

**OTHER TEST METHOD 28** – April 15, 2009

**DRY IMPINGER METHOD FOR DETERMINING CONDENSABLE PARTICULATE  
EMISSIONS FROM STATIONARY SOURCES**

**1. Scope and Applicability**

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

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

1.3 Responsibility. You are responsible for obtaining the equipment and supplies you will need to use this method. You must also develop your own procedures for following this method

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and any additional procedures to ensure accurate sampling and analytical measurements.

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

- (a) Method 1 - Sample and Velocity Traverses for Stationary Sources.
- (b) Method 2 - Determination of Stack Gas Velocity and Volumetric Flow Rate (Type S Pitot Tube).
- (c) Method 3 - Gas Analysis for the Determination of Dry Molecular Weight.
- (d) Method 4 - Determination of Moisture Content in Stack Gases.
- (e) Method 5 - Determination of Particulate Matter Emissions from Stationary Sources.
- (f) Method 17 - Determination of Particulate Matter Emissions from Stationary Sources (in-stack filtration method).
- (g) Method 201A - Determination of  $PM_{10}$  Emissions from Stationary Sources (Constant Sampling Rate Procedure) or Other Test Method 27 (OTM 27) - Determination of  $PM_{10}$  and  $PM_{2.5}$  Emissions from Stationary Sources (Constant Sampling Rate Procedure)

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1.5 Additional Methods. You will need additional test methods to measure filterable particulate matter. You may use this method to collect CPM in conjunction with Method 5 or 17 of Appendices A-1 through A-3 and A-6 to Part 60, Method 201A of Appendix M to Part 51 or, OTM 27. The sample train operation and front end recovery and analysis are conducted according to the filterable particulate method you choose. This method addresses the equipment, preparation, and analysis necessary to measure only CPM.

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

1.7 Conditions. You must maintain isokinetic sampling conditions to meet the requirements of the filterable particulate method used in conjunction with this method. You must sample at the required number of sampling points specified in Method 5, 17, 201A or OTM 27. Also, if you are using this method as an alternative to a required performance test method, you must receive approval from the appropriate authorities prior to conducting the test.

## **2.0 Summary of Method**

2.1 Summary. The CPM is collected in dry impingers after

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

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

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

### 3.0 Definitions

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

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

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

3.4 Primary PM<sub>2.5</sub>. Primary PM<sub>2.5</sub> (also known as direct PM<sub>2.5</sub>, total PM<sub>2.5</sub>, PM<sub>2.5</sub>, or filterable PM<sub>2.5</sub>, and condensable PM, individually) means solid particles emitted directly from an air emissions source or activity, or gaseous emissions or liquid

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droplets from an air emissions source or activity that condense to form particulate matter at ambient temperatures. Direct PM<sub>2.5</sub> emissions include elemental carbon, directly emitted organic carbon, directly emitted sulfate, directly emitted nitrate, and other inorganic particles (including but not limited to crustal material, metals, and sea salt).

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

#### **4.0 Interferences** [Reserved]

#### **5.0 Safety**

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

#### **6.0 Equipment and Supplies**

The equipment used in the filterable particulate portion of the sampling train is described in Methods 5 and 17 of Appendix

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A-1 through A-3 and A-6 to Part 60, Method 201A in Appendix M to Part 51 and OTM 27. The equipment used in the CPM portion of the train is described in this section.

#### 6.1 Condensable Particulate Sampling Train Components.

The sampling train for this method is consistent with the sampling train for collecting filterable particulate using Method 5, 17, 201A or OTM 27 with the following exceptions or additions:

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

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

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stream.

6.1.3 Long Stem Impinger Insert. You will need a long stem modified Greenburg Smith impinger insert for the dropout impinger to perform the nitrogen purge of the sampling train.

