BOILER NUMBER ONE PELLET TRIALS TEST PROGRAM EMISSIONS TEST REPORT

Prepared for:



L'ANSE WARDEN ELECTRIC COMPANY, LLC.

157 South Main Street L'Anse, Michigan 49946

Prepared by:

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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 was previously a coal, oil, and gas-fired steam generating station and has been converted to burn biomass. The facility currently operates under the State of Michigan Renewable Operating Permit (ROP) No. MI-ROP-B4260-2011 and the Michigan Department of Environmental Quality (MDEQ) Air Quality Division (AQD) Permit to Install (PTI) 53-17, issued 18 August 2017, which allowed for a temporary (180 calendar day) trial use of engineered fuel pellets as part of LWEC's fuel stream. An extension to the PTI (53-17A) was received on 12 April 2018, which provided an additional 180 days for LWEC to burn engineered fuel pellets and conduct further testing.

The objective of this test program was to determine concentrations and emission rates of particulate matter (PM), particulate matter ≤ 10 microns (PM₁₀), metals (As, Pb, Mn, Ni), hydrogen chloride (HCl), nitrogen oxides (NOx), and sulfur dioxide (SO₂) during two additional pellet fuel trial tests as required by the PTI. LWEC conducted two initial pellet trial tests in December 2017, with the results reported to MDEQ in February 2018. The additional testing will again be used to determine the maximum percentage of pellets in the fuel mix, and the minimum required rate of sorbent injection.

WESTON's Integrated Air Services (IAS) group completed all required testing during 20-21 June 2018. A representative of the MDEQ was present throughout the testing.

1.1 PLANT INFORMATION

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1.2 TESTING FIRM INFORMATION

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1.3 ANALYTICAL LABORATORIES

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1.4 SUMMARY OF TEST PARAMETERS

All testing was performed pursuant to WESTON's Emissions Test Protocol submitted in May 2018. Table 1-1 provides the test parameters, associated test methods, and reporting units for this test program.

Following this introduction, Section 2 provides a summary of the test results. Section 3 provides a description of the process and sampling locations. Section 4 provides a description of the sampling and analytical procedures. Section 5 outlines the fuel processing, fuel sampling and analytical procedures to be used during the test program. Section 6 provides quality assurance and quality control procedures (QA/QC). Appendix A provides detailed test results. Raw test data, boiler operating data, laboratory reports, fuel sample results, quality control records, example calculations, and listing of project participants are provided in Appendices B through H, respectively.

No of Test Conditions ¹	No of Test Runs and Duration	Analytical Parameters and Test Method Reporting Units ²		PTI 53-17 Emissions Limits
2	6 runs on pellets, 84 min.	PM-Metals/EPA 5 & 29	gr/dscf, lb/MMBtu, lb/hr	19.2 lb/hr (PM) 0.06 lb/MMBtu (PM) 0.02 lb/hr (Pb)
	6 runs on pellets, 98.5-106 min.	PM10/EPA 201A-202	gr/dscf, 1b/hr	15.4 lb/hr
	6 runs on pellets, 60 min.	HCl/Modified EPA 26A	ppmvd, lb/hr	2.17 lb/hr
	6 runs on pellets, 60-126 min.	NOx, EPA 7E	ppmvd, lb/hr	145 lb/hr
	6 runs on pellets, 60-126 min.	SO ₂ , EPA 6C	ppmvd, lb/hr	290 lb/hr
	6 runs on pellets, 60-126 min.	O ₂ /CO ₂ , EPA 3A	%	

Table 1-1Summary of Test Parameters

1 Two separate pellet fuel firing conditions at ~ 6 and 5.1 tons/hr (~ 32 and 26% fuel mix ratio).

2 lb/MMBtu emission factors calculated by EPA 19 and LWEC provided F_d-factor from the CO CEMS.

2. SUMMARY OF TEST RESULTS

2.1 TEST RESULTS DISCUSSION

Tables 2-1 through 2-2 of this section provide summaries of the compliance test results for each pollutant parameter. 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.

It should be noted the first two HCl test runs of Condition 2 were believed not representative and an additional two test runs were performed (five total). WESTON determined these runs were not representative due to a negative pressure leak located in the outer probe sheath causing dilution of the stack gas around the probe tip. As a result, the stack gas moisture pickups and HCl concentrations were lower for these runs. The data and results from all five test runs are included in the appendices, but only test runs three through five are included in the average result of Summary Table 2-2 for the purpose of reporting HCl mass emissions.

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 Condition One – 6 TPH Pellets, 285 lb/hr Reagent

Pollutent		PTI 53-17A			
Fonutant	1	2	3	Average	Emissions Limit
Particulate Matter (PM) (lb/hr)	1.65	2.18	0.94	1.59	19.2 lb/hr
Particulate Matter (PM) (lb/MMBtu)	0.006	0.008	0.004	0.006	0.06 lb/MMBtu
Particulate Matter ≤ 10 microns (PM ₁₀) (lb/hr)	2.61	3.96	2.75	3.11	15.4 lb/hr
Particulate Matter ≤ 10 microns (PM ₁₀) (lb/MMBtu)	0.010	0.014	0.010	0.011	
Lead (Pb) (lb/hr)	1.64E-03	2.92E-03	9.63E-04	1.84E-03	0.02 lb/hr
Arsenic (As) (lb/hr)	1.79E-04	1.46E-04	< 1.21E-04	≤1.49E-04	
Manganese (Mn) (lb/hr)	1.64E-02	3.44E-03	5.68E-03	8.50E-03	
Nickel (Ni) (lb/hr)	4.35E-04	6.76E-04	4.71E-04	5.27E-04	
Hydrogen Chloride (HCl) (lb/hr)	1.43	1.45	1.00	1.29	2.17 lb/hr
Nitrogen Oxides (NOx) (lb/hr)	68.9	65.2	64.4	66.2	145 lb/hr
Sulfur Dioxide (SO ₂) (lb/hr)	10.0	7.1	5.7	7.6	290 lb/hr

Table 2-2

Boiler No.1

Summary of Test Results Condition Two – 5.1 TPH Pellets, 225 lb/hr Reagent

Pollutort	Test Run Number						PTI 53-17A
Fonutant	1	2	3	4	5	Average ¹	Emissions Limit
Particulate Matter (PM) (lb/hr)	1.17	0.42	0.75			0.78	19.2 lb/hr
Particulate Matter (PM) (lb/MMBtu)	0.004	0.001	0.003			0.003	0.06 lb/MMBtu
Particulate Matter ≤ 10 microns (PM ₁₀) (lb/hr)	2.84	3.29	2.51			2.88	15.4 lb/hr
Particulate Matter ≤ 10 microns (PM ₁₀) (lb/MMBtu)	0.010	0.012	0.008			0.010	
Lead (Pb) (lb/hr)	1.07E-03	1.16E-03	8.21E-04			1.02E-03	0.02 lb/hr
Arsenic (As) (lb/hr)	1.23E-04	1.30E-04	<1.18E-04			≤1.23E-04	
Manganese (Mn) (lb/hr)	2.60E-03	3.53E-03	2.27E-03			2.80E-03	
Nickel (Ni) (lb/hr)	7.03E-04	7.89E-04	2.07E-04			5.66E-04	
Hydrogen Chloride (HCl) (lb/hr)	0.69	1.00	1.41	1.38	1.25	1.35	2.17 lb/hr
Nitrogen Oxides (NOx) (lb/hr)	65.1	63.2	65.8			64.7	145 lb/hr
Sulfur Dioxide (SO ₂) (lb/hr)	7.6	6.7	7.0			7.1	290 lb/hr

1 HCl Run Nos.1 and 2 are not included in the average result.

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 14 to 17.7 megawatts per hour (MW/hr).

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.

The boiler load was maintained at 90% of capacity during the test program.

3.1.3 Test Program Fuel Mix and Firing Rates

The fuel mix during the testing consisted of wood, creosote treated wood derived fuel (CDF), and engineered pellets. The firing rates for each of the fuels were within the range consistent for safe normal operations.

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.

In order to comply the HCl emission limits set forth in the PTI while burning pellets, a dry sorbent injection system (DSI), provided by Nol-Tec Systems, was installed at the plant. This system is designed to inject reagent into the flue gas exhaust duct. The DSI system includes a super sack test system (bulk bag unloader and injection system) for use during the short-term trial, bulk silo for longer-term storage and use of the sorbent material, and control skid connected into LWEC's control room. The DSI system delivers reagent into the flue gas exhaust duct prior to the ESP and is capable of delivering up to 1000 lb/hr of reagent to the duct.

As per PTI 53-17A, the DSI system must be running at all times while burning pellets and operating in a satisfactory manner.

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.



FIGURE 3-1 SAMPLE PORT AND TRAVERSE POINT LOCATIONS

3.3.1 Flue Gas Parameters

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

Temperature: approximately 400-450 °F, load dependent Moisture: approximately 15% v/v, fuel moisture dependent Volumetric Flow Rate: Up to about 150,000 ACFM, load dependent

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 six test runs (three per fuel firing condition) was performed for each parameter at each test condition. 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 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-alumel (type-K) thermocouples.

