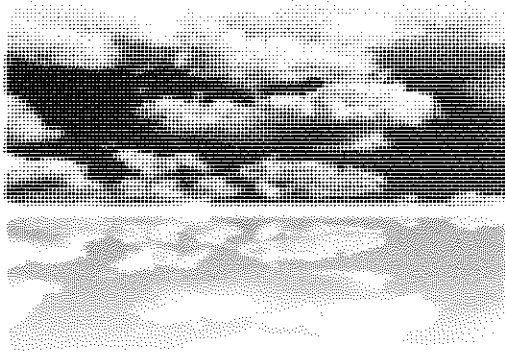




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REPORT ON COMPLIANCE
TESTING

Zug Island EES Coke Battery, LLC
Underfire Combustion Stack

DTE Energy
1400 Zug Island
River Rouge, Michigan 48218
Client Reference No. 4701118953

CleanAir Project No. 13297-3
STAC Certificate No. 2007.002.0113.1217
Revision 0, Final Report
Submittal Date – November 17, 2017

1. PROJECT OVERVIEW

Test Program Summary

DTE Energy contracted CleanAir Engineering (CleanAir) to complete testing on the Underfire Combustion Stack at the Zug Island EES Coke Battery, LLC located in River Rouge, Michigan. The test program included the following objectives:

- Perform total particulate matter (TPM), filterable and condensable particulate matter testing to demonstrate compliance with Michigan Permit to Install (MI-PTI) No. 51-08C.
- Perform nonsulfate filterable particulate matter testing to demonstrate compliance with Michigan Permit to Install (MI-PTI) No. 51-08C.
- Perform volatile organic compound (VOC) testing to demonstrate compliance with Michigan Permit to Install (MI-PTI) No. 51-08C.

A summary of the test program results is presented below. Section 2 Results provides a more detailed account of the test conditions and data analysis. Test program information, including the test parameters, on-site schedule and a project discussion, begin on page 2.

**Table 1-1:
Summary of Results**

Source Constituent	Sampling Method	Average Emission	Permit Limit ¹
<u>Underfire Combustion Stack</u>			
PM (lb/hr)	EPA M5F	0.10	25.7 ²
PM (gr/dscf)	EPA M5F	0.000083	0.012 ²
PM (lb/1000 lb exhaust gas @50% EA)	EPA M5/202	0.009	0.095
PM ₁₀ (lb/hr) ⁴	EPA M5/202	30.2	73.3
PM _{2.5} (lb/hr) ⁴	EPA M5/202	30.2	73.0
VOC (lb/hr)	EPA M25A	12.7	43.1 ³
VOC (lb/MMBtu, heat input)	EPA M25A	0.0181	0.0956 ³

¹ Permit limits obtained from Michigan Permit to Install (MI-PTI) No. 51-08C.

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² Excludes sulfates

³ Excludes Methane concentrations

⁴ Total PM from Method 5/202 were compared to PM10 and PM2.5 limits.

Test Program Details

Parameters

The test program included the following measurements:

- total particulate matter (TPM), filterable and condensable particulate matter reported as:
 - particulate matter less than 10 microns in diameter (PM₁₀)
 - particulate matter less than 2.5 microns in diameter (PM_{2.5})
- nonsulfate particulate matter (NSPM)
- volatile organic compounds (VOC), excluding methane (CH₄)
- flue gas composition (e.g., O₂, CO₂, H₂O)
- flue gas temperature
- flue gas flow rate

Schedule

Testing was performed the week of September 25th, 2017. The on-site schedule followed during the test program is outlined in Table 1-2.

