BOILER NUMBER ONE EPA SECTION 114 INFORMATION REQUEST EMISSIONS TEST REPORT



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L'ANSE WARDEN ELECTRIC COMPANY, LLC. 157 South Main Street

L'Anse, Michigan 49946

August, 2016

W.O. No. 14464.007.004

IASDATA\LWEC\14464.007.004\EPA 114 LETTER REPORT-LW



MICHIGAN DEPARTMENT OF ENVIRONMENTAL QUALITY AIR QUALITY DIVISION

RENEWABLE OPERATING PERMIT REPORT CERTIFICATION

Authorized by 1994 P.A. 451, as amended. Failure to provide this information may result in civil and/or criminal penalties.

Reports submitted pursuant to R 336.1213 (Rule 213), subrules (3)(c) and/or (4)(c), of Michigan's Renewable Operating (RO) Permit program must be certified by a responsible official. Additional information regarding the reports and documentation listed below must be kept on file for at least 5 years, as described in General Condition No. 22 in the RO Permit and be made available to the Department of Environmental Quality, Air Quality Division upon request.

Source Name L'Anse Warden Electric Company LLC	County Baraga					
Source Address _ 157 S. Main Street	City _L'Anse					
AQD Source ID (SRN) B4260 RO Permit No. MI-ROP-B4260-2011	RO Permit Section No.					
Please check the appropriate box(es):						
Annual Compliance Certification (General Condition No. 28 and No. 29 of the I	RO Permit)					
Reporting period (provide inclusive dates): From To						
1. During the entire reporting period, this source was in compliance with ALL terms and conditions contained in the RO Permit, each term and condition of which is identified and included by this reference. The method(s) used to determine compliance is/are the method(s) specified in the RO Permit.						
2. During the entire reporting period this source was in compliance with all terms each term and condition of which is identified and included by this reference, E enclosed deviation report(s). The method used to determine compliance for each term the RO Permit, unless otherwise indicated and described on the enclosed deviation	and conditions contained in the RO Permit, XCEPT for the deviations identified on the erm and condition is the method specified in report(s).					
Semi-Annual (or More Frequent) Report Certification (General Condition No. 2	23 of the RO Permit)					
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the supporting enclosures are true, accurate and complete.

Steve Walsh	Chief Executive Officer	906-885-7910
Name of Responsible, Official (print or type)	Title	Phone Number
Steve Walsh		4 Aug 2016
Signature of Responsible Official		Date

1. INTRODUCTION

Weston Solutions, Inc. (WESTON) was retained by L'Anse Warden Electric Company, LLC (LWEC) to perform an emissions testing program on the Boiler No. 1 exhaust duct at the LWEC facility located in L'Anse, Baraga County, Michigan. Boiler No. 1, which is permitted to operate on several different biomass or renewable fuels, was previously a coal, oil, and gas-fired steam generating unit. The objective of this test program was to satisfy the requirements of the U.S. Environmental Protection Agency (EPA) Region V Section 114 Information Request submitted on 1 April 2016. Boiler No. 1 is identified as EUBOILER No. 1, and the facility currently operates under the State of Michigan Renewable Operating Permit (ROP) No. MI-ROP-B4260-2011 and Permit to Install (PTI) 168-07D.

The EPA Region V 114 letter initially requested emissions testing under two operating conditions. Test condition one included a typical fuel mix, under the existing permitting, of wood, tire derived fuel (TDF), wood from creosote treated railroad ties and pentachlorophenol (PCP) treated railroad ties. Test condition two was the same as test condition one but excluded the use of PCP ties. However, LWEC has discontinued the use of PCP tie fuel and has submitted a permit application (PTI Application No. 67-16) to the Michigan Department of Environmental Quality (MDEQ) to remove PCP ties as an authorized fuel. As LWEC no longer had PCP ties available for combustion and submitted a permit application to remove PCP ties as a fuel, EPA Region V modified its 1 April 2016 request to include only test condition two. The resulting Section 114 Test Program was conducted pursuant to the EPA Region V approved test protocol submitted May 17, 2016 and the test protocol addendum submitted June 22, 2016.

WESTON's Integrated Air Services (IAS) group completed all required testing during 6-7 July, 2016. A representative of the MDEQ was present throughout the testing.

1.1 PLANT INFORMATION

L'Anse Warden Electric Company, LLC 157 South Main Street L'Anse, Michigan 49946 Mr. JR Richardson, Technical Manager Phone: 906-885-7187

Test Parameter ⁽¹⁾	Test Method ⁽²⁾	Reporting Units ⁽³⁾
Total Particulate (filterable)	EPA M5	gr/dscf, lb/MMBtu, lb/hr
	(combined with EPA M29)	
PM ₁₀ /PM _{2.5} (filterable and condensable)	EPA M201A/202	gr/dscf, lb/MMBtu, lb/hr
Metals (nickel, lead, arsenic, manganese)	EPA M29	ug/m³, lb/hr
Polychlorinated Dibenzo-p-dioxins/ Polychlorinated Dibenzofurans (PCDD/PCDF)	EPA M23	ug/m ³ @ 7% O ₂ TEQ, lb/hr TEQ
Cresol Isomers	EPA SW846 M0010 (combined with EPA 23)	ug/m ³ , lb/hr
Hydrogen Chloride/Chlorine	EPA M26A (modified)	ppmvd, lb/hr
Volatile Organic Compounds (VOCs) as methane	EPA M25A	ppmvd @ 7% O ₂ , lb/hr
Opacity	EPA M9	%

Table 1-1Summary of Test Parameters

1. Cresol isomers include m-cresol, o-cresol and p-cresol.

2. EPA Method 26A was modified by collecting the sample non-isokinetically from a single traverse point (similar to EPA Method 26).

3. The exhaust gas O₂ concentration (diluent gas) and a facility provided F-factor (F_d 9561) were used to calculate emission rates in terms of lb/MMBtu.

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2. SUMMARY OF TEST RESULTS

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2.1 TEST RESULTS DISCUSSION

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Table 2-1 of this section provide a summary of the compliance test results for each pollutant parameter [particulate matter (PM), particulate matter ≤ 10 microns and PM ≤ 2.5 microns (PM₁₀/PM_{2.5}), metals (includes Pb, As, Mn, and Ni), hydrogen chloride (HCl) and chlorine (Cl), polychlorinated dibenzo-p-dioxins/polychlorinated dibenzo furans (PCDD/PCDF) as 2,3,7,8-TCDD Toxic Equivalent, cresol isomers, volatile organic compounds (VOC)], and opacity. Any differences in the test results summary tables and detailed test results shown in the appendices are due to rounding the results for presentation purposes.

As discussed in the Addendum to Emissions Test Protocol (Revision 2, June 2016), WESTON calculated the stack exit velocity and recorded the gas temperature at the stack inlet duct. The stack exit velocity (in terms of ft/s) and stack inlet duct temperature data can be found in the detailed results tables presented in Appendix A.