Sample	No. of Test Runs	Sampling Duration	Sampling Method	Sample Size	Analytical Parameters	Preparation Method	Analytical Method
Stack Gas	6 (3 x 2 conditions)	60-min. composite sample per run	Modified M26A	40-45 ft ³	HCl	NA	Ion Chromatography (SW846-9057)
		84-min. composite sample per run	M5/29	50-60 ft ³	Particulate Metals	Desiccation followed by acid digestion (SW-846-3050A)	Gravimetric (EPA Method 5) ICPMS (SW-846-6010A)
		98.5-106 min. composite sample per run	M201A/202	30-35 ft ³	PM ₁₀	Desiccation	Gravimetric (EPA Method 5)
		Continuous	M3A	NA	CO ₂ /O ₂	NA	CEM
		Continuous	M7E	NA	NOx	NA	CEM
		Continuous	M6C	NA	SO ₂	NA	CEM
					Moisture	NA	Gravimetric
		Concurrent	M1-4	NA	Temperature	NA	Temperature
					Velocity	NA	Pitot Tube

Table 4-1 **Summary of Sampling and Analytical Methods**

Notes:

M5/M29 Combined Method 5 and Method 29 sampling train. = ICPMS -----Inductively Coupled Argon Plasma Mass Spectrometry. Metals

=

Pb, Ni, As, Mn

Velocity measurements and stack gas temperatures were incorporated in the isokinetic sampling trains which traverse across the stack diameter. 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. Velocity and volumetric flow rate were used for calculating the parameter mass emission rates. For HCl test runs 4 and 5, an independent velocity and volumetric flow rate was conducted by EPA Method 2 procedures to calculate mass rates. For all other HCl test runs the mass rate calculations were derived from velocity and volumetric flow rates from the corresponding particulate/metals sample trains.

The gas stream composition [oxygen (O₂) and carbon dioxide content (CO₂)] of the flue gas was measured according to EPA Method 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 $(248\pm25^{\circ}F)$ borosilicate probe. The probe was connected to a heated $(248\pm25^{\circ}F)$ 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 optional empty moisture knockout impinger was not used. The first and second impingers each contained 100 ml of nitric acid (HNO₃)/hydrogen peroxide (H₂O₂) solution, the third impinger was empty, and the fourth impinger contained 300 grams (g) of dry silica gel. The second impingers 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.



FIGURE 4-1 EPA METHOD 5/29 PARTICULATE AND METALS SAMPLING TRAIN

IASDATA\LWEC\\14464.007.007\FIGURE 4-1 EPA5- 29

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 the isokinetic criteria of \pm 10 percent. Flue gas temperature was monitored at each point with a calibrated pyrometer and thermocouple.

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 HNO₃/H₂O₂ 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 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. The sample volume was reduced to 50 ml on a hot plate then brought to 300 ml final volume and analyzed for metals by Inductively Coupled Argon Plasma Mass Spectrometry (ICPMS).

Following digestion, the metals samples were ready for analysis by ICPMS.

4.4 EPA METHOD 26A (MODIFIED) – HYDROGEN CHLORIDE 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 non-isokinetic sampling from a single traverse point similar to EPA Method 26. A heated (248-273°F) borosilicate probe was attached to a heated (248-273°F) borosilicate filter holder containing a 9-cm quartz filter. The filter folder was connected to the first of four 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 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 exit gas temperatures were monitored with a calibrated direct read-out pyrometer equipped with chromel-alumel thermocouples.

Sampling was conducted in conjunction with the isokinetic sample trains and resulting volumetric flow rates for determination of HCl mass rates.



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

IASDATA\LWEC\14464.007.007\FIGURE 4-2 EPA 26

4.4.1 Hydrogen Chloride 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 was removed from its holder with tweezers and discarded.
- 2. The total liquid content of impingers one, two and three $(0.1 \text{ N H}_2\text{SO}_4)$ 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 silica gel impinger was immediately weighed to the nearest 0.5 g.
- 4. 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 analytical laboratory. Sample integrity was assured by maintaining chain-of-custody records.

4.4.2 Hydrogen Chloride Analysis

The samples from the H₂SO₄ impingers were analyzed for chloride (Cl⁻) by the procedures outlined in EPA SW-846 Method 9057 (ion chromatography) and reported as HCl.

4.5 PARTICULATE MATTER ≤ 10 MICRON SAMPLING TRAIN

The PM_{10} sampling was performed using EPA Method 201A combined with EPA Method 202 (see Figure 4-3).

The sampling train consisted of the following components:

A stainless steel nozzle with an inside diameter sized to sample isokinetically connected to a PM_{10} cyclone separator.



FIGURE 4-3 EPA METHOD 201A/202 PARTICULATE (PM10) / CONDENSABLES SAMPLING TRAIN

- A heated borosilicate probe (stack temperature) 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 borosilicate filter holder (stack temperature) containing a tared quartz fiber filter followed by a water cooled coil condenser.
- An impinger train consisting of four impingers. The first and second impingers were empty and the third impinger contained 100 ml of distilled water. The fourth impinger contained 300 grams of 6-16 mesh dry silica gel. The first impinger had a shortened stem and served as a moisture drop out. The second, third, and fourth impingers were of a modified design. A glass filter holder containing a Teflon filter were placed between the second and third impingers. The filter exit temperature was monitored and maintained at 65°F to 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, 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

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 PM₁₀ cyclone was performed at test completion. This minimized particle bypass through the cyclone during the leak check.

4.5.1 PM₁₀ Sample Recovery

At the conclusion of each PM₁₀ 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 condenser and impinger portion of the EPA 202 train were purged with ultra-high purity nitrogen for one hour at a rate of at least 14 liters per minute to expel dissolved sulfur dioxide. Prior to the purge, the short stem impinger in the moisture dropout was replaced with a long stem impinger and if necessary a known volume of DI water was added so that the water level was at least 1 cm above the impinger tip.

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 original containers (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 cyclone inlet was rinsed with acetone into a borosilicate container while brushing a minimum of three times with acetone until no visible particulate remains. 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 front half acetone No. 1).
- 3. The particulate adhering to the internal surfaces of the cyclone to filter holder connecting tube (cyclone exit) and filter holder was rinsed with acetone into a borosilicate container while brushing a minimum of three times until no visible particulate remains. 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 front half acetone No. 2).
- 4. Following completion of the nitrogen purge, the total liquid content of impingers one, two and three was measured volumetrically and the sample placed in a borosilicate container (sample type 4).
- 5. The condenser, first and second impingers, front half of the Teflon filter holder, and connectors were rinsed two times with degassed (with nitrogen) distilled water. The rinsate was added to sample type 4.
- 6. Following the water rinses, the condenser, first and second impingers, front half of the Teflon filter holder, and connectors were rinsed once with acetone and then two times with hexane. The rinses were placed in a borosilicate container (sample type 5).
- 7. The silica gel was removed from the last impinger and immediately weighed to the nearest one-tenth g. The weight gain was recorded.
- 8. Acetone, PM₁₀ filter, Teflon filter, distilled water and hexane blank samples were placed into a borosilicate/Teflon container or petri dish and sealed for gravimetric analysis.

In addition and as required by EPA 202, a blank train was set up, recovered, and analyzed with the source samples.

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 leakage occurred during transport.

4.5.2 PM₁₀ Sample Analysis

- 1. 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. As an alternative, the filters may be heated to 105°C and desiccated prior to the first weighing. This option is an alternative procedure per EPA Method 5.
- 2. The front-half acetone wash samples (nozzle/cyclone rinse and cyclone 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. Since the acetone No. 1 sample collects particulate greater than PM₁₀, analysis of this sample is optional.
- 3. The contents of sample type 4 was mixed with approximately 30 ml of hexane in a separatory funnel. After mixing, the organic phase was removed and retained in a tared beaker. Two separate additions of 30 ml of hexane were added to the separatory funnel and removed (following mixing and separation) to the tared beaker. The organic extract from Sample Type 4 was combined with the organic train rinse in sample type 4. The organic fraction was evaporated at room temperature (not to exceed 85°F) to approximately 10 ml. The resulting liquid was transferred to a preweighed tin, evaporated to dryness at room temperature (not to exceed 85°F), desiccated for 24 hours and weighed to a constant \pm 0.5 mg to the nearest 0.1 mg.
- 4. 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 not less than 10 ml in an oven at 105°C, then allowed to evaporate to dryness at room temperature. After obtaining dryness, the residue was re-dissolved in 100 ml distilled water. The sample was titrated to a pH of 7.0 using NH₄OH (of known normality). The volume of titrant was recorded. The solution was then evaporated to approximately 10 ml. The resulting liquid was transferred to a preweighed tin, evaporated to dryness at room temperature (not to exceed 85°F), desiccated for 24 hours and weighed to a constant \pm 0.5 mg to the nearest 0.1 mg.
- 5. The water soluble condensable particulate matter from the Teflon filter was extracted from the filter using ultra-filtered water in an extraction tube and sonication bath. The aqueous extract was combined with the contents of Sample Type 4. The organic soluble condensable particulate matter from the Teflon filter

was extracted from the filter using methylene chloride in an extraction tube and sonication bath. The organic extract was combined with the contents of Sample Type 5.

6. The field blank train and blank samples of acetone, distilled water and hexane were analyzed as described above.

The total of the organic and inorganic fractions represents the condensable particulate catch. The PM_{10} includes the filterable PM_{10} particulate catch (front-half acetone sample No. 2 and filter) plus the organic and inorganic condensable.

4.6 REFERENCE METHOD GASEOUS MONITORING SYSTEM

A continuous emission monitoring trailer equipped with instrumental analyzers was used to measure concentrations of oxygen O₂/CO₂, SO₂, and NO_x (see Figure 4-4). A description of each instrumental analyzer is provided below:

Pollutant	EPA Reference Method	Operating Principle
O ₂	3A	Paramagnetic
CO ₂	3A	Single beam, single wavelength infrared
SO ₂	6C	Ultraviolet
NOx	7E	Chemiluminescent

Stack gas was withdrawn from the stack through a heated stainless steel probe and heated filter via a heated sample line maintaining a temperature > 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 analyzers via a Teflon® sample line. A separate Teflon® line was used for introduction of O_2/CO_2 , SO_2 , and NOx bias gases to the probe outlet.

4.6.1 NOx, SO₂ and O₂/CO₂ Monitoring Procedures

The analyzers were calibrated daily by direct introduction of EPA Protocol calibration gases to the analyzers. These gases are prepared with a balance of nitrogen and nitrogen is also used as the zero gas. After the analyzer calibration, a system bias check was conducted by introducing the zero gas and one selected O_2/CO_2 , SO₂, and NOx calibration gas to the sample probe outlet.



FIGURE 4-4 WESTON SAMPLING SYSTEM 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 instrumental analyzers were previously performed (December 2014) in accordance with EPA Method 7E and were not repeated for this test program.

Additionally, an O₂ stratification check was performed prior to the test effort in accordance with EPA Method 7E – Section 8.1.2. Based on the stratification test results, no more than \pm 5.0% difference of the average for each traverse point, the WESTON system sampled from a single point during all formal test runs.

Gas stream moisture content and stack volumetric flow rate data from the corresponding isokinetic testing were used to calculate NOx and SO₂ mass emission rates.

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 used to compute the average concentration for the test run.

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.

Although not required in the PTI, composite samples of each fuel type were submitted for chlorine, moisture, and heat content analysis. This approach remains consistent with the most recent HCl semi-annual test program. 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 collected fuel samples twice per run (approximately beginning and mid-point). Table 5-1 provides a summary of the fuel sample analytical methods.

Fuel Type	Required Analysis	Analytical Methods	Minimum Detection Level
Wood, Creosote Treated	Moisture Content	<u>ASTM D3173</u> , "Standard Test Method for Moisture in the Analysis Sample of Coal and Coke"	Not Applicable
Wood Derived Fuel (CDF) & Pellets	Chlorine Concentration	EPA 5050/9056, "Determination of Inorganic Anions by Ion Chromatography"	~50 ppm
	Heat Content	<u>ASTM D5865</u> , "Standard Test Method for Gross Calorific Value of Coal and Coke"	Not Applicable

27

Table 5-1Fuel Sample Analytical Methods

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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:

29

• 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 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 \pm 10 percent of the true isokinetic (100 %) rate.
- Points by point isokinetic sampling rates (± 20 percent) were calculated for the PM₁₀ sampling train.
- 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 O₂ stratification check 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 an accredited Stationary Source Audit Sample (SSAS) provider [Sigma-Aldrich RTC, Inc. (RTC)] in accordance with the EPA SSAS program. The audit samples were analyzed in conjunction with the stack samples.

It should be noted the analytical laboratory [Maxxam Analytics (MAXXAM)] reported a result slightly below the acceptable range for the arsenic on filter paper audit sample, whereas the arsenic in solution result was reported within the provider's acceptance window. MAXXAM requested a second arsenic filter sample from the same lot number, but at the time of this report writing MAXXAM had not been contacted by RTC. WESTON telephoned Mr. Tom Gasloli of the MDEQ-AQD regarding the issue and it was concluded that the arsenic results would be acceptable in this case since the facility does not have a specific emissions limit for arsenic. All other audit sample values listed in the provider evaluation report indicate passing results for each sample submitted.