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Air Emissions Test Report Detroit Water and Sewerage Department Multiple Hearth Incinerator Nos. 9 and 10 State Registration Number (SRN) B2103 Permit-to-Install Application ID# 61-13 Detroit, Michigan

Prepared for:

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Prepared by:

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December 2015

GamAir Project No.: 753-1508

REPORT CERTIFICATION

I have personally examined and am familiar with the information submitted in this document and all attachments thereto, and I certify, based on reasonable investigation, including my inquiry of those individuals responsible for obtaining the information, that the submitted information is true, accurate, and complete to the best of my knowledge and belief.

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10 December 2015

Leigh A. Gammie, QSTI Principal Gammie Air Monitoring, LLC Date

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1.0 INTRODUCTION

1.1 Summary of Test Program

Gammie Air Monitoring, LLC (GamAir) was retained by Walsh Construction Company (Walsh) to perform an air emissions compliance test program on two multiple hearth incinerators (MHI) Nos. 9 and 10 which are owned and operated by the Detroit Water and Sewerage Department (DWSD). Testing occurred on the scrubber exhaust duct of each MHI. The purpose of this source test program was to quantify the controlled emissions of filterable particulate matter (FPM), multiple metals (cadmium, lead, and mercury), hydrogen chloride (HCl), fluoride (F), sulfuric acid (H₂SO₄), particulate matter less than 10 microns and 2.5 microns (PM10/2.5), polychlorinated dibenzo-p-dioxins (PCDD)/polychlorinated dibenzofurans (PCDF), sulfur dioxide, nitrogen oxides, carbon monoxide, volatile organic compounds, carbon dioxide, and oxygen. The applicable USEPA MACT 129 emission limits are as follows.

Parameter	Units	USEPA MACT 129 Emission Limits
Carbon Monoxide (CO)	ppmvd @ 7% O2	3,800
Sulfur Dioxide (SO ₂)	ppmvd @ 7% O ₂	26
Oxides of Nitrogen (NO _x)	ppmvd @ 7% O ₂	220
Filterable Particulate Matter (FPM)	milligrams per dry standard cubic meter (a) 7% O ₂	80
Hydrogen Chloride (HCl)	ppmvd @ 7% O ₂	1.2
PCDD/PCDF	nanograms per dry standard cubic meter @ 7% O_2 (total mass basis) or nanograms per dry standard cubic meter @ 7% O_2 (toxic equivalency basis)	0.32
Cadmium	milligrams per dry standard cubic meter @ 7% O ₂	0.095
Lead	milligrams per dry standard cubic meter @ $7\% O_2$	0.30
Mercury	milligrams per dry standard cubic meter @ 7% O ₂	0.28
Fugitive Emissions from Ash Handling	% Opacity	5

The applicable Michigan Department of Environmental Quality (MDEQ) emission limits are as follows.

Parameter	Units	MDEQ Emission Limits
PM 10 particulate matter	pounds per hour	1.2
PM 2.5 particulate matter	pounds per hour	1.2
Volatile Organic Compounds (VOC)	pounds per hour	3.2
Fluoride (F)	pounds per hour	1.73
Sulfuric acid (H ₂ SO ₄)	pounds per hour	1.3

Compliance emission tests focused on the parameters listed above. The tests were conducted in accordance with the conditions and monitoring requirements for compliance testing as set forth in the State of Michigan Department of Environmental Quality (MDEQ) and United States Environmental Protection Agency (USEPA) Part 60, Subpart MMMM - Emission Guidelines for Existing Sewage Sludge Incineration Units (Model Rule).

Compliance tests were conducted in accordance with EPA Methods 1-5, 6C, 7E, 8, 10, 13B, 22, 23, 25A, 26A, 29, 201A, and 202 as published in Title 40, <u>Code of Federal Regulations</u>, Parts 51 and 60. The test program for each MHI was conducted over a two day period. Unit No. 9 was tested on 10 & 11 October 2015 and Unit No. 10 was tested on 8, 9 & 12 October 2015. Representatives from Walsh and DWSD were responsible for coordinating the testing with the MDEQ. Mr. Thomas Maza served on the onsite representative from MDEQ. DWSD was responsible for collecting all process data, collecting and analyzing all biosolid samples. Gammie Air Monitoring, LLC (GamAir) was responsible for collecting all air emission samples and the respective analysis of those samples.

Section 2.0 of this report presents a description of each source and describes the sampling locations. Section 3.0 summarizes the test results. Section 4.0 describes the sampling and analysis methodologies. Section 5.0 provides the quality assurance and quality control (QA/QC) procedures specific to this test program.

1.2 Test Program Organization

The following is a list of those individuals responsible for the organization of this test program.

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2.0 SOURCE AND SAMPLE LOCATION DESCRIPTIONS

2.1 Process and Air Pollution Control Description

DWSD operated two identical multiple hearth incinerators (MHI), identified at Nos. 9 and 10, which are 22 foot in diameter and are equipped with 12-hearths. Sludge was dewatered with centrifuges and conveyed to the multiple hearth furnaces with belt conveyors. The sludge conveyors were equipped with nuclear weigh scales for continuous monitoring of the amount of sludge being incinerated. During the compliance test program, each incinerator operated at a minimum 85 percent of rated capacity. The furnace is equipped with auxiliary natural gas burners at hearths 2, 4, 6, 8, 10, and 12. The firing rate of the burners is modulated by a central control system to sustain the desired hearth temperatures. Each air pollution control system is comprised of a double zero hearth afterburner section of Hearths 1 and 2, a quench section, and EnviroCare® Venturi-Pak (venturi throat sections and mist eliminator) scrubber system. Individual process monitoring for each incinerator are shown in Tables 2-1 through 2-4.

2.2 Process Monitoring

2.2.1 Unit No. 9

During the test program MHI No. 9 operated at an optimized feed rate. The following two tables summarize the process conditions during the PCDD/PCDF/metals tests and the FPM/HCl tests, respectively. Sludge feed rate ranged between 2.42 to 2.73 dry tons per hour.

Table 2-1 Summary of Process Data PCDD/PCDF and Metals Tests Detroit Water and Sewerage Department - Incinerator No. 9 Detroit, Michigan 10 October 2015							
Method/Component Units Run 4 Run 5 Run 6 Average 0945-1145 1215-1415 1440-2040							
Biosolids Feed Rate	wet tons/hour	11.4	11.3	10.1	10.93		
Biosolids Cake Solids	%	22	23	27	24.0		
Biosolids Feed Rate	dry tons/hour	2.51	2.60	2.73	2.61		
Afterburner Exit Temp.	°F	1171	1163	1241	1192		
Total Scrubber Water Flow	gallon/minute	1380	1384	1407	1390		
Total Scrubber Pressure Drop	inches w.c.	21.5	23.7	24.5	23.2		
Scrubber Water Outlet	pН	6.00	5.94	5.89	5.94		

% - percent

inches w.c. - inches water column

Table 2-2Summary of Process DataFPM and HCl TestsDetroit Water and Sewerage Department - Incinerator No. 9Detroit, Michigan11 October 2015							
Method/Component Units Run 4 Run 5 Run 6 Average 0835-1005 1010-1127 1300-1430							
Biosolids Feed Rate	wet tons/hour	10.53	10.86	11.27	10.89		
Biosolids Cake Solids	%	23	25	23	23.7.7		
Biosolids Feed Rate	dry tons/hour	2.42	2.72	2.59	2.58		
Afterburner Exit Temp.	°F	1183	1180	1208	1190		
Total Scrubber Water Flow	gallon/minute	1377	1363	1380	1373		
Total Scrubber Pressure Drop	inches w.c.	22.2	24.4	22.9	23.2		
Scrubber Water Outlet	pН	6.02	5.94	5.91	5.96		

% - percent

inches w.c. - inches water column

2.2.2 Unit No. 10

During the test program MHI No. 10 operated at an optimized feed rate. Tables 2-3 and 2-4 summarize the process conditions during the PCDD/PCDF/metals tests and the FPM/HCl tests, respectively. Sludge feed rate ranged between 1.76 to 2.93 dry tons per hour. Additional process monitoring data is contained in Appendix D.

