PM test method unless

otherwise specified by an applicable subpart.

#### F. Required Use of Method 202

Condensable PM is formed from gaseous materials that condense and/or react upon cooling and dilution in the ambient air. Method 202 requires the use of a particulate sampling method (e.g., Method 5, 17, or 201A) to separately collect the filterable PM from CPM.

Filterable PM methods that collect particulate out-of-stack have specified filter temperature requirements and require the addition of a Method 202 sampling train to collect CPM. Filterable PM methods that employ in-stack filters collect particulate material at the source gas temperature.

If the temperature of the filterable PM sampling equipment, including the filter, meets Method 202 temperature requirements (i.e., ≤30 °C (85 °F)), both filterable and CPM are collected together on the filter and CPM is not quantified independently but rather as total particulate, total PM<sub>10</sub>, or total PM<sub>2.5</sub> depending on the filterable collection method.

In this action, we propose to revise section 1.2 to clearly state that, if the sample gas filtration temperature never exceeds 30 °C (85 °F), then Method 202 is not required to measure total primary PM because the CPM would be collected with the filterable PM.

#### G. Sample Container Material

Currently, section 6.2.1(d) of Method 202 specifies the use of amber glass sample bottles for sample recovery. In this action, we propose to revise section 6.2.1(d) to allow the use of sample containers made from other non-reactive materials (e.g., high density polyethylene (HDPE), polytetrafluoroethylene (PTFE)) as an alternative to amber glass bottles for inorganic (aqueous) samples. We also propose to revise sections 6.2.1(d), 8.5.5.3, 8.5.5.5, and 8.5.5.7 to require cleaning of all sample containers according to the procedures in section 8.4 prior to use.

Although we are proposing to revise the method to allow use of polymer or glass sample containers for inorganic samples, we continue to require glass containers for organic samples. The proposed revisions would provide testers with an alternative for storing inorganic samples to avoid this potential source of contamination.

#### H. Weighing Containers

Currently, section 6.2.2(b) of Method 202 specifies that glass evaporation vials, fluoropolymer beaker liners, or

aluminum weighing tins can be used for final sample evaporation and weighing. In this action, we propose to include a list of acceptable weighing containers that includes fluoropolymer beaker liners and other vessels that have low mass and are unreactive to the sample and the atmosphere. Laboratories have reported that aluminum weighing tins may oxidize in contact with some sample matrices. The heavier weight of some glass beakers or containers may cause difficulty with measurement of trace amounts of residual mass. We propose to revise sections 6.2.2(b), 11.2.2.3, 11.2.3, 11.2.4, 11.2.5, and 11.2.6 to remove the connotation of sampling “tin” as an implicit approval of aluminum tins.

#### I. Laboratory Analytical Balance Requirements

We propose additional quality control requirements for analytical balance use. Currently, section 9.6 of Method 202 requires calibration of the analytical balance on each day that samples are weighed, and section 10.3 of the Method 202 Best Practices Handbook provides additional steps that stack testers can use to improve consistency in analytical balance measurements. In this action, we propose to amend section 9.6 to specify the correct mass standard to use for the Analytical Calibration Check, specifications for the temperature and humidity control in weighing areas and requirements for balance calibration checks that approximately match the sample measurements to include the following requirements:

- The laboratory analytical balance must be maintained at a constant temperature of 20 °C ± 3 °C (68 °F ± 5 °F).
- The relative humidity at the location of the laboratory analytical balance must be maintained at 35 to 50 percent, with the exception that if the relative humidity is lower than 35 percent, the relative humidity must be maintained within ±10 percent during sample weighing.
- The results of the calibration check of the laboratory analytical balance must be within 0.05 percent of the applicable certified weight.
- The laboratory analytical balance must be checked each day it is used for gravimetric measurements by weighing at least one ASTM E617–13 Class 2 tolerance (or better) calibration weight that corresponds to 50 to 150 percent of the weight of one filter or between 1 gram (g) and 5 g. If the scale cannot reproduce the value of the calibration weight to within 0.5 mg of the certified mass, perform corrective measures and

conduct the multipoint calibration before use.

#### *J. Field Balance Requirements*

In this action, we propose to correct section 9.4 to specify the mass standard with which to conduct the field balance calibration check. We believe that this additional requirement is necessary to increase consistency of Method 202 moisture sample measurements. We propose the requirement that the field balance calibration check be performed daily with an ASTM E617–13 Class 6 (or better) weight.

#### *K. pH Measurement*

In sections 6.2.2(h) and 11.2.2.2 of the current method, pH measurement by pH meter or colorimetric pH indicator is allowable for the titration procedure. While the use of a colorimetric (*e.g.*, Phenolphthalein) indicator is an acceptable technique for accurately determining the end-point of an acid-base titration, we are concerned that determining the pH using colorimetric pH indicators may introduce additional error in the measurement of CPM due to over-titration.

In this action, we propose to amend sections 6.2.2(h) and 11.2.2.2 to remove the option of using a colorimetric pH indicator and require the use of a pH meter whose calibration has been checked immediately prior to the titration step. We also propose to correct the CPM Sample Processing Flow Chart for sample analysis (Figure 8). We believe these revisions will increase the consistency and comparability of Method 202 results between source tests.

#### *L. Glassware Cleaning Procedures*

To obtain reliable CPM data using Method 202 for PSD and NSR permits, residual mass from sampling and analysis equipment must be minimized.

In this action, we propose the following amendments to clarify equipment and glassware cleaning in section 8.4 of Method 202, including:

- Adding a specification that all glassware used in the implementation of Method 202, including the impinger train and sample containers, should be cleaned sufficiently to meet the blank correction maximum limit of 2.0 mg in section 9.9.
- Removing the statement referencing cleaning silicone grease so that it is not mistakenly viewed as acceptable to use such grease in Method 202 sampling trains.
- Removing the requirement that glassware must be baked after cleaning (although the EPA is proposing to remove the baking requirement, we

highly recommended baking of glassware as discussed in the EPA Method 202 Best Practices Handbook).

- Removing the option to use the field train proof blank as an alternative to baking since the field train proof blank is being proposed as a requirement of Method 202.
- Adding a recommended procedure for cleaning the probe liners by heating for a period of at least 3 hours at the maximum practical temperature.

These proposed revisions make the glassware cleaning procedures performance-based, clarify the requirements, and provide testers with an additional method for ensuring cleanliness of the probe liners.

#### *M. Reagent Blanks*

Currently, Method 202 specifies a volume of 150 milliliters (mL) for performing reagent blank analyses and specifies that field reagent blanks are optional. In this action, we propose to revise section 9.7 to specify a minimum volume of 200 mL for these field reagent blank volumes and to revise section 9.8 to require analysis of field reagent blanks in the performance of Method 202. We also propose to make accompanying revisions to sections 8.5.5.5, 8.5.5.6, 8.5.5.7, 11.2.4, 11.2.5, and 11.2.6.

The original solvent blank volume was intended to represent amounts typically used during sample recovery. A larger reagent blank volume is necessary to quantify residual mass using the analytical balance specified in Method 202 with a sensitivity of 0.0001 g (0.1 mg). These proposed revisions are based on recommendations received from state agencies. This change to the method quality control quantifies any addition to the sample mass from gross contamination originating from the use of reagents in the field.

#### *N. Nitrogen Purge Requirements*

Method 202, as promulgated in 2010, includes two approaches for performing the post-test nitrogen purge: (1) A negative pressure purge using the pump and meter box from the sampling train or (2) a positive pressure purge using the gas cylinder pressure to propel the nitrogen gas through the CPM collection components.

The intent of the multiple purge options was to allow the testing contractors to either purge the sampling train on or near the sampling location or to transport the train components to a controlled environment less susceptible to sources of contamination. We now believe that a post-test nitrogen purge of the sampling train using the meter box and a vacuum pump adds steps that

could potentially contaminate samples and outweigh the advantages of train purges done immediately following the sampling. In this action, we propose to revise section 8.5.4 to eliminate the option for performing the post-test nitrogen purge using the meter box and vacuum pump. We also propose to make accompanying revisions in sections 8.5.4.1, 8.5.4.2, 8.5.4.4 and 8.5.4.5.

#### *O. Data Record Requirements*

In this action, we propose the following amendments to Method 202 sections to record and report test information that were either absent or undefined in the current promulgated method:

- Record the pre- and post-test weights of the impingers, as well as the color of the indicating silica gel, at the completion of sampling (sections 8.4.5 and 8.5.3).
- Record the results of the pre- and post-test leak checks of the sampling train (sections 8.4.6 and 8.5.2).
- Record the time (hh:mm), nitrogen flowrate, CPM filter temperature, and moisture trap temperature (if applicable) during the post-test nitrogen purge (section 8.5.4.4).
- Record the results of the field and laboratory analytical balance calibration checks (sections 9.4 and 9.6.4).
- Record the temperature and relative humidity conditions of the laboratory analytical balance (section 9.6.3).

