DRAFT METHOD: DRY IMPINGER METHOD FOR DETERMINING CONDENSABLE PARTICULATE EMISSIONS FROM STATIONARY SOURCES

1. Scope and Application

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 may use this method only for stationary source emission measurement.

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

1.4 Results. To obtain reliable results, you must have a thorough knowledge of the following test methods which are found in Appendix A to Part 60:

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

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

(c) Method 3 - Gas Analysis for Carbon Dioxide, Oxygen, Excess Air, and Dry Molecular Weight.

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

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

1.5 Additional Methods. You will need additional test methods to measure and/or remove filterable particulate matter. Use this method to collect CPM in conjunction with Method 5 or 17 of Appendix A to Part 60 or Method 201A of Appendix M to Part 51 to collect filterable particulate matter. 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 temperature to cause water droplets sampled through the probe to become gaseous.

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

2.0 Summary of Method

2.1 Summary. The CPM is collected in impingers after filterable particulate material has been collected using Method 5, 17, or 201A. The organic, aqueous, and ambient filter fractions 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 includes the addition of a condenser, followed by a water dropout impinger immediately after the final in-stack or heated filter. One modified Greenburg Smith impinger and an ambient temperature filter follow 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, modified Greenburg Smith impinger and ambient filter portion of the sampling train as described in this method. The impinger contents are purged with nitrogen (N_2) immediately after sample collection to remove dissolved sulfur dioxide (SO_2)

gases from the impinger. The impinger solution is then extracted with methylene chloride ($MeCl_2$). The organic and aqueous fractions are dried and the residues weighed. The total of the aqueous, organic, and ambient filter fractions represents the CPM.

2.1.2 Dry Impinger and Additional Filter. The potential artifacts from SO_2 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 is placed between the second and third impinger.

3.0 Definitions

3.1 *PM10* means particulate matter less than 10 microns in aerodynamic diameter.

3.2 *PM2.5* means particulate matter less than 2.5 microns in aerodynamic diameter.

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 found in Methods 5 and 17 of Appendix A to Part 60 and Method 201A in Appendix M to Part 51. The equipment used in the CPM portion of the train is described in this section.

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

6.1.1 Condenser and Impingers. You must add the following components to the filterable particulate sampling train: A Method 23 condenser as described in Section 2.1.2 of Method 23 of Appendix A 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 Ambient Temperature Filter Holder. The modified Greenburg-Smith impinger is followed by a filter holder that is either glass, stainless steel (316 or equivalent), or Teflon-coated stainless steel. Commercial size filter holders are available depending on project requirements. Use a commercial filter holder capable of supporting 25 mm, 47 mm, 63 mm, and 110 mm diameter filters. Commercial size filter holders contain a Teflon O-ring, stainless steel, ceramic or Teflon filter support and a final Teflon O-ring. At the exit of the ambient temperature filter, install a Teflon-coated or stainless steel encased thermocouple that is in contact with the gas stream.

6.1.3 Long Stem Impinger Insert. You will need a long stem modified Greenburg Smith impinger insert 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. Inert tubing and fittings must be capable of delivering at least 30 liters/min of nitrogen gas to the impinger train from a standard gas cylinder (see Figure 2 of Section 18). Standard 0.95 cm (3/8-in.) plastic tubing and compression fittings in conjunction with an adjustable pressure regulator and needle valve may be used.

6.2.1.2 Rotameter. Rotameter must be capable of measuring gas flow up to 30 L/min. 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 30 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 to 350 mL.

6.3.3 Drying Equipment. Hot plate and oven with temperature control.

6.3.4 Pipets. 5 mL.

6.3.5 Burette. Glass, 0 to 100 mL in 1.0 mL graduations.

6.3.6 Analytical Balance. Analytical balance capable of weighing 0.0001 g. For extremely low emission sources, a balance capable of weighing 0.00001 g may be required.

7.0 Reagents and Standards

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

7.1.1 Filter. You must use a glass fiber, quartz, or Teflon filter that does not have an organic binder. The filter must also have an efficiency of at least 99.95 percent (<0.05 percent penetration) on 0.3 micron dioctyl phthalate smoke particles. Conduct the filter efficiency test in accordance with

ASTM Method D2986-71, 78, 95a (incorporated by reference). Alternatively, you may use test data from the supplier's quality control program. If the source you are sampling has SO_2 or sulfur trioxide (SO_3) emissions, you must use a filter that will not react with SO_2 or SO_3. Depending on your application and project data quality objectives (DQOs), filters are commercially available in 25-, 47-, 63-, and 110-mm sizes.

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

7.1.3 Water. Use deionized distilled water (to conform to ASTM D1193-77, 91 Type 3) to recover material caught in the impinger, if required. If you use water to recover this material, then you must run blanks 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.

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

7.2 Sample Recovery and Analytical Reagents. You will need acetone, $MeCl_2$, anhydrous sodium sulfate, ammonia hydroxide (NH_4OH), and deionized water for the sample analysis. Unless otherwise indicated, all reagents must conform to the specifications established by the Committee on Analytical Reagents of the American Chemical Society. If such specifications are not available, 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. In no case shall a blank value of greater than 1.6E-06 of the weight of $MeCl_2$ used in sample recovery and extraction be subtracted from the sample weight (i.e., the maximum blank correction is 0.2 mg per 100 mL of $MeCl_2$ used to recover samples).

7.2.3 Water. Use deionized distilled water (to conform to ASTM D1193-77, 91 Type 3) to recover material caught in the impinger, if required.

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

7.2.5 Ammonium Hydroxide. Use NIST traceable or equivalent

(0.1 N) NH_4OH.

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. 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) Determine the sampling site location and traverse points.

(b) Calculate probe/cyclone blockage.

(c) Verify the absence of cyclonic flow.

(d) Complete a preliminary velocity profile and select a nozzle(s).

8.3.1 Sampling Site Location and Traverse Point Determination. Follow the standard procedures in Method 1 of Appendix A to Part 60 to select the appropriate sampling site. Then 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. The recommended maximum number of traverse points at any location can be found in Methods 5, 17, or 201A, whichever is applicable to your test requirements. Prevent the disturbance and capture of any solids accumulated on the inner wall surfaces by maintaining a 1 inch distance from the stack wall (½ inch for sampling locations less than 24 inches in diameter).

8.4 Sampling Train Preparation. A schematic of the sampling train used in this method is shown in Figure 1 of Section 18. All sampling train glassware must be cleaned prior to the test with soap and water, and rinsed using tap water, deionized water, acetone, and finally, $MeCl_2$. It is important to completely remove all silicone grease from areas that will be exposed to the $MeCl_2$ during sample recovery.

8.4.1 Condenser. Add a condenser and a condensate dropout impinger without bubbler tube after the final in-stack or out-of-stack hot filter assembly. A Method 23 type stack gas condenser as described in Section 2.1.2 of Method 23 has been found adequate for this purpose. It must be capable of cooling the stack gas to less than 30°C (85°F).

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

8.4.3 Ambient Filter. A filter holder and tared filter meeting the requirements in Section 6.1.2 follow the ≤ 30°C (≤ 85°F) modified Greenburg-Smith impinger. The connection between the ambient filter and the moisture trap impinger includes a thermocouple fitting that provides a leak-free seal between the thermocouple and the stack gas. (Note: A thermocouple well is not sufficient for this purpose because the Teflon or steel encased thermocouple must be in contact with the sample gas.)

8.4.4 Moisture Traps. An empty modified Greenburg-Smith impinger or alternatives described in Method 5 followed by an impinger containing silica gel must be used to collect moisture that passes through the ambient 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. 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. Use the procedures outlined in Section 8.4 of Method 5 to leak check the entire sampling system. Specifically, perform the following procedures:

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

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.

8.4.6.3 Ambient Temperature Dropout and Impinger. Ambient temperature water is added to the first impinger section/box. Heat or cool the water to maintain ≤ 30°C (≤85°F) at the exit of the ambient temperature filter.

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

8.5.1 Ambient Filter Assembly. Record the ambient filter temperature readings at the beginning and end of each sample time increment and when sampling is halted. Maintain the ambient filter ≤ 30°C (≤85°F) during sample collection. Do not leak-check the sampling system during port changes.