Table 2-3Summary of Process DataPCDD/PCDF and Metals TestsDetroit Water and Sewerage Department - Incinerator No. 10Detroit, Michigan8 October 2015							
Method/Component Units Run 1 Run 2 Run 3 Average 1005-1220 1250-1450 1635-1841							
Biosolids Feed Rate	wet tons/hour	8.29	8.00	13.90	10.06		
Biosolids Cake Solids	%	24	22	20	22.0		
Biosolids Feed Rate	dry tons/hour	1.99	1.76	2.78	2.18		
Afterburner Exit Temp.	٥F	1169	1106	1187	1154		
Total Scrubber Water Flow	gallon/minute	1317	1346	1320	1328		
Total Scrubber Pressure Drop	inches w.c.	23.6	26.6	25.8	25.3		
Scrubber Water Outlet	pН	6.08	6.28	6.18	6.18		

Table 2-4Summary of Process DataFPM and HCl TestsDetroit Water and Sewerage Department - Incinerator No. 10Detroit, Michigan9 October 2015							
Method/Component Units Run 1 Run 2 Run 3 Average 0905-1035 1055-1225 1415-1545							
Biosolids Feed Rate	wet tons/hour	13.9	10.73	9.15	11.27		
Biosolids Cake Solids	%	21	24	22	22.3		
Biosolids Feed Rate	dry tons/hour	2.93	2.58	2.01	2.51		
Afterburner Exit Temp.	°F	1167	1167	1153	1162		
Total Scrubber Water Flow	gallon/minute	1359	1301	1318	1326		
Total Scrubber Pressure Drop	inches w.c.	27.5	25.3	25.8	26.2		
Scrubber Water Outlet*	pH	6.07	5.98	6.00	6.02		

2.3 Outlet Flue Gas Sampling Locations

MHI Nos. 9 and 10 sampling locations were identical. Outlet flue gas sampling occurred at a location that is between the scrubber exhaust and induced draft fan. The inside diameter of the exhaust duct is 54 inches. Two test ports, spaced 90° apart, were located 120 inches (2.2 duct diameters) to the nearest upstream disturbance and 108 inches (2.0 duct diameters) to the nearest downstream disturbance. In accordance with EPA Method 1, twenty four (24) traverse points (12 per port) were used for isokinetic sampling and volumetric flowrate determinations. Continuous emissions monitoring (CEM) took place through a single port that was located adjacent to the DWSD total hydrocarbons (THC) sampling probe (same elevation). All continuous emissions monitoring (CEM) took place at the following three traverse points 9", 26", and 44". The EPA Method 1 traverse points are shown in Appendix A.

3.0 SUMMARY AND DISCUSSION OF RESULTS

3.1 Objectives and Test Matrix

The purpose of this test program was to measure air emissions from two MHI specifically Nos.9 and 10. Emissions testing was conducted in accordance with EPA approved test procedures. Three emissions tests were conducted on each MHI for each parameter, with the average result of the three tests reported. Table 3-1 lists the compliance parameters measured, the EPA reference methods used, and the sampling times for each test. The specific objectives for each MHI were to:

- Measure fluoride emissions from the outlet stack in accordance with EPA Method 13B.
- Measure multiple metals (cadmium, lead, and mercury) emissions from the outlet stack in accordance with EPA Method 29.
- Measure PCDD/PCDF emissions from the outlet stack in accordance with EPA Method 23.
- Measure filterable particulate matter (PM) emissions from the outlet stack in accordance with EPA Method 5 and measure PM10/2.5 emissions in accordance with EPA Methods 201A and 202.
- Measure fugitive emissions (VE) from the ash handling system in accordance with EPA Method 22.
- Measure hydrogen chloride (HCl) emissions from the outlet stack in accordance with EPA Methods 5 and 26A.
- Measure O₂, CO₂, SO₂, NO_x, CO, and VOC from the outlet stack in accordance with EPA Methods 3A, 6C, 7E, 10, and 25A.
- Measure sulfuric acid emissions from the outlet stack in accordance with EPA Method 8
- Determine percent solids from collected sludge samples in accordance with Method SM2540B (Standard Methods) and ASTM Method D3684-01, respectively.
- Monitor and record scrubber pressure drop, auxiliary fuel consumption use, hearth operating temperatures, scrubber water pH, and sludge feed rate.

3.2 Test Matrix

Table 3-1 presents the sampling and analytical matrix used at each of the scrubber outlet sampling locations.

TABLE 3-1 Test Matrix Compliance Test Program Detroit Water and Sewerage Department Two Multiple Hearth Incinerators - Nos. 9 & 10 Detroit, Michigan							
Sampling	No. of	Pollutant	Sampling	Sampling	Sample Run	Analytical	Analytical
Location	Runs ^a	Type ^b	Method ^c	Organization	Time (min.)	Method ^d	Laboratory
Outlet Stack CEMS Port	3	O ₂ /CO ₂ & SO ₂ NO _x , CO, VOC	EPA M3A & 6C 7E,10, 25A	GamAir	60	CEM	GamAir
Outlet EPA Ports ^e	3	Metals	EPA M29	GamAir	120	ICP/MS	Maxxam
Outlet EPA Ports	3	PCDD/PCDF	EPA M23	GamAir	120	HRGC/ HRMS	Maxxam
Outlet EPA Ports	3	HC1	EPA M26A	GamAir	84	IC	Maxxam
		FPM	EPA M1-5	GamAir	84	Gravimetric	Maxxam
Outlet EPA Ports	3	H_2SO_4	EPA M8	GamAir	84	Titration	Maxxam
Outlet EPA Ports	3	PM10/2.5	EPA 201A/202	GamAir	84	Gravimetric	Maxxam
Outlet EPA Ports	3	Fluoride	EPA 13B	GamAir	84	IC	Maxxam
Ash Handling	3	Visible Emissions	EPA M22	GamAir	60	NA	GamAir
Process Feed	3	Percent Solids	Grab	BWSD	Grab	Gravimetric	Maxxam

° M-EPA Method.

a number of test runs
 b HCl and FPM tests will be conducted using the same M1-5/26A sampling train.
 c M - EPA 1
 d CEM - continuous emissions monitoring; ICP/MS - inductively coupled plasma/mass spectrometry; IC - ion chromatography
 EPA Ports - two ports spaced 90° apart.

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3.2 Field Test Changes

During the first EPA Method 201A/202 test run on MHI No. 10 the unit encountered unstable operating conditions. This was made apparent when particulate matter "broke through" the instack filter due to a higher than normal particulate mass. After consulting with Mr. Tom Maza, the onsite MDEQ representative, it was agreed that the second EPA Method 201A/202 test would be reduced from 84 minutes to 60 minutes. During the second run a similar high particulate loading occurred as the sample train vacuum rose quickly, starting at 2.5 inches of vacuum to 6 inches within 5 minutes. As a result of the unstable operating conditions a third test run was not attempted. This decision was approved by Mr. Tom Maza. Both EPA Method 201A/202 test runs were recovered and submitted for analysis. After further consultation with the Detroit Water and Sewerage Department (DWSD) project manager three additional EPA Method 201A/202 test runs would be conducted on 12 October 2015. Each of the three additional tests, identified as Runs 7, 8, and 9, were performed with a total test time of 60 minutes. The additional testing was approved by Mr. Tom Maza with the stipulation that all five tests would be reported. For Unit No. 10 a total of five EPA Method 201A/202 test run were submitted for analysis. Results from the five test runs are detailed in Appendix B. EPA Method 201A/202 test results from the two test runs, conducted on 9 October 2015, showed compliance with the MDEQ emission limit as did the three tests performed on 12 October 2015.