#### *P. Method Detection Limits*

In this action, we propose to revise section 13.0 regarding method performance. We updated method detection limit values based on a formal study submitted to the EPA by NCASI that evaluated the zero bias of Method 202 when Method 202 Best Practices were implemented. A copy of this study titled, "Method 202 Zero Bias Study When Incorporating Draft Best Practices Developed by the US EPA," (NCASI 2017) is available in the docket (EPA–HQ–OAR–2016–0456–005).

#### *Q. Alternative Blank Procedure and Correction Value*

While the EPA believes that field train proof blank results of 2.0 mg or less are achievable, we recognize there may be certain instances when the environment surrounding the sampling location may significantly contribute to the systematic bias of the method results as measured by the field train proof blank. This proposed alternative procedure would account for the uncontrollable environmental bias associated with measurements collected in problematic sampling locations.

In this action, we are proposing to amend section 16.1 of Method 202 to allow the combined results from multiple field train proof blanks to be used as the basis for blank correction up to 3.9 mg when approved by the regulatory authority. The 3.9 mg value is based on the Upper Prediction Limit (UPL) of the NCASI field study used to update the method detection limit (NCASI 2017). In this procedure, we have included conditions and criteria that a facility must satisfy in order to demonstrate need for the alternative procedure.

#### IV. Request for Comments

The EPA is requesting public comments on all of the proposed editorial and technical amendments to Method 202. For the convenience of the reader, we include in this notice the entire text of Method 202, including proposed revisions, but the scope of this rulemaking is limited to the proposed revisions and does not include any unchanged provisions.

#### V. Statutory and Executive Order Reviews

Additional information about these statutes and Executive Orders can be found at <http://www2.epa.gov/laws-regulations/laws-and-executive-orders>.

##### A. Executive Order 12866: Regulatory Planning and Review and Executive Order 13563: Improving Regulation and Regulatory Review

This action is not a significant regulatory action and was, therefore, not submitted to the Office of Management and Budget (OMB) for review.

##### B. Executive Order 13771: Reducing Regulations and Controlling Regulatory Costs

This action is not expected to be an Executive Order 13771 regulatory action because this action is not significant under Executive Order 12866.

##### C. Paperwork Reduction Act (PRA)

This action does not impose an information collection burden under the PRA. The revisions being proposed in this action do not add information collection requirements, but make corrections and updates to existing testing methodology.

##### D. Regulatory Flexibility Act (RFA)

I certify that this action will not have a significant economic impact on a substantial number of small entities under the RFA. This action will not impose any requirements on small entities. The proposed revisions to Method 202 neither impose any

requirements on regulated entities beyond those specified in the current regulations, nor do they change any emission standard.

##### E. Unfunded Mandates Reform Act (UMRA)

This action does not contain any unfunded mandate of \$100 million or more as described in UMRA, 2 U.S.C. 1531–1538, and does not significantly or uniquely affect small governments. The action imposes no enforceable duty on any state, local or tribal governments or the private sector.

##### F. Executive Order 13132: Federalism

This action 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.

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

This action does not have tribal implications, as specified in Executive Order 13175. This action proposes corrections and updates to the existing procedures specified in Method 202. Thus, Executive Order 13175 does not apply to this action.

##### H. Executive Order 13045: Protection of Children From Environmental Health Risks and Safety Risks

The EPA interprets Executive Order 13045 as applying only to those regulatory actions that concern environmental health or safety risks that the EPA has reason to believe may disproportionately affect children, per the definition of “covered regulatory action” in section 2–202 of the Executive Order. This action is not subject to Executive Order 13045 because it does not concern an environmental health risk or safety risk.

##### I. Executive Order 13211: Actions That Significantly Affect Energy Supply, Distribution, or Use

This action is not subject to Executive Order 13211, because it is not a significant regulatory action under Executive Order 12866.

##### J. National Technology Transfer and Advancement Act (NTTAA)

This rulemaking does not involve technical standards.

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

The EPA believes that this action is not subject to Executive Order 12898 (59 FR 7629, February 16, 1994) because it does not establish an environmental health or safety standard. This action makes corrections and updates to existing testing methodology and does not have any impact on human health or the environment.

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

Administrative practice and procedure, Air pollution control, EPA Method 202, Incorporation by reference, Particulate matter, Reporting and recordkeeping requirements, Sulfur dioxide.

Dated: August 23, 2017.

**E. Scott Pruitt,**  
Administrator.

For the reasons stated in the preamble, the Environmental Protection Agency proposes to amend title 40, chapter I of the Code of Federal Regulations as follows:

#### PART 51—REQUIREMENTS FOR PREPARATION, ADOPTION, AND SUBMITTAL OF IMPLEMENTATION PLANS

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

Authority: 42 U.S.C. 7401, *et seq.*

##### Subpart BB—Data Requirements for Characterizing Air Quality for the Primary SO<sub>2</sub> NAAQS

■ 2. In appendix M to part 51—Recommended Test Methods for State Implementation Plans, revise Method 202 to read as follows:

##### 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.** This method addresses the equipment, preparation, and analysis necessary to measure only CPM. You can use this method only for stationary source emission measurements. You can use this method to measure CPM from stationary source

emissions after filterable particulate matter (PM) has been removed.

Condensable PM is measured in the emissions after removal from the stack and after passing through a filter.

(a) If you are required to measure total primary (direct) PM<sub>2.5</sub> and/or PM<sub>10</sub>, then you must combine the procedures in this method with the procedures in Method 201A of appendix M to this part. If you are required to measure both the filterable and condensable components of total primary (direct) PM emissions to the atmosphere, then you may use Method 5 of appendix A–3 to part 60, or Method 17 of appendix A–6 to part 60.

**Note:** If Method 17 of appendix A–6 to part 60 is attempted in conjunction with Method 202 to measure total primary PM, and the constant weight requirements for the filterable fractions cannot be met, it may be necessary to conduct additional test runs using an applicable filterable PM method that requires a heated filter temperature.

(b) If the gas filtration temperature of the filterable PM method used does not exceed 30 °C (85 °F), then use of this method is not necessary to measure primary PM, as the CPM is collected as filterable PM.

**Note:** For those methods that require in-stack filtration (*i.e.*, Method 17 and 201A), the measured stack temperature is considered the filtration temperature.

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

**1.4 Additional Methods.** To obtain reliable results, you should 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 this part:

(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).

(h) In addition to Method 5, it is also acceptable to use Method 5A, 5D or 5I to collect filterable PM from stationary sources.

**Note:** You must maintain the gas filtration temperature of the filterable PM method as specified in the method, unless otherwise specified by an applicable subpart.

**1.5 Limitations.** You can use this method to measure emissions in stacks that have entrained droplets only when this method is combined with a filterable PM test method that operates at high enough temperatures to cause water droplets sampled through the probe to become vaporous.

**Note:** The EPA recommends that under these conditions or any other conditions, when moisture collection is expected to be in excess of 2 percent, the testing periods be limited to no greater than 2 hours.

**1.6 Conditions.** You must maintain isokinetic sampling conditions to meet the requirements of the filterable PM test method used in conjunction with this method. You must sample at the required number of sampling points specified in the filterable PM test method used in conjunction with this method. Also, if you are using this method as an alternative to a required performance test method, you must receive approval from the regulatory authority that established the requirement to use this test method prior to conducting the test.

## 2.0 Summary of Method

**2.1 Summary.** The CPM is collected in dry impingers after filterable PM has been collected on a filter maintained as specified in either Method 5 of appendix A–3 to part 60, Method 17 of appendix A–6 to part 60, or Method 201A of appendix M to this part. The organic and aqueous sample fractions from the impingers and an out-of-stack CPM filter are then taken to dryness and weighed. The total mass collected from the impinger fractions and the CPM filter represents the CPM. Compared to the version of Method 202 that was promulgated on December 17, 1991, this method eliminates the use of water as the collection media in impingers and includes the addition of a condenser followed by a water dropout impinger after the final in-stack or heated filter. This method also includes the addition of one modified Greenburg-Smith impinger (backup 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 PM.** Condensable PM 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 as soon as possible after the post-test leak check to remove dissolved sulfur dioxide (SO<sub>2</sub>) gases from the impingers. The impinger solutions are collected and the glassware is rinsed with water, acetone, and hexane. The CPM filter is extracted with water and hexane; the extracted liquid is then combined with the hexane and water fractions from the impingers. The aqueous impinger solution is then extracted with hexane. The organic and aqueous fractions are evaporated to dryness 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 vertical condenser and water dropout impinger to separate CPM from reactive gases. No water is added to the water dropout and backup 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 Condensable PM (CPM)** means material that is vapor phase at stack conditions, but 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.

**3.2 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.

**3.3 Field Train Proof Blank.** A field train proof blank for each source category tested is recovered on-site from a clean, fully-assembled sampling train.