6.2 Sample Recovery Equipment.

6.2.1 Condensable Particulate Matter Recovery.

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

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

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

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

6.3.1 Separatory Funnel. Glass, 1 liter.

6.3.2 Weighing Tins. 50 mL.

6.3.3 Glass Beakers. 300 to 500 mL.



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

6.3.5 Pipets. 5 mL.

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

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

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

## **7.0 Reagents and Standards**

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

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

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project data quality objectives (DQOs), filters are commercially available in 47 mm and larger sizes.

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

7.1.3 Water. Use deionized distilled ultra-filtered water (to conform to ASTM D1193-06, Type 1 water or equivalent) (incorporated by reference) to recover material caught in the impinger, if required. The Director of the Federal Register approves this incorporation by reference in accordance with 5 U.S.C. 552(a) and 1 CFR part 51. You may obtain a copy from American Society for Testing and Materials (ASTM), 100 Barr Harbor Drive, Post Office Box C700, West Conshohocken, PA 19428-2959. You may inspect a copy at the Office of Federal Register, 800 North Capitol Street, NW., Suite 700, Washington, DC.

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

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

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7.2 Sample Recovery and Analytical Reagents. You will need acetone,  $\text{MeCl}_2$ , anhydrous sodium sulfate, ammonia hydroxide ( $\text{NH}_4\text{OH}$ ), and deionized water for the sample recovery and analysis. Unless otherwise indicated, all reagents must conform to the specifications established by the Committee on Analytical Reagents of the American Chemical Society. If such specifications are not available, then use the best available grade. Find additional information on each of these items in the following paragraphs:

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

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

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

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

7.2.5 Ammonium Hydroxide. Use NIST traceable or

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equivalent (0.1 N)  $\text{NH}_4\text{OH}$ .

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

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

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

8.2 Preparations. You must clean glassware prior to field tests as described in Section 8.4, including baking glassware at  $300^\circ\text{C}$  for 6 hours prior to use. Cleaned, baked glassware is used at the start of each new source category tested. Analyze reagent blanks (water, acetone, and methylene chloride) before field tests to verify low blank concentrations. Follow the pretest preparation instructions in Section 8.1 of Method 5.

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

- (a) Determining the sampling site location and traverse points.
- (b) Calculating probe/cyclone blockage.
- (c) Verifying the absence of cyclonic flow.
- (d) Completing a preliminary velocity profile, and selecting a nozzle(s).

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### 8.3.1 Sampling Site Location and Traverse Point.

Determination. Follow the standard procedures in Method 1 of Appendix A-1 to Part 60 to select the appropriate sampling site. Then you must do all of the following:

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

8.3.1.2 Traverse points. Use the recommended maximum number of traverse points at any location, as found in Methods 5, 17, 201A, or OTM 27, whichever is applicable to your test requirements. You must prevent the disturbance and capture of any solids accumulated on the inner wall surfaces by maintaining a 1-inch distance from the stack wall ( $\frac{1}{2}$  inch for sampling locations less than 24 inches in diameter).

8.4 Sampling Train Preparation. A schematic of the sampling train used in this method is shown in Figure 1 of Section 18. All sampling train glassware must be cleaned prior to the test with soap and water, and rinsed using tap water, deionized water, acetone, and finally,  $\text{MeCl}_2$ . It is important to completely remove all silicone grease from areas that will be exposed to the  $\text{MeCl}_2$  rinse during sample recovery. After cleaning, you must bake glassware at  $300^\circ\text{C}$  for 6 hours prior to each source type sampled. Prior to each sampling run, the train glassware used to collect condensable particulate matter must be rinsed thoroughly with deionized, distilled ultra-filtered water

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that conforms to ASTM D1193-06, Type 1 or equivalent (incorporated by reference).

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

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

8.4.3 CPM Filter. Place a filter holder with a filter meeting the requirements in Section 6.1.2 following the modified Greenburg-Smith impinger. The connection between the CPM filter and the moisture trap impinger includes a thermocouple fitting that provides a leak-free seal between the thermocouple and the stack gas. (**Note:** A thermocouple well is not sufficient for this purpose because the Teflon<sup>®</sup> or steel encased thermocouple must be in contact with the sample gas).