**Table 1-2:
Test Schedule**

Run Number	Location	Method	Analyte	Date	Start Time	End Time
1	Underfire Combustion Stack	USEPA Method 2	Velocity	09/27/17	15:04	15:40
2	Underfire Combustion Stack	USEPA Method 2	Velocity	09/27/17	16:18	16:55
3	Underfire Combustion Stack	USEPA Method 2	Velocity	09/27/17	17:32	18:09
1	Underfire Combustion Stack	USEPA Method 4	Moisture	09/27/17	15:04	15:39
2	Underfire Combustion Stack	USEPA Method 4	Moisture	09/27/17	16:16	16:51
3	Underfire Combustion Stack	USEPA Method 4	Moisture	09/27/17	17:32	18:07
1	Underfire Combustion Stack	Method 3A, 25A	O ₂ , CO ₂ , VOC	09/27/17	14:57	15:57
2	Underfire Combustion Stack	Method 3A, 25A	O ₂ , CO ₂ , VOC	09/27/17	16:14	17:14
3	Underfire Combustion Stack	Method 3A, 25A	O ₂ , CO ₂ , VOC	09/27/17	17:31	18:31
2	Underfire Combustion Stack	USEPA Method 5F	Nonsulfate PM	09/28/17	08:12	11:02
3	Underfire Combustion Stack	USEPA Method 5F	Nonsulfate PM	09/28/17	12:50	15:27
4	Underfire Combustion Stack	USEPA Method 5F	Nonsulfate PM	09/28/17	16:38	19:03
2	Underfire Combustion Stack	USEPA Method 5/202	FPM/CPM	09/28/17	08:12	11:02
3	Underfire Combustion Stack	USEPA Method 5/202	FPM/CPM	09/28/17	12:50	15:27
4	Underfire Combustion Stack	USEPA Method 5/202	FPM/CPM	09/29/17	08:00	10:29

Discussion

Project Synopsis

Permit Requirements

PM₁₀/PM_{2.5}

Appendix A of Permit 51-08C states that requirements for PM₁₀ and PM_{2.5} shall follow EPA Method 201A/202. The duration and sample volume was 120 minutes and 60 dscf, respectively. CleanAir proposed the use of Method 5 in lieu of Method 201A.

The test ports at the sample location were not an adequate size to accommodate the PM₁₀/PM_{2.5} cyclone head. The 2015 test program encountered many issues with broken glass due to the narrow and long test ports. In the past, CleanAir performed Method 201A/202 versus Method 5/202 and analysis of the results indicated an acceptable comparison between the results. For example, the 3-run average (gr/dscf) of TPM for Methods 201A/202 and 5/202 were 0.0466 and 0.0484, respectively. The Method 5/202 results were approximately 3.7% higher than the 201A/202 results.

The appendix states that any changes to the testing method must be approved by the AQD District Supervisor. Test data from the 2015 compliance program highlight a similarity between the readings with the Method 5/202 results being biased slightly higher than the 201A/202 results. Total PM (TPM) is defined as the sum of filterable and condensable particulate matter. Method 5/202 does not provide unique values for PM₁₀ and PM_{2.5} and TPM will instead be used to determine PM₁₀ and PM_{2.5} emissions.

Non-Sulfate PM

Appendix A of Permit 51-08C states that requirements for particulate matter determinations (excluding sulfates) must be conducted per EPA Method 5 corrected for sulfate. However, CleanAir discussions with EES and MDEQ led to using a modification of EPA Method 5F. Details from that call are outlined in the Modifications to Test Methodology section. The permit also states a sample time of 60 minutes and minimum volume of 30 dscf. CleanAir performed a longer test based on results from the 2015 compliance test campaign.

VOC

VOC emission rates from the Underfire Combustion Stack were measured during this test program. Testing followed EPA Method 25A and a total of three 1-hour tests were performed at a single point. VOC results were reported on a propane-basis. The methodology section of this protocol provides additional information on the approach to VOC determination.

Invalidated Test Runs

CleanAir had no invalidated Method 25A tests.

During the test program, a total of four tests for Methods 5F and 5/202 were performed. The first test run for each was invalidated. CleanAir's test leader was in contact with the on-site MDEQ representative, Mark Dziadosz, regarding the tests. The Method 5/202 test train experienced a vacuum issue which limited the test's ability to pull isokinetically. After experiencing a long delay, all the PM test runs were restarted, per MDEQ. The three consecutive runs used for the report were runs 2-4.

Modifications to Test Methodology

Determination of Nonsulfate Particulate Matter – EPA Method 5F

A conference call between EES, MDEQ and CleanAir representatives was held on Monday, January 26, 2015 to discuss the best methodology for the determination of sulfate free particulate emissions at the Underfire Combustion Stack. It was agreed upon to perform EPA Method 5F for the sulfate-free particulate matter (nonsulfate PM) measurements. This method is contained in Appendix A of 40 CFR 60.