It should be noted WESTON experienced sampling difficulties during the first $PM_{10}/PM_{2.5}$ run conducted on 6 July. Due to a misaligned pitot tube/PM sampling head assembly, the measured stack gas velocity head (ΔP) readings were lower than the preliminary traverse readings resulting in a low-biased calculation of volumetric flow rate and subsequent PM mass rate in terms of lb/hr. Since it was believed the results may not be representative and biased low, WESTON elected not to analyze the sample and attempted a repeat of the run on 7 July. During the repeated run WESTON inadvertently broke the glass sample probe while changing test ports and after a discussion with Mr. Tom Gasloli of the MDEQ, a decision was made to scrap the run and start over. WESTON successfully repeated the run later that morning and completed all $PM_{10}/PM_{2.5}$ testing on 7 July 2016. Please note the PM runs are numbered Runs 2-4 throughout the report vs.1-3 for all other sample trains.

There were no other sampling or operational issues that impacted the field testing and the results presented are believed to be representative of the emissions encountered during the test periods.

Table 2-1 Boiler No.1 Summary of Test Results

Dellutert	Test Run Number					PTI 168-07D
ronutant	1	2	3	4	Average	Emissions Limit
Particulate Matter (PM) (lb/hr)	0.8	1.9	1.2		1.3	19.2 lb/hr
Particulate Matter (PM) (lb/MMBtu)	0.003	0.006	0.004		0.004	0.06 lb/MMBtu
Particulate Matter ≤ 10 microns (PM ₁₀) (lb/hr)		5.3	8.0	8.3	7.2	15.4 lb/hr
Particulate Matter ≤ 10 microns (PM ₁₀) (lb/MMBtu)		0.020	0.029	0.030	0.026	
Particulate Matter ≤ 2.5 microns (PM _{2.5}) (lb/hr)		4.7	7.3	7.1	6.3	
Particulate Matter ≤ 2.5 microns (PM _{2.5}) (lb/MMBtu)		0.018	0.027	0.026	0.023	
Lead (Pb) (lb/hr)	1.19E-03	1.00E-03	1.13E-03		1.10E-03	0.02 lb/hr
Arsenic (As) (lb/hr)	< 1.24E-04	1.41E-04	1.43E-04		≤ 1.36E-04	
Manganese (Mn) (lb/hr)	1.51E-03	2.88E-03	2.87E-03		2.42E-03	
Nickel (Ni) (lb/hr)	1.20E-03	4.70E-04	6.04E-04		7.60E-04	
Hydrogen Chloride (HCl) (lb/hr)	1.73	1.91	1.61		1.75	2.17 lb/hr
Chlorine (Cl ₂) (lb/hr)	< 0.25	< 0.26	< 0.26		< 0.26	
2,3,7,8-TCDD Toxic Equivalent (µg/dscm @ 7% O ₂)	7.72E-06	6.35E-06	5.70E-06		6.59E-06	
2,3,7,8-TCDD Toxic Equivalent (lb/hr)	2.06E-09	1.66E-09	1.54E-09		1.75E-09	wam
Volatile Organic Compounds (ppmvd @ 7% O_2) as methane	< 0.12	< 0.12	< 0.12		< 0.12	50 ppmvd @ 7% O ₂
Volatile Organic Compounds (lb/hr) as methane	< 0.02	< 0.02	< 0.02		< 0.02	9.1 lb/hr
Cresol Isomers (lb/hr)	< 7.77E-04	< 8.44E-04	< 8.15E-04		< 8.12E-04	
Opacity	0	0	0		0	
Average Stack Exit Velocity, 6 July (ft/s) ¹	57.6					
Average Stack Exit Velocity, 7 July (ft/s) ¹	56.1					
Average Stack Inlet Duct Temp, 6 July (°F) ¹	442.3					
Average Stack Inlet Duct Temp, 7 July (°F) ¹	439.4					

1. See Appendix A for detailed exit velocity and temperature data.

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3. DESCRIPTION OF PROCESS AND SAMPLING LOCATIONS

3.1 PROCESS OVERVIEW

LWEC is a cogeneration facility, consisting of a single boiler generating process steam and electric power to the grid firing primarily biomass materials. The boiler typically produces steam at 180,000 lbs/hr and maximum gross power generation from 15.9 to 16.4 megawatts (MW).

3.1.1 Basic Operating Parameters

The fuel feed to the boiler is regulated to meet process steam and electrical generation requirements. The fuel blend and excess air may be modified to improve combustion characteristics. Adjustments to air, fuel blend or load will be made as necessary to conform to emissions monitoring limits.

3.1.2 Test Program Boiler Load

The hourly boiler operating limit is 324 million British thermal units (MMBtu). The maximum annual heat input is 2,656,800 MMBtu, based on 8,200 hours of operation per year.

As noted in the Addendum to Emissions Test Protocol (Revision 2, June 2016), the boiler "maximum rate of electricity production" for the stack test was determined by calculating an average gross annualized MW range for the years 2012 to 2015 (ranging from 15.92 to 16.37 MW). The boiler load was maintained within this range during the Section 114 Information Request Test Program.

3.1.3 Test Program Fuel Mix and Firing Rates

The fuel mix during the Section 114 Test Program consisted of wood, creosote treated railroad ties, and TDF at a target feed rate of 15 tons per hour for creosote treated railroad ties and 7.5 tons per hour for wood (i.e. at a 2:1 ratio of creosote treated railroad ties to wood). As required by the 114 Request, fuel samples were collected during the test program during each test run in accordance with 40 CFR 63 Subpart 7521(c and d). However, as noted in the Addendum to Emissions Test Protocol (Revision 2, June 2016), due to safety and operational necessity the belt

was not stopped to collect fuel samples; LWEC designated personnel collected fuel samples from a point where each fuel drops onto the conveyor belt feeding the boiler.

To calculate the feed rates during the Section 114 Test Program, LWEC:

- 1. Established a fixed indicator line across where the three cables that raise and lower the fuel feed rakes are located on the south side of the Fuel Storage Building.
- 2. At the start of testing, a mark was put on each cable at the indicator line, signifying the elevation of each fuel feed rake at that time.
- 3. Individual bins were filled with the separate fuel types (one bin with wood, the other two bins with creosote treated railroad ties) and the tonnage of fuel added to each bin was recorded. As the fuel was added, the rakes were raised up toward the top of each pile. The fuel weights as received at the fuel bins were determined based upon the fuel weights determined at the Fuel Aggregation Facility before delivery to the power plant and boiler.
- 4. Stack testing proceeded for the specified run times.
- 5. When the rakes once again reached the elevation where they started, signified by the mark on each cable re-aligning with the indicator, the respective times were recorded.
- 6. The known tonnage added to each bin was then divided by the difference in times to yield a tons per hour value for each bin over the course of the testing day.
- 7. The above procedure was repeated for the second day of testing.