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

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

8.5.3.1 If no water was collected before the ambient filter, 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 ASTM D1193-77, 91 Type 3 or equivalent 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 drop out impinger to correct the moisture content of the effluent gas. (Note: 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 it 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 ΔH@, and (2) a positive overflow rate through the rotameter of less than 2 L/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. Continue the purge under these conditions for 1 hour, checking the rotameter and ΔH 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.

8.5.3.5 If a balance is available in the field, weight 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.

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 method selected to measure filterable particulate matter (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 ambient filter into a clean sample bottle (glass or plastic). Rinse each impinger and the connecting glassware, including probe extension, condenser, and front half of the ambient 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 each impinger and all of the connecting glassware from the condenser to the ambient filter with an acetone rinse, then repeat the entire procedure with two rinses of $MeCl_2$ 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, Ambient Filter Sample. Use tweezers and/or clean disposable surgical gloves to remove the filter from the ambient filter holder. Place the filter in the petri dish identified as CPM Container #3.

8.5.4.5 CPM Container #4, Cold Impinger Water. If the water from the cold impinger has been weighed in the field, then it can be discarded. Otherwise, quantitatively transfer liquid from the cold impinger that follows the ambient filter into a clean sample bottle (glass or plastic). Mark the liquid level on the bottle. This container holds the remainder of the liquid water from the emission gases. The contents of CPM Container #4 are weighed during sample analysis (see Section 11.2.4).

8.5.4.6 CPM Container #5, Silica Gel Absorbent. Transfer the silica gel to its original container and seal. A funnel may make it easier to pour the silica gel without spilling. A rubber policeman may be used as an aid in removing the silica gel from the impinger. It is not necessary to remove the small amount of dust particles that may adhere to the impinger wall and are difficult to remove. Since the gain in weight is to be used for moisture calculations, do not use any water or other liquids to transfer the silica gel. If the silica gel has been weighed in the field to measure water content, then it can be discarded. Otherwise, the contents of CPM Container #5 are weighed during sample analysis (see Section 11.2.5).

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.

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.

8.5.4.9 CPM Container #8, Methylene Chloride Rinse Blank. Take 150 mL of the $MeCl_2$ directly from the wash bottle you used, and place it in CPM Container #8, labeled Methylene Chloride Rinse Blank.

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.

9.0 Quality Control

9.1 Daily Quality Checks. You must perform daily quality checks using data quality indicators that require review of (1) recording and transfer of raw data, (2) calculations, and (3) documentation of testing procedures.

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

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

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

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

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

9.7 Analytical Balance. Check the calibration of your analytical balance each day you weigh filters or 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 used for sample analysis. Analyze three samples of reagent water 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. You must run acetone blanks prior to field use to confirm low blank values. In no case shall a blank value of greater than 1.0 micrograms be subtracted from the sample weight.

9.9 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.

9.10 Audit Samples. As of the publication date of this test method, audit materials are not available. If audit materials become available, then 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 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 http://www.epa.gov/ttn/emc/email.html#qaqc. The request for the audit sample must be made at least 30 days prior to the scheduled compliance sample analysis.

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

10.2 Ammonium Hydroxide. The 0.1 N NH_4OH used for titrations in this method is made as follows: Add 7 mL of concentrated (14.8 M) NH_4OH to l liter of water. Standardize against standardized 0.1 N H2SO4 and calculate the exact normality using a procedure parallel to that described in Section 5.5 of Method 6 of Appendix A to 40 CFR part 60. Alternatively, purchase 0.1 N NH_4OH that has been standardized against a NIST reference material.

11.0 Analytical Procedures

11.1 Analytical data sheet. Record all data on the analytical data sheet. Obtain the data sheet from Figure 5-6 of Method 5. Alternatively, data may be recorded electronically using software applications such as the Electronic Reporting Tool (ERT) http://www.epa.gov/ttn/chief/ert/ert_tool.html.

11.2 Condensable Particulate Matter Analysis.

11.2.1 CPM Container #1, Aqueous Liquid Impinger Contents. You must analyze the water soluble CPM in Container 1 as described in this section. See the flow chart in Figure 4 of Section 18. First, note the level of liquid in each container, and confirm on the analysis sheet whether leakage occurred during transport. If a noticeable amount of leakage has occurred, either void the sample or use methods, subject to the approval of the Administrator, to correct the final results. Measure the liquid in this container either volumetrically to ± 1 mL or gravimetrically to ± 0.5 g. Place the contents of Container #1 into a separatory funnel. Add approximately 30 mL of $MeCl_2$ to the funnel, mix well, and drain off the lower organic phase. Repeat this procedure twice with 30 mL of $MeCl_2$ each time. Each time, leave a small amount of the organic/$MeCl_2$ 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.

11.2.1.1 CPM Container #2. Combine the contents of Container #2 with the organic extract from Container 1.

11.2.1.2 Organic Fraction Weight Determination. Place the organic phase in a clean glass beaker. Evaporate the organic extract at room temperature (not to exceed 30°C (85°F)) and pressure in a laboratory hood to not less than 10 mL. Quantitatively transfer the beaker contents to a 50-mL preweighed tin and evaporate to dryness at room temperature (not to exceed 30°C (85°F)) and pressure in a laboratory hood. Following evaporation, desiccate the organic fraction for 24 hours in a desiccator containing anhydrous calcium sulfate. Weigh and report the results to the nearest 0.1 mg.

11.2.1.3 Inorganic Fraction Weight Determination. Transfer the aqueous fraction from the extraction to a 250-mL beaker and evaporate to no less than 10 mL liquid on a hot plate or in the oven at 105 °C and then allow to air dry at ambient temperature. 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 ASTM D1193-77, 91 Type 3 water or equivalent. (Note: Do not evaporate the aqueous fraction containing CPM to dryness at an elevated temperature. Final evaporation must be performed a room temperature (not to exceed 30°C (85°F)) for valid results.)

11.2.1.4 Use titration to neutralize acid in the sample and remove water of hydration. Titrate the sample with 0.1N NH_4OH to a pH of 7.0, as indicated by a pH meter. Record the volume of titrant used.

11.2.1.5 Using a hot plate or an oven at 105°C, evaporate the aqueous phase to approximately 10 mL. Quantitatively transfer the beaker contents to a 50-mL preweighed tin and evaporate to dryness at room temperature (not to exceed 30°C (85°F)) and pressure in a laboratory hood. Following evaporation, desiccate the residue for 24 hours in a desiccator containing anhydrous calcium sulfate. Weigh and report the results to the nearest 0.1 mg.

11.2.1.6 Calculate the correction factor to subtract the $NH4^+$ retained in the sample using Equation 1 in Section 12.

11.2.2 Container #3, Ambient Filter Sample. Dry the filter recovered from the ambient temperature portion of the train until it reaches constant weight. The filter may be dried at room temperature (not to exceed 30°C (85°F)) in a laboratory hood until condensed water has evaporated. Following evaporation, desiccate the filter for 24 hours in a desiccator containing anhydrous indication calcium sulfate. Weigh and report the results to the nearest 0.1 mg.

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 analysis sheet whether leakage occurred during transport. If a noticeable amount of leakage has occurred, either void the sample or use methods, subject to the approval of the Administrator, to correct the final results. Measure the liquid in this container either volumetrically to ± 1 mL or gravimetrically to ± 0.5 g.

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.

11.2.5 Container #6, Acetone Rinse Blank. Use 100 mL of acetone from the blank container for this analysis. If insufficient liquid is available or if the acetone has been lost due to container breakage, either void the sample or use methods, subject to the approval of the Administrator, to correct the final results. Transfer 100 mL of the acetone to a clean 250-mL beaker. Evaporate the acetone at room temperature (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 and report the results to the nearest 0.1 mg.