**3.4 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.

**3.5 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.

**3.6 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

combined filterable PM<sub>2.5</sub> and condensable PM) means PM with an aerodynamic diameter less than or equal to 2.5 micrometers. These solid particles are emitted directly from an air emissions source or activity, or are the gaseous emissions or liquid droplets from an air emissions source or activity that condense to form PM 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).

**3.7 Primary PM<sub>10</sub>** (also known as direct PM<sub>10</sub>, total PM<sub>10</sub>, PM<sub>10</sub>, or the combination of filterable PM<sub>10</sub> and condensable PM) means PM with an aerodynamic diameter equal to or less than 10 micrometers.

**3.8 ASTM E617-13.** ASTM E617-13 "Standard Specification for Laboratory Weights and Precisions Mass Standards," approved May 1, 2013, was developed and adopted by the American Society for Testing and Materials (ASTM). The standards cover weights and mass standards used in laboratories for specific classes. The ASTM E617-13 standard has been approved for incorporation by reference by the Director of the Office of the Federal Register in accordance with 5 U.S.C. 552(a) and 1 CFR part 51. The standard may be obtained from <http://www.astm.org> or from the ASTM at 100 Barr Harbor Drive, P.O. Box C700, West Conshohocken, PA 19428-2959. All approved material is available for inspection at the EPA Docket Office, EPA WJC West Building, Room 3334, 1301 Constitution Avenue NW., Washington, DC 20460, telephone number (202) 566-1744. It is also available for inspection at the National Archives and Records Administration (NARA). For information on the availability of this material at NARA, call 202-741-6030 or go to [http://www.archives.gov/federal\\_register/code\\_of\\_federal\\_regulations/ibr\\_locations.html](http://www.archives.gov/federal_register/code_of_federal_regulations/ibr_locations.html).

#### 4.0 Interferences

[Reserved]

#### 5.0 Safety

*Disclaimer.* Because the performance of this method may require the use of hazardous materials, operations, and equipment, you should develop a health and safety plan to ensure the safety of your employees who are on site conducting the particulate emission test. Your plan should conform with all applicable Occupational Safety and Health Administration, Mine Safety and

Health Administration, and Department of Transportation regulatory requirements. Because of the unique situations at some facilities and because some facilities may have more stringent requirements than is required by state or federal laws, you may have to develop procedures to conform to the plant health and safety requirements.

#### 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 of appendix M to this part. 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 used in addition to filterable particulate collection using Method 5 of appendix A-3 to part 60, Method 17 of appendix A-6 to part 60, or Method 201A of appendix M to this part. This method includes the following exceptions or additions:

**6.1.1 Probe Extension and Liner.** The probe extension between the filterable particulate filter and the condenser must be glass- or fluoropolymer-lined. Follow the specifications for the probe liner specified in section 6.1.1.2 of Method 5 of appendix A-3 to part 60.

**6.1.2 Condenser and Impingers.** You must add the following components to the filterable particulate sampling train: A vertical condenser, followed by a water dropout impinger or flask, followed by a modified Greenburg-Smith impinger (backup impinger) with an open tube tip as described in section 6.1.1.8 of Method 5 of appendix A-3 to part 60.

**6.1.3 Dropout Impinger Insert for Nitrogen Purge.** You must use a leak-free ground glass fitting with a long glass or PTFE stem (*e.g.*, modified Greenburg-Smith impinger insert or purge stem, etc.) for the water dropout impinger to perform the nitrogen purge of the sampling train. The glass stem must be designed so that the tip of the stem is 1/2" from the bottom of the impinger.

**6.1.4 CPM Filter Holder.** The modified Greenburg-Smith impinger is followed by a filter holder that is either glass, stainless steel (316 or equivalent), or fluoropolymer-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 filter. Commercial size filter holders contain a fluoropolymer O-ring, stainless steel, ceramic or fluoropolymer filter support and a final

fluoropolymer O-ring. At the exit of the CPM filter, install a fluoropolymer-coated or stainless steel encased thermocouple that is in direct contact with the gas stream.

#### 6.2 Sample Recovery Equipment

**6.2.1 Condensable PM Recovery.** Use the following equipment to quantitatively determine the amount of CPM recovered from the sampling train.

(a) Nitrogen purge line. You must use inert tubing and fittings capable of delivering at least 14 liters/min of nitrogen gas to the impinger train from a standard gas cylinder (*see* Figures 2 and 3 of section 18). You may use standard 0.6 centimeters (1/4 inch) tubing and compression fittings in conjunction with an adjustable pressure regulator and needle valve.

(b) Rotameter. You must use a rotameter capable of measuring gas flow up to 20 liters/min. The rotameter must be accurate to five percent of full scale.

(c) Nitrogen gas purging system. Compressed ultra-pure nitrogen, regulator, and filter must be capable of providing at least 14 liters/min purge gas for one hour through the sampling train.

(d) Sample bottles (500 ml). You must use amber glass bottles or other non-reactive bottles (*e.g.*, High Density Linear Polyethylene (HDLPE), or PTFE) pre-cleaned sample bottles for inorganic samples. Amber glass bottles are required for organic samples and must be prepared according to section 8.4 of this method.

**6.2.2 Analysis Equipment.** The following equipment is necessary for CPM sample analysis:

(a) Separatory Funnel. Glass, 1 liter.

(b) Weighing Containers. Fluoropolymer beaker liners or other low-mass vessels which are unreactive to the sample or atmosphere.

**Note:** The use of an anti-static device(s) during gravimetric analysis to prevent static from interfering with the analysis is recommended when using Fluoropolymer or similar beaker liners.

(c) Glass Beakers. 300 to 500 ml.

(d) Drying Equipment. A desiccator containing anhydrous calcium sulfate that is maintained below 10 percent relative humidity, and a hot plate or oven equipped with temperature control.

(e) Glass Pipets. 5 ml.

(f) Burette. Glass, 0 to 100 ml in 0.1 ml graduations.

(g) Analytical Balance. Analytical balance capable of weighing at least 0.0001 g (0.1 mg).

(h) pH Meter. The pH meter must be capable of determining the acidity of liquid within 0.1 pH units.

(i) Sonication Device. The device must have a minimum sonication frequency of 20 kHz and be approximately four to six inches deep to accommodate the sample extractor tube.

(j) Leak-Proof Sample Containers. Containers used for sample and blank recovery must not contribute more than 0.05 mg of residual mass to the CPM measurements.

(k) Wash bottles. Any container material is acceptable, but wash bottles used for sample and blank recovery must not contribute more than 0.1 mg of residual mass to the CPM measurements.

## 7.0 Reagents and Standards

**7.1 Sample Collection.** To collect a sample, you will need a CPM 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 CPM Filter.** You must use a nonreactive, non-disintegrating polymer filter that does not have an organic binder and does not contribute more than 0.5 mg of residual mass to the CPM measurements. The CPM filter must also have an efficiency of at least 99.95 percent (less than 0.05 percent penetration) on 0.3 micrometer dioctyl phthalate particles. You may use test data from the supplier's quality control program to document the CPM filter efficiency.

**7.1.2 Silica Gel.** Use an indicating-type silica gel of 6 to 16 mesh. You must obtain approval of the Administrator for 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 if the indicator shows the silica gel is active for moisture collection.

**7.1.3 Water.** Use deionized, ultra-filtered water that contains 1.0 parts per million by weight (ppmw) (1 mg/L) residual mass or less to recover and extract samples.

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

**7.1.5 Nitrogen Gas.** Use Ultra-High Purity 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 parts per million by volume (ppmv) oxygen, 1 ppmv total hydrocarbons as carbon, and 2 ppmv moisture. The compressed nitrogen must not contribute more than 0.1 mg of residual mass per purge.

**7.2 Sample Recovery and Analytical Reagents.** You will need acetone, hexane, anhydrous calcium sulfate,

ammonia hydroxide, 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. 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 residual mass in the laboratory and field reagent blanks. You must use acetone that has a blank value less than 1.0 ppmw (0.1 mg/100 g) residue.

**7.2.2 Hexane, American Chemical Society Grade or Equivalent.** You must use hexane that has a blank residual mass value less than 1.0 ppmw (0.1 mg/100 g) residue.

**7.2.3 Water.** Use deionized, ultra-filtered water that contains 1.0 ppmw (1.0 mg/L) residual mass or less to recover material caught in the impinger.

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

**7.2.5 Ammonium Hydroxide.** Use National Institute of Standards and Technology (NIST)-traceable or equivalent (0.1 N) ammonium hydroxide (NH<sub>4</sub>OH).

**7.2.6 Standard Buffer Solutions.** Use one buffer solution with a neutral pH and a second buffer solution with an acid pH of no less than 4.

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

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

**8.2 Preparations.** Clean all glassware used to collect and analyze samples prior to field tests as described in Section 8.4 prior to use. Cleaned glassware must be used at the start of each new source category tested at a single facility. You must analyze laboratory reagent blanks (water, acetone, and hexane) before field tests to verify low blank concentrations for the reagent lot(s) used. Follow the pretest preparation instructions in Section 8.1 of Method 5.

**8.3 Site Setup.** You must follow the procedures required in Methods 5, 17, or 201A, whichever is applicable to your test requirements including:

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

(b) Calculating probe/cyclone blockage (as appropriate).

(c) Verifying the absence of cyclonic flow.

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

**8.3.1 Sampling Site Location.** Follow the standard procedures in Method 1 of appendix A-1 to part 60 to select the appropriate sampling site. Choose a location that maximizes the distance from upstream and downstream flow disturbances.

**8.3.2 Traverse Points.** Use the required number of traverse points at any location, as found in in the method used to collect the filterable particulate. 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 (0.5 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 glassware that is used to collect and analyze samples should be cleaned sufficiently to meet the maximum field train proof blank contribution to be subtracted from the test results in section 9.9 (0.002g or 2.0 mg). Cleaning glassware prior to the test with soap and water, then rinsing with tap water, followed by deionized water, acetone, and finally, hexane is recommended. After cleaning, you should bake glassware at 300 °C for 6 hours prior to beginning tests at each source category sampled at a facility. Prior to each sampling run, the train glassware used to collect condensable PM must be rinsed thoroughly with acetone, hexane, and then deionized, ultra-filtered water that contains 1 ppmw (1 mg/L) residual mass or less.

**Note:** Due the length of most probes, it is not practical to heat them in an oven. After cleaning the probe liners, it is recommended to heat the probe to the maximum temperature practical for the probe sheath for a period of at least 3 hours. Then rinse thoroughly with acetone, hexane, and deionized, ultra-filtered water.

**8.4.1 Condenser and Water Dropout Impinger.** Add a vertical condenser and a water dropout impinger without bubbler tube after the final probe extension that connects the in-stack or out-of-stack hot filter assembly with the CPM sampling train. This vertical condenser must be constructed in a manner that prevents the pooling of the condensate liquid within the condenser and be capable of cooling the stack gas to less than or equal to 30 °C (85 °F).

At the start of the tests, the condenser and water dropout impingers must be clean, without any water or reagent added.

**8.4.2 Backup Impinger.** The water dropout impinger is followed by a modified Greenburg-Smith impinger (backup impinger) with no taper (*see* Figure 1 of section 18). Place the water dropout and backup impingers in an insulated box with water at less than or equal to 30 °C (less than or equal to 85 °F). At the start of the tests, the backup impinger must be free of any residual solvents from the recovery or glassware preparation.

**8.4.3 CPM Filter.** Place a filter holder with a filter meeting the requirements in section 7.1.1 after the backup impinger. The connection between the CPM filter and the moisture trap impinger must include a thermocouple fitting that provides a leak-free seal between the thermocouple and the stack 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 of appendix A-3 to part 60, followed by an impinger containing 200 to 300 g of indicating-type 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 Weighing of Impingers (Pretest).** Weigh each impinger to 0.1 g, including the silica gel impinger prior to train assembly using the field balance. Record the weights of each impinger on the CPM Impinger Data Sheet (Figure 4).

**8.4.6 Leak-Check (Pretest).** Use the procedures outlined in Method 5 of appendix A-3 to part 60, Method 17 of appendix A-6 to part 60, or Method 201A of appendix M to this part 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 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. Record the leak-check results on the field test data sheet (*see* Figure 5). (**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 of appendix A-3 to part 60, Method 17 of appendix A-6 to part 60, or Method 201A of appendix M to this part) with the following additions or exceptions:

#### 8.5.1 Impinger and CPM Filter Assembly

**8.5.1.1** During sampling, monitor the moisture condensation in the water dropout impinger and backup impinger. If the accumulated water from moisture condensation overwhelms (*i.e.*, the water level is more than approximately one-half the capacity of the water dropout impinger) the water dropout impinger, or if water accumulates in the backup impinger sufficient to cover the impinger insert tip, then you must interrupt the sampling run, leak check the Method 202 portion of the sampling train, replace the water dropout and/or backup impingers with new pre-weighed impinger(s), reassemble, leak check the sampling train, and then resume the sampling run. Weigh the impingers removed from the sampling train and purge the water collected as soon as practical following the procedures in section 8.5.3.

**8.5.1.2** You must include the weight of the moisture in your moisture calculation and you must combine the recovered water with the appropriate sample fraction for subsequent CPM analysis.

**8.5.1.3** Use the field data sheet to record the CPM filter temperature readings at the beginning of each sample time increment and when sampling is halted. Maintain the CPM filter greater than 20 °C (greater than 65 °F) but less than or equal to 30 °C (less than or equal to 85 °F) during sample collection.

**8.5.2 Leak-Check (Post-Test).** Conduct the leak rate check according to the filterable particulate sampling method used during sampling. Conduct the leak-check at a vacuum equal to or greater than the maximum vacuum achieved during the test run. Record the leak-check results on the field test data sheet. If the leak rate of the sampling train exceeds 0.02 actual cubic feet per minute 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 Weighing of Impingers (Post-test).** You must weigh each impinger to 0.1 g after the completion of the testing and prior to the post-test nitrogen purge and record these weights on the CPM Impinger data sheet. Alternatively, you

may choose to weigh each impinger after completion of the post-test nitrogen purge. If this option is chosen, you must do the following in addition to the procedures of section 8.5.4. Purge the sampling train from the water dropout impinger to the exhaust of the moisture traps (*see* Figure 2). You must maintain the temperature of the moisture traps following the CPM filter 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).

**Note:** You should also note the color of the indicating silica gel to determine whether it has been completely spent, and record its condition on the CPM Impinger Data Sheet.

**8.5.4 Post-Test Nitrogen Purge.** As soon as possible after the post-test leak-check, conduct the nitrogen purge. If no water was collected before the CPM filter, then you may skip the remaining purge steps and proceed with sample recovery (*see* section 8.5.5). If any water was collected before the CPM filter, you must purge the CPM sampling train.

**8.5.4.1** You may purge the entire CPM sample collection train from the water dropout impinger through the CPM filter holder outlet *or* you may quantitatively transfer the water collected in the water dropout impinger to the backup impinger and purge only the backup impinger and the CPM filter and holder (*see* Figure 3).

**8.5.4.2** If you choose to conduct a purge of the entire CPM sampling train, you must place the dropout impinger insert into the water dropout impinger, and the impinger tip must extend at least 1 centimeter below the water level of the impinger catch.

**8.5.4.3** If the tip of the impinger insert does not extend below the water level (including the water transferred from the water dropout impinger if this option was chosen), you must add a measured amount of degassed, deionized ultra-filtered water that contains 1 ppmw (1 mg/L) residual mass or less until the impinger tip is at least 1 centimeter below the surface of the water. You must record the amount of water added to the water dropout impinger ( $V_p$ ) (*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.)

**8.5.4.4** To perform the nitrogen purge, you must start with no flow of gas running through the clean purge line and fittings. Connect the purge nitrogen in-line filter outlet to the input of the

impinger train to be purged. Increase the nitrogen flow gradually to avoid over-pressurizing the impinger array. You must purge the CPM train at a minimum of 14 liters per minute. Record the time (hh:mm), nitrogen flowrate, and the temperature(s) of the CPM filter and moisture trap (if applicable) at the start of the nitrogen purge on the CPM Impinger Data Sheet.

**8.5.4.5** During the purge procedure, maintain the gas temperature measured at the exit of the CPM filter greater than 20 °C (65 °F), but less than or equal to 30 °C (85 °F). Continue the purge under these conditions for at least 1 hour, recording the CPM temperature and nitrogen rotameter value every 10 minutes. At the conclusion of the purge, turn off the nitrogen delivery system. Record the time (hh:mm) of the purge and the temperature of the CPM filter at the start of the nitrogen purge on the CPM Impinger Data Sheet.

### 8.5.5 Sample Recovery

**8.5.5.1** Filterable PM samples. Recovery of the filterable PM samples involves the quantitative transfer of PM according to the filterable particulate sampling method used (*i.e.*, Method 5 of appendix A–3 to part 60, Method 17 of appendix A–6 to part 60, or Method 201A of appendix M to this part).

**8.5.5.2** CPM Container #1, Aqueous liquid impinger contents. Quantitatively transfer liquid from the dropout and the backup impingers prior to the CPM filter into a clean, leak-proof container labeled with test identification and “CPM Container #1, Aqueous Liquid Impinger Contents.” Rinse all sampling train components including the back half of the filterable PM filter holder, the probe extension (if applicable), 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 CPM Container #1. Mark the liquid level on the container.

**8.5.5.3** CPM Container #2, Organic rinses. Follow the water rinses of the back half of the filterable PM filter holder, probe extension (if applicable), condenser, each impinger, and all of the connecting glassware and front half of the CPM filter with an acetone rinse. Recover the acetone rinse into a clean, leak-proof amber glass container labeled with test identification and “CPM Container #2, Organic Rinses.” Then repeat the entire rinse procedure with two rinses of hexane, and save the hexane rinses in the same container as the acetone rinse (CPM Container #2). Mark the liquid level on the container.