8.4.4 Moisture Traps. You must use a modified Greenburg-

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

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

8.4.6 Leak-Check (Pretest). Use the procedures outlined in Method 5, 17, 201A, or OTM 27, as appropriate to leak check the entire sampling system. Specifically, perform the following procedures:

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

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

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

8.5 Sampling Train Operation. Operate the sampling train as described in the filterable particulate sampling method (i.e., Method 5, 17, 201A, or OTM 27) with the following additions or exceptions:

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

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



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8.5.3 Post-Test Nitrogen Purge. As soon as possible after the post-test leak-check, detach the probe, any cyclones, and in-stack or hot filters from the condenser and impinger train. Leave the ice in the second impinger box to prevent removal of moisture during the purge. If necessary, add more ice during the purge to maintain the gas temperature measured at the exit of the silica gel impinger below 20°C (68°F).

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

8.5.3.2 Replace the short stem impinger insert with a modified Greenberg Smith impinger insert. The impinger tip length must extend below the water level in the impinger catch. If insufficient water was collected, you must add a measured amount of degassed deionized, distilled ultra-filtered ASTM D1193-06, Type 1 or equivalent) (incorporated by reference) water until the impinger tip is at least 1 cm below the surface of the water. You must record the amount of water added to the dropout impinger (see Figure 4 of Section 18) to correct the moisture content of the effluent gas. (**Note:** Prior to use, water must be degassed using a nitrogen purge bubbled through the water for at least 15 minutes to remove dissolved oxygen).

8.5.3.3 With no flow of gas through the clean purge line and fittings, attach the line to a purged inline filter.

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Connect the filter outlet to the input of the impinger train (see Figure 2 of Section 18). To avoid over- or under-pressurizing the impinger array, slowly commence the nitrogen gas flow through the line while simultaneously opening the meter box pump valve(s). Adjust the pump bypass and nitrogen delivery rates to obtain the following conditions: (1) 20 liters/min or  $\Delta H\theta$ , and (2) a positive overflow rate through the rotameter of less than 2 liters/min. Condition (2) guarantees that the nitrogen delivery system is operating at greater than ambient pressure and prevents the possibility of passing ambient air (rather than nitrogen) through the impingers. During the purge, continue operation of the condenser recirculation pump, and heat or cool the water surrounding the first two impingers to maintain the gas temperature measured at the exit of the CPM filter below 30°C (85°F). Continue the purge under these conditions for 1 hour, checking the rotameter and  $\Delta H$  value(s) periodically. After 1 hour, simultaneously turn off the delivery and pumping systems.

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

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the field log notebook.

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

#### 8.5.4 Sample Recovery.

##### 8.5.4.1 Recovery of Filterable Particulate Matter.

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

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

8.5.4.3 CPM Container #2, Organic Rinses. Follow the water rinses of the probe extension, condenser, each impinger and all of the connecting glassware and front half of the CPM filter with an acetone rinse. Then repeat the entire procedure

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with two rinses of  $\text{MeCl}_2$ , and save both solvents in a separate glass container identified as CPM Container #2. Mark the liquid level on the jar.

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

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

8.5.4.6 CPM Container #5, Silica Gel Absorbent. You must weigh the contents of CPM Container #5 in the field or during sample analysis (see Section 11.2.4). If the silica gel has been weighed in the field to measure water content, then it can be discarded. Otherwise, transfer the silica gel to its original container and seal. A funnel may make it easier to pour the silica gel without spilling. A rubber policeman may be used as an aid in removing the silica gel from the impinger. It

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is not necessary to remove the small amount of silica gel dust particles that may adhere to the impinger wall and are difficult to remove. Since the gain in weight is to be used for moisture calculations, do not use any water or other liquids to transfer the silica gel.

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

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

8.5.4.9 CPM Container #8, Methylene Chloride Rinse Blank. Take 150 mL of the  $\text{MeCl}_2$  directly from the wash bottle you used, and place it in CPM Container #8, labeled Methylene Chloride Rinse Blank (see Section 11.2.7 for analysis). Mark the liquid level on the bottle.

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

## **9.0 Quality Control**

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

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

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

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

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requirements. The procedures must also conform to the plant health and safety requirements.

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

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

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

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

9.9 Field Reagent Blanks. You must run at least one field blank of water, acetone, and methylene chloride you use for field recovery. Running independent reagent field blanks will verify that low blank concentrations were maintained during field solvent use and demonstrate that reagents have not been

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contaminated during field tests.

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

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

9.12 Audit Samples. As of the publication date of this



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

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

## **10.0 Calibration and Standardization**

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

10.1 Thermocouple Calibration. You must calibrate the thermocouples using the procedures described in

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Section 10.1.4.1.2 of Method 2 of Appendix A-1 to Part 60. Calibrate each temperature sensor at a minimum of three points over the anticipated range of use against an NIST-traceable mercury-in-glass thermometer.

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

## **11.0 Analytical Procedures**

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

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

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

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

11.2.1.1 Extract the water soluble (aqueous or inorganic) CPM from the CPM filter as described in this section. Fold the CPM filter in quarters, and place it into a 50 mL extraction tube. Add sufficient deionized ultra-filtered water to cover the filter (e.g., 10 mL of water). Place the extractor tube into a sonication bath and extract the water soluble material

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for a minimum of 2 minutes. Combine the aqueous extract with the contents of Container #1. Repeat this extraction step twice for a total of three extractions.

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

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

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

11.2.2.2 Organic Fraction Weight Determination. Place the organic phase in a clean glass beaker. Evaporate the organic

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extract at room temperature (not to exceed 30°C (85°F)) and pressure in a laboratory hood to not less than 10 mL.

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

#### 11.2.2.3 Inorganic Fraction Weight Determination.

Transfer the aqueous fraction from the extraction to a clean 500-mL or smaller beaker. Evaporate to 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)). You must ensure that water and volatile acids have completely evaporated before neutralizing nonvolatile acids in the sample. Redissolve the residue in 100 mL of deionized distilled ultra-filtered water (ASTM D1193-06, Type 1 water or equivalent) (incorporated by reference).

#### 11.2.2.4 Use titration to neutralize acid in the sample and remove water of hydration. Calibrate the pH meter with the neutral and acid buffer solutions; then titrate the sample with

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0.1N  $\text{NH}_4\text{OH}$  to a pH of 7.0, as indicated by the pH meter. Record the volume of titrant used on the Condensable Particulate Matter Work Table (see Figure 5 of Section 18).

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

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

11.2.3 CPM Container #4, Cold Impinger Water. If the amount of water has not been determined in the field, note the level of liquid in the container, and confirm on the filterable particulate analytical data sheet whether leakage occurred during transport. If a noticeable amount of leakage has occurred, either void the sample or use methods, subject to the approval of the Administrator, to correct the final results. Measure the liquid in Container #4 either volumetrically to  $\pm 1$

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mL or gravimetrically to  $\pm 0.5$  g, and record the volume or weight on the filterable particulate analytical data sheet of the filterable particulate matter test method.

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

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

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

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



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

## 12.0 Calculations and Data Analysis

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

$\Delta H_{\theta}$	=	Pressure drop across orifice at flow rate of 0.75 SCFM at standard conditions, in. W.C. [ <b>Note:</b> specific to each orifice and meter box].
17.03	=	mg/milliequivalents for ammonium ion.
ACFM	=	Actual cubic feet per minute.
$C_{\text{cpm}}$	=	Concentration of the condensable particulate matter in the stack gas, dry basis, corrected to standard conditions, milligrams/dry standard cubic foot.
$m_{\text{c}}$	=	Mass of the $\text{NH}_4^+$ added to sample to form ammonium sulfate, mg.
$m_{\text{cpm}}$	=	Mass of the total condensable particulate matter, mg.
$m_{\text{fb}}$	=	Mass of field train total CPM blank, mg
$m_{\text{i}}$	=	Mass of inorganic CPM matter, mg.
$m_{\text{ib}}$	=	Mass of field train inorganic CPM blank, mg.
$m_{\text{o}}$	=	Mass of organic CPM, mg.
$m_{\text{ob}}$	=	Mass of organic field train blank, mg.
$m_{\text{r}}$	=	Mass of dried sample from inorganic fraction, mg.
N	=	Normality of ammonium hydroxide titrant.
$V_{\text{m(Std)}}$	=	Volume of gas sample measured by the dry gas meter, corrected to standard conditions, dry

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standard cubic meter (dscm) or dry standard cubic foot (dscf) as defined in Equation 5-1 of Method 5.

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

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

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

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

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

12.2.2 Mass of the Field Blank (mg). Per Section 9.10, the mass of the field blank,  $m_{fb}$ , 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).

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

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

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

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

(c) Flowchart of the process or processes tested.

(d) Sketch of the sampling location.

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

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

(g) Laboratory analytical sheets and case narratives.

(h) Pretest and post test reagent blank results.

(i) Sample calculations.

(j) Pretest and post-test calibration data.

(k) Chain of custody forms.

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

**13.0 Method Performance** [Reserved]

**14.0 Pollution Prevention** [Reserved]

**15.0 Waste Management**

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

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sampling equipment should be managed as RCRA organic waste.

#### **16.0 Alternative Procedures** [Reserved]

#### **17.0 References**

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

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

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

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

5. Texas Air Control Board, Laboratory Division.

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

6. Nothstein, Greg. Masters Thesis. University of Washington. Department of Environmental Health. Seattle, Washington.

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

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

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

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

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

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

18.0 Tables, Diagrams, Flowcharts, and Validation Data

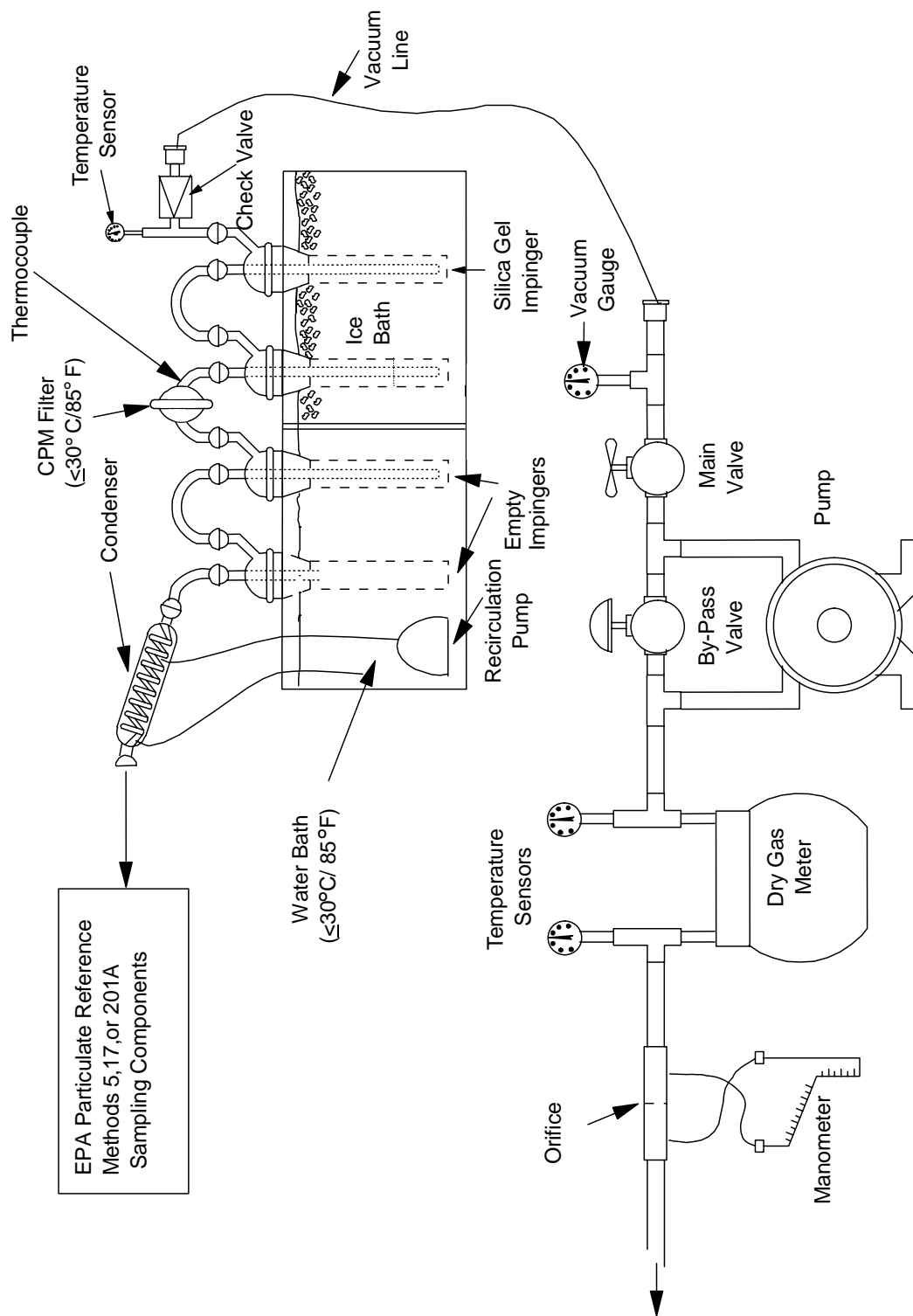


Figure 1. Schematic of Condensable Particulate Sampling Train

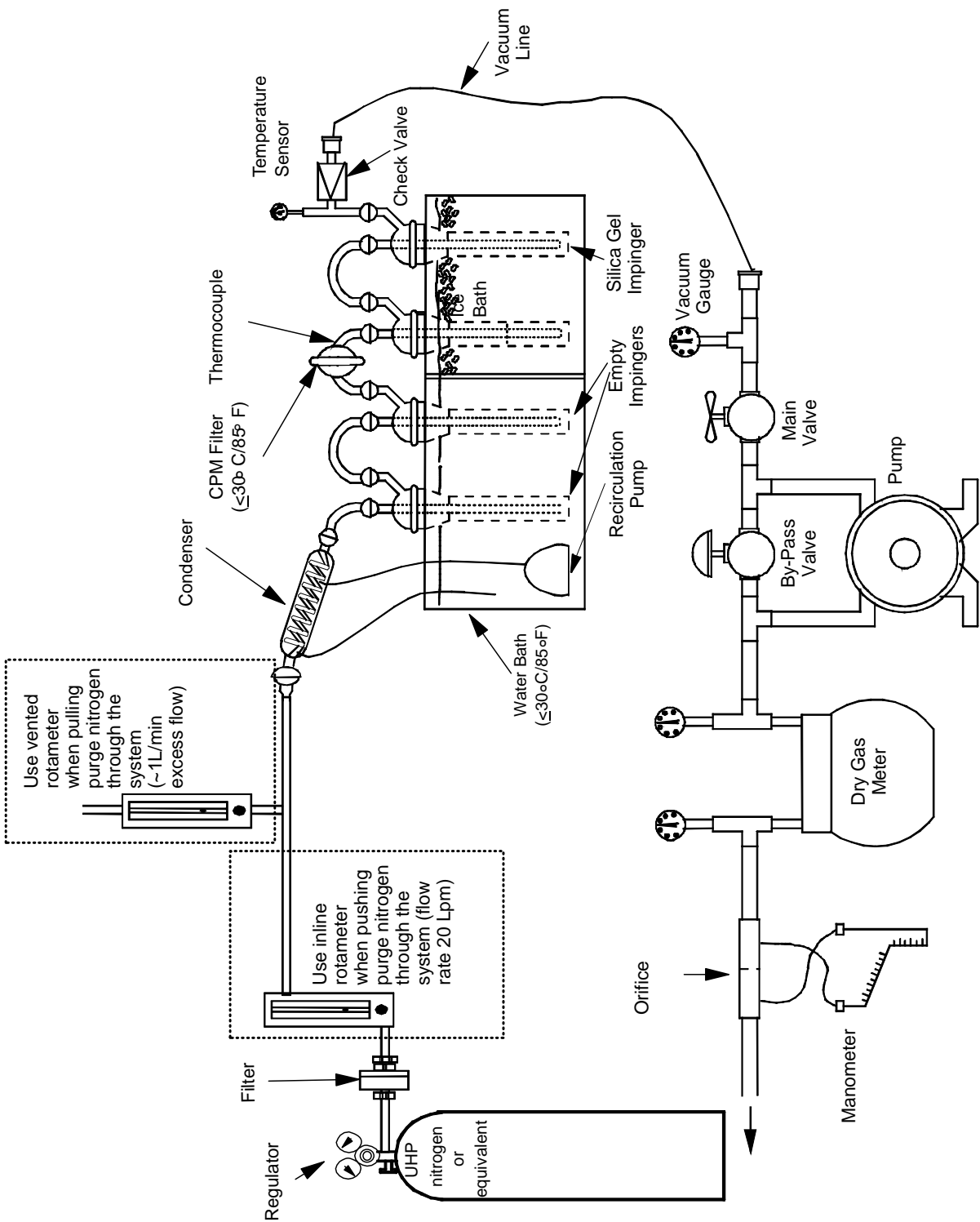


Figure 2. Nitrogen Purge Train Configuration



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<b>Field Train Blank Condensable Particulate Calculations</b>	
Plant	
Date	
Blank No.	
CPM Filter No.	
Water volume added to purge train ( $V_p$ )	ml
<b>Field Reagent Blank Mass</b>	
Water (Section 11.2.6)	mg
Acetone (Section 11.2.5)	mg
Methylene Chloride (Section 11.2.7)	mg
<b>Field Train Reagent Blank Mass</b>	
Mass of Organic CPM ( $m_{ob}$ ) (Section 11.2.2.2)	mg
Mass of Inorganic CPM ( $m_{ib}$ ) (Equation 3)	mg
Mass of the Field Train Blank (not to exceed 2.0 mg) (Equation 2)	mg

**Figure 3. Field Train Blank Condensable Particulate Calculations**

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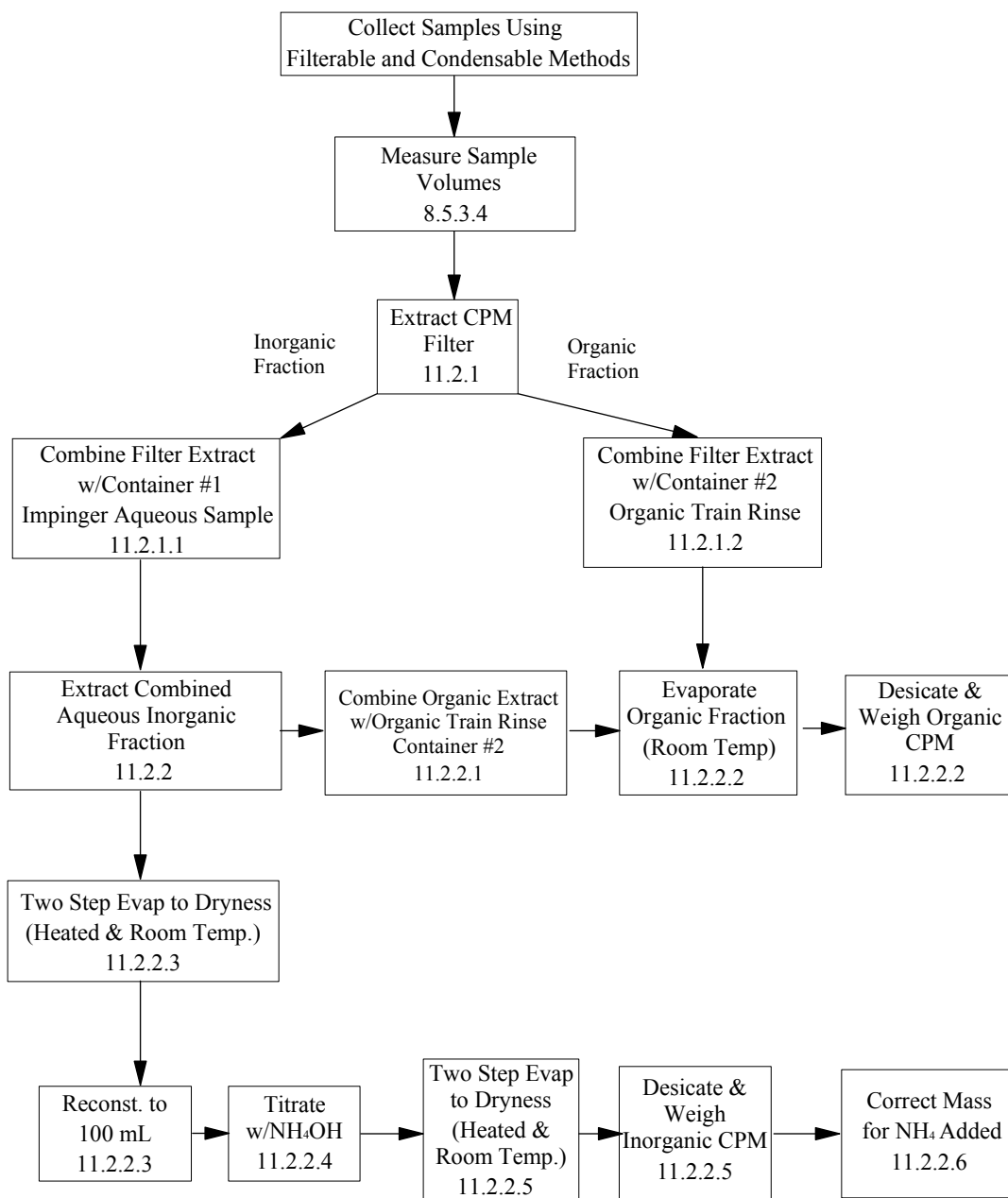
<b>Other Field Train Sample Condensable Particulate Data</b>	
Plant	
Date	
Run No.	
CPM Filter No.	
Water volume added to purge train [max 50 mL] ( $V_p$ )	ml
Date	
Run No.	
CPM Filter No.	
Water volume added to purge train [max 50 mL] ( $V_p$ )	ml
Date	
Run No.	
CPM Filter No.	
Water volume added to purge train [max 50 mL] ( $V_p$ )	ml

**Figure 4. Other Field Train Sample Condensable Particulate Data**

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<b>Calculations for Recovery of Condensable Particulate Matter (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 11.2.2.4)	_____	mL
Mass of $\text{NH}_4$ added ( $m_c$ ) (Equation 1)	_____	mg
<b>For CPM Blank Weights</b>		
Inorganic Train Field Blank Mass ( $m_{ib}$ ) (Section 9.9)	_____	mg
Organic Train Field blank Mass ( $m_{ob}$ ) (Section 9.9)	_____	mg
Mass of Train Field Blank ( $M_{fb}$ ) (max. 2 mg) (Equation 2)	_____	mg
<b>For CPM Train Weights</b>		
Mass of Organic CPM ( $m_o$ ) (Section 11.2.2.2)	_____	mg
Mass of Inorganic CPM ( $m_i$ ) (Equation 3)	_____	mg
Total CPM Mass ( $m_{cpm}$ ) (Equation 4)	_____	mg

**Figure 5. Condensable Particulate Matter Work Table**



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