11.2.6 Water Rinse 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 and report the results to the nearest 0.1 mg.

11.2.7 Methylene Chloride Field Blank, Container #8. Use 100 mL of $MeCl_2$ from the blank container for this analysis. Transfer 100 mL of the $MeCl_2$ to a clean 250-mL beaker. Evaporate the methylene chloride at room temperature (not to exceed 30°C (85°F)) and pressure in a laboratory hood to approximately 10 mL. Quantitatively transfer the beaker contents to a 50-mL preweighed tin and evaporate to dryness at room temperature (not to exceed 30°C (85°F)) and pressure in a laboratory hood. Following evaporation, desiccate the residue for 24 hours in a desiccator containing anhydrous calcium sulfate. Weigh and report the results to the nearest 0.1 mg.

12.0 Calculations and Data Analysis

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

ACFM = Actual cubic feet per minute.

C_{cpm} = Concentration of the condensable particulate matter in the stack gas, dry basis, corrected to standard conditions, g/dscf.

m_c = Mass of the NH_4^+ added to sample to form ammonium sulfate, mg.

m_{cpm} = Mass of the condensable particulate matter in the stack gas, dry basis, corrected to standard conditions, g/dscf.

m_f = Mass of the condensable particulate found on the ambient filter.

m_{fb} = Sum of the mass of the water, $MeCl_2$ and acetone field blanks, after evaporation, mg.

m_i = Mass of inorganic CPM matter, mg.

m_o = Mass of organic CPM, mg.

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

M_c =

N =

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

V_t = Volume of NH_4OH titrant, mL.

18.03 = mg/meq. for ammonium ion.

12.2 Calculations. Perform all of the calculations found in Table 1 of Section 18. Table 1 of Section 18 also provides instructions and references for the calculations.

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

12.2.1 Correction for Ammonia added during titration of 100 mL aqueous CPM sample.

$$M_c = 18.03\, V_t\, N \qquad \text{Equation 1}$$

12.2.2 Mass of Inorganic CPM (mg).

$$m_i = m_r - m_c \qquad \text{Equation 2}$$

12.2.3 Mass of organic CPM (mg).

$$m_{cpm} = m_i + m_o + m_f - m_{fb}$$

Equation 3

12.2.4 Concentration of CPM (mg).

$$C_{cpm} = \frac{m_{cpm}}{V_{ms}}$$

Equation 4

12.3 Analyses. Analyze the organic and inorganic condensable particulate mass by weighing the residual mass to a sensitivity of 0.0001 g.

12.4 Emission 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.

(g) Laboratory analytical sheets and case narratives.

(h) Sample calculations.

(i) Pretest and post-test calibration data.

(j) Chain of custody forms.

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

13.0 Method Performance [Reserved]

14.0 Pollution Prevention [Reserved]

15.0 Waste Management

Solvent and water are evaporated in a laboratory hood during analysis. No liquid waste is generated in the performance of this method. Organic solvents used to clean sampling equipment should be managed as RCRA organic waste.

16.0 Alternative Procedures [Reserved]

17.0 References

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

2. Richards, J.R. “Test protocol: PCA PM10/PM2.5 Emission Factor Chemical Characterization Testing,” PCA R&D Serial No. 2081, Portland Cement Association, 1996.

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

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

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

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

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

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

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

10. U.S. Environmental Protection Agency, "Laboratory Evaluation of Method 202 to Determine Fate of SO2 in Impinger Water," EPA Contract No. 68-D-02-061, Work Assignment 3-14,

September 30, 2005.

11. [Placeholder: ERG report on evaluation of improved dry impinger modifications to Method 202.]

12. [Placeholder: EPRI report on evaluation of improved dry impinger modifications to Method 202 at conditions representative of electric power generating facilities.]

18.0 Tables, Diagrams, Flowcharts, and Validation Data

Table 1. Calculations for Recovery of Condensable Particulate Matter (CPM)

Calculations	Instructions and References
Correction for ammonia added to neutralize aqueous samples.	Use Equation 1.
Mass of Inorganic CPM	Use Equation 2.
Mass of Organic CPM	Use Equation 3.
Concentration of CPM	Use Equation 4.

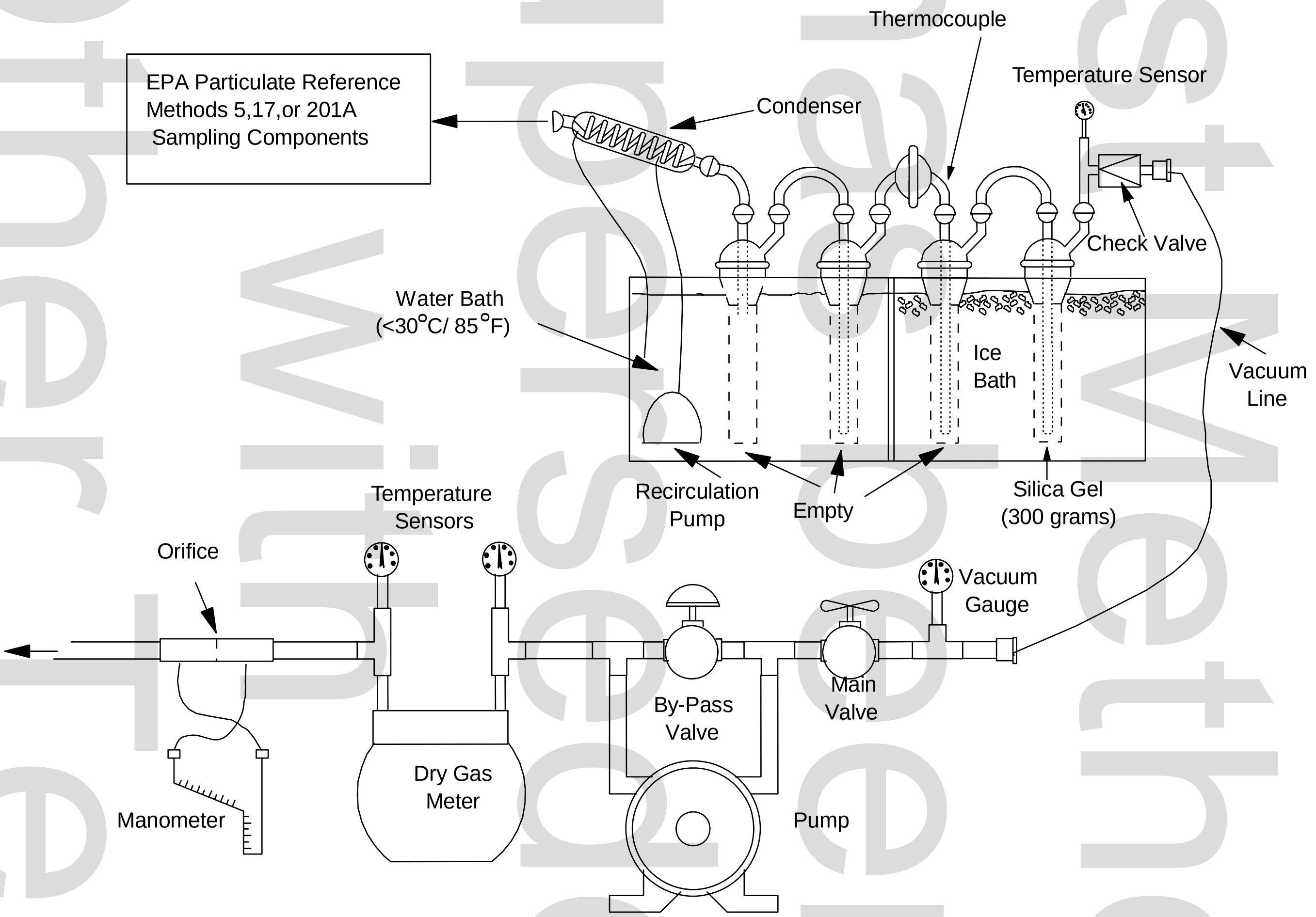

Figure 1. Schematic of Condensable Particulate Sampling Train

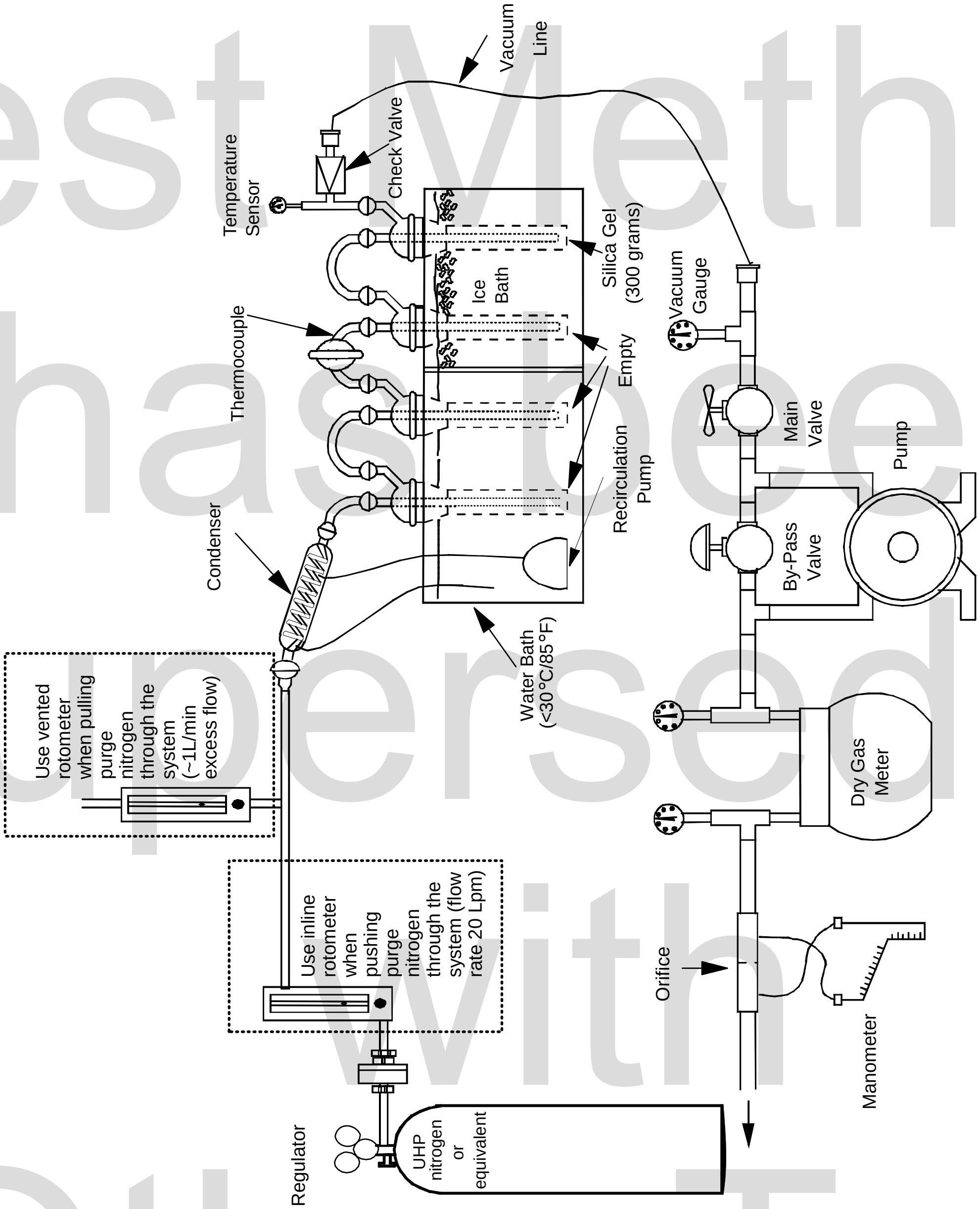

Figure 2. Nitrogen Purge Train Configuration

Sample Preparation (CPM Container No. 1, 2, and 3)

Amount of liquid lost during transport________________ mL
Final volume ____________________________________ mL
pH of sample prior to analysis_______________________
Addition of NH_4OH required? ______________________
Sample extracted 3X with 30 mL methylene chloride? ______________

For Titration of Sulfate

Normality of NH_4OH ________________________________ N
Volume of sample titrated ____________________________ mL
Volume of titrant ___________________________________ mL
Mass of NH_4 added (from equation 1) _________________________________ mg

Sample Analysis

Condensable Particulate Work Table			
Container Number	**Weight of Condensable Particulate (mg)**		
	Final Weight (corrected for NH_4)	**Tare Weight**	**Weight Gain**
a) Inorganic after extraction			
b) Organic extract			
c) Ambient filter weight			
d) Field blank (average of 3)			
Total CPM		**a+b+c-d**	

Figure 3. Analytical Data Sheet

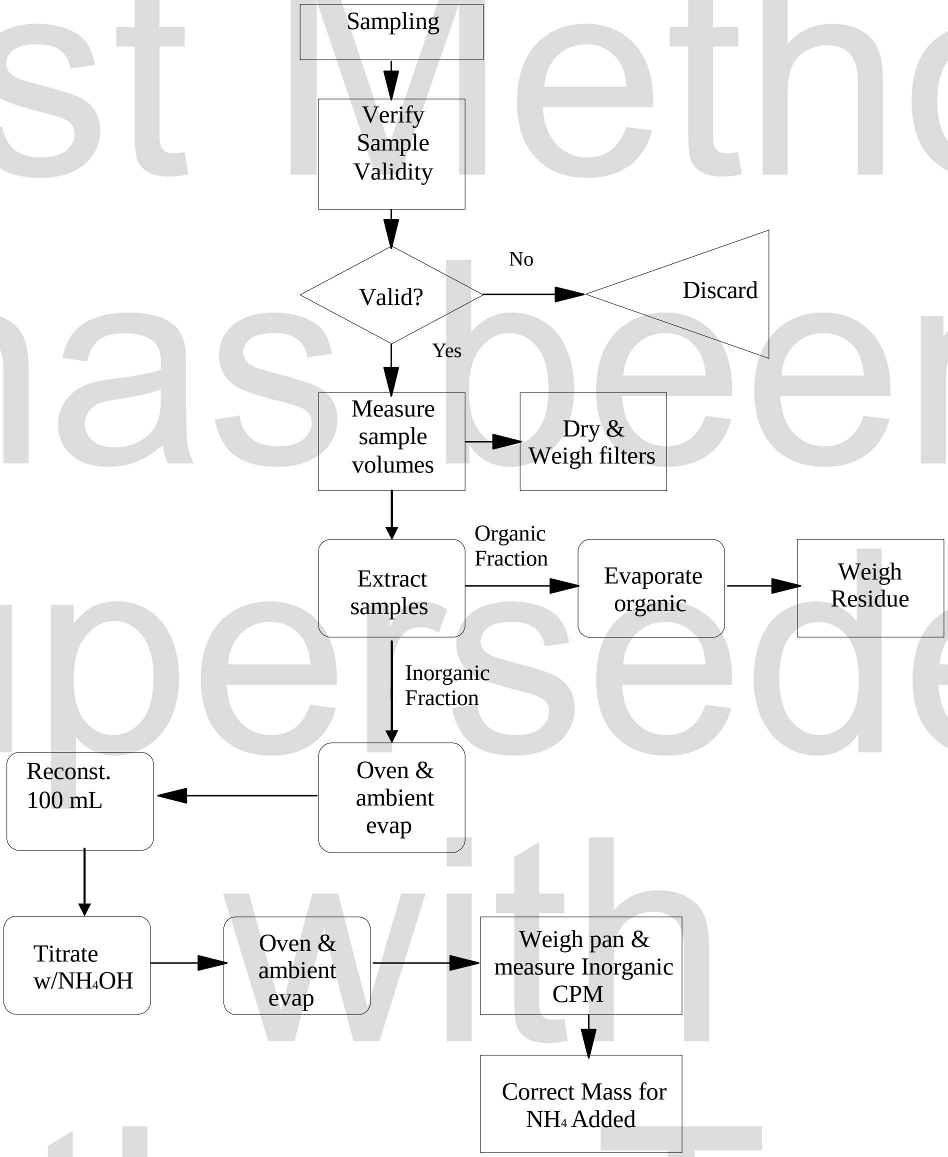

Figure 4. Flow Chart