**8.5.5.4** 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 labeled with test identification and “CPM Container #3, Filter Sample.”

**8.5.5.5** CPM Container #4, Acetone field reagent blank. Take a minimum of 200 ml of the acetone directly from the wash bottle you used for sample recovery and place it in a clean, leak-proof amber glass container labeled with test identification and “CPM Container #4, Acetone Field Reagent Blank” (*see* section 11.2.6 for analysis). Mark the liquid level on the container. Collect one acetone field reagent blank from each lot of acetone used for the test.

**8.5.5.6** CPM Container #5, Water field reagent blank. Take a minimum of 200 ml of the water directly from the wash bottle you used for sample recovery and place it in a clean, leak-proof container labeled with test identification and “CPM Container #5, Water Field Reagent Blank” (*see* section 11.2.7 for analysis). Mark the liquid level on the container. Collect one water field reagent blank from each lot of water used for the test.

**8.5.5.7** CPM Container #6, Hexane field reagent blank. Take a minimum of 200 ml of the hexane directly from the wash bottle you used for sample recovery and place it in a clean, leak-proof amber glass container labeled with test identification and “CPM Container #6, Hexane Field Reagent Blank” (*see* section 11.2.8 for analysis). Mark the liquid level on the container. Collect one hexane field reagent blank from each lot of hexane used for the test.

**8.5.5.8** Field train proof blank. To demonstrate the cleanliness of sampling train glassware, you must prepare a full sampling train to serve as a field train proof blank just as it would be prepared for sampling, including the filterable PM method front half, probe extension (if applicable), condenser, impingers, CPM filter, and transfer line. Transport and assemble the field train proof blank sample train to the sampling location and perform a pre-test leak check as if it were an actual sample train. Hold this train at the sampling location for the same amount of time as a test run unless otherwise specified by the Administrator, and perform a post-test leak check on this train at the end of the actual test sampling time. After the post-test leak check, you must conduct a nitrogen purge of the field train proof blank sample as specified in section 8.5.4. For the nitrogen purge, you must add 100 ml of deionized ultra-filtered water and replicate the nitrogen purge procedures that you will use for the test runs. After conducting the nitrogen purge, recover the field train proof blank

as described in sections 8.5.5.8.1 through 8.5.5.8.3.

**8.5.5.8.1** CPM Container #7, Field train proof blank, inorganic rinses. 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 place it in a clean, leak-proof container labeled with test identification and “CPM Container #7, Field Train Proof Blank, Inorganic Rinses.” Mark the liquid level on the container.

**8.5.5.8.2** CPM Container #8, Field train proof blank, organic rinses. Follow the water rinse of the probe extension, condenser, each impinger and the connecting glassware, and the front half of the CPM filter housing with an acetone rinse. Recover the acetone rinse into a clean, leak-proof container labeled with test identification and “CPM Container #8, Field Train Proof Blank, Organic Rinses.” Then repeat the entire rinse procedure with two rinses of hexane and recover the hexane rinses into the same container as the acetone rinse (CPM Container #10). Mark the liquid level on the container.

**8.5.5.8.3** CPM Container #9, Field train proof blank, 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 labeled with test identification and “CPM Container #9, Field Train Proof Blank, Filter Sample.”

**8.5.6** *Sample 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 should 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 notebooks 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 notebook 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 Field Balance Calibration

**Check.** Record the results of the calibration check procedures on field balances each day that they are used as required in section 10.3.

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

#### 9.6 Laboratory Analytical Balance

9.6.1 Maintain the location of the analytical balance (*i.e.*, weighing room) at  $20\text{ }^{\circ}\text{C} \pm 3\text{ }^{\circ}\text{C}$  ( $68\text{ }^{\circ}\text{F} \pm 5\text{ }^{\circ}\text{F}$ ).

9.6.2 *Maintain the location the analytical balance (i.e., weighing room) at 35 to 50 percent relative humidity.* Alternatively, it is acceptable for the percent relative humidity to be less than 35 percent. In either case, you should maintain the relative humidity within  $\pm 10$  percent relative humidity for sampling weighings.

9.6.3 Record and report the temperature and relative humidity of the analytical balance location for each measurement performed.

9.6.4 *Calibration Check.* Record the calibration check of your laboratory analytical balance at least once each day that you weigh CPM samples. Audit the balance using at least one ASTM E617–13 Class 2 tolerance (or better) calibration weight, within 1 g to 5 g of the weight of the sample plus container you will be weighing.

9.7 *Laboratory Reagent Blanks.* You should analyze blanks of water, acetone, and hexane used for field recovery and sample analysis. Analyze and report at least one sample (500 ml minimum) of each lot of reagents that you plan to use for sample recovery and analysis. These blanks are not required by the test method, but analyzing reagent blanks before field use is recommended to verify low reagent blank concentrations.

9.8 *Field Reagent Blanks.* You must analyze and report the results of each lot of reagent used for the field test.

9.9 *Field Train Proof Blank.* You must recover a minimum of one field train proof blank for each new source category at a single facility using glassware prepped according to section 8.4. You must assemble the sampling train as it will be used for testing, including the filterable PM method front half, CPM filter, and transfer line. You must prepare and recover the field train proof blank as described in section

8.5.5.8. From each field sample weight, you will subtract the condensable particulate mass you determine with this field train proof blank or 0.002 g (2.0 mg), whichever is less, unless otherwise specified by the regulatory authority.

### 10.0 Calibration and Standardization

Maintain a field log notebook 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 10.3.1 of Method 2 of appendix A–1 to part 60 or Alternative Method 2, Thermocouple Calibration (ALT–011) (<https://www.epa.gov/emc>). Calibrate each temperature sensor at a minimum of three points over the anticipated range of use against a NIST-traceable thermometer. Alternatively, a reference thermocouple and potentiometer calibrated against NIST standards can be used.

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 certified standard of 0.1 N  $\text{H}_2\text{SO}_4$ , and calculate the exact normality using a procedure parallel to that described in section 10.5 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 CPM Work Table (*see* Figure 6 of section 18).

10.3 *Field Balance Calibration Check.* Check the calibration of the balance used to weigh impingers with a weight that is at least 500 g or within 50 g of a loaded impinger. The weight must be ASTM E617–13 “Standard Specification for Laboratory Weights and Precision Mass Standards” Class 6 (or better). Daily, before use, the field balance must measure the weight within  $\pm 0.5$  g of the certified mass and record the results. If the balance calibration check fails, perform corrective measures and repeat the check before using balance.

10.4 *Analytical Balance Calibration.* Perform a multipoint calibration (at least five points spanning the operational range) of the analytical balance before the first use, and semiannually thereafter. The calibration of the analytical balance must be conducted using ASTM E617–13 “Standard Specification for Laboratory Weights and Precision Mass Standards” Class 2 (or better) tolerance weights. Audit the balance each day it is used for

gravimetric measurements by weighing at least one ASTM E617–13 Class 2 tolerance (or better) calibration weight that corresponds to 50 to 150 percent of the weight of one filter or between 1 g and 5 g and record the results. If the scale cannot reproduce the value of the calibration weight to within 0.5 mg of the certified mass, perform corrective measures and conduct the multipoint calibration before use.

### 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. Record the condensable particulate data on the CPM Work Table (*see* Figure 7 of section 18).

(b) Visually inspect the liquid level mark on each sample container and record on the CPM Work Table 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 PM Analysis.* See the flow chart in Figure 8 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 CPM filter as described in this section.

11.2.1.1 Extract the water soluble (aqueous or inorganic) CPM from the CPM filter by placing it into a clean extraction container or flask. Add sufficient deionized, ultra-filtered water to cover the filter (*e.g.*, 10 ml of water). Place the extractor container 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 by adding sufficient hexane to cover the filter (*e.g.*, 10 ml of hexane). Place the extractor tube into a sonication bath and extract the organic soluble material for a minimum of two 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 hexane to the funnel, mix well, and pour off the upper organic phase. Repeat this procedure twice with 30 ml

of hexane each time combining the organic phase from each extraction. Each time, leave a small amount of the organic/hexane 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. Combine the organic extract from Container #1 with the organic train rinse in Container #2.

**11.2.2.1** Determine the inorganic fraction weight. Transfer the aqueous fraction from the extraction to a clean 500 ml or smaller beaker. Evaporate to no 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)). 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. (See section 3.0 for a definition of constant weight.) Report results to the nearest 0.1 mg on the CPM Work Table (see Figure 6 of section 18) and proceed directly to section 11.2.3. If the residue cannot be weighed to constant weight, re-dissolve the residue in 100 ml of deionized distilled ultra-filtered water that contains 1 ppmw (1 mg/L) residual mass or less and continue to section 11.2.2.2.

**11.2.2.2** You must ensure that water and volatile acids have completely evaporated before neutralizing nonvolatile acids in the sample. Only after failure to reach constant weight and rehydration, per section 11.2.2.1, use titration to neutralize acid in the sample and remove water of hydration. Calibrate the pH meter with the neutral and acid buffer solutions immediately prior to the titration of the samples. Then titrate the sample with 0.1 N NH<sub>4</sub>OH to a pH of 7.0, as indicated by the pH meter. Record the volume of titrant used on the CPM Work Table (see Figure 6 of section 18).

**11.2.2.3** 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 clean, 50 ml pre-tared weighing container 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. (See section 3.0 for a definition of constant weight.) Report results to the nearest 0.1 mg on the CPM Work Table (see Figure 6 of section 18).

**11.2.2.4** Calculate the correction factor to subtract the NH<sub>4</sub><sup>+</sup> retained in the sample using Equation 1 in section 12.

**11.2.3 CPM Container #2, Organic Fraction Weight Determination.** Analyze the organic soluble CPM in Container #2 as described in this section. 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 clean 50 ml pre-tared weighing container 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.*, less than or equal to 0.5 mg change from previous weighing), and report results to the nearest 0.1 mg on the CPM Work Table (see Figure 6 of section 18).

**11.2.4 Container #4, Acetone Field Reagent Blank.** Use 200 ml of acetone from the blank container used for this analysis. Transfer 200 ml of the acetone field reagent blank 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 clean pre-tared weighing container, 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.*, less than or equal to 0.5 mg change from previous weighing), and report results to the nearest 0.1 mg on Figure 5 of section 19.

**11.2.5 Container #5, Water Field Reagent Blank.** Use 200 ml of the water from the blank container for this analysis. 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 50 ml pre-tared weighing container 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.*, less than or equal to 0.5 mg change from previous weighing) and report results to the nearest 0.1 mg on Figure 5 of section 18.

**11.2.6 Container #6, Hexane Field Reagent Blank.** Use 200 ml of hexane from the blank container for this analysis. Transfer 150 ml of the hexane

to a clean 250 ml beaker. Evaporate the hexane 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 clean 50 ml pre-tared weighing container 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.*, less than or equal to 0.5 mg change from previous weighing), and report results to the nearest 0.1 mg on Figure 5 of section 18.

## 12.0 Calculations and Data Analysis

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

$\Delta H_{\odot}$  = Pressure drop across orifice at flow rate of 0.75 SCFM at standard conditions, inches of water column (**Note** Specific to each orifice and meter box).  
17.03 = mg/milliequivalents for ammonium ion.

ACFM = Actual cubic feet per minute.  
 $C_{cpm}$  = Concentration of the condensable PM in the stack gas, dry basis, corrected to standard conditions, milligrams/dry standard cubic foot.

$m_c$  = Mass of the NH<sub>4</sub><sup>+</sup> added to sample to form ammonium sulfate, mg.

$m_{cpm}$  = Mass of the total condensable PM, mg.

$m_{tb}$  = Mass of total CPM in field train proof blank, mg.

mg = Milligrams.

mg/dscf = Milligrams per dry standard cubic foot.

mg/L = Milligrams per liter.

$m_i$  = Mass of inorganic CPM, mg.

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

$m_o$  = Mass of organic CPM, mg.

$m_{ob}$  = Mass of organic CPM in field train proof blank, mg.

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

N = Normality of ammonium hydroxide titrant.

ppmv = Parts per million by volume.

ppmw = Parts per million by weight.

$V_{m(st,d)}$  = 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 NH<sub>4</sub>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 CPM Work Table (see Figure 7 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 Train Proof Blank (mg).* Per section 9.9, the mass of the field train proof blank,  $m_b$ , shall not exceed 2.0 mg.

$$M_{fb} = M_{ib} + M_{ob} \quad (\text{Eq. 2})$$

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).*

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

12.2.5 *Concentration of CPM (mg/dscf).*

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

12.3 *Emissions Test Report.* You must prepare a test report following the guidance in EPA Guideline Document 043.

### 13.0 Method Performance

A field evaluation (NCASI 2017) of Method 202 incorporating Best Practices showed that the detection limit was 1.6 for total CPM; consisting of approximately 1.0 mg for organic CPM and approximately 0.6 mg for inorganic CPM. This field evaluation also demonstrated that the expected blank value of the field train proof blank was less than 1.8 mg.

### 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 Resource Conservation and Recovery Act organic waste.

### 16.0 Alternative Procedures

16.1 *Alternative Field Train Proof Blank Procedure.* The following procedure may be utilized with approval by the regulatory authority at stationary sources with environments with significant ambient PM concentrations that could positively bias the results of the Method 202 samples collected. This procedure would permit you to subtract up to 0.0039 g (3.9 mg) from the measured condensable particulate mass.

16.1.1 The facility must request this alternative prior to the test program, and the request must be approved by the regulatory authority prior to the testing. The request may include the following elements:

(1) Documented adherence to the Best Practices for Method 202 by the tester. This documentation may include:

- (a) Tester's Method 202 standard operating procedure (SOP);
- (b) Residual mass of the laboratory reagent blanks (Reagent ID, Manufacturer, Lot Number);
- (c) Tester-specific Method Detection Limit;
- (d) Training records.

(2) Justification by the facility that the environment around the sampling location is likely to bias the CPM results. This justification may include:

- (a) Schematic of the facility identifying locations that may contribute to environmental bias;
- (b) Ambient PM concentration (mg/m<sup>3</sup>);
- (c) Previous test results (*i.e.*, field train proof blank results).

16.1.2 Upon the regulatory authority approval, you will recover a minimum of two field train proof blanks for each source category tested at the subject facility using glassware prepped according to section 8.4 of this method. You must perform the field train proof blank evaluations as described in section 9.9 of this method.

16.1.3 From each field sample weight, you will subtract the average condensable particulate mass you determine with all of the duplicate field train proof blank trains or 0.0039 g (3.9 mg), whichever is less unless the difference between highest and lowest values of the field train proof blanks is >1.0 mg. If the agreement is >1.0 mg, then you must subtract the lowest

condensable particulate mass values you determine with the field train proof blank trains or 0.002 g (2.0 mg), whichever is less, unless otherwise specified by the regulatory authority.

#### 16.2 Alternative Method 2.

Thermocouple Calibration (ALT-011) for the thermocouple calibration can be found at <http://www3.epa.gov/ttn/emc/approalt/alt-011.pdf>.

### 17.0 References

- (1) Commonwealth of Pennsylvania, Department of Environmental Resources. 1960. 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.
- (2) DeWees, W.D. and K.C. Steinsberger. 1989. "Method Development and Evaluation of Draft Protocol for Measurement of Condensable Particulate Emissions." Draft Report. November 17, 1989.
- (3) DeWees, W.D., K.C. Steinsberger, G.M. Plummer, L.T. Lay, G.D. McAlister, and R.T. Shigehara. 1989. "Laboratory and Field Evaluation of 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.
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### 18.0 Tables, Diagrams, Flowcharts, and Validation Data

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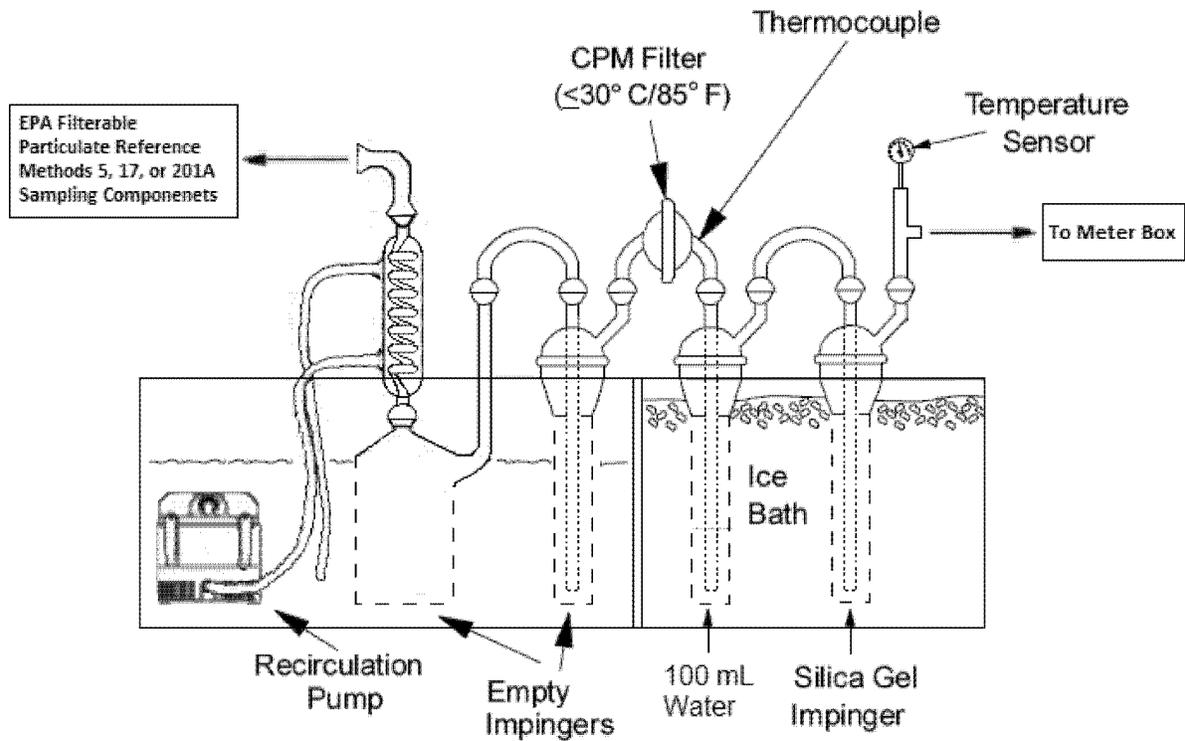


Figure 1. Schematic of Condensable Particulate Sampling Train

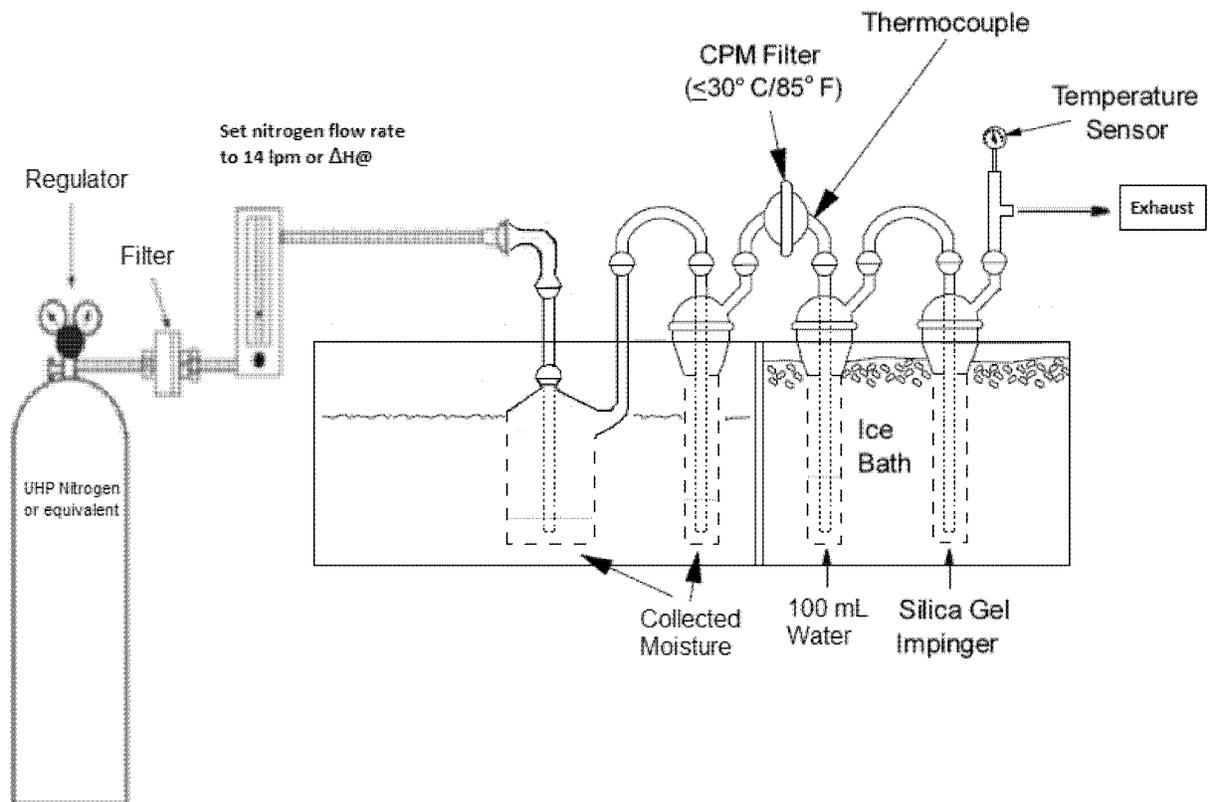


Figure 2. Nitrogen Purge (Entire CPM Train)

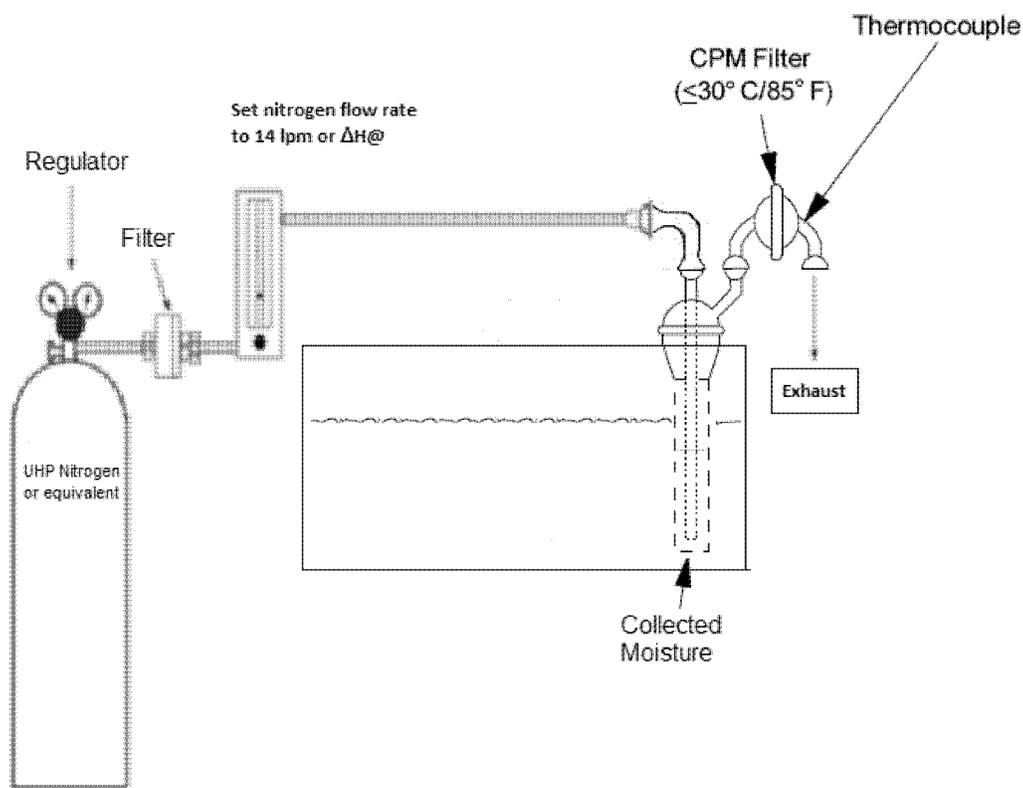
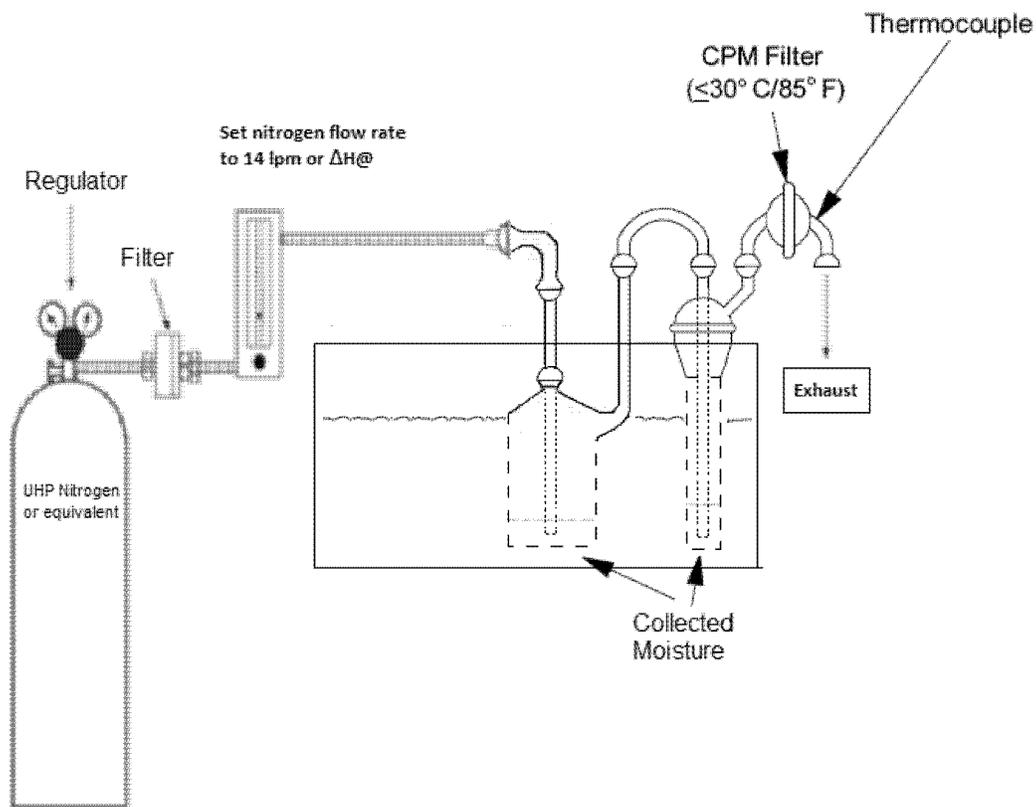


Figure 3. Nitrogen Purge (Alternative Configurations)

| CPM Impinger Field Data Page                                  |                          |                       |                       |                                       |
|---|--------------------------|-----------------------|-----------------------|---------------------------------------|
| Plant   |                          |                       |                       |                                       |
| Date  |                          |                       |                       |                                       |
| Source ID   |                          |                       |                       |                                       |
| Run Number  |                          |                       |                       |                                       |
|   | Mass of Liquid Collected |                       |                       |                                       |
|   | CPM Impingers            |                       | Moisture Trap         |                                       |
|   | Dropout Impinger         | Back-up Impinger      | Impinger              | Silica Gel                            |
| Final - g   |                          |                       |                       |                                       |
| Initial - g   |                          |                       |                       |                                       |
| V <sub>p</sub> - Water added to purge train - ml <sup>1</sup> |                          |                       | ----                  | ----                                  |
| Mass of water collected <sup>2</sup>                          |                          |                       |                       |                                       |
| Total mass of water collected                                 |                          |                       |                       |                                       |
| Condition of Silica Gel                                       |                          |                       |                       |                                       |
| Post-Test Purge   |                          |                       |                       |                                       |
|   | Time (HH:MM)             | Nitrogen Flowrate-lpm | CPM Filter Temp. (°F) | Moisture Trap Temp. <sup>3</sup> (°F) |
| Start   |                          |                       |                       |                                       |
|   |                          |                       |                       |                                       |
|   |                          |                       |                       |                                       |
|   |                          |                       |                       |                                       |
| End   |                          |                       |                       |                                       |

<sup>1</sup> Convert volume of water to mass by multiplying volume by density of water (1g/ml)

<sup>2</sup> Final Mass – (Initial mass – water added for purge)

<sup>3</sup> If applicable

**Figure 4. CPM Impinger Data Sheet**



| <b>Field Train Proof Blank Condensable Particulate Calculations</b>     |    |
|---|----|
| Plant   |    |
| Date  |    |
| Blank No.   |    |
| <b>Field Reagent Blank Mass</b>   |    |
| Water (Section 11.2.7)  | mg |
| Acetone (Section 11.2.6)  | mg |
| Hexane (Section 11.2.8)   | mg |
| <b>Field Train Proof Blank Mass</b>                                     |    |
| Mass of Organic CPM ( $m_{ob}$ )(Section 11.2.3)                        | mg |
| Mass of Inorganic CPM ( $m_{ib}$ )(Equation 3)                          | mg |
| Mass of the Field Train Proof Blank (not to exceed 2.0 mg) (Equation 2) | mg |

**Figure 6. Field Train Proof Blank Condensable Particulate Calculations**

| <b>Calculations for Recovery of Condensable PM (CPM)</b>                 |       |    |
|--|-------|----|
| Plant  | _____ |    |
| Date   | _____ |    |
| Run No.  | _____ |    |
| <b>Sample Preparation - CPM Containers No. 1 and 2 (Section 11.1)</b>    |       |    |
| Was significant volume of water lost during transport?                   | _____ |    |
| Yes or No  |       |    |
| If Yes, measure the volume received.                                     | _____ |    |
| Estimate the volume lost during transport.                               | _____ | ml |
| Was significant volume of organic rinse lost during transport? Yes or No | _____ |    |
| If Yes, measure the volume received.                                     | _____ |    |
| Estimate the volume lost during transport.                               | _____ | ml |
| <b>For Titration</b>   |       |    |
| Normality of $\text{NH}_4\text{OH}$ (N)                                  |       |    |
| (Section 10.2)   |       | N  |
| Volume of titrant ( $V_t$ )  | _____ |    |
| (Section 11.2.2.2)   |       | ml |
| Mass of $\text{NH}_4$ added ( $m_c$ )                                    | _____ |    |
| (Equation 1)   |       | mg |
| <b>For CPM Blank Weights</b>   |       |    |
| Inorganic Field Train Proof Blank Mass ( $m_{ib}$ ) (Section 9.9)        | _____ | mg |
| Organic Field Train Proof Blank Mass ( $m_{ob}$ ) (Section 9.9)          | _____ | mg |
| Mass of Field Train Proof Blank ( $M_{fb}$ ) (max. 2 mg)                 |       |    |
| (Equation 2)   |       | mg |
| <b>For CPM Train Weights</b>   |       |    |
| Mass of Organic CPM ( $m_o$ ) (Section 11.2.3)                           | _____ | mg |
| Mass of Inorganic CPM ( $m_i$ ) (Equation 3)                             | _____ | mg |
| Total CPM Mass ( $m_{cpm}$ ) (Equation 4)                                | _____ | mg |

Figure 7. CPM Work Table

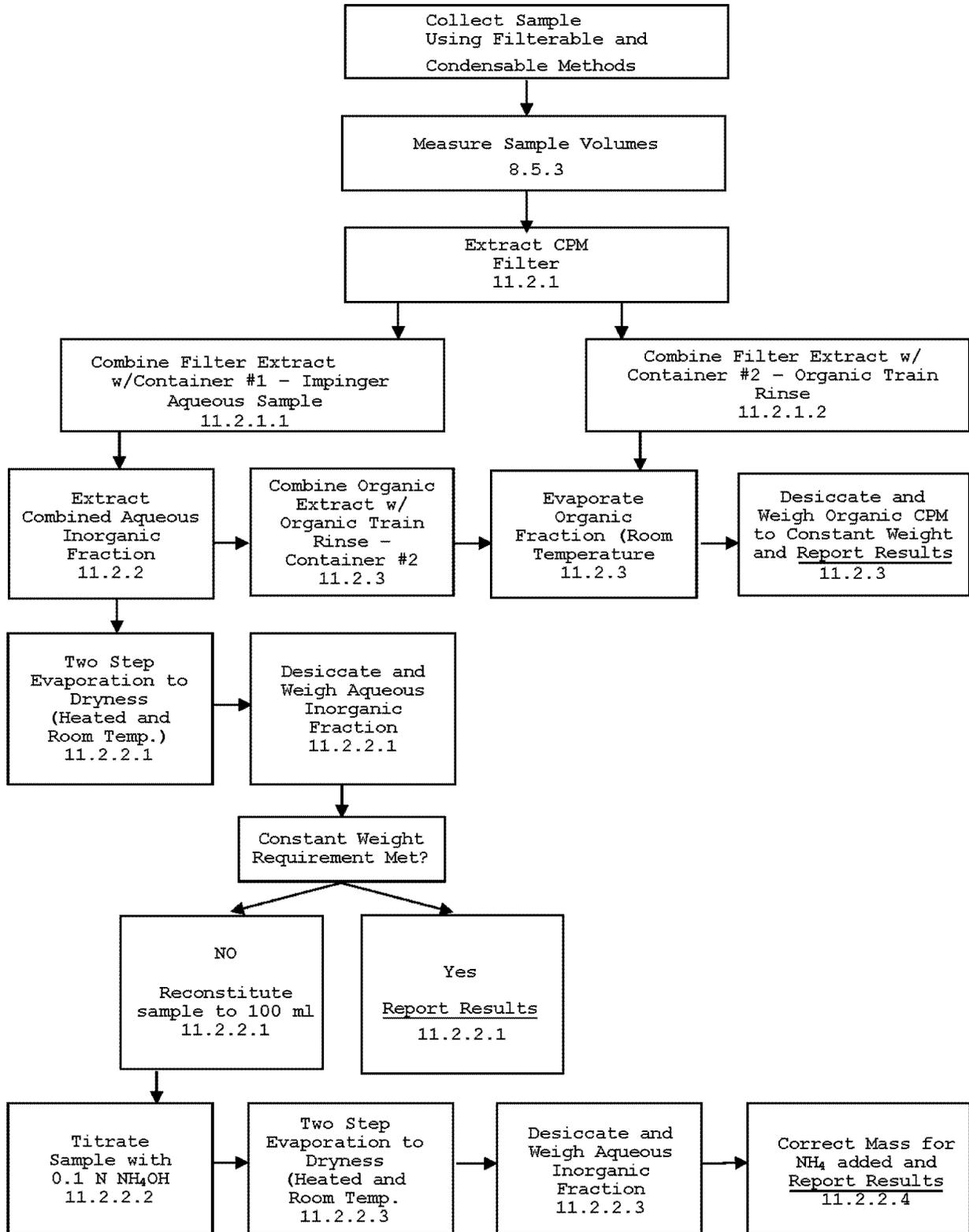


Figure 8. CPM Sample Processing Flow Chart