

I. INTRODUCTION

Midland Cogeneration Venture (MCV) contracted Spectrum Systems, Inc., to perform particulate emissions testing on Units 16-21 (EUBOILER 1 – EUBOILER 6) located at 100 Progress Place in Midland, Michigan.

Testing was performed to satisfy the requirements contained in the Michigan Department of Environmental Quality (MDEQ) Renewable Operating Permit (ROP) No. MI-ROP-B6527-2014. Particulate emissions were quantified using U.S. Environmental Protection Agency (EPA), Title 40 of the Code of Federal Regulations, Part 60 (40CFR60), Appendix A, Reference Methods 1, 2, 3, 4, 5 and RM 202.

The testing was conducted October 27-30, 2014 by James Garrett, Rick Artybridge, Wayne Smith, Sean Myrick and Gary Martin of Spectrum Systems, Inc. (SSI). Ms. Barbara Vanderkelen of MCV coordinated the test events and Mr. Jim Lazzaro collected the process data. A representative from MDEQ was on site to observe the testing.

| Affiliation | Address | Contact Info |
|-------------------------|--|--|
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Contact Information



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II. SUMMARY OF RESULTS

Particulate emissions testing results are summarized in the tables below Midland Cogeneration Venture Boilers 16-21. Included are figures for Emission rate in lb/mmbtu and the Allowable Emission rate in lb/mmbtu. Specific supporting data is placed in the Appendices of this report.

| Run No. | Load >90% | Emission Rate (lb/mmbtu) | Allowable (lb/mmbtu) |
|---------|--------------|-----------------------------|-------------------------|
| Run 1 | >90% | .0017 | .0075 |
| Run 2 | >90% | .0026 | .0075 |
| Run 3 | . >90% | .0015 | .0075 |
| Avg. | >90% | .0019 | .0075 |

Unit 16 October 27-28, 2014

Unit 17 October 27-28, 2014

| Run No. | Load >90% | Emission Rate (lb/mmbtu) | Allowable (lb/mmbtu) |
|---------|--------------|-----------------------------|-------------------------|
| Run 1 | >90% | .0018 | .0075 |
| Run 2 | >90% | .0054 | .0075 |
| Run 3 | >90% | .0024 | .0075 |
| Avg. | >90% | .0032 | .0075 |



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Unit 18 October 28-29, 2014

| Run No. | Load >90% | Emission Rate (lb/mmbtu) | Allowable (lb/mmbtu) |
|---------|--------------|-----------------------------|-------------------------|
| Run 1 | >90% | .0019 | .0075 |
| Run 2 | >90% | .0020 | .0075 |
| Run 3 | >90% | .0015 | .0075 |
| Avg. | >90% | .0018 | .0075 |

Unit 19 October 28-29, 2014

| Run No. | Load >90% | Emission Rate (lb/mmbtu) | Allowable (lb/mmbtu) |
|---------|--------------|-----------------------------|-------------------------|
| Run 1 | >90% | .0017 | .0075 |
| Run 2 | >90% | .0022 | .0075 |
| Run 3 | >90% | .0039 | .0075 |
| Avg. | >90% | .0026 | .0075 |



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Unit 20 October 29-30, 2014

| Run No. | Load | Emission Rate (lb/mmbtu) | Allowable (lb/mmbtu) |
|---------|------|-----------------------------|-------------------------|
| Run 1 | >90% | .0021 | .0075 |
| Run 2 | >90% | .0023 | .0075 |
| Run 3 | >90% | .0017 | .0075 |
| Avg. | >90% | .0020 | .0075 |

Unit 21 October 29-30, 2014

| Run No. | Load >90% | Emission Rate (lb/mmbtu) | Allowable (lb/mmbtu) |
|---------|--------------|-----------------------------|-------------------------|
| Run 1 | >90% | .0016 | .0075 |
| Run 2 | >90% | .0021 | .0075 |
| Run 3 | >90% | .0019 | .0075 |
| Avg. | >90% | .0018 | .0075 |



III. Test Methods and Sampling Procedures

The sampling train that was used for execution of the particulate testing was manufactured by APEX Instruments, in Apex, North Carolina. The train meets all specifications as outlined by the U.S. Environmental Protection Agency.

As specified in the various test methods, all necessary calibrations on the dry gas meter, sampling tip, pitot tubes, thermocouples, and aneroid barometer were performed. The results of the calibrations are included in the Appendix.

The number of sample points and the sampling location were determined according to EPA Method 1. The test location and nearest disturbances (upstream and downstream) from the test site can be found in detail on the preliminary sample traverse data sheet in the Appendix.

EPA Method 2 was used to determine the stack gas velocity and volumetric flow rate. An S- type pitot tube was used in conjunction with an inclined oil gauge manometer. After each test repetition, a leak check was performed on the pitot manometer assembly and indicated no leak for 15 seconds.

Dry molecular weight was determined according to EPA Method 3A during each repetition by analysis of integrated gas samples. The results of these analyses were reported on the field data sheets and in the Appendix.

US EPA Method 5, Determination of Particulate Emissions from Stationary Sources, was used to determine particulate concentrations. The sampling train consisted of a calibrated stainless steel nozzle, probe with heated glass liner, glass fiber filter and filter holder in which the filter outlet temperature could be monitored, four impinges 120' umbilical cord, pump, dry gas meter, and orifice.

Before each test, the probe and filter holder assemblies are secured in the filter box.

After the probe and filter heaters warmed up to the specified operating temperature, the probe was inserted into the stack at the first sample traverse point. The stack gas parameters were recorded on the field data sheet, the pump turned on, and the sampling rate set at the isokinetic rate. At the end of the sampling period for the first point, the probe was moved to the next traverse point, and the sampling rate adjusted to maintain the isokinetic rate for the measured gas parameters at that point. This procedure was followed until all of the traverse points had been sampled. At each point, the following information was measured and recorded on the field data sheet: dry gas meter volume, stack gas velocity pressure differential, orifice meter pressure differential, stack gas temperature, sample train filtration temperature, filter outlet temperature, impinger train exit temperature, dry gas meter inlet and outlet temperature, and sample train system vacuum. After all of the points had been sampled, the pump was turned off and the probe was removed from the stack.

Immediately following each repetition, a leak check was performed on the sample train and indicated less than the allowable 0.02 CFM. The train was then disassembled. The filter holder assembly was first removed and recovered for later weighing. Next the probe and nozzle was flushed with acetone and the catch stored. Next, the moisture catch from impingers one, two, and three were measured with a graduated cylinder. The silica gel from the fourth impinger was removed and weighed. The impingers were then reloaded as previously described.

After the test was completed, the filter and catches were returned to the laboratory. The filter containers were desiccated for a minimum of 24 hours, then weighed to a constant weight. The contents of each probe wash bottle were transferred to tared beakers. The containers were then rinsed with either acetone or water to ensure that all of the particulate matter was recovered. Each wash was evaporated at ambient temperature and pressure in the SSI laboratory. Each beaker, with residue, was desiccated and weighed to the nearest 0.1 milligram.

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

1.0 Scope and Applicability

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

1.2 Applicability. This method addresses the equipment, preparation, and analysis necessary to measure only CPM. You can use this method only for stationary source emission measurements. You can use this method to measure CPM from stationary source emissions after filterable particulate matter (PM) has been removed. CPM is measured in the emissions after removal from the stack and after passing through a filter.

- (a) If the gas filtration temperature exceeds 30°C (85°F) and you must measure both the filterable and condensable (material that condenses after passing through a filter) components of total primary (direct) PM emissions to the atmosphere, then you must combine the procedures in this method with the procedures in Method 201A of appendix M to this part for measuring filterable PM. However, if the gas filtration temperature never exceeds 30°C (85°F), then use of this method is not required to measure total primary PM.
- (b) If Method 17 of appendix A-6 to part 60 is used in conjunction with this method and constant weight requirements for the in-stack filter cannot be met, the Method 17 filter and sampling nozzle rinse must be treated as described in Sections 8.5.4.4 and 11.2.1 of this method.



(See Section 3.0 for a definition of constant weight.) Extracts resulting from the use of this procedure must be filtered to remove filter fragments before the filter is processed and weighed.

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

1.4 Additional Methods. To obtain reliable results, you should have a thorough knowledge of the following test methods that are found in appendices A-1 through A-3 and A- 6 to part 60, and in appendix M to this part:

- (a) Method 1 Sample and velocity traverses for stationary sources.
- (b) Method 2 Determination of stack gas velocity and volumetric flow rate (Type S pitot tube).
- (c) Method 3 Gas analysis for the determination of dry molecular weight.
- (d) Method 4 Determination of moisture content in stack gases.
- (e) Method 5 Determination of particulate matter emissions from stationary sources.
- (f) Method 17 Determination of particulate matter emissions from stationary sources (instack filtration method).
- (g) Method 201A Determination of PM10 and PM2.5 emissions from stationary sources (Constant sampling rate procedure).
- (h) You will need additional test methods to measure filterable PM. You may use Method 5 (including Method 5A, 5D and 5I but not 5B, 5E, 5F, 5G, or 5H) of appendix A-3 to part 60, or Method 17 of appendix A-6 to part 60, or Method 201A of appendix M to this part to collect filterable PM from stationary sources with temperatures above 30°C (85°F) in conjunction with this method. However, if the gas filtration temperature never

exceeds 30°C (85°F), then use of this method is not required to measure total primary PM.

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

1.6 Conditions. You must maintain isokinetic sampling conditions to meet the requirements of the filterable PM test method used in conjunction with this method. You must sample at the required number of sampling points specified in Method 5 of appendix A-3 to part 60, Method 17 of appendix A-6 to part 60, or Method 201A of appendix M to this part. Also, if you are using this method as an alternative to a required performance test method, you must receive approval from the regulatory authority that established the requirement to use this test method prior to conducting the test.

2.0 Summary of Method

2.1 Summary. The CPM is collected in dry impingers after filterable PM has been collected on a filter maintained as specified in either Method 5 of appendix A-3 to part 60, Method 17 of appendix A-6 to part 60, or Method 201A of appendix M to this part. The organic and aqueous fractions of the impingers and an out-of-stack CPM filter are then taken to dryness and weighed. The total of the impinger fractions and the CPM filter represents the CPM. Compared to the version of Method 202 that was promulgated on December 17, 1991, this method eliminates the use of water as the collection media in impingers and includes the addition of a condenser followed by a water dropout impinger immediately after the final in-stack or heated filter. This method also includes the addition of one modified Greenburg Smith impinger (backup impinger)

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and a CPM filter following the water dropout impinger. Figure 1 of Section 18 presents the schematic of the sampling train configured with these changes.

2.1.1 Condensable PM. 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 immediately after sample collection to remove dissolved sulfur dioxide (SO₂) gases from the impinger. The CPM filter is extracted with water and hexane. The impinger solution is then extracted with hexane. The organic and aqueous fractions are dried and the residues are weighed. The total of the aqueous and organic fractions represents the CPM.

2.1.2 Dry Impinger and Additional Filter. The potential artifacts from SO₂ are reduced using a condenser and water 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 <u>Condensable PM (CPM)</u> means material that is vapor phase at stack conditions, but condenses and/or reacts upon cooling and dilution in the ambient air to form solid or liquid PM immediately after discharge from the stack. Note that all condensable PM is assumed to be in the PM2.5 size fraction.

3.2 <u>Constant weight</u> means a difference of no more than 0.5 mg or one percent of total weight less tare weight, whichever is greater, between two consecutive weighings, with no less than six hours of desiccation time between weighings.

3.3 <u>Field Train Proof Blank</u>. A field train proof blank is recovered on site from a clean, fullyassembled sampling train prior to conducting the first emissions test.

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Midland Cogeneration Venture Units 16-21 3.4 <u>Filterable PM</u> means particles that are emitted directly by a source as a solid or liquid at stack or release conditions and captured on the filter of a stack test train.

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

3.6 <u>Primary PM2.5</u> (also known as direct PM2.5, total PM2.5, PM2.5, or combined filterable PM2.5 and condensable PM) means PM with an aerodynamic diameter less than or equal to 2.5 micrometers. These solid particles are emitted directly from an air emissions source or activity, or are the gaseous emissions or liquid droplets from an air emissions source or activity that condense to form PM at ambient temperatures. Direct PM2.5 emissions include elemental carbon, directly emitted organic carbon, directly emitted sulfate, directly emitted nitrate, and other inorganic particles (including but not limited to crustal material, metals, and sea salt).

3.7 <u>Primary PM10</u> (also known as direct PM10, total PM10, PM10, or the combination of filterable PM10 and condensable PM) means PM with an aerodynamic diameter equal to or less than 10 micrometers.

4.0 Interferences

[Reserved]

5.0 Safety

Disclaimer. Because the performance of this method may require the use of hazardous materials, ' operations, and equipment, you should develop a health and safety plan to ensure the safety of your employees who are on site conducting the particulate emission test. Your plan should conform with all applicable Occupational Safety and Health Administration, Mine Safety and Health Administration, and Department of Transportation regulatory requirements. Because of the unique situations at some facilities and because some facilities may have more stringent

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requirements than is required by State or federal laws, you may have to develop procedures to conform to the plant health and safety requirements.

6.0 Equipment and Supplies

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

6.1 Condensable Particulate Sampling Train Components. The sampling train for this method is used in addition to filterable particulate collection using Method 5 of appendix A-3 to part 60, Method 17 of appendix A-6 to part 60, or Method 201A of appendix M to this part. This method includes the following exceptions or additions:

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

6.1.2 Condenser and Impingers. You must add the following components to the filterable particulate sampling train: A Method 23 type condenser as described in Section 2.1.2 of Method 23 of appendix A-8 to part 60, followed by a water dropout impinger or flask, followed by a modified Greenburg-Smith impinger (backup impinger) with an open tube tip as described in Section 6.1.1.8 of Method 5 of appendix A-3 to part 60,

6.1.3 CPM Filter Holder. The modified Greenburg- Smith impinger is followed by a filter holder that is either glass, stainless steel (316 or equivalent), or fluoropolymer-coated stainless steel. Commercial size filter holders are available depending on project requirements. Use a commercial filter holder capable of supporting 47 mm or greater diameter filters. Commercial



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size filter holders contain a fluoropolymer O-ring, stainless steel, ceramic or fluoropolymer filter support and a final fluoropolymer O-ring. A filter that meets the requirements specified in Section 7.1.1 may be placed behind the CPM filter to reduce the pressure drop across the CPM filter. This support filter is not part of the PM sample and is not recovered with the CPM filter. At the exit of the CPM filter, install a fluoropolymer-coated or stainless steel encased thermocouple that is in contact with the gas stream.

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

6.2 Sample Recovery Equipment.

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

- (a) Nitrogen purge line. You must use inert tubing and fittings capable of delivering at least 14 liters/min of nitrogen gas to the impinger train from a standard gas cylinder (see Figures 2 and 3 of Section 18). You may use standard 0.6 centimeters (1/4 inch) tubing and compression fittings in conjunction with an adjustable pressure regulator and needle valve.
- (b) Rotameter. You must use a rotameter capable of measuring gas flow up to 20 L/min. The rotameter must be accurate to five percent of full scale.
- (c) Nitrogen gas purging system. Compressed ultrapure nitrogen, regulator, and filter must be capable of providing at least 14 L/min purge gas for one hour through the sampling train.
- (d) Amber glass bottles (500 ml).

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- 6.2.2 Analysis Equipment. The following equipment is necessary for CPM sample analysis:
 - (a) Separatory Funnel. Glass, 1 liter.
 - (b) Weighing Tins. 50 ml. Glass evaporation vials, fluoropolymer beaker liners, or aluminum weighing tins can be used.
 - (c) Glass Beakers. 300 to 500 ml.
 - (d) Drying Equipment. A desiccator containing anhydrous calcium sulfate that is maintained below 10 percent relative humidity, and a hot plate or oven equipped with temperature control.
 - (e) Glass Pipets. 5 ml.
 - (f) Burette. Glass, 0 to 100 ml in 0.1 ml graduations.
 - (g) Analytical Balance. Analytical balance capable of weighing at least 0.0001 g (0.1 mg).
 - (h) pH Meter or Colormetric pH Indicator. The pH meter or colormetric pH indicator (e.g., phenolphthalein) must be capable of determining the acidity of liquid within 0.1 pH units.
 - Sonication Device. The device must have a minimum sonication frequency of 20 kHz and be approximately four to six inches deep to accommodate the sample extractor tube.
 - (j) Leak-Proof Sample Containers. Containers used for sample and blank recovery must not contribute more than 0.05 mg of residual mass to the CPM measurements.
 - (k) Wash bottles. Any container material is acceptable, but wash bottles used for sample and blank recovery must not contribute more than 0.1 mg of residual mass to the CPM measurements.



7.0 Reagents and Standards

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

7.1.1 CPM Filter. You must use a nonreactive, nondisintegrating polymer filter that does not have an organic binder and does not contribute more than 0.5 mg of residual mass to the CPM measurements. The CPM filter must also have an efficiency of at least 99.95 percent (less than 0.05 percent penetration) on 0.3 micrometer dioctyl phthalate particles. You may use test data from the supplier's quality control program to document the CPM filter efficiency.

7.1.2 Silica Gel. Use an indicating-type silica gel of six to 16 mesh. You must obtain approval of the Administrator for other types of desiccants (equivalent or better) before you use them. Allow the silica gel to dry for two hours at 175°C (350°F) if it is being reused. You do not have to dry new silica gel if the indicator shows the silica gel is active for moisture collection.

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

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

7.1.5 Nitrogen Gas. Use Ultra-High Purity compressed nitrogen or equivalent to purge the sampling train. The compressed nitrogen you use to purge the sampling train must contain no more than 1 parts per million by volume (ppmv) oxygen, 1 ppmv total hydrocarbons as carbon, and 2 ppmv moisture. The compressed nitrogen must not contribute more than 0.1 mg of residual mass per purge.

7.2 Sample Recovery and Analytical Reagents. You will need acetone, hexane, anhydrous calcium sulfate, ammonia hydroxide, and deionized water for the sample recovery and analysis.

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

7.2.1 Acetone. Use acetone that is stored in a glass bottle. Do not use acetone from a metal container because it normally produces a high residual mass in the laboratory and field reagent blanks. You must use acetone that has a blank value less than 1.0 ppmw (0.1 mg/100 ml) residue.

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

7.2.3 Water. Use deionized, ultra-filtered water that contains 1 ppmw (1 mg/L) residual mass or less to recover material caught in the impinger.

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

7.2.5 Ammonium Hydroxide. Use National Institute of Standards and Technology-traceable or equivalent (0.1 N) NH₄OH.

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

8.0 Sample Collection, Preservation, Storage, and Transport

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

8.2 Preparations. You must clean all glassware used to collect and analyze samples prior to field tests as described in Section 8.4 prior to use. Cleaned glassware must be used at the start of each new source category tested at a single facility. Analyze laboratory reagent blanks (water, acetone, and hexane) 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 in Methods 5, 17, or 201A, whichever is applicable to your test requirements including:

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

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

(c) Verifying the absence of cyclonic flow.

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

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

8.3.2 Traverse points. Use the required number of traverse points at any location, as found in Methods 5, 17, or 201A, whichever is applicable to your test requirements. You must prevent the disturbance and capture of any solids accumulated on the inner wall surfaces by maintaining a 1-inch distance from the stack wall (0.5 inch for sampling locations less than 24 inches in diameter).

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

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glassware at 300°C for six hours prior to beginning tests at each source category sampled at a facility. As an alternative to baking glassware, a field train proof blank, as specified in Section 8.5.4.10, can be performed on the sampling train glassware that is used to collect CPM samples. Prior to each sampling run, the train glassware used to collect condensable PM must be rinsed thoroughly with deionized, ultra-filtered water that that contains 1 ppmw (1 mg/L) residual mass or less.

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

8.4.2 Backup Impinger. The water dropout impinger is followed by a modified Greenburg Smith impinger (backup impinger) with no taper (see Figure 1 of Section 18). Place the water dropout and backup impingers in an insulated box with water at less than or equal to 30°C (less than or equal to 85°F). At the start of the tests, the water dropout and backup impingers 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 7.1.1 after the backup impinger. The connection between the CPM filter and the moisture trap impinger must include a thermocouple fitting that provides a leak-free seal between the thermocouple and the stack gas. (Note: A thermocouple well is not sufficient for this purpose because the fluoropolymer- or steel-encased thermocouple must be in contact with the sample gas).

8.4.4 Moisture Traps. You must use a modified Greenburg-Smith impinger containing 100 ml of water, or the alternative described in Method 5 of appendix A-3 to part 60, followed by an

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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 pre-weighed, 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 of appendix A-3 to part 60, Method 17 of appendix A-6 to part 60, or Method 201A of appendix M to this part as appropriate to leak check the entire sampling system. Specifically, perform the following procedures:

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

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

8.5 Sampling Train Operation. Operate the sampling train as described in the filterable particulate sampling method (i.e., Method 5 of appendix A-3 to part 60, Method 17 of appendix A-6 to part 60, or Method 201A of appendix M to this part) with the following additions or exceptions:

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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 greater than 20° C (greater than 65° F) but less than or equal to 30° C (less than or equal to 85° F) during sample collection. (Note: Maintain the temperature of the CPM filter assembly as close to 30° C (85° F) as feasible.)

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 actual cubic feet per minute or four 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. 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). You may purge the CPM sampling train using the sampling system meter box and vacuum pump or by passing nitrogen through the train under pressure. For either type of purge, you must first attach the nitrogen supply line to a purged inline filter.

8.5.3.1 If you choose to conduct a pressurized nitrogen purge on the complete CPM sampling train, you must quantitatively transfer the water collected in the condenser and the water dropout impinger to the backup impinger. You must measure the water combined in the backup impinger and record the volume or weight as part of the moisture collected during sampling as specified in Section 8.5.3.4.



- (a) You must conduct the purge on the condenser, backup impinger, and CPM filter. If the tip of the backup impinger insert does not extend below the water level (including the water transferred from the first impinger), you must add a measured amount of degassed, deionized ultra-filtered water that contains 1 ppmw (1 mg/L) residual mass or less until the impinger tip is at least 1 centimeter below the surface of the water. You must record the amount of water added to the water dropout impinger (Vp) (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).
- (b) To perform the nitrogen purge using positive pressure nitrogen flow, you must start with no flow of gas through the clean purge line and fittings. Connect the filter outlet to the input of the impinger train and disconnect the vacuum line from the exit of the silica moisture collection impinger (see Figure 3 of Section 18). You may purge only the CPM train by disconnecting the moisture train components if you measure moisture in the field prior to the nitrogen purge. You must increase the nitrogen flow gradually to avoid over-pressurizing the impinger array. You must purge the CPM train at a minimum of 14 liters per minute for at least one hour. At the conclusion of the purge, turn off the nitrogen delivery system.

8.5.3.2 If you choose to conduct a nitrogen purge on the complete CPM sampling train using the sampling system meter box and vacuum pump, 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.

(a) You must conduct the purge on the complete CPM sampling train starting at the inlet of the condenser. If insufficient water was collected, you must add a measured amount of degassed, deionized ultra-filtered water that contains 1 ppmw (1 mg/L) residual mass or

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less until the impinger tip is at least 1 centimeter below the surface of the water. You must record the amount of water added to the water dropout impinger (Vp) (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).

(b) You must start the purge using the sampling train vacuum pump with no flow of gas through the clean purge line and fittings. 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/or nitrogen delivery rates to obtain the following conditions: 14 liters/min or ΔH@ and a positive overflow rate through the rotameter of less than 2 liters/min. The presence of a positive overflow rate 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 at least one hour, checking the rotameter and ΔH@ value(s) at least every 15 minutes. At the conclusion of the purge, simultaneously turn off the delivery and pumping systems.

8.5.3.3 During either purge procedure, 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 greater than 20° C (greater than 65° F), but less than or equal to 30° C (less than or equal to 85° F). If the volume of liquid collected in the moisture traps has not been determined prior to conducting the nitrogen purge, maintain the temperature of the moisture traps following the CPM filter to prevent removal of moisture during the purge. If necessary, add more ice during the purge to maintain the gas temperature measured at the exit of the silica gel impinger below 20° C (68° F). Continue the purge under these

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. . .

conditions for at least one hour, checking the rotameter and ΔH @ value(s) periodically. At the conclusion of the purge, 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 if this has not been done prior to purging the sampling train. Measure the liquid in the water dropout impinger to within 1 ml using a clean graduated cylinder or by weighing it to within 0.5g using a balance. Record the volume or weight of liquid present to be used to calculate the moisture content of the effluent gas in the field log notebook.

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

8.5.4 Sample Recovery.

8.5.4.1 Recovery of filterable PM. Recovery of filterable PM involves the quantitative transfer of particles according to the filterable particulate sampling method (i.e., Method 5 of appendix A-3 to part 60, Method 17 of appendix A-6 to part 60, or Method 201A of appendix M to this part).

8.5.4.2 CPM Container #1, Aqueous liquid impinger contents. Quantitatively transfer liquid from the dropout and the backup impingers prior to the CPM filter into a clean, leak-proof container labeled with test identification and "CPM Container #1, Aqueous Liquid Impinger Contents." Rinse all sampling train components including the back half of the filterable PM filter holder, the probe extension, condenser, each impinger and the connecting glassware, and the front half of the CPM filter housing twice with water. Recover the rinse water, and add it to CPM Container #1. Mark the liquid level on the container.



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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. Recover the acetone rinse into a clean, leak-proof container labeled with test identification and "CPM Container #2, Organic Rinses." Then repeat the entire rinse procedure with two rinses of hexane, and save the hexane rinses in the same container as the acetone rinse (CPM Container #2). Mark the liquid level on the 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 labeled with test identification and "CPM Container #3, Filter Sample."

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 11.2.4). 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, leakproof container labeled with test identification and "CPM Container #4, Cold Water Impinger." Mark the liquid level on the container. CPM Container #4 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 11.2.5). If the silica gel has been weighed in the field to measure water content, then it can be discarded or recovered for reuse. Otherwise, transfer the silica gel to its original container labeled with test identification and "CPM Container #5, Silica Gel Absorbent" and seal. You may use a funnel to make it easier to pour the silica gel without spilling. You may also use a rubber policeman as an aid in removing the silica gel from the impinger. It is not necessary to remove the small amount of silica gel dust particles that may adhere to the impinger wall and are difficult to remove. Since the gain in

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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 field reagent blank. Take approximately 200 ml of the acetone directly from the wash bottle you used for sample recovery and place it in a clean, leak-proof container labeled with test identification and "CPM Container #6, Acetone Field Reagent Blank" (see Section 11.2.6 for analysis). Mark the liquid level on the container. Collect one acetone field reagent blank from the lot(s) of solvent used for the test.

8.5.4.8 CPM Container #7, Water field reagent blank. Take approximately 200 ml of the water directly from the wash bottle you used for sample recovery and place it in a clean, leak-proof container labeled with test identification and "CPM Container #7, Water Field Reagent Blank" (see Section 11.2.7 for analysis). Mark the liquid level on the container. Collect one water field reagent blank from the lot(s) of water used for the test.

8.5.4.9 CPM Container #8, Hexane field reagent blank. Take approximately 200 ml of the hexane directly from the wash bottle you used for sample recovery and place it in a clean, leak-proof container labeled with test identification and "CPM Container #8, Hexane Field Reagent Blank" (see Section 11.2.8 for analysis). Mark the liquid level on the container. Collect one hexane field reagent blank from the lot(s) of solvent used for the test.

8.5.4.10 Field train proof blank. If you did not bake the sampling train glassware as specified in Section 8.4, you must conduct a field train proof blank as specified in Sections
8.5.4.11 and 8.5.4.12 to demonstrate the cleanliness of sampling train glassware.

8.5.4.11 CPM Container #9, Field train proof blank, inorganic rinses. Prior to conducting the emission test, 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

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and place it in a clean, leak-proof container labeled with test identification and "CPM Container #9, Field Train Proof Blank, Inorganic Rinses." Mark the liquid level on the container.

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

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

9.0 Quality Control

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

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



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

9.4 Field Analytical Balance Calibration Check. Perform calibration check procedures on field analytical balances each day that they are used. You must use National Institute of Standards and Technology (NIST)- traceable weights at a mass approximately equal to the weight of the sample plus container you will weigh.

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

9.6 Laboratory Analytical Balance Calibration Check. Check the calibration of your laboratory analytical balance each day that 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.

9.7 Laboratory Reagent Blanks. You should run blanks of water, acetone, and hexane used for field recovery and sample analysis. Analyze at least one sample (150 ml minimum) of each lot of reagents that you plan to use for sample recovery and analysis before you begin testing. These blanks are not required by the test method, but running blanks before field use is advisable to verify low blank concentrations, thereby reducing the potential for a high field blank on test samples.

9.8 Field Reagent Blanks. You should run at least one field reagent blank of water, acetone, and hexane you use for field recovery. These blanks are not required by the test method, but running independent field reagent blanks is advisable to verify that low blank concentrations



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were maintained during field solvent use and demonstrate that reagents have not been contaminated during field tests.

9.9 Field Train Proof Blank. If you are not baking glassware as specified in Section 8.4, you must recover a minimum of one field train proof blank for the sampling train used for testing each new source category at a single facility. You must assemble the sampling train as it will be used for testing. You must recover the field train proof blank samples as described in Section 8.5.4.11 and 8.5.4.12.

9.10 Field Train Recovery Blank. You must recover a minimum of one field train blank for each source category tested at the facility. You must recover the field train blank after the first or second run of the test. 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 4. 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.

10.0 Calibration and Standardization

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

10.1 Thermocouple Calibration. You must calibrate the thermocouples using the procedures described in Section 10.3.1 of Method 2 of appendix A-1 to part 60 or Alternative Method 2, Thermocouple Calibration (ALT-011) (http://www.epa.gov/ttn/emc). Calibrate each temperature sensor at a minimum of three points over the anticipated range of use against a NIST-traceable thermometer. Alternatively, a reference thermocouple and potentiometer calibrated against NIST standards can be used.

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10.2 Ammonium Hydroxide. The 0.1 N NH₄OH used for titrations in this method is made as follows: Add 7 ml of concentrated (14.8 M) NH₄OH to 1 liter of water. Standardize against standardized 0.1 N H₂SO₄, and calculate the exact normality using a procedure parallel to that described in Section 10.5 of Method 6 of appendix A-4 to 40CFR part 60. Alternatively, purchase 0.1 N NH₄OH that has been standardized against a NIST reference material. Record the normality on the CPM Work Table (see Figure 6 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 available at http://www.epa.gov/ttn/chief/ert/ert_tool.html. Record the condensable particulate data on the CPM Work Table (see Figure 6 of Section 18).
 - (b) Measure the liquid in all containers either volumetrically to ± 1 ml or gravimetrically to ± 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 PM Analysis. See the flow chart in Figure 7 of Section 18 for the steps to process and combine fractions from the CPM train.

11.2.1 Container #3, CPM Filter Sample. If the sample was collected by Method 17 or Method 201A with a stack temperature below 30°C (85°F) and the filter can be brought to a constant weight, transfer the filter and any loose PM from the sample container to a tared glass

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weighing dish. (See Section 3.0 for a definition of constant weight.) Desiccate the sample for 24 hours in a desiccator containing anhydrous calcium sulfate. Weigh to a constant weigh and report the results to the nearest 0.1 mg. If the filter cannot be brought to constant weight using this procedure, you must follow the extraction and weighing procedures in this section. (See Section 3.0 for a definition of constant weight.) 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 in Containers 1 and 2, respectively. Extract the CPM filter as follows:

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

11.2.1.2 Extract the organic soluble CPM from the CPM filter by adding sufficient hexane to cover the filter (e.g., 10 ml of hexane). Place the extractor tube into a sonication bath and extract the organic soluble material for a minimum of two minutes. Combine the organic extract with the contents of Container #2. Repeat this extraction step twice for a total of three extractions.

11.2.2 CPM Container #1, Aqueous Liquid Impinger Contents. Analyze the water soluble CPM in Container 1 as described in this section. Place the contents of Container #1 into a separatory funnel. Add approximately 30 ml of hexane to the funnel, mix well, and drain off the lower organic phase. Repeat this procedure twice with 30 ml of hexane each time combining the organic phase from each extraction. Each time, leave a small amount of the organic/hexane phase in the separatory funnel, ensuring that no water is collected in the organic phase. This extraction

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should yield about 90 ml of organic extract. Combine the organic extract from Container #1 with the organic train rinse in Container 2.

11.2.2.1 Determine the inorganic fraction weight. Transfer the aqueous fraction from the extraction to a clean 500-ml or smaller beaker. Evaporate to no less than 10 ml liquid on a hot plate or in the oven at 105°C and allow to dry at room temperature (not to exceed 30°C (85°F)). You must ensure that water and volatile acids have completely evaporated before neutralizing nonvolatile acids in the sample. Following evaporation, desiccate the residue for 24 hours in a desiccator containing anhydrous calcium sulfate. Weigh at intervals of at least six hours to a constant weight. (See Section 3.0 for a definition of Constant weight.) Report results to the nearest 0.1 mg on the CPM Work Table (see Figure 6 of Section 18) and proceed directly to Section 11.2.3. If the residue cannot be weighed to constant weight, redissolve the residue in 100 ml of deionized distilled ultra-filtered water that contains 1 ppmw (1 mg/L) residual mass or less and continue to Section 11.2.2.2.

11.2.2.2 Use titration to neutralize acid in the sample and remove water of hydration. If used, calibrate the pH meter with the neutral and acid buffer solutions. Then titrate the sample with $0.1N NH_4OH$ to a pH of 7.0, as indicated by the pH meter or colorimetric indicator. Record the volume of titrant used on the CPM Work Table (see Figure 6 of Section 18).

11.2.2.3 Using a hot plate or an oven at 105°C, evaporate the aqueous phase to approximately 10 ml. Quantitatively transfer the beaker contents to a clean, 50-ml pre-tared weighing 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 six hours to a constant weight. (See Section 3.0 for a definition of Constant weight.) Report results to the nearest 0.1 mg on the CPM Work Table (see Figure 6 of Section 18).



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11.2.2.4 Calculate the correction factor to subtract the NH_4 + retained in the sample using Equation 1 in Section 12.

11.2.3 CPM Container #2, Organic Fraction Weight Determination. Analyze the organic soluble CPM in Container #2 as described in this section. Place the organic phase in a clean glass beaker. Evaporate the organic extract at room temperature (not to exceed 30°C (85°F)) and pressure in a laboratory hood to not less than 10 ml. Quantitatively transfer the beaker contents to a clean 50-ml pre-tared weighing 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 six hours to a constant weight (i.e., less than or equal to 0.5 mg change from previous weighing), and report results to the nearest 0.1 mg on the CPM Work Table (see Figure 6 of Section 18).

11.2.4 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 ± 1 ml or gravimetrically to ± 0.5 g, and record the volume or weight on the filterable particulate analytical data sheet of the filterable PM test method.

11.2.5 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 PM test method.

11.2.6 Container #6, Acetone Field Reagent Blank. Use 150 ml of acetone from the blank container used for this analysis. Transfer 150 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 clean 50-ml pre-tared weighing 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 six hours to a constant weight (i.e., less than or equal to 0.5 mg change from previous weighing), and report results to the nearest 0.1 mg on Figure 4 of Section 19.

11.2.7 Water Field Reagent Blank, Container #7. Use 150 ml of the water from the blank container for this analysis. Transfer the water to a clean 250-ml beaker, and evaporate to approximately 10 ml liquid in the oven at 105°C. Quantitatively transfer the beaker contents to a clean 50 ml pre-tared weighing 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 six hours to a constant weight (i.e., less than or equal to 0.5 mg change from previous weighing) and report results to the nearest 0.1 mg on Figure 4 of Section 18.

11.2.8 Hexane Field Reagent Blank, Container #8. Use 150 ml of hexane from the blank container for this analysis. Transfer 150 ml of the hexane to a clean 250-ml beaker. Evaporate the hexane at room temperature (not to exceed 30°C (85°F)) and pressure in a laboratory hood to approximately 10 ml. Quantitatively transfer the beaker contents to a clean 50-ml pre-tared weighing 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 six hours to a constant weight (i.e., less than or equal to 0.5 mg change from previous weighing), and report results to the nearest 0.1 mg on Figure 4 of Section 18.









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Figure 3. Nitrogen Purge Train Configuration (Pressure Purge)



IV. Statement of Authenticity

The sampling and analysis for this report was carried out under my direction. I have reviewed the testing details and results of this report and hereby certify that the data contained within is authentic and accurate to the best of my knowledge.

Verified and Reviewed by:

Date: January 9th, 2015

Signature: James Garrett Testing Engineer



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Units 16-21

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