Particulate matter was withdrawn isokinetically and collected on a filter maintained at a temperature in the range of $320 \pm 25^\circ\text{F}$. A minimum of 60 dry standard cubic feet (dscf) of sample gas was collected over a 120-minute test period for each run.

During the conference call between MDEQ, EES and CleanAir on January 26, 2015 MDEQ elected for Method 5F testing over Method 5B. However, concern was raised by MDEQ that the recovery of the probe with a water rinse would not be adequate and requested a change to acetone. The following deviations to the method were agreed upon during the conference call and were performed on-site:

1. The sample train nozzle, probe liner and front-half filter holder were rinsed and recovered with acetone (Method 5F outlines the use of deionized distilled water; ASTM D1193-77 or 91 Type 3).
2. Due to the use of acetone, additional analytical steps were taken by the CleanAir Analytical laboratory, located in Palatine Illinois, during the first recovery step:
 - a. The acetone was evaporated in a tared FEP beaker liner while the filter was being digested.
 - b. The acetone residue was combined with the filter digestate and brought to volume in a 500mL flask.
 - c. The flask could settle and an aliquot was removed for sulfate determinations via Ion Chromatography (IC).
 - d. The solution was re-evaporated in the original tared FEP beaker liner, and then the normal analytical steps, as outlined in Method 5F, were followed.

Note: The above approach to Method 5F was followed during the compliance testing in 2015 and 2017.

Particulate Testing – EPA Method 5F and 5/202

During the compliance test program of 2015, CleanAir experienced several invalidated tests as a result of broken glassware. This was a product of both the large stack diameter and long test ports which required long test probes. As this location experiences high winds, it increases the likelihood of broken glassware during port changes.

An excerpt from Section 6.1.1.2 of EPA Method 5 reads:

Alternatively, metal liners (e.g., 316 stainless steel, Incoloy 825 or other corrosion resistant metals) made of seamless tubing may be used, subject to the approval of the Administrator.

CleanAir requested and obtained approval to use stainless steel-lined probes and nozzles in lieu of glass, or quartz, liners for NSPM and TPM testing during the 2017 compliance campaign.

2. RESULTS

This section summarizes the test program results. Additional results are available in the report appendices, specifically Appendix C Parameters.

**Table 2-1:
Underfire Combustion Stack –TPM, Method 5/202**

Run No.		2	3	4	Average
Date (2017)		Sep 28	Sep 28	Sep 29	
Start Time (approx.)		08:12	12:50	08:00	
Stop Time (approx.)		11:02	15:27	10:29	
Process Conditions					
Cap	Capacity factor (hours/year)	8,760	8,760	8,760	8,760
Gas Conditions					
O ₂	Oxygen (dry volume %)	10.6	10.2	9.8	10.2
CO ₂	Carbon dioxide (dry volume %)	4.9	4.9	5.1	5.0
T _s	Sample temperature (°F)	528	528	526	527
B _w	Actual water vapor in gas (% by volume)	14.2	13.9	15.1	14.4
Gas Flow Rate					
Q _a	Volumetric flow rate, actual (acfm)	286,000	288,000	300,000	291,000
Q _s	Volumetric flow rate, standard (scfm)	150,000	151,000	158,000	153,000
Q _{std}	Volumetric flow rate, dry standard (dscfm)	128,000	130,000	134,000	131,000
Sampling Data					
V _{std}	Volume metered, standard (dscf)	72.76	73.65	76.12	74.18
%I	Isokinetic sampling (%)	96.0	96.0	96.4	96.1
Laboratory Data					
m _n	Total FPM (g)	0.04673	0.04316	0.04013	
m _{CPM}	Total CPM (g)	0.09273	0.10004	0.06553	
m _{Part}	Total particulate matter (g)	0.13946	0.14320	0.10566	
n _{MDL}	Number of non-detectable fractions	N/A	N/A	N/A	
DLC	Detection level classification	ADL	ADL	ADL	
FPM Results					
C _{sd}	Particulate Concentration (lb/dscf)	1.416E-06	1.292E-06	1.162E-06	1.290E-06
C _{sd}	Particulate Concentration (gr/dscf)	0.0099	0.0090	0.0081	0.0090
E _{lb/yr}	Particulate Rate (lb/hr)	10.9	10.1	9.3	10.1
E _{T/yr}	Particulate Rate (Ton/yr)	47.8	44.2	40.9	44.3
CPM Results					
C _{sd}	Particulate Concentration (lb/dscf)	2.810E-06	2.995E-06	1.898E-06	2.568E-06
C _{sd}	Particulate Concentration (gr/dscf)	0.0197	0.0210	0.0133	0.0180
E _{lb/yr}	Particulate Rate (lb/hr)	21.7	23.4	15.2	20.1
E _{T/yr}	Particulate Rate (Ton/yr)	94.9	102.4	66.7	88.0
Total Particulate Matter Results					
C _{sd}	Particulate Concentration (lb/dscf)	4.227E-06	4.287E-06	3.061E-06	3.858E-06
C _{sd}	Particulate Concentration (gr/dscf)	0.0296	0.0300	0.0214	0.0270
E _{lb/yr}	Particulate Rate (lb/hr)	32.6	33.5	24.6	30.2
E _{T/yr}	Particulate Rate (Ton/yr)	142.7	146.5	107.6	132.3
E _{EA50%}	Particulate Rate (lb per 1000lb exhaust gas at 50% EA)	0.0102	0.0086	0.0071	0.0087