The fuel feed rates were calculated and the creosote treated railroad tie to wood ratio was determined on a dry basis using average moisture contents by fuel per day supplied by the laboratory from analysis of the collected fuel samples. The fuel feed rate ratios were 2.14 and 2.48 tons of creosote treated railroad ties to tons of wood for 6 July 2016 and 7 July 2016, respectively. LWEC utilized a professional engineer (Mr. Jed Chrestensen from Mannik Smith Group) to assist with the fuel accounting method and perform quality control of the calculations.

Data and the calculation methodology are provided in Appendix E.

3.2 AIR POLLUTION CONTROL EQUIPMENT

Particulate emissions are controlled by a multi-cyclone followed by a single chamber, three-field electrostatic precipitator (ESP).

3.2.1 ESP Operating Parameters

The precipitator electrical controls and rapping sequence, intensity and frequency are set for optimum performance and are not generally modified after this optimization exercise unless emissions issues are observed.

3.3 REFERENCE METHOD TEST LOCATION

The reference method sample ports (two sets) are located on a section of rectangular ductwork that runs horizontally from the exit of the ESP prior to the exhaust stack. The rectangular ductwork is six feet by six feet six inches (6' x $6\frac{1}{2}$) and has a straight run of fifty-seven feet (57'). All dimensions and port locations were verified prior to testing.

A second set of four sample ports are installed approximately 2 feet downstream from the primary sample ports and allows for additional sample trains to be operated simultaneously. Air flow disturbances in the secondary sample ports were minimized by port selection and placement of the upstream sampling equipment. Additionally, a third set of sample ports located on top of the ESP outlet ductwork was used for single point sampling (continuous emissions monitoring). All dimensions and port locations were verified prior to testing.

Figure 3-1 presents a diagram of the sample port and traverse point location.

3.3.1 Flue Gas Parameters

The expected flue gas parameters at this location are as follows:

Temperature: approximately 370-450 °F, load dependent Moisture: approximately 15% v/v, fuel moisture dependent Volumetric Flow Rate: Up to about 150,000 ACFM, load dependent



FIGURE 3-1 SAMPLE PORT AND TRAVERSE POINT LOCATIONS

4. SAMPLING AND ANALYTICAL PROCEDURES

The purpose of this section is to detail the stack sampling and analytical procedures utilized during the test program. Table 4-1 summarizes the sampling and analytical methods.

4.1 PRE-TEST DETERMINATIONS

Preliminary test data was obtained at the sampling location. Geometry measurements were measured and recorded, and traverse point distances verified. A preliminary velocity traverse was performed utilizing a calibrated "S" type pitot tube and a Dwyer inclined manometer to determine velocity profiles. Flue gas temperatures were observed with a calibrated direct readout pyrometer equipped with a chromel-alumel thermocouple. Water vapor content was based on previous test data (preliminary only).

A check for the presence or absence of cyclonic flow was conducted at the test location. The results demonstrated the location was suitable for testing with no significant turbulent flow (< 20° average flow angle) noted. Preliminary test data was used for nozzle sizing and sampling rate determinations for isokinetic sampling procedures.

Pre-test calibration of probe nozzles, pitot tubes, metering systems, and temperature measurement devices were performed as specified in Section 5 of EPA Method 5 test procedures.

4.2 FORMAL TESTING

4.2.1 Gas Volumetric Flow Rate

A series of three test runs was performed for each parameter. The gas velocity was measured using EPA Methods 1 and 2. Velocity measurements were performed using an "S-type" pitot tube fastened alongside the EPA Methods 5/29, 23/0010 and 201A/202 sample probes. The stack gas pressure differential was measured with inclined manometers. Flue gas temperatures were measured with calibrated digital temperature readouts equipped with chromel-alume1 (type-K) thermocouples.

Sample	No. of Test Runs	Sampling Duration	Sampling Method	Sample Size	Analytical Parameters	Preparation Method	Analytical Method
	<u></u>	1-hr composite sample per run	Modified M26A	30-50 ft ³	HCl/Cl ₂	NA	Ion Chromatography (SW846-9057)
		1 to 1.5-hr composite sample per run	M 5/29	30-50 ft ³	Particulate Metals	Desiccation Acid digestion (SW-846- 3050A)	Gravimetric (EPA Method 5) ICP and AAS (SW-846-6010A)
		1 to 1.5-hr composite sample per run	M201A/202	30-50 ft ³	PM10/PM2.5	Desiccation	Gravimetric (EPA Method 5)
Stack Gas	3	3-hr composite sample per run	M23/M0010	> 90 ft ³	PCDD-PCDF/ Cresol Isomers	Extraction	M23/SW 846-8270
		Continuous	M3A	NA	CO_2/O_2	NA	CEM
]		Continuous	M25A	NA	VOC	NA	CEM
					Moisture	NA	Gravimetric
		Concurrent	M1-4	NA	Temperature	NA	Temperature
					Velocity	NA	Pitot Tube
		1-hour observation per run	M9	NA	Opacity	NA	NA

Table 4-1Summary of Sampling and Analytical Methods

Notes:

=	Combined Method 5 and Method 29 sampling train.
=	Inductively coupled plasma emission spectroscopy.
25	Atomic absorption spectroscopy.
=	Pb, Ni, As, Mn
=	Combined Method 23 and Method 0010 sampling train.
	= = = =

Velocity measurements and stack gas temperatures were incorporated in the isokinetic sampling trains which traverse across the stack diameter. Velocity and volumetric flow rate were used for determining the parameter mass rate calculations. Likewise moisture content was determined concurrently with each test. The moisture content of the gas stream was determined by the volume increase of the impinger water rand weight increase of the silica gel in comparison to the volume of gas sampled.

The gas stream composition [oxygen (O_2) and carbon dioxide content (CO_2)] of the flue gas was measured according to EPA Method 3A or 3/3A procedures using a Reference Method Continuous Emission Monitoring (CEM) system.

4.3 PARTICULATE AND METALS SAMPLING TRAIN

The sampling train utilized to perform the particulate and metals sampling was an EPA Reference Method 5/29 train (see Figure 4-1).

A calibrated glass nozzle was attached to a heated (~250°F) borosilicate probe. The probe was connected to a heated (~250°F) borosilicate filter holder containing a 9-centimeter (cm) quartz filter (preweighed to a constant 0.1 milligram (mg) weight). The filter holder was connected to the first of four impingers by means of rigid glass connectors. The first moisture knockout impinger (if used) was dry. The second and third impingers each contained 100 ml of nitric acid (HNO₃)/hydrogen peroxide (H₂O₂) solution, and the fourth impinger contained 300 grams (g) of dry silica gel. The third impinger was a standard Greensburg-Smith type, while all other impingers were of a modified design. All impingers were maintained in an ice bath. A control console with a leakless vacuum pump, a calibrated dry gas meter, a calibrated orifice, and inclined manometers were connected to the final impinger via an umbilical cord to complete the train.

During particulate/metals sampling, gas stream velocities were measured by inserting a calibrated "S"-type pitot tube into the gas stream adjacent to the sampling nozzle. The velocity pressure differential was observed immediately after positioning the nozzle at each traverse point, and the sampling rate was adjusted to maintain isokineticity \pm 10 percent. Flue gas temperature was monitored at each point with a calibrated pyrometer and thermocouple.



FIGURE 4-1 EPA METHOD 5/29 PARTICULATE AND METALS SAMPLING TRAIN Probe, filter box, and impinger exit gas temperatures were monitored with a calibrated direct readout pyrometer equipped with chromel-alumel thermocouples positioned in the heated filter chamber and in the sample gas stream after the last impinger.

Isokinetic test data was recorded at each traverse point during all test periods. Leak checks were performed on the sampling apparatus according to reference method instructions, prior to and following each run, and/or component change.

4.3.1 Particulate and Metals Sample Recovery

At the conclusion of each test, the sampling train was dismantled, the openings sealed, and the components transported to the field laboratory.

A consistent procedure was employed for sample recovery as follows:

- 1. The quartz fiber filter(s) was removed from its holder with tweezers and placed in its original container (petri dish), along with any loose particulate and filter fragments (Sample type 1).
- 2. The probe and nozzle were separated and the particulate rinsed with acetone into a borosilicate container with a Teflon-lined closure while brushing with a non-metallic (Teflon) brush a minimum of three times. Particulate adhering to the brush was rinsed with acetone into the same container. The front-half of the filter holder and connecting glassware were rinsed with acetone while brushing a minimum of three times. The acetone rinses were combined in a borosilicate container and sealed with a Teflon-lined closure (Sample type 2). A separate 0.1N HNO₃ acid rinse of the probe, nozzle, front-half of the filter holder and connecting glassware was performed after the acetone rinse. The 0.1N HNO₃ rinses were combined and sealed with a Teflon-lined closure (Sample type 3).
- 3. The total volume of HNO_3/H_2O_2 and condensate in impingers 1, 2 and 3 was measured to the nearest ml and the value recorded. The liquid was then placed in a borosilicate container along with a 100-ml HNO₃ rinse of the impingers, connectors, and back half of the filter holder. The container was sealed with a Teflon-lined closure (sample type 4).
- 4. The silica gel was removed from the last impinger and immediately weighed to the nearest 0.1 g.
- 5. Samples of acetone and 0.1 N HNO₃ acid and HNO₃/H₂O₂ were retained for blank analysis.

Each sample bottle was labeled to clearly identify its contents. The height of the fluid level was marked on each bottle. Sample integrity was assured by maintaining chain-of-custody records.

4.3.2 Particulate Analysis

The particulate analysis proceeded as follows:

- 1. The filters (Sample type 1) and any loose fragments were desiccated for 24-hours and weighed to the nearest 0.1 mg to a constant (\pm 0.5 mg) weight.
- 2. The front-half acetone wash samples (Sample type 2) and an acetone blank were evaporated at ambient temperature and pressure in tared beakers, then desiccated and weighed to constant 0.5-mg weight.

The total weight of material measured in the acetone-rinse fraction plus the weight of material collected on the quartz filter represents the total particulate catch. Blank corrections were made where appropriate for all sample weights.

Following the gravimetric particulate analysis of the filter, the sample was analyzed for metals. Likewise upon completion of the gravimetric analysis of the front-half acetone samples, the residue was resolubilized with 0.1 N HNO₃ and combined with the front half nitric sample for metals analysis.

4.3.3 Metals Analysis

Samples collected for metals analysis were contained in three different media:

- Front Half Nitric Acid (including resolubilized particulate residue for front-half acetone samples)
- Filter (following particulate analysis)
- Back Half Nitric Acid

The front half nitric acid and particulate filter samples were combined with the back half nitric acid impingers and condensate in the laboratory for analysis. The metals were solubilized by the addition of nitric acid and 30% H₂O₂. Sample volume was reduced to 50 ml on a hot plate. The sample was brought to 300 ml final volume and analyzed for Atomic Absorption Spectroscopy (AAS) and Inductively Coupled Argon Plasma (ICP) metals.

Following digestion, the metals samples were ready for analysis by ICP-AAS.

4.4 EPA METHOD 26A (MODIFIED) – HYDROGEN CHLORIDE/CHLORINE SAMPLING TRAIN

The sampling train utilized to perform the hydrogen chloride sampling was configured as an EPA Reference Method 26A full-size sampling train except there was no borosilicate nozzle attached to the sample probe (see Figure 4-2). This modification was implemented to allow nonisokinetic sampling from a single traverse point similar to EPA Method 26. A heated ($\geq 248^{\circ}$ F) borosilicate probe was attached to a heated ($\geq 248^{\circ}$ F) borosilicate filter holder containing a 9-cm quartz filter. The filter folder was connected to the first of six impingers by means of rigid glass connectors. The first moisture knockout impinger contained 50 ml of 0.1 normal sulfuric acid. The second and third impingers each contained 100 ml of 0.1 N sulfuric acid. The fourth and fifth impingers each contained 100 ml of 0.1 N sodium hydroxide, and the sixth impinger contained 300 grams of dry silica gel. The second and third impingers were a standard Greenburg-Smith type and all other impingers were of a modified design. All impingers were maintained in an ice bath. A control console with a leakless vacuum pump, a calibrated dry gas meter, a calibrated orifice, and inclined manometers was connected to the final impinger via an umbilical cord to complete the train. Probe, filter box, and impinger exit gas temperatures were monitored with a calibrated direct read-out pyrometer equipped with a chromel-alumel thermocouples.

Sampling was conducted in conjunction with the isokinetic sample trains and continuous monitoring parameters, and these stack gas velocities and stack gas composition $(O_2/CO_2$ content) were used to determine hydrogen chloride/chlorine mass rates.

4.4.1 Hydrogen Chloride/Chlorine Sample Recovery

At the conclusion of each test, the sampling train is dismantled, the openings sealed, and the components are transported to the field laboratory.

A consistent procedure was employed for sample recovery as follows:

1. The quartz fiber filter or thimble was removed from its holder with tweezers and discarded.



FIGURE 4-2 EPA METHOD 26A (MODIFIED) HYDROGEN CHLORIDE AND CHLORINE SAMPLING TRAIN

- 2. The total liquid content of impingers one, two and three (0.1 N H₂SO₄) was measured and the sample placed in a polyethylene container fitted with a Teflonlined closure (Sample type 1). Also included in this sample was distilled water rinse of the impingers and connectors. The sample was labeled for chloride analysis.
- 3. The total liquid content of impingers four and five (0.1 N NaOH) were measured and the sample placed in a polyethylene container fitted with a Teflon-lined closure (Sample type 2). Also included in this sample was a distilled water rinse of the impingers and connectors. The sample was labeled for chlorine analysis. Sodium thiosulfate was added to the NaOH samples as a preservative per Method 26A procedures.
- 4. The silica gel impinger was immediately weighed to the nearest 0.5 g.
- 5. Samples of sulfuric acid, sodium hydroxide and distilled water used for this program were retained for blank analysis.

Each sample bottle was labeled to clearly identify its contents. The height of the fluid level was marked on each bottle. The samples were then transported to the subcontract laboratories. Sample integrity was assured by maintaining chain-of-custody records.

4.4.2 Hydrogen Chloride Analysis

The samples from the H_2SO_4 impingers were analyzed for chloride (Cl⁻) by the procedures outlined in EPA SW-846 Method 9057 (ion chromatography) and reported as HCl. The samples from the NaOH impingers were analyzed for chlorine (Cl₂) by the procedures outlined in EPA SW846 Method 9057 (ion chromatography) and reported as chlorine.

4.5 EPA METHOD 23/EPA SW846 METHOD 0010 - PCDD/PCDF AND CRESOL SAMPLING TRAIN

The test train utilized to perform the polychlorinated dibenzo-p-dioxins/polychlorinated dibenzo furans (PCDD/PCDF) and the cresol isomers sampling was conducted using a combined EPA Method 23 and EPA SW846 Method 0010 sample train (see Figure 4-3).

A borosilicate nozzle was attached to a heated (~250°F) borosilicate probe. The probe was connected directly to a heated borosilicate filter holder containing a solvent extracted



FIGURE 4-3 EPA METHOD 23/ EPA SW846 METHOD 0010 PCDD/PCDFAND CRESOL SAMPLING TRAIN

glass fiber filter. A section of borosilicate tubing joined the filter holder exit to a spiral type ice water-cooled condenser, an ice water-jacketed sorbent module containing approximately 40 g of 30/60 mesh XAD-2 resin. A thermowell is located on the outlet of the condenser so the XAD module inlet temperature is monitored. The XAD module was connected to a condensate trap followed by a series of three impingers. The first two impingers each contained 100-ml of high purity distilled water. The final impinger contained 300 g of dry pre-weighed silica gel. All impingers and the condensate trap were maintained in an ice bath. A control console with a leakless vacuum pump, a calibrated orifice, and dual inclined manometers was connected to the final impinger via an umbilical cord to complete the sample train.

During PCDD/PCDF and cresol sampling, gas stream velocities were measured by inserting a calibrated "S"-type pitot tube into the gas stream adjacent to the sampling nozzle. The velocity pressure differential was observed immediately after positioning the nozzle at each traverse point, and the sampling rate was adjusted to maintain isokineticity \pm 10 percent. Flue gas temperature was monitored at each point with a calibrated pyrometer and thermocouple. Probe, filter box, XAD module, and impinger exit gas temperatures were monitored with a calibrated direct readout pyrometer equipped with chromel-alumel thermocouples. The thermocouples were positioned in the heated filter chamber and between the condenser and XAD module and after the last impinger.

Isokinetic test data was recorded at each traverse point during all test periods. Leak checks were performed on the sampling apparatus according to reference method instructions, prior to and following each run, and/or component change.

4.5.1 EPA Method 23/EPA SW846 Method 0010 - PCDD/PCDF and Cresol Sample Recovery

At the conclusion of each test, the sampling train was dismantled, the openings sealed, and the components transported to the field laboratory.

A consistent procedure was employed for sample recovery:

1. The foil covered XAD-2 module was sealed, labeled, and placed in an ice-cooled chest (sample type 1).

- 2. The glass fiber filter was removed from its holder with tweezers and placed in a borosilicate container with a Teflon-lined closure along with any loose particulate and filter fragments (sample type 2).
- 3. The particulate adhering to the internal surfaces of the nozzle, probe and front half of the filter holder were rinsed with acetone into a borosilicate container while brushing a minimum of three times until no visible particulate remained. Particulate adhering to the brush was rinsed with acetone into the same container. The container was sealed with a Teflon®-lined closure (sample type 3).
- 4. The components from the aforementioned step were rinsed with methylene chloride while brushing. The solvent was added to Sample Type 3.
- 5. The volume of liquid collected in the condensate trap was measured, the value recorded, and the contents poured into a glass sample bottle along with deionized water rinse of the back-half of the filter holder, connectors, condenser coil and condensate trap. The borosilicate sample container was capped with a Teflon-lined closure (sample type 4). The train components in the aforementioned step were washed with acetone followed by methylene chloride and the solvent rinses placed in a separate borosilicate container with a Teflon-lined closure (sample type 5).
- 6. The volume of liquid in impingers one and two was measured, the values recorded.
- 7. All Method 23 test train components up to the exit of the condenser were rinsed with toluene. The toluene rinse was placed in a borosilicate sample container capped with a Teflon lined closure (sample type 6).
- 8. The silica gel in the third and final impinger was weighed and the weight gain value recorded.
- 9. Site blank samples of the solvents, XAD-2 module, filter, and distilled water were retained for analysis.

Each container was labeled to clearly identify its contents. The height of the fluid level was marked on the container of each liquid sample to provide a reference point for a leakage check after transport.

4.5.2 EPA Method 23 - PCDD/PCDF Sample Analysis

The front-half solvent wash, filter, XAD-2 resin, back-half solvent and toluene rinse contents were extracted. The extracts were combined into a train total composite extract and analyzed as per the procedures outlined in EPA Method 23 utilizing high resolution capillary column GC/high resolution mass spectrometry (MS) procedures.

4.5.3 EPA SW846 Method 0010 – Cresol Sample Analysis

General analysis for cresol isomers followed the analytical procedures summarized below. Refer to SW 846 Method 8270 for detailed specifications of this analysis procedure. Analysis was limited to three target cresol isomers; m-cresol, o-cresol and p-cresol.

First, each front-half wash sample is concentrated to 1-5 ml using a rotary evaporator apparatus. The sample container is rinsed three times with methylene chloride, added to the concentrated solution, and concentrated further to near dryness.

The above concentrate is added to the filter and XAD-2 resin in a soxhlet apparatus that contained a precleaned glass extraction thimble and silica gel. Internal standards are added, covered with a plug of precleaned glass wool and refluxed with toluene for 16 hours. The extract is transferred using three 10-ml rinses of toluene to a rotary evaporator, concentrated to approximately 8 ml, and reduced to 1 ml under nitrogen stream. The sample is split in half, one split is analyzed, and the second archived.

The back-half impinger solvent rinse is concentrated to 2 ml using a rotary evaporator, then added to the impinger water/condensate sample. Following solvent addition, the sample is spiked with the appropriate internal standards. A liquid extraction is then conducted using methylene chloride. The extract is combined with the front-half soxhlet extract for cleanup and analysis. The remaining extract is analyzed for the targeted cresol isomers utilizing GC with low-resolution MS.

Site blanks and laboratory blanks are analyzed with each group of source samples using the above procedure as QC, contamination or performance checks, as appropriate. All GC/MS analyses include analysis of method blank, a method blank spike, a matrix spike, and a laboratory control standard. In addition, appropriate surrogate compounds for the cresols are spiked into each XAD-2 module. Recoveries from method spikes and surrogate compounds are calculated and recorded on control charts to maintain a history of system performance.

4.6 PM₁₀/PM_{2.5} SAMPLING TRAIN

Particle size $(PM_{10}/PM_{2.5})$ was collected using EPA Method 201A. The sampling train also incorporated the revision to EPA 202 procedures for determination of condensible particulate also referred to as the dry impinger method (see Figure 4-4).

The sampling train consisted of the following components:

- A stainless steel nozzle with an inside diameter sized to sample isokinetically connected to a cyclonic separator.
- A PM₁₀/PM_{2.5} dual stage sampling cyclone.
- A borosilicate probe equipped with a calibrated thermocouple to measure flue gas temperature and a calibrated S-type Pitot tube to measure flue gas velocity pressure.
- A heated (at stack temperature) borosilicate filter holder containing a tared quartz fiber filter.
- The pitot tube tip mounted slightly beyond the combined cyclone head assembly and at least one inch off the gas flow path to the cyclone nozzle.
- A section of borosilicate connections from the outlet of the filter holder to the water cooled coil condenser. The outlet of the condenser is connected to the first impinger.
- An impinger train consisting of four impingers. The first two impingers were empty and have a short stem and modified stem, respectively. The third impinger was of a standard design and contained 100 ml of distilled water. The fourth impinger contained 300 grams of dry preweighed silica gel.
- An untared Teflon filter and glass filter holder was located between the second (dry) impinger and the third impinger. The filter exit temperature was monitored and maintained between 65°F and 85°F.
- A vacuum hose with adapter to connect the outlet of the impinger train to a control module.
- A control module containing a 3-cfm carbon vane vacuum pump (sample gas mover), a calibrated dry gas meter (sample gas volume measurement device), a calibrated orifice (sample gas flow rate monitor), and inclined manometers (orifice and gas stream pressure indicators).
- A switchable calibrated digital pyrometer to monitor flue and sample gas temperatures.



FIGURE 4-4 EPA METHOD 201A (PM10/PM2.5) / 202 SAMPLING TRAIN

Leak checks of the entire sampling train were performed prior to sampling. At test completion, a final leak check was performed at the sample probe inlet. Per EPA 201A procedures, no leak check of the $PM_{10}/PM_{2.5}$ cyclone and filter housing was performed at test completion. This is to minimize particle bypass through the cyclone during the leak check.

During PM₁₀/PM_{2.5}, flue gas velocity was measured with a calibrated S-type pitot tube (provided with extensions) fastened slightly beyond the combined cyclone head and at least one inch from nozzle. Flue gas temperature was monitored with a calibrated direct readout pyrometer equipped with a chromel-alumel (Type K) thermocouple positioned near the sampling nozzle. The probe, filter box, CPM filter exit, and impinger exit gas temperatures were monitored with a calibrated direct readout pyrometer equipped with Type K thermocouples. The PM₁₀/PM_{2.5} sample was collected at a constant rate based on stack gas conditions. The sampling time at each traverse point was adjusted based on the stack velocity measured at each point to ensure the sample is collected isokinetically.

4.6.1 PM₁₀/PM_{2.5} SAMPLE RECOVERY

At the conclusion of each $PM_{10}/PM_{2.5}$ test, the sampling train was dismantled. The openings sealed and the components transported to the field laboratory.

Following test completion and prior to the start of sample recovery the impinger portion of the EPA 201A/202 train was purged with nitrogen at a minimum of 14 liters per minute for 60 minutes. The CPM filter was maintained between 65°F and 85°F during the purge. This purge is to expel any dissolved sulfur dioxide.

A consistent procedure was employed for sample recovery:

- 1. The pre-weighed quartz fiber filter was removed from the borosilicate filter housing with tweezers and placed in its original container (petri dish) along with any loose particulate and filter fragments (sample type 1).
- 2. The particulate adhering to the internal surfaces of the nozzle and PM_{10} cyclone were rinsed with acetone into a borosilicate container while brushing a minimum of three times until no visible particulate remained. Particulate adhering to the brush was rinsed with acetone into the same container. The container was sealed with a Teflon lined closure (sample type 2-PM greater than 10 μ m).

- 3. The particulate adhering to the internal surfaces of the PM_{10} cyclone exit connecting tube and the internal surfaces of the $PM_{2.5}$ cylcone was rinsed with acetone into a borosilicate container while brushing a minimum of three times until no visible particulate remained. Particulate adhering to the brush was rinsed with acetone into the same container. The container was sealed with a Teflon lined closure (sample type 3-PM less than 10 µm but greater than 2.5 µm).
- 4. The particulate adhering to the internal surfaces of the $PM_{2.5}$ cyclone to filter holder connecting tube ($PM_{2.5}$ cyclone exit) and filter holder was rinsed with acetone into a borosilicate container while brushing a minimum of three times with no visible particulate remained. Particulate adhering to the brush was rinsed with acetone into the same container. The container was sealed with a Teflon-lined closure (sample type 4-PM less than 2.5 μ m).
- 5. Following completion of the nitrogen purge, the total liquid content of impingers one and two were measured volumetrically or gravimetrically and the sample placed in a borosilicate container (sample type 5).
- 6. The coil condenser, the first two impingers, the back half of the filterable particulate filter holder, the front half of the condensable filter housing, and the connectors were rinsed twice with distilled water. The rinsate was added to sample type 5.
- 7. The coil condenser, the first two impingers, the back half of the filterable particulate filter holder, the front half of the condensable filter housing, and the connectors were rinsed twice with acetone and hexane. The rinses were placed in a borosilicate container with Teflon-lined closure (sample type 6).
- 8. The Teflon filter (CPM filter) located between impingers 2 and 3 was removed from its filter holder and placed into a petri dish or borosilicate container (sample type 7).
- 9. The total liquid content of impinger three was measured volumetrically and discarded.
- 10. The silica gel was removed from the last impinger and immediately weighed to the nearest one-tenth gram. The weight gain was recorded.
- 11. Acetone, PM_{2.5} filter, distilled water and hexane blank samples were placed into a borosilicate/Teflon container or petri dish and sealed for gravimetric analysis.

Each container was labeled to clearly identify its contents. The height of the fluid level was marked on the container of each liquid sample to determine whether or not leakage occurred during transport.

4.6.2 Filterable PM₁₀/PM_{2.5} (EPA 201A) Analysis

- The filters and any loose fragments were desiccated for 24 hours and weighed to the nearest 0.1 mg to a constant weight of no more than 0.5 mg between 2 consecutive weighings with no less than six hours of desiccation time between weighings.
- The front-half acetone wash samples (nozzle/PM₁₀ cyclone rinse, PM₁₀ cyclone exit/PM_{2.5} cyclone rinse and PM_{2.5} exit/filter holder rinse) were evaporated at ambient temperature and pressure in tared beakers, and then desiccated to constant weight to the nearest 0.1 mg.
- A blank sample of acetone and a filter was analyzed along with the PM₁₀/PM_{2.5} source samples.

The residue weight of the nozzle PM_{10} /cyclone rinse sample represents the particulate catch greater than 10 microns (>PM_{10}). The PM cyclone exit PM_{2.5} cyclone rinses represent the particulate catch less than 2.5 microns (< PM_{10}), The PM_{2.5} filter holder rinse sample plus the filter residue represents the filterable particulate catch less than and equal to 2.5 microns (PM_{2.5}).

4.6.3 Condensable Particulate (EPA 202) Analysis

- The total volume of sample type 5 was measured.
- The Teflon filter was extracted (rinsed).
- The remaining contents of sample type 5 and the acetone/hexane rinse (sample type 6) were combined in a separatory funnel. After mixing, the organic phase was removed and retained in a tared beaker. Two separate additions of 75 ml of hexane were added to the separatory funnel and removed (following mixing and separation) to the tared beaker. The organic fraction was evaporated at room temperature and desiccated to the nearest 0.1 mg to a constant weight.
- The resulting water (inorganic fraction) was placed in a tared beaker and taken to near dryness (~ 50 ml) on a hot plate and then evaporated to dryness in an oven at 105°C.

The total of the organic and inorganic fractions represent the condensible particulate catch. The total $PM_{10}/PM_{2.5}$ includes the filterable $PM_{10}/PM_{2.5}$ catch plus the organic and inorganic condensables.

4.7 CONTINUOUS EMISSIONS MONITORING SYSTEM

A diagram of the reference method sampling system used to measure VOC and O_2/CO_2 is presented in Figure 4-5. The system conformed to the requirements of EPA Reference Methods 25A and 3A. A flame ionization analyzer was used to measure VOC concentrations. A non-



FIGURE 4-5 WESTON CEM SAMPLING SYSTEM dispersive infrared (NDIR) analyzer was used to measure CO_2 and a paramagnetic analyzer was used to measure O_2 concentrations.

Stack gas was withdrawn from the stack through a heated stainless steel probe and heated filter via a heated sample line maintaining a temperature of 250°F. The probe was inserted into a dedicated sample port at a single point in the gas stream. The outlet of the heated sample line was connected to a sample conditioning system for moisture removal. The clean, dried sample was then transported to the O_2 and CO_2 analyzers via a Teflon_® sample line. The VOC sample was drawn directly to the flame ionization analyzer from a "T" located before the sample conditioners. The flame ionization analyzers measures VOC on a wet basis. A separate Teflon_® line was used for introduction of VOC and O_2/CO_2 bias gases to the probe outlet.

4.7.1 VOC and O₂/CO₂ Monitoring Procedures

The VOC and O_2/CO_2 analyzers were calibrated daily by introduction of EPA Protocol calibration gases to the analyzers. After the analyzer calibration, a system bias check was conducted by introducing a zero gas (zero air or nitrogen) and one selected VOC and O_2/CO_2 , calibration gas to the sample probe outlet. The bias check was repeated at the end of each test run to determine sampling system bias and instrument drift for each analyzer.

The interference checks on WESTON's O_2/CO_2 instrumental analyzers were previously performed (December 2014) in accordance with EPA Method 7E and were not repeated for this test program.

Additionally, an O_2 stratification check was performed prior to the test effort in accordance with EPA Method 7E – Section 8.1.2. Sampling during formal testing was performed at a single point based on the results of the stratification test (< 5% difference for each traverse point compared to the average result).

Three formal test runs of one hour or longer duration coincided with the isokinetic sample runs in order to correct wet concentrations to a dry basis and calculate mass rates in terms of lb/hr.

The output from the analyzers was directed to a data acquisition system and recorded by a computer equipped with data reduction software designed by WESTON. The software calculated

the average one-minute measured concentrations which were used to compute an average concentration for the test run.

4.8 OPACITY

Opacity was determined by a certified visible emissions (VE) evaluator pursuant to EPA Reference Method 9. A 60-minute opacity observation (3 total) was conducted in conjunction with each EPA 5/29 and 201A/202 test train pairing. General procedures related to EPA 9 are presented below:

- A qualified observer stood at a distance to provide a clear view of the emissions with the sun oriented in the 140° sector to his/her back.
- The observers' line of vision was perpendicular to the plume direction.
- The observer recorded all pertinent atmospheric conditions and pertinent site information.
- Opacity observations were made at the point of greatest opacity of the plume and at a point without condensed water vapor.
- The exhaust plume was observed in 15 second intervals to make a reading for a minimum of 240 readings per 60-minute period. The reported % opacity was calculated as the average of the 240 consecutive observations.

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5. FUEL SAMPLING AND ANALYSIS

LWEC fuel is supplied by M.A. Energy Resources LLC (MAER). MAER operates a fuel aggregation facility where raw materials are processed then conveyed to the facility.

As required by the 114 Request, fuel samples were collected during the test program during each test run in accordance with 40 CFR 63 Subpart 7521(c and d). LWEC designated personnel to collect fuel samples at least twice per run (approximately beginning and mid-point) from a point where each fuel drops onto the conveyor belt feeding the boiler. A composite sample of each fuel type per test run was submitted for analysis.

Prior to the stack test program, LWEC personnel collected samples of each fuel fired in the boiler on fifteen separate occasions (19 May - 2 June, 2016).

The stack test composites and all fuel samples collected prior to formal testing were submitted for analysis as listed in Table 5-1.

Euel Type	Required Analysis	Analytical Methods	Minimum Detection Level
TDF	Moisture Content	<u>ASTM D3173</u> , "Standard Test Method for Moisture in the Analysis Sample of Coal and Coke"	Not Applicable
	Chlorine Concentration	EPA 5050/9056, "Determination of Inorganic Anions by Ion Chromatography"	~50 ppm
	Sulfur Concentration	ASTM D4239, "Standard Test Method for Sulfur in the Analysis Sample of Coal and Coke Using High-Temperature Tube Furnace Combustion"	0.02 weight %
Wood	Moisture Content	<u>ASTM D3173</u> , "Standard Test Method for Moisture in the Analysis Sample of Coal and Coke"	Not Applicable
	Chlorine Concentration	EPA 5050/9056, "Determination of Inorganic Anions by Ion Chromatography"	~50 ppm
	Sulfur Concentration	ASTM D4239, "Standard Test Method for Sulfur in the Analysis Sample of Coal and Coke Using High-Temperature Tube Furnace Combustion"	0.02 weight %
Creosote Ties	Moisture Content	<u>ASTM D3173</u> , "Standard Test Method for Moisture in the Analysis Sample of Coal and Coke"	Not Applicable
	Chlorine Concentration	EPA 5050/9056, "Determination of Inorganic Anions by Ion Chromatography"	~50 ppm
	Sulfur Concentration	ASTM D4239, "Standard Test Method for Sulfur in the Analysis Sample of Coal and Coke Using High-Temperature Tube Furnace Combustion"	0.02 weight %

Table 5-1Fuel Sample Analytical Methods

6. QUALITY ASSURANCE/QUALITY CONTROL

6.1 QUALITY CONTROL PROCEDURES

As part of the compliance test, WESTON implemented a QA/QC program. QA and QC are defined as follows:

- <u>Quality Control</u>: The overall system of activities whose purpose is to provide a quality product or service: for example, the routine application of procedures for obtaining prescribed standards of performance in the monitoring and measurement process.
- <u>Quality Assurance</u>: A system of activities whose purpose is to provide assurance that the overall quality control is being done effectively. Further,

The field team manager for stack sampling was responsible for implementation of field QA/QC procedures. Individual laboratory managers were responsible for implementation of analytical QA/QC procedures. The overall project manager oversaw all QA/QC procedures to ensure that sampling and analyses met the QA/QC requirements and that accurate data resulted from the test program.

6.2 GAS STREAM SAMPLING QA PROCEDURES

General QA checks were conducted during testing and apply to all methods including the following:

- Performance of leak checks.
- Use of standardized forms, labels and checklists.
- Maintenance of sample traceability.
- Collection of appropriate blanks.
- Use of calibrated instrumentation.
- Review of data sheets in the field to verify completeness.
- Use of validated spreadsheets for calculation of results.

The following section details specific QA procedures applied to the isokinetic methods.

6.2.1 Stack Gas Velocity/Volumetric Flow Rate QA Procedures

The QA procedures followed for velocity/volumetric flow rate determinations followed guidelines set forth by EPA Method 2. Incorporated into this method, were sample point determinations by EPA Method 1, and gas moisture content determination by EPA Method 4. QA procedures for Methods 1 and 2 are discussed below.

Volumetric flow rates were determined during the isokinetic flue gas tests. The following QC steps were followed during these tests:

- The S-type pitot tube was visually inspected before sampling.
- Both legs of the pitot tube were leak checked before sampling.
- Proper orientation of the S-type tube was maintained while making measurements. The yaw and pitch axes of the S-type pitot tube were maintained at 90° to the flow.
- The manometer oil was leveled and zeroed before each run.
- Pitot tube coefficients were determined based on physical measurement techniques as delineated in Method 2.

6.2.2 Moisture and Sample Gas Volume QA Procedures

Gas stream moisture was determined as part of the isokinetic test trains. The following QA procedures were followed in determining the volume of moisture collected:

- Preliminary impinger train tare weights were weighed or measured volumetrically to the nearest 0.1 g or 1.0 ml.
- The balance was leveled and placed in a clean, motionless, environment for weighing.
- The indicating silica gel was fresh for each run and periodically inspected and replaced during runs if needed.
- The silica gel impinger gas temperature was maintained below 68°F.

The QA procedures that were followed in regards to accurate sample gas volume determination were:

• The dry gas meter was fully calibrated annually using an EPA approved intermediate standard device.

- Pre-test, port-change, and post-test leak-checks were completed (must be less than 0.02 cfm or 4 percent of the average sample rate).
- The gas meter was read to the thousandth of a cubic foot for all initial and final readings.
- Readings of the dry gas meter, meter orifice pressure (Delta H) and meter temperatures were taken at every sampling point.
- Accurate barometric pressures were recorded at least once per day.
- Pre- and Post-test dry gas meter checks were completed to verify the accuracy of the meter calibration constant (Y).

6.2.3 Isokinetic Sampling Train QA Procedures

The Quality Assurance procedures outlined in this section were designed to ensure collection of representative, high quality test parameter (HCl/HF) concentrations and mass emissions data. The sampling QA procedures followed to ensure representative measurements were:

- All glassware was prepared per reference method procedures.
- The sample rates were within ± 10 percent of the true isokinetic (100 percent) rate.
- All sampling nozzles were manufactured and calibrated according to EPA standards.
- Recovery procedures were completed in a clean environment.
- Sample containers for liquids and filters were constructed of borosilicate or polyethylene with Teflon®-lined lids.
- At least one reagent blank of each type of solution or filter was retained and analyzed.
- All test train components from the nozzle through the last impinger were constructed of glass (with the exception of the filter support pad which is Teflon®).
- All recovery equipment (i.e., brushes, graduated cylinders, etc.) were non-metallic.

6.2.4 Sample Identification and Custody

Sample custody procedures for this program were based on EPA recommended procedures. Since samples were analyzed at remote laboratories, the custody procedures emphasized careful documentation of sample collection and field analytical data and the use of chain-of-custody records for samples being transferred. These procedures are discussed below. The Field Team Manager was responsible for ensuring that all stack samples taken were accounted for and that all proper custody and documentation procedures were followed for the field sampling and field analytical efforts. The Field Team Manager was assisted in this effort by key sampling personnel involved in sample recovery.

Following sample collection, all stack samples were given a unique sample identification code. Stack sample labels were completed and affixed to the sample container. The sample volumes were determined and recorded and the liquid levels on each bottle were marked. Sample bottle lids were sealed on the outside with Teflon® tape to prevent leakage. Additionally, the samples were stored in a secure area until they are shipped.

As the samples were packed for travel, chain-of-custody forms were completed for each shipment. The chain-of-custody forms specifying the treatment of each sample were also enclosed in the sample shipment container.

6.2.5 Data Reduction and Validation QC Checks

All data and/or calculations for flow rates, moisture contents, and isokinetic rates, were made using a computer software program validated by an independent check. In addition, all calculations were spot checked for accuracy and completeness by the Field Team Leader.

In general, all measurement data was validated based on the following criteria:

- Process conditions during sampling or testing.
- Acceptable sample collection procedures.
- Consistency with expected or other results.
- Adherence to prescribed QC procedures.

Any suspect data was flagged and identified with respect to the nature of the problem and potential effect on the data quality.

6.3 REFERENCE METHOD CEMS QA/QC CHECKS

 Continuous emissions monitoring system (probe to sample conditioner) were checked for leaks prior to the testing.

- Pre and post-test calibration bias tests were performed as required by the reference methods.
- Prior to formal testing, a three point stratification check using O₂/CO₂ was performed pursuant to Section 8.1.2 of EPA Method 7E. The three points (16.7, 50 and 83.3 percent of the stack diameter) were each sampled for a minimum of two times the system response. Based on the stratification test results (each point compared to the mean difference was no more than ± 5.0 %), all sampling was performed at a single point at the stack midpoint.
- A permanent data record of analyzer response was made using computer software designed by WESTON.
- All calibration gases used met EPA Protocol standards.

6.4 LABORATORY AUDIT SAMPLES

Laboratory audit samples for metals (Pb, Ni, As, Mn) and HCl were obtained from a Stationary Source Audit Sample (SSAS) provider in accordance with the EPA SSAS program. The audit samples were analyzed in conjunction with the stack samples and the laboratory report indicates passing results for all audit samples submitted.

5/17/2016