3.3 Summary of Results

3.3.1 Unit No. 9

All Unit No. 9 test results are summarized in Table 3-2. The three tests are shown as Run 4, Run 5, and Run 6 as Unit No. 9 was the second unit tested during this week long test program. The average FPM concentration was 13.20 milligrams per dry standard cubic meter corrected to 7% oxygen (mg/dscm at 7% O₂). The FPM concentration is below the EPA limit of 80.0 mg/dscm at 7% O₂ and represents 16.5 percent of the limit. HCl emissions averaged <0.12 parts per million corrected to 7% oxygen (ppm at 7% O₂). The HCl emissions are below the EPA limit of 1.2 ppm at 7% O₂ and represent 10 percent of the limit. PCDD/PCDF emissions were below both EPA limits of 5.0 nanograms per dry standard cubic meter corrected to 7% oxygen (ng/dscm at 7% O₂), total weight basis and 0.32 nanograms per dry standard cubic meter corrected to 7% oxygen (ng/dscm at 7% O_2), toxic equivalency factor basis. The average PCDD/PCDF concentration of 2.32 ng/dscm at 7% O2 (total weight basis) represents 46.4 percent of the limit and the TEF concentration represents 17.8 percent of the limit. The average cadmium concentration of 0.011 mg/dscm at 7% O₂ is below the limit of 0.095 mg/dscm at 7% O_2 and is 11.6 percent of the limit. The average lead concentration of 0.037 mg/dscm at 7% O₂ is below the limit of 0.30 mg/dscm at 7% O₂ and is 12.3 percent of the limit. The average mercury concentration of 0.046 mg/dscm at 7% O_2 is below the limit of 0.28 mg/dscm at 7% O₂ and is 16.4 percent of the limit. All sulfur dioxide, oxides of nitrogen, and carbon monoxide gaseous pollutants were below their respective EPA limits as shown in Table 3-2.

All Michigan DEQ targeted pollutants were below their respective emission limits. The average sulfuric acid emission rate of 0.061 pounds per hour is below the emission limit of 1.3 pounds per hour. The average fluoride emission rate of less than (\leq) 0.004 pounds per hour is below the emission limit of 1.73 pounds per hour. The average VOC emission rate of 1.41 pounds per hour is below the emission limit of 3.2 pounds per hour. Both the PM10 and PM2.5 average emission rates of 0.79 and 0.66, respectively were below the emission limit of 1.2 pounds per hour.

Test results are further detailed in Appendix B. An example calculation is also contained in Appendix B. Copies of field data sheets are shown in Appendix C. Process monitoring data is contained in Appendix. D. Appendix E contains all laboratory analytical reports and Appendix F displays all equipment calibration data.

		LE 3-2	······································					
Summary of Emissions Data								
	Compliance							
Detroit	Detroit Water and Sewerage Department - Incinerator No. 9							
		Michigan						
	······	ober 2015						
Average Test USEPA MACT 129								
Parameter	Concentration*	or MDEQ Emission	Compliance Status					
·	or Emission Rate	Limit*						
	USEPA MACT	129 Pollutants						
Sulfur Dioxide	0.7 ppmvd	26 ppmvd	Yes					
Oxides of Nitrogen	195.4 ppmvd	220 ppmvd	Yes					
Carbon Monoxide								
Filterable Particulate	13.20 mg/dscm	80.0 mg/dscm	Yes					
Matter								
Hydrochloric Acid	<0.12 ppmvd	1.2 ppmvd	Yes					
Cadmium	0.011 mg/dscm	0.095 mg/dscm	Yes					
Lead	0.037 mg/dscm	0.30 mg/dscm	Yes					
Mercury	0.046 mg/dscm	0.28 mg/dscm	Yes					
PCDD/PCDF	2.32 ng/dscm	5.0 ng/dscm	Yes					
	0.057 ng/dscm TEF	0.32 ng/dscm TEF	Yes					
Fugitive Emission	0.0%	5%	Yes					
Michigan DEQ Pollutants								
PM2.5 and CPM	0.66 pounds/hour	1.2 pounds/hour	Yes					
PM10 and CPM	0.79 pounds/hour	1.2 pounds/hour	Yes					
Sulfuric Acid	0.061 pounds/hour	1.3 pounds/hour	Yes					
Fluoride	<0.004 pounds/hour	1.73 pounds/hour	Yes					
VOC	1.41 pounds/hour	3.2 pounds/hour	Yes					

*All MACT 129 concentrations are corrected to 7 percent oxygen (@ 7% O2).

mg/dscm – milligrams per dry standard cubic meter. ng/dscm – nanograms per dry standard cubic meter.

ng/dscm TEF - with toxic equivalency factor.

ppmvd – parts per million, volume dry basis.

3.3.2 Unit No. 10

All Unit No. 10 test results are summarized in Table 3-3. The average FPM concentration was 14.93 milligrams per dry standard cubic meter corrected to 7% oxygen (mg/dscm at 7% O_2). The FPM concentration is below the EPA limit of 80.0 mg/dscm at 7% O_2 and represents 18.7 percent of the limit. HCl emissions averaged 0.23 parts per million corrected to 7% oxygen (ppm at 7% O_2). The HCl emissions are below the EPA limit of 1.2 ppm at 7% O_2 and represent 19.2 percent of the limit. PCDD/PCDF emissions were below the EPA limit of 0.32 nanograms per dry standard cubic meter corrected to 7% oxygen (ng/dscm at 7% O_2), toxic equivalency factor (TEF) basis. The average PCDD/PCDF concentration of 0.239 ng/dscm at 7% O_2 (TEF basis) represents 74.7 percent of the limit. The average cadmium concentration of 0.008 mg/dscm at 7% O_2 is below the limit of 0.095 mg/dscm at 7% O_2 and is 8.4 percent of the limit. The average lead concentration of 0.016 mg/dscm at 7% O_2 is below the limit of 0.38 mg/dscm at 7% O_2 and is 5.3 percent of the limit. The average mercury concentration of 0.038 mg/dscm at 7% O_2 is below the limit of 0.28 mg/dscm at 7% O_2 and is 13.6 percent of the limit. All sulfur dioxide, oxides of nitrogen, and carbon monoxide gaseous pollutants were below their respective EPA limits as shown in Table 3-3.

All Michigan DEQ targeted pollutants were below their respective emission limits. The average sulfuric acid emission rate of 0.073 pounds per hour is below the emission limit of 1.3 pounds per hour. The average fluoride emission rate of less than (<) 0.0032 pounds per hour is below the emission limit of 1.73 pounds per hour. The average VOC emission rate of 2.73 pounds per hour is below the emission limit of 3.2 pounds per hour. Both the PM10 and PM2.5 average emission rates of 0.71 and 0.70, respectively were below the emission limit of 1.2 pounds per hour.

TABLE 3-3Summary of Emission DataCompliance Test ProgramDetroit Water and Sewerage Department - Incinerator No. 10Detroit, Michigan8, 9 & 12 October 2015						
Average TestUSEPA MACT 129ParameterConcentration*or MDEQ EmissionCompliance Statusor Emission RateLimit*						
	USEPA MACT	129 Pollutants				
Sulfur Dioxide	1.1 ppmvd	26 ppmvd	Yes			
Oxides of Nitrogen						
Carbon Monoxide						
Filterable Particulate Matter	0					
Hydrochloric Acid	0.23 ppmvd	1.2 ppmvd	Yes			
Cadmium	0.008 mg/dscm	0.095 mg/dscm	Yes			
Lead	0.016 mg/dscm	0.30 mg/dscm	Yes			
Mercury	0.038 mg/dscm	0.28 mg/dscm	Yes			
PCDD/PCDF	0.24 ng/dscm TEF	0.32 ng/dscm TEF	Yes			
Fugitive Emissions	0%	5%	Yes			
	MDEQ F	Pollutants				
PM2.5 and CPM	0.70 pounds/hour	1.2 pounds/hour	Yes			
PM10 and CPM	0.71 pounds/hour	1.2 pounds/hour	Yes			
Sulfuric Acid	0.073 pounds/hour	1.3 pounds/hour	Yes			
Fluoride	<0.0032 pounds/hour	1.73 pounds/hour	Yes			
VOC	2.73 pounds/hour	3.2 pounds/hour	Yes			

*All MACT 129 concentrations are corrected to 7 percent oxygen (@ 7% O2).

ppmvd - parts per million, volume dry basis.

mg/dscm – milligrams per dry standard cubic meter. ng/dscm – nanograms per dry standard cubic meter. ng/dscm TEF – with toxic equivalency factor

4.0 SAMPLING AND ANALYTICAL METHODOLOGY

The following EPA test methods were utilized during this emissions test program:

EPA Method 3APitot tube)EPA Method 3ADetermination of oxygen and carbon dioxide concentrations in emissions from stationary sources (Instrumental analyzer procedur Determination of moisture content in stack gasesEPA Method 4Determination of particulate emissions from stationary sources	e)
EPA Method 5 Determination of particulate emissions from stationary sources	
EPA Method 6C Determination of sulfur dioxide emissions from stationary sources (instrumental analyzer procedure)	
EPA Method 7E Determination of oxides of nitrogen emissions from stationary sou (instrumental analyzer procedure)	rces
EPA Method 8 Determination of sulfuric acid from stationary sources	
EPA Method 10 Determination of carbon monoxide emissions from stationary sour (instrumental analyzer procedure)	ces
EPA Method 13B Determination of total fluoride emissions from stationary sources	
EPA Method 22 Visual determination of fugitive emissions from material sources	
EPA Method 23 Determination of polychlorinated dibenzo-p-dioxins (PCDD) and polychlorinated dibenzofurans (PCDF) emissions from stationary sources	
EPA Method 25A Determination of total gaseous organic concentration using a flamionization analyzer	3
EPA Method 26A EPA Method 29Determination of hydrogen chloride emissions from stationary sourcesEPA Method 201A EPA Method 202Determination of PM10 and PM2.5 emissions from stationary sourcesDry impinger method for determining condensable particulate emissions from stationary sources	

The following sections describe the sampling and analytical procedures utilized during this emissions test program. All tests were performed in triplicate unless otherwise noted. All sampling and analytical procedures followed those outlined in Title 40, <u>CFR</u>, Part 60; any deviations for this test program are addressed in the following sections.

4.1 Volumetric Flow Rate Tests

The volumetric flowrate of the exhaust gases, at the scrubber outlet test locations, were determined using EPA Methods 1-4. In accordance with EPA Method 1 twenty four (24) traverse points were utilized for flue gas velocity measurements during the isokinetic tests. The locations of the traverse points, as determined by EPA Method 1, are listed in Appendix A. Flue gas velocity measurements were taken at each traverse point during each test run using an S-type Pitot tube and an inclined water manometer in accordance with EPA Method 2. Stack temperature measurements was taken at each traverse point using a Type-K thermocouple and digital temperature readout. The stack static pressure was measured during

each test run using the Pitot tube and manometer setup. Pitot tube leak checks were performed before and after each test run in accordance with EPA Method 2.

The molecular weight of the outlet exhaust gases was determined by measuring the oxygen (O_2) and carbon dioxide (CO_2) concentrations using EPA Method 3A in conjunction with collected an integrated gas sample in 25 liter Tedlar bags. When applicable carbon dioxide and oxygen concentrations were determined from integrated gaseous samples collected during each test run using the sample train described in EPA Method 3B. The flue gas moisture content was determined from the moisture catch of each isokinetic sampling train during the emissions testing in accordance with EPA Method 4. A minimum sample volume of 35 cubic feet was collected during each moisture test run. The moisture content of the flue gas was calculated as the ratio of the moisture catch corrected to standard conditions to the sum of the dry sample volume and the moisture catch, both corrected to standard conditions.

4.2 Hydrogen Chloride and Filterable Particulate Matter Tests

The hydrogen chloride (HCl) and filterable particulate matter (FPM) emissions were sampled and analyzed in accordance with EPA Methods 5 and 26A. Three 84 minute tests were performed for compliance determination on each MHI. This sampling train is shown in Appendix A. The Method 5/26A sampling train consists of a Pyrex® glass nozzle, a heated glass lined sample probe, a heated Teflon filter (tared), a set of four tared glass impingers connected in series in an ice bath, a control module consisting of a leak free sampling pump, a calibrated critical orifice, an inclined manometer, and a calibrated dry gas meter.

The first two impingers each contained 100 ml of 0.1 N sulfuric acid (H₂SO₄) while the third impinger contained 100 ml of sodium hydroxide (NaOH). The fourth impinger contained a known quantity of silica gel. The sampling train glassware was cleaned prior to testing with soap and water and rinsed thoroughly with water. The sample probe and oven box were maintained at a temperature of $248\pm25^{\circ}$ F during sampling to prevent moisture condensation. The impinger outlet temperature was maintained below 68°F during sampling by adding ice to the cold box. A vacuum line is connected from the outlet of the fourth impinger to the control module.

Before each test, the sampling train was leak checked to ensure no leakage greater than 0.02 cubic feet per minute at 10" Hg vacuum. The probe was then placed in the stack and stack gas was withdrawn isokinetically for an equal period of time at each traverse point with a sampling rate not exceeding 1.0 cfm.

The velocity differential pressure, critical orifice differential pressure, dry gas meter volume, dry gas meter outlet temperatures, probe temperature, stack temperature, oven box temperature, impinger outlet temperature, and sample vacuum were recorded at each traverse point during sampling. At the end of each test, the sampling train was leak checked to ensure no leakage greater than 0.02 cubic feet per minute at the highest recorded test vacuum.

Sample recovery consisted of rinsing the nozzle, probe, and filter holder front half three times with acetone. These acetone rinses were collected in Container 2. The tared Teflon filter was recovered in a clean laboratory area and placed in Container 1. Both the acetone and filter were desiccated and weighed (constant weight) in accordance with EPA Method 5. The contents of the four impingers was measured gravimetrically for moisture gain then

transferred to sample Container 3. Impingers 1 and 2 and their connecting glassware were rinsed with deionized distilled (DI) water twice. These rinses will be added to Container 3. The third impinger contents were discarded. The sample containers were sealed and the liquid levels marked. The HCl samples were shipped to Maxxam Analytics, Inc. for analysis. The HCl analysis was performed by ion chromatography (IC) as described in EPA Method 26A.

4.3 Multiple Metals Tests

Multiple metals (cadmium, lead, and mercury) were sampled and analyzed in accordance with EPA Method 29. These metals tests were performed in triplicate on each MHI at the exhaust stack location during 120-minutes test runs. A schematic of the multiple metals sampling train is shown in Appendix A. The following is a description of the metals sampling train and the procedures used to quantify multiple-metals during the test program. The multiple-metals sampling train consisted of a glass nozzle, a heated glass lined probe, a quartz filter/holder in a heated box, a set of six glass impingers connected in series, a control module consisting of a leak free sampling pump, a calibrated critical orifice, an inclined manometer, and a calibrated dry gas meter. A Teflon fitting connected the nozzle to the probe liner. All of the sampling train glassware underwent the cleaning and nitric acid soaking procedure described in EPA Method 29 prior to testing. The sample probe and oven box were maintained at a temperature of 248+25⁰F during sampling to prevent moisture condensation. The first and second impingers each contained 100 ml of 5% nitric acid/10% hydrogen peroxide (5%HNO₃/10%H₂O₂). The third impinger was empty. The fourth and fifth impingers contained 100 ml of 4% potassium permanganate/10% sulfuric acid (4%KMnO₄/10%H₂SO₄). The acidic permanganate solution was prepared fresh on-site daily. The sixth impinger contained a known quantity of silica gel. The second impinger was a Greenburg-Smith impinger with a standard tip, while the other impingers had modified tips. The temperature at the outlet of the sixth impinger was maintained below 68°F during sampling by adding ice to the water bath. A vacuum line connected the outlet of the sixth impinger to the control module.

Before each test and after each test run, the sampling train was leak checked to ensure no leakage greater than 0.02 cubic feet per minute at 15" Hg vacuum. The probe was then placed in the stack and stack gas was withdrawn isokinetically for an equal period of time at each traverse point. The velocity differential pressure, critical orifice differential pressure, dry gas meter volume, dry gas meter inlet and outlet temperatures, probe temperature, stack temperature, oven box temperature, impinger outlet temperature, and sample vacuum were recorded at each traverse point during sampling. At the completion of each test, the sampling train was leak checked to ensure no leakage greater than 0.02 cubic feet per minute at the highest recorded test vacuum. After the post-test leak check, the sampling train was disassembled, all open ends were sealed, and the sampling train components were moved to the cleanup area for recovery. The recovery procedure for the multiple-metals sampling train is described as follows. The filter was carefully removed from the filter holder with Teflon coated forceps and placed in a labeled plastic Petri dish (Container 1). Any particulate matter or filter fragments that adhered to the filter holder gasket were transferred to the Petri dish using a dry, acid cleaned nylon bristle brush. The Petri dish was sealed for transport to the

laboratory.

The nozzle, probe liner, and filter holder front half were rinsed and brushed thoroughly with 100 ml of 0.1 N nitric acid (HNO₃) using a Teflon coated brush. These rinses were collected in a labeled glass sample jar (Container 3). The sample jars were sealed and the liquid level marked. The nozzle, probe liner, and filter holder front half were then rinsed with deionized water followed by acetone. These rinses were discarded. The moisture gain in the first two impingers was measured gravimetrically and their contents transferred to a labeled glass sample jar (Container 4). The first two impingers, the filter support, the back half of the filter holder, and the connecting glassware between the back half of the filter holder and the second impinger were rinsed with 100 ml of 0.1 N HNO₃. These rinses were combined with the impinger contents and the sample jar was sealed and the liquid level marked.

The moisture gain in the third impinger was measured gravimetrically and its contents transferred to a labeled glass sample jar (Container 5A). This impinger was then rinsed with 100 ml of 0.1 N HNO₃. The rinses were combined with the impinger contents and the sample jar was sealed and the liquid level marked.

The moisture gain in the permanganate impingers was measured gravimetrically and the contents transferred to a labeled glass sample jar (Container 5B). The impinger and connecting glassware was then rinsed with 100 ml of fresh 4%KMnO₄/10%H₂SO₄ followed by a rinse with 100 ml of deionized water. The permanganate and deionized water rinses were combined with the impinger contents and the sample jar was sealed and the liquid level marked. This sample jar was not completely filled and was vented to relieve excess pressure. The permanganate impingers were rinsed with a total of 25 ml of 8N HCl. The walls and stem of the permanganate impingers were rinsed and collected in a labeled sample jar containing 200 ml of deionized water (Container 5C). The sample jar was sealed and the liquid level marked. The silica gel impinger was weighed for moisture gain. The silica gel was then returned to its original storage container to be dried for reuse. The following is a list of the sample recovery containers.

Container 1	filter is removed from filter holder and stored in sealed Petri dish
Container 2	not used in this procedure, used for FPM procedure
Container 3	nozzle, sample probe, and front half of filter housing are brushed and
	rinsed three times with 100 ml of 0.1N HNO3 and save
Container 4	measure contents of impingers 1 and 2 and save; filter support, back
	half of filter housing, and flexible Teflon line are rinsed three times
	with 100 ml of 0.1N HNO ₃ and save
Container 5A	measure contents of impinger 3 then rinse three times with 100 ml of
	0.1N HNO ₃ and save
Container 5B	measure contents of impingers 4&5 then rinse three times with 100 ml
	of acidified KMnO ₄ and 100 ml of deionized distilled water and save.
Container 5C	rinse impingers 4&5 with 25 ml of 8N HCl solution followed by 100
	ml of deionized distilled water and save
Container 6	weigh contents of impinger 6 for moisture gain

Container 7not used in this procedureContainer 8A0.1N HNO3 reagent blank (300 ml)Container 8Bdeionized distilled water blank (100 ml)Container 9HNO3/H2O2 reagent blank (200 ml)Container 10acidified KMnO4 reagent blank (100 ml)Container 118N HCl reagent blank (200 ml)Container 12filter blanks (3)

Two unused filter and aliquots of each of the impinger and rinse solutions, in the volumes specified in Method 29, were collected and submitted with the field samples as reagent blanks. Metals results were not reagent blank corrected. Front half and back half fractions were combined before analysis. Containers 1 through 4 were digested in concentrated acid before being analyzed for the target metals (except mercury) by inductively coupled plasma-mass spectrometry (ICP/MS). The mercury analysis (Containers 1-4, 5A, B, and C) were conducted on each of the sample fractions following digestion with acid and permanganate by cold vapor atomic absorption spectroscopy (CVAAS). All collected reagent blanks were analyzed using the same methodologies as the collected samples.

4.4 Polychlorinated Dibenzo-p-dioxins and Dibenzofurans Tests

Polychlorinated dibenzo-p-dioxins and polychlorinated dibenzofurans (PCDD/PCDF) sampling was conducted in accordance with EPA Method 23. Three PCDD/PCDF tests were performed on each MHI with each test being 120-minutes in duration. The Method 23 sampling train is shown in Appendix A. The following is a description of the sampling train and the procedures used to quantify PCDD and PCDF.

The sampling train consisted of precleaned acid soaked, DI water soaked, and tap water rinsed glassware, a Pyrex® glass button hook nozzle, a heated glass lined sample probe, a heated filter in a glass filter holder, a water cooled glass coil condenser, a water cooled adsorbent module (spiked pre-weighed XAD-2 trap), a set of four glass impingers connected in series in an ice bath, and a control module consisting of a leak free sampling pump, a calibrated critical orifice, an inclined manometer, and a calibrated dry gas meter. A Teflon fitting connected the nozzle to the probe liner. The probe and oven box were maintained at a temperature of 248+25°F during sampling to prevent moisture condensation. The first impinger was empty with the next two impingers each containing 100 ml of deionized water. The fourth impinger contained a known quantity of silica gel. The second impinger used a standard Greenburg-Smith tip, while the other impingers have modified straight tips. The glass components of the sampling train including the adsorbent module were cleaned in strict accordance with the procedures of EPA Method 23. Silicone grease is not used on any GamAir sampling trains. The spiked adsorbent traps were loaded with XAD resin by the subcontracted analytical laboratory prior to being shipped to the test site. The fourth impinger outlet temperature was maintained below 68°F during sampling by adding ice. The coil condenser was cooled by circulating water to and from the cold box using a peristaltic pump. The adsorbent module was also maintained at a temperature less than 68°F during sampling.

Before each test the sampling train was leak checked to ensure no leakage greater than 0.02 cubic feet per minute at 10" Hg vacuum. The probe was placed in the stack and stack gas was

withdrawn isokinetically for an equal period of time at each traverse point. The velocity differential pressure, critical orifice differential pressure, dry gas meter volume, dry gas meter outlet temperatures, probe temperature, stack temperature, oven box temperature, impinger outlet temperature, and sample vacuum were recorded at each traverse point during sampling. At the completion of each test, the sampling train was leak checked to ensure no leakage greater than 0.02 cubic feet per minute at the highest recorded test vacuum.

Following the post test leak check, the train was disassembled and all open ends are sealed with cleaned Teflon tape then again with hexane rinsed aluminum foil. The sample recovery and analysis procedures are as follows. The filter was carefully removed from the filter holder with tweezers and placed in a labeled glass sample container (Container 1). Any particulate matter or filter fragments, which adhered to the filter holder gasket, were placed into Container 1. The adsorbent module (Container IA) was post weighed, wrapped in Teflon tape and hexane rinsed aluminum foil and stored on ice for transport to the analytical laboratory. The nozzle, probe liner, and filter holder front half were rinsed three times with acetone (Container 2) then toluene (Container 2). These rinses were collected in a labeled sample jar (Container 2). The filter holder back half, the connecting glassware between the filter and the condenser, and the coil condenser were soaked with three separate rinses of toluene for 5 minutes each. These rinses were combined in Container 2, which was sealed and the liquid level marked.

The moisture gain was determined by gravimetrically weighing the XAD module and the first three impingers prior to their recovery. The silica gel impinger was also weighed for moisture gain. The collected samples were submitted to Maxxam Analytics, Inc. for analysis. The combined solvent rinses (acetone and toluene), filters and XAD resins were extracted prior to analysis. The PCDD's and PCDF's were separated by high resolution gas chromatography (HRGC) and measured by high resolution mass spectrometry (HRMS).

4.5 Biosolids Tests

At the completion of each test run biosolid sample containers were labeled with the test number and sampling period. A biosolids sample was collected a half hour prior to the start of the test, at the start of the test, at the mid-point of the test, and at the end of the test. At the end of each test run the sample was composited into one biosolids sample. Each composited sample was analyzed for total solids content in accordance with Standard Method 2540B. A total of three biosolids samples will be analyzed for the three PCDD/PCDF/metals tests and three biosolids samples will be analyzed for the three HCl/FPM tests. The percent solids and the average wet biosolids feed rate, for each 84 or 120 minute test run, was used in calculating the dry tons per hour feed rate for that particular test run.

4.6 Fugitive Emissions Tests

Three 60-minute measurements of fugitive emissions from the ash handling area were performed in accordance with EPA Method 22. The accumulated emissions time of fugitive emissions was determined by observing the process area(s) during normal operations for a pre-determined observation period (60 minutes). This method does not require that the opacity of emissions be determined, but rather the length of time any fugitive emissions are visible. If any fugitive emissions were observed during the observation period, the length of time that the emissions were visible was quantified using a stopwatch. The total accumulated time of fugitive emissions is then used to determine compliance.

4.7 Fluoride Tests

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Fluoride (F⁻) emissions were sampled and analyzed in accordance with EPA Method 13B. Three 84 minute tests were performed for compliance determination on Unit No. 8 while one 84 minute test was performed on Unit No. 7 (see section 3.2 for further explanation). This sampling train is shown in Appendix A. The Method 13B sampling train consists of a Pyrex® glass nozzle, a heated glass lined sample probe, a heated quartz filter (untared), a set of four tared glass impingers connected in series in an ice bath, a control module consisting of a leak free sampling pump, a calibrated critical orifice, an inclined manometer, and a calibrated dry gas meter.

The first two impingers each contained 100 ml of deionized distilled water while the third impinger was empty. The fourth impinger contained a known quantity of silica gel. The sampling train glassware was cleaned prior to testing with soap and water and rinsed thoroughly with water. The sample probe and oven box were maintained at a temperature of $248\pm25^{\circ}$ F during sampling to prevent moisture condensation. The impinger outlet temperature was maintained below 68°F during sampling by adding ice to the cold box. A vacuum line is connected from the outlet of the fourth impinger to the control module.

Before each test, the sampling train was leak checked to ensure no leakage greater than 0.02 cubic feet per minute at 10" Hg vacuum. The probe was then placed in the stack and stack gas was withdrawn isokinetically for an equal period of time at each traverse point with a sampling rate not exceeding 1.0 cfm.

The velocity differential pressure, critical orifice differential pressure, dry gas meter volume, dry gas meter outlet temperatures, probe temperature, stack temperature, oven box temperature, impinger outlet temperature, and sample vacuum were recorded at each traverse point during sampling. At the end of each test, the sampling train was leak checked to ensure no leakage greater than 0.02 cubic feet per minute at the highest recorded test vacuum.

Sample recovery consisted of brushing and rinsing the nozzle, probe, and filter holder front half three times with deionized distilled water. These water rinses were collected in Container 1. The untared quartz filter was recovered in a clean laboratory area and placed in Container 1. The first three impingers and connecting glassware were also rinsed at least three times with deionized distilled water. These rinses were also collected in Container 1. The sample containers were sealed and the liquid levels marked. The fluoride samples were shipped to Maxxam Analytics, Inc. for analysis. Fluoride analysis was performed by specific ion electrode ion (SIE) as described in EPA Method 13B.

4.8 Continuous Emissions Monitoring Tests

Carbon monoxide (CO), oxides of nitrogen (NO_x), carbon dioxide (CO₂), volatile organic compounds (VOC), sulfur dioxide (SO₂), and oxygen (O₂) were measured with a properly calibrated, transportable, continuous emissions monitoring system (CEMS). Sampling and analysis occurred at the outlet stack from each MHI. A daily instrument calibration check and

individual test run system calibration check was performed on each analyzer in accordance with EPA Methods 3A, 6C, 7E, 10, and 25A. A schematic diagram of the CEM sampling train is shown in the Appendix. Triplicate 60-minute test runs for SO₂, NO_x, CO, VSC, CO₂ and O₂ were performed with the data recorded in one-minute averages. The average JAN concentration over the 60-minute test period constituted a single test run. 0

The NO_x analyzer was a Teledyne/API Model T200H. This NO_x analyzer is based of the principles of chemiluminescence and was equipped with a NO₂ to NO converter.

4 2018 The carbon dioxide analyzer was a Teledyne/API Model T300M. The theory of operation for the CO₂ analyzer is based on the principle that every gas has a unique absorption line spectrum in the infrared region. The instrument consists of an infrared light source, a chopper, a measurement cell, and a detector.

The VOC analyzer was a VIG Industries, Inc. Model 20. The VIG analyzer was a heated hydrocarbon analyzer, which detects concentrations of total hydrocarbons (THC) in a sample stream by using a flame ionization detector (FID). THC in the gas stream was ionized by burning them in a hydrogen/helium flame. The burner tip was positioned between two highly charged (approximately300 VDC) plates. Ions were produced from the combustion of the THC in the gas stream which creates a current through migration of the ions between the highly charged plates. The current created was directly proportional to the concentrations of hydrocarbons present in the gas stream. Volatile organic compounds consisted of the total hydrocarbon content. Any methane present in the exhaust gas was measured as total. Methane was not subtracted from the total hydrocarbon content.

Continuous oxygen measurements were performed with a Teledyne/API Model T200H analyzer. The Model T200H oxygen analyzer is housed in the same instrument case as the NO_x analyzer and uses a paramagnetic concept where oxygen molecules are attracted by a magnetic field.

The CO analyzer was a Teledyne/API Model T300M. The Model T200H carbon monoxide analyzer is housed in the same instrument case as the CO₂ analyzer. The Model T300M employs gas filter correlation (GFC) to measure CO by infrared (IR) absorption. GFC uses a correlation wheel consisting of two hemispherical cells, one filled with CO and the other filled with nitrogen (N₂). Radiation from the IR source was chopped and passed through the correlation wheel, alternating between the CO cell and the N2 cell. Radiation then passed through a narrow bandpass interference filter and enters a multiple optical pass sample cell, where absorption by the sample gas occurred. Other gases in the sample do not cause modulation of the detector signal, since they absorb the reference and measurements beams equally.

A Teledyne/API Model 100H fluorescent analyzer was used to measure SO₂. The instrument operates on the principle of fluorescence, by excitation of sulfur dioxide molecules by pulsing high intensity ultraviolet (UV) light. The resulting fluorescence is measured by a photo multiplier tube (PMT) sensitive in the near UV. The signal generated is an analog voltage

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linearly proportional to the sulfur dioxide concentration in the gas stream.

Instrument calibrations were performed with NIST certified compressed gas mixtures of SO₂, CO, NO_x, CO₂, methane, and O₂ in nitrogen. A three or four point analyzer calibration error (<2%) check was conducted, prior to testing, by injecting the calibration gases directly into the gas analyzers and recording the responses. Zero and upscale calibration checks were conducted both before and after each test run in order to quantify measurement system calibration drift (<3%) and sampling system bias (<5%). During these checks, the calibration gases were introduced into the sampling system at the probe outlet so that the calibration gases were analyzed in the same manner as the flue gas samples. Drift was the difference between the pre- and post-test run calibration check responses. Sampling system bias was the difference between the test run calibration check responses (direct analyzer calibration) and the initial calibration error responses (direct analyzer calibration) to the zero and upscale calibration gases. For a test run to be valid, the system bias and calibration drift results must meet the specifications in EPA Method 7E and 25A. The NO_x stratification test was performed at three sample points.

С	Continuous Emissions Monitoring Instrumentation				
Parameter	Manufacturer	Model Number	Span Values		
O ₂	API	Т200Н	21.44 %		
CO ₂	API	T300M	17.98 %		
VOC	VIG	20	89 ppm		
NO _x	API	T200H	448 ppm		
SO ₂	API	100H	91 ppm		
СО	API	T300M	4721 ppm		

Instrument spans are shown in the following table.

Using Equation 7E-5 (Title 40, <u>CFR</u>, Part 60, Appendix A), the parts per million (ppm) and percent values were corrected to account for the zero and span instrument drift as follows:

$$C_{gas} = (\overline{C} - C_o) x \frac{C_{ma}}{C_m - C_o},$$

Where:

= emissions concentration (ppm or %)

= average emissions reading (ppm or %)

- $C_o = average zero reading (ppm or %)$
- C_m = average span reading (ppm or %)

Ceas

 $\overline{\mathbf{C}}$

C_{ma} = span gas concentration (ppm or %)

EPA Method 25A was used to calculate emissions on a methane and propane (concentration and pound per hour) basis.

4.9 Particulate Matter Less Than 10 Microns and Less Than 2.5 Microns

Testing for particulate matter less than 10 microns (PM10), particulate matter less than 2.5 microns (PM2.5) and condensable particulate matter (CPM) was performed in accordance with EPA Test Methods 201A and 202, respectively. Three 120-minute isokinetic tests were performed on Unit No. 8 while one test was performed on Unit No. 7 (see section 3.2 for further explanation). Nozzle selection was of the proper size to maintain a cut diameter size of 10.0 microns/2.5 microns and isokinetics between 80% to 120%. Flue gases were extracted at a constant flowrate (Δ H) through the PM10/2.5 sizing device. Sampling dwell times at each of the twelve sampling points was calculated from the preliminary velocity traverse and Equation 23 of Method 201A. A schematic of the EPA Method 201A/202 sampling train is shown in Appendix A. The PM10/2.5 sampling train consisted of a stainless steel nozzle, in-stack PM10/2.5 cyclone sizing device, in-stack tare weighed fiberglass filter, and heated (248°F±25°) glass lined probe. The moisture removal portion of this sample train collected the CPM and consisted of the following components. Exiting the heated probe was a heated filter bypass, a water jacketed glass condenser, two empty impingers, an unheated 81mm Teflon filter (equipped with a thermocouple to monitor exit gas temperature), two impingers immersed in an ice bath, a vacuum pump, a calibrated dry gas meter, and calibrated orifice. The four impingers contained the following solutions: 1st and 2nd impingers were empty, 3rd impinger 100 milliliters of DI water, and the 4th impinger contained 250 grams of silica gel. Sampling train operating parameters were recorded at each traverse point on preprinted data sheets as shown in Appendix C.

Prior to the start of the first PM10/2.5 test a full 12 point velocity traverse was conducted with the entire PM10/2.5 sampling head and S-type Pitot tube assembly. Velocity data collected from this preliminary traverse allowed for the proper set-up and sampling scheme for this test method. Once the sizing device had reached stack temperature conditions sampling began. Pre and posttest sample train leak checks were performed on each test run. The post test leak check requires that the PM10/2.5 sizing device be removed prior to the leak check. The post test leak check was then conducted from the Teflon union at the end of the sample probe. This procedure ensured that fine particulate matter was not disturbed or altered within the sampling device. A successful leak check was less than 0.02 cubic feet in one minute. At the completion of each test run the impingers and PM10/2.5 cyclone/filter assembly were removed to the sample recovery area. The entire impinger train was then purged with zero grade nitrogen for a period of 1 hour at an approximate flow rate of 14 liters per minute. At the completion of the nitrogen purge the contents of the 4 impingers were measured for moisture gain. The recovery process for CPM then began.

PM10 and PM2.5 sample recovery procedures require the sizing device cyclones, and throat be brushed and rinsed three times with acetone. The rinses were collected in the following container.

- Container 1 glass fiber filter and any loose particulate matter (< PM2.5 fraction)
- Container 2 acetone rinses from PM10 turn around cup, downcomer throat, connector to 2.5 cyclone, and 2.5 cyclone internal surfaces, (<10 PM10 and >PM2.5 fraction).
- Container 3 acetone rinses from the 2.5 cyclone exit tube and front half of the filter holder (< PM2.5 fraction).

Sample containers 1-3 were brought back to the GamAir laboratory for gravimetric analysis per EPA Method 5 procedures. A minimum of duplicate weighings was performed on each sample fraction in order to achieve constant weight conditions. If weighings differed by more than 0.0005 grams from one weighing to the next the procedure was repeated until two consecutive weighings were no more than 0.0005 grams apart (per EPA Method 5 specifications). Upon completion of all final acetone residue and filter weighings the PM10 and PM2.5 fractions were reported. The PM10 fraction consisted of the sum of the net weight gain from Containers 1, 2, & 3. PM2.5 fraction consists of the combined net weight gain from Containers 1 and 3.

CPM sample recovery procedures consisted of collecting the moisture gained in impingers 1 and 2, the unheated filter, the acetone, hexane, and DI water rinses of the probe, connecting glassware, condenser, first two impingers, filter holder front half section, and connecting glassware. The following is a summary of the CPM sample recovery containers.

- Container 1 contents of impingers 1 and 2 plus deionized/distilled water rinses from the connecting glassware, spiral condenser, impingers, and filter holder front half.
- Container 2 duplicate rinses of both acetone and hexane from the connecting glassware, spiral condenser, impingers, and filter holder front half.
- Container 3 unheated Teflon filter

Containers 1, 2, and 3 were shipped to Maxxam Analytics, Inc. for EPA Method 202 condensable particulate matter gravimetric analysis. The CPM was reported as two separate fractions; inorganic condensables consisted of the residue collected in Container 1; organic condensables consisted of the residue collected in Containers 2 and 3. Total condensable particulate matter was reported as the sum of the inorganic condensables and the organic condensables. The results were not blank corrected. For reporting purposes total PM2.5 emissions equal the sum of the PM2.5 and CPM. Total PM10 emissions equal the sum of the PM10, PM2.5, and CPM.

4.10 Sulfuric Acid Mist Tests

The sulfuric acid mist (H₂SO₄) emissions were sampled and analyzed in accordance with EPA Method 8. Three 84-minutes tests were performed for compliance determination. This sampling train is shown in Appendix A. The Method 8 sampling train consisted of a Pyrex® glass nozzle, a heated glass lined sample probe, a set of four tared glass impingers connected

in series in an ice bath, an unheated filter (glass wool) between the first and second impingers, a control module consisting of a leak free sampling pump, a calibrated critical orifice, an inclined manometer, and a calibrated dry gas meter.

The first impinger contained 100 ml of 80 percent isopropanol (IPA) while the second and third impingers each contained 100 ml of 3 percent hydrogen peroxide. The fourth impinger contained a known quantity of silica gel. The sampling train glassware was cleaned prior to testing with soap and water and rinsed thoroughly with water. The IPA was checked for peroxide impurities by the potassium iodide procedure identified in EPA Method 8 prior to field use. The IPA solution did not contain any peroxide impurities. The sample probe and oven box were maintained at a temperature of $248\pm25^{\circ}$ F during sampling to prevent moisture condensation. The impinger outlet temperature was maintained below 68°F during sampling by adding ice to the cold box. A vacuum line was connected from the outlet of the fourth impinger to the control module.

Before each test, the sampling train was leak checked to ensure no leakage greater than 0.02 cubic feet per minute at 10" Hg vacuum. The probe was then placed in the stack and stack gas was withdrawn isokinetically for an equal period of time at each traverse point with a sampling rate not exceeding 1.0 cfm.

The velocity differential pressure, critical orifice differential pressure, dry gas meter volume, dry gas meter outlet temperatures, probe temperature, stack temperature, oven box temperature, impinger outlet temperature, and sample vacuum were recorded at each traverse point during sampling. At the end of each test, the sampling train was leak checked to ensure no leakage greater than 0.02 cubic feet per minute at the highest recorded test vacuum. After completion of sampling the sample probe was disconnected from the train and clean ambient air was drawn through the impingers for 15 minutes at the average flow rate used for sampling.

Sample recovery consisted of rinsing the nozzle, probe, and filter holder front half three times with 80% IPA. These rinses were collected in Container1. The contents of the first impinger was measured gravimetrically for moisture gain then transferred to sample Container 1. The first impinger and first U-tube connector were rinsed with 80% IPA and added to the impinger catch (Container 1). The sample was then diluted to 250-ml with 80% IPA with the unheated glass wool filter added to Container 1. The second and third impingers were measured gravimetrically for moisture gain then discarded. The sample containers were sealed and the liquid levels marked. The silica gel impinger was also weighed for moisture gain. The silica gel was returned to its original storage container to be dried for reuse. The samples were shipped to Maxxam Analytics, Inc. for analysis. Container 1 was analyzed for sulfuric acid mist by the barium-thorin titration method as described in EPA Method 8.

4.11 Sample Identification and Custody

Sample custody procedures for this program were based on EPA recommended guidelines. The project manager was responsible for ensuring that proper custody and documentation procedures were followed for the field sampling and field analytical efforts. The project manager was assisted in this effort by key sampling personnel involved in sample recovery.

All sampling data, including information regarding sampling times, locations, and any

specific considerations associated with sample acquisition were recorded in black ink on pre-formatted data sheets. A master sample logbook was used to document all sample collection activities.

Following sample collection, all samples were given a unique alphanumeric sample identification code. Sample labels and integrity seals were completed and affixed to the sample container. The sample volumes were recorded and the liquid level on each bottle marked. All samples were packed and shipped according to Department of Transportation (DOT) guidelines. As the samples were packed for shipment to the appropriate laboratories, chain-of-custody forms were completed for each shipment box, these forms were enclosed in each respective box.

5.0 QUALITY ASSURANCE/QUALITY CONTROL PROCEDURES

5.1 QC Procedures

Specific quality control (QC) procedures were followed to ensure the continuous production of useful and valid data throughout the course of this test program. The QC checks and procedures described in this section represent an integral part of the overall sampling and analytical scheme. Pre-printed data sheets were used for each individual test method. These individual data sheets acted as QC check lists and were verified for completeness by the project manager. A discussion of both the sampling and analytical QC checks that were utilized during this program is presented below.

5.2 Sampling Equipment Calibrations

Emissions sampling equipment requiring calibration included sampling nozzles, Pitot tubes, pressure gauges, thermometers, dry gas meters, and dry gas meter orifice. Post-test calibration of the dry gas meters were all within their acceptable limit (Y = 1.00 + 0.05). Copies of all equipment calibrations are contained in Appendix F.

5.3 Sampling and Laboratory Analytical QA/QC

All analyses for this program were performed using accepted laboratory procedures in accordance with the specified analytical protocols. Adherence to prescribed QC procedures for the EPA methods used for this test program ensured data of consistent and measurable quality. Analytical QC focused on the use of control standards to provide a measure of analytical precision and accuracy. Laboratory method blanks, spike recovery, and duplicate analyses were performed where applicable and met their respective targeted ranges. A summary of the quality assurance and quality control results for this test program are shown in Table 5-1. Both sampling and analytical QA/QC results are shown

5.3.1 Continuous Emission Monitoring QA/QC

Calibration gases of nitric oxide, carbon monoxide, sulfur dioxide, propane, carbon dioxide, and oxygen were analyzed and certified per EPA Protocol Mixture Procedure No. G1 (traceable to the National Institute of Standards and Technology). Each analyzer met their respective calibration error (<2%), system bias error (<5%), and system drift (<3%) for each of the individual test runs. NO₂ converter efficiency checks for both units were greater than the required 90% efficiency.

5.3.2 PM10/2.5 and CPM QA/QC

All PM2.5 filters and acetone rinses were analyzed by GamAir in accordance with EPA Method 5 procedures (constant weight). All daily weight calibrations met their limits. Acetone residues and filters were weighed to a constant weight per EPA Method 5 procedures. The blank deionized distilled water (inorganic) result showed 1.8 milligrams (mg) while the blank acetone/hexane (organic) result showed 4.0 milligrams. Total inorganic analytical results ranged from 1.5 mg to 3.8 mg. Test results were not blank corrected.

Table 5-1		
Quality Assurance/Quality Control Sum	mary	
Detroit Water and Sewerage Department - Unit	Nos. 9 and 10	
Detroit, Michigan		

Detroit, Michigan					
EPA Test Method	QC Criteria	Actual QC Results			
Methods 3A, 6C, 7E, and 10		Unit 9/Unit 10			
Calibration Gases	EPA Traceability Protocol	Yes/Yes			
Analyzer Calibration Error	Less than 2% of the span	Yes/Yes			
System Calibration Bias	Less than 5% of span	Yes/Yes			
Instrument Drift	Less than 3% of span	Yes/Yes			
Instrument Drift	Less than 3% of span	Yes/Yes			
NO ₂ -NO Conversion Eff.	Greater than 90%	93.2% & 98.4%			
Method 26A					
Matrix Spike Recovery	80-120%	94%			
Spiked Blank Recovery	90-110%	98%			
Method Blank	Below detection limit	<200 micrograms			
Isokinetics	90% < I < 110%	Yes/Yes			
Method 29 (Cd, Pb, Hg)					
Method Blanks	At or below detection limits	Yes for all 3 metals			
Matrix Spike Recovery	85-115%	Yes within range			
Spiked Blank Recovery	90-110%	Yes within range			
Relative Percent Difference	Less than 20%	Yes for all metal fractions			
Isokinetics	90% < I < 110%	Yes/Yes			
Method 23					
Spiked Blank Recovery	CDF 40-130%	68 to 121%			
	CDD 80-140%	91 to 133%			
Method Blanks	At or below detection limits	Yes all CDD and CDF			
Isokinetics	90% < I < 110%	Yes/Yes			
Method 201A/202 (PM10/2.5)					
Isokinetics	80% < I < 120%	All within +/- 10% of 100			
Cut Diameter Range 10 µm	9.0 $\mu m \le D_{50} \le 11.0 \ \mu m$	10.56 μm to 11.21 μm			
Cut Diameter Range 2.5 µm	$2.0 \ \mu m \le D_{50} \le 3.0 \ \mu m$	2.27 μm to 2.58 μm			
Spiked Blank	70-130%	95%			
Sulfuric Acid					
Matrix Spike Recovery	80-120%	99%			
Spiked Blank Recovery	90-110%	101%			
Method Blank	Below detection limit	<0.2 milligrams			
Isokinetics	90% < I < 110%	Yes/Yes			

5.3.3 Hydrochloric Acid QA/QC

One HCl audit was submitted with the six samples. Spiked blank recoveries were all within their respective QC limits. Method blanks were all below the reportable detection limits.

5.3.4 Multiple Metals QA/QC

Three metals were analyzed per EPA Method 29. Two metals (cadmium and lead) were analyzed by ICP/MS (Method 6020); mercury fractions were analyzed by CVAA (Method 29 and 0060). Two audit samples were submitted with the six samples. Each matrix spike was within the acceptable QC limit of 85-115%. Spiked blanks were also within acceptable QC limit of 85-115%. Method blanks were below their reportable detection limits (RDL) and the highest relative percent difference of 7.1 percent was below the QC limit of less than 20%.

5.3.5 PCDD/PCDF QA/QC

Confirmation of 2,3,7,8 tetra CDF was performed with the DB-225 column for all six samples. 2,3,7,8 tetra CDF results are from the confirmation analysis not the regular GC analysis. Surrogate spike recoveries were within the acceptable range of 40-130% and 80-140%. Relative percent differences (RPD) were all below the 20% limit. Method blanks were below their respective limits of detection.

5.3.6 Sulfuric Acid QA/QC

Spiked blank recoveries were all within their respective QC limits. The method blank was the reportable detection limits of < 0.2 milligrams.

5.4 Calculations Quality Control Procedures

Calculations for determining flow rates, moisture contents, isokinetics, particulate and gaseous concentrations were made using spreadsheets developed by GamAir. The spreadsheets utilize the calculation procedures and equations specified in EPA Methods 2, 4, 5, 7E and 201A. The spreadsheets have been successfully used for numerous test efforts and have been validated by independent performance audits. Sample calculations were performed and a definition of terms is provided in the final report.

5.5 QA Audits

There were four audit samples presented for analysis: fluoride (Cat #1441) ERA Project #091515F, metals (Cat #1425), mercury (Cat #1427), and hydrogen halides (Cat #1440) ERA Project #091815O. Audit samples were provided by ERA through the Stationary Source Audit Testing program. All audits were acceptable and met ERA requirements. All audit results are shown in the Maxxam Analytics, Inc. reports shown in Appendix E.

5.6 QA/QC Checks of Data Reduction

The project manager ran an independent check (using validated spreadsheets) of the calculations with predetermined data before the field test. This ensured that calculations done in the field were accurate.

Appendix A

Figures and Diagrams