Average includes 3 runs.

**Table 2-2:
Underfire Combustion Stack – NSPM, Method 5F**

Run No.	2	3	4	Average
Date (2017)	Sep 28	Sep 28	Sep 28	
Start Time (approx.)	08:12	12:50	16:38	
Stop Time (approx.)	11:02	15:27	19:03	
Process Conditions				
Cap Capacity factor (hours/year)	8,760	8,760	8,760	8,760
Gas Conditions				
O ₂ Oxygen (dry volume %)	10.6	9.7	10.1	10.1
CO ₂ Carbon dioxide (dry volume %)	4.9	5.2	4.8	5.0
T _s Sample temperature (°F)	524	526	529	526
B _w Actual water vapor in gas (% by volume)	14.5	14.3	14.2	14.3
Gas Flow Rate				
Q _a Volumetric flow rate, actual (acfm)	325,000	314,000	315,000	318,000
Q _s Volumetric flow rate, standard (scfm)	171,000	165,000	165,000	167,000
Q _{std} Volumetric flow rate, dry standard (dscfm)	146,000	141,000	142,000	143,000
Sampling Data				
V _{mstd} Volume metered, standard (dscf)	85.48	83.28	81.73	83.49
%I Isokinetic sampling (%)	99.0	100.9	98.9	99.6
Laboratory Data				
m _n Total NSFPM (g)	0.00045	0.00045	0.00045	
n _{MDL} Number of non-detectable fractions	N/A	N/A	N/A	
DLC Detection level classification	ADL	ADL	ADL	
NSFPM Results				
C _{sd} Particulate Concentration (lb/dscf)	1.16E-08	1.19E-08	1.21E-08	1.19E-08
C _{sd} Particulate Concentration (gr/dscf)	0.000081	0.000083	0.000085	0.000083
E _{lb/hr} Particulate Rate (lb/hr)	0.1019	0.1011	0.1032	0.1021
E _{T/yr} Particulate Rate (Ton/yr)	0.4465	0.4430	0.4519	0.4471

Average includes 3 runs.

**Table 2-3:
 Underfire Combustion Stack – VOC, Method 25A**

Run No.	1	2	3	Average
Date (2017)	Sep 27	Sep 27	Sep 27	
Start Time	14:57	16:14	17:31	
End Time	15:57	17:14	18:31	
Elapsed Time	1:00	1:00	1:00	
Process Conditions				
Actual Gas Flow Rate - Underfire Combustion Stack (acfm)	285,882	288,161	299,867	291,303
Standard Gas Flow Rate - Underfire Combustion Stack (scfm)	149,791	151,062	157,551	152,801
Dry Standard Gas Flow Rate - Underfire Combustion Stack (dscfm)	128,469	130,040	106,816	130,757
H2O - Underfire Combustion Stack (%)	14.85	14.05	14.90	14.60
Gas Conditions				
Oxygen (O2) - Underfire Combustion Stack (%dv)	10.56	10.65	10.51	10.58
Carbon Dioxide (CO2) - Underfire Combustion Stack (%dv)	4.95	4.88	5.03	4.95
Non-Methane Hydrocarbons (NMHC) - Underfire Combustion Stack				
Concentration (ppmwv)	10.45	8.77	9.23	9.48
Mass Rate (lb/hr)	10.83	9.11	7.96	9.30
Mass Rate (lb/MMBtu) - Heat Input	0.021	0.018	0.015	0.018

Note:
 Runs 1-3 Process Conditions taken from M5/202

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End of Section

3. DESCRIPTION OF INSTALLATION

Process Description

EES Coke Battery, LLC is a DTE Energy Service facility located on Zug Island in River Rouge, Michigan. The testing described in this document was performed at the Underfire Combustion Stack location.

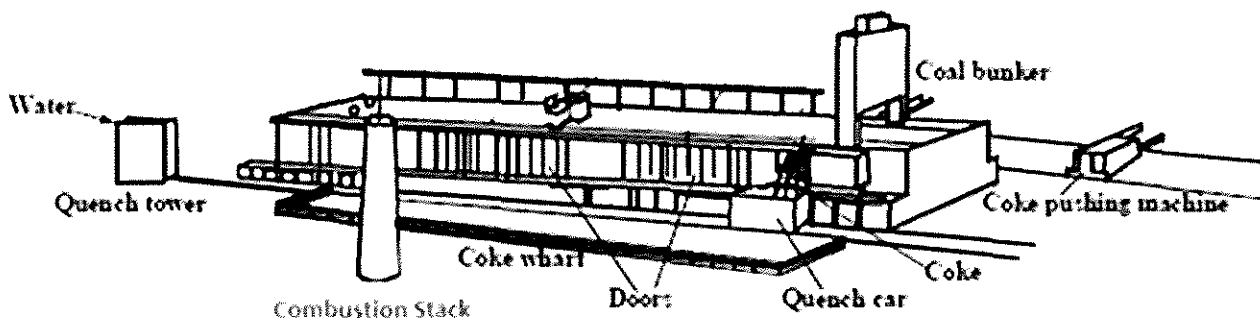
The No. 5 Coke Battery consists of 85 six-meter-high ovens producing furnace coke. A coal blend is used to charge each oven on timed intervals depending on the current production of the battery. Coking of the coal occurs in an oxygen free environment for 17 to 30 hours and the gases produced are collected, cleaned, and used to under fire the battery, supply fuel for other site sources, and sold to permitted off-site utilities.

The current permit limits allow for the charging of up to 1.420 million dry tons of coal. The design capacity heating requirement of the battery is approximately 375 MMBtu per hour. Also, the heating requirements of the battery at the current production rate are approximately 325 MMBtu per hour.

Process source description information above was taken directly from written information provided by DTE Energy.

A schematic of the process, indicating proposed sampling locations, is shown in Figure 3-1.

Figure 3-1:
Process Schematic



Note: The EES Coke Battery Combustion Stack is located on the other side of the battery as depicted in the drawing.

Test Location

EPA Method 1 specifications determined the sample point locations. Table 3-1 presents the sampling information for the test location. The figure shown on page 10 represents the layout of the test location.

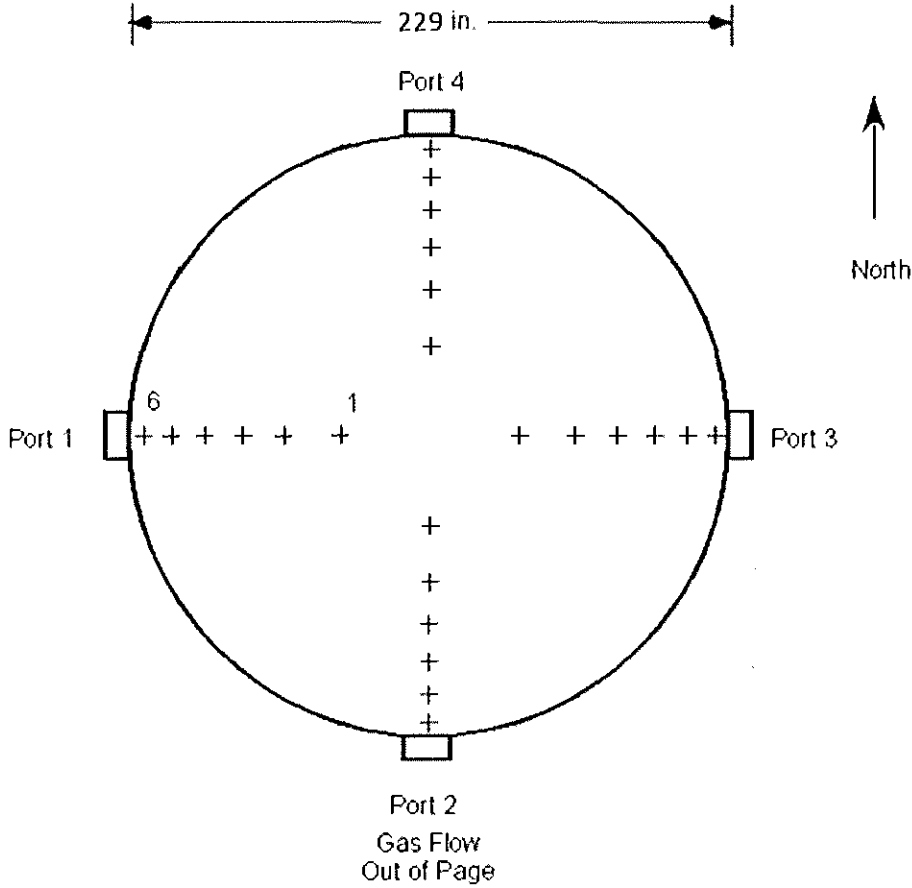
**Table 3-1:
Sampling Information**

Source	Method	Run No.	Ports	Points per Port	Minutes per Point	Total Minutes	Figure
<u>Underfire Combustion Stack</u>							
NSPM	EPA M5F	1-3	4	6	5	120	3-2
TPM	EPA M5/202	1-4	4	6	5	120	3-2
VOC ¹	EPAM25A	1-3	1	1	60	60	N/A

¹ VOC measurements were collected from a single point.

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**Figure 3-2:
 Underfire Combustion Stack Sample Point Layout (EPA Method 1)**



Sampling Point	% of Stack Diameter	Port to Point Distance (inches)
1	2.1	4.8
2	6.7	15.3
3	11.8	27.0
4	17.7	40.5
5	25.0	57.3
6	35.6	81.5

Duct diameters upstream from flow disturbance (A): 10.9
 Duct diameters downstream from flow disturbance (B): 2.7

Limit: 0.5
 Limit: 2.0

4. METHODOLOGY

Procedures and Regulations

The test program sampling measurements followed procedures and regulations outlined by the USEPA and State Agency Name. These methods appear in detail in Title 40 of the CFR and at <https://www.epa.gov/emc>. Appendix A includes diagrams of the sampling apparatus, as well as specifications for sampling, recovery and analytical procedures.

CleanAir follows specific QA/QC procedures outlined in the individual methods and in USEPA "Quality Assurance Handbook for Air Pollution Measurement Systems: Volume III Stationary Source-Specific Methods," EPA/600/R-94/038C. Appendix D contains additional QA/QC measures, as outlined in CleanAir's internal Quality Manual.

Title 40 CFR Part 60, Appendix A

- Method 1 "Sample and Velocity Traverses for Stationary Sources"
- Method 2 "Determination of Stack Gas Velocity and Volumetric Flow Rate (Type S Pitot Tube)"
- Method 3A "Determination of Oxygen and Carbon Dioxide Concentrations in Emissions from Stationary Sources (Instrumental Analyzer Procedure)"
- Method 4 "Determination of Moisture Content in Stack Gases"
- Method 5 "Determination of Particulate Matter Emissions from Stationary Sources"
- Method 5F "Determination of Nonsulfate Particulate Matter Emissions from Stationary Sources"
- Method 25A "Determination of Total Gaseous Organic Concentration Using a Flame Ionization Analyzer"

Title 40 CFR Part 51, Appendix M

- Method 202 "Dry Impinger Method for Determining Condensable Particulate Emissions from Stationary Sources"

Methodology Discussion

Verification of the Absence of Cyclonic Flow – EPA Method 1

A cyclonic flow check was performed in accordance with EPA Method 1, Section 2.4 during the compliance test program in 2015. The results of that test indicated an absence of cyclonic flow. This test was not repeated and results are available as an appendix to the test report.

The cyclonic flow check procedure was referred to as the "nulling" technique. An S-type pitot tube connected to an inclined manometer was used in this method. This was the same apparatus as referenced in EPA Method 2.

The pitot tube was positioned at each of the EPA Method 1 traverse point locations so that the face openings of the pitot tube were orientated perpendicular to the stack or duct cross-sectional plane. This position was referenced as the "0° reference." The velocity pressure (ΔP) measurement at this position was recorded.

If the ΔP reading was zero, a cyclonic angle of 0° was recorded. If the ΔP reading was not zero, the pitot tube was rotated clockwise (positive) or counter-clockwise (negative), as required, to obtain a zero ΔP reading. The angle required to obtain the zero reading was measured using a digital protractor (± 0.1 degree) attached to the pitot tube.

Determination of Flue Gas Composition – Methods 1-4

CleanAir measured flow rates using S-type pitot tubes following sampling point requirements of EPA Methods 1 and 2. The testing occurred in 4 test ports at 6 points per port for a total of 24 points. The pitot tube measurements were used to determine the stack gas velocity and volumetric flow rate. EPA Method 3A was followed to determine the oxygen and carbon dioxide content of the flue gas. Values were obtained via continuous extraction of CleanAir CEMS or via grab samples. EPA Method 4 was followed to determine the moisture content of the sample.

The methods mentioned above were utilized to determine the flue gas volumetric flow rate and composition.

Nonsulfate Particulate Matter – Method 5F (modified)

EPA Method 5F, "Determination of Nonsulfate Particulate Matter Emissions from Stationary Sources," was used for the nonsulfate particulate matter (NSPM) measurements. This method was contained in Appendix A of 40 CFR 60.

Particulate matter was withdrawn isokinetically from the source and collected on a quartz fiber filter maintained at a temperature of $160^{\circ}\text{C} \pm 14^{\circ}\text{C}$ ($320^{\circ}\text{F} \pm 25^{\circ}\text{F}$). A minimum of 60 dry standard cubic feet of sample gas was collected over a two-hour test period for each run. Flue gas volumetric flow rate, moisture concentration and flue gas molecular weight were also determined as part of the sample method. The previously agreed upon method of analysis, discussed in the Modifications to Test Methodology section, was followed.

Total Particulate Matter Determination – Method 5/202

EPA Method 5, "Determination of Particulate Emissions from Stationary Sources," was followed for the filterable particulate matter (FPM) measurements. This method's contained in Appendix A of 40 CFR 60. Method 5 defines particulate matter as any material that is collected before or on the surface of a quartz fiber filter.

Stack gas was isokinetically withdrawn through a temperature-controlled probe and high-efficiency quartz fiber filter. A minimum of 60 dry standard cubic feet of sample gas was collected over a two-hour test period for each run. The mass of particulate collected on the filter and in the sampling probe was determined gravimetrically.

The back-half impinger catch was analyzed in accordance with EPA Method 202. The impinger solution was extracted and the organic and aqueous fractions were taken to dryness and the residues weighed. The total of both fractions represented the condensable particulate matter (CPM). The combined FPM and CPM fractions result in total filterable particulate matter (TPM).

Volatile Organic Compounds, Excluding Methane – Method 25A

The flue gas was continuously sampled and for temperature and total hydrocarbons (THCs). THC concentrations were collected on a wet-basis as the analyzer measured the sample prior to conditioning. The flue gas sample was maintained at a temperature sufficient to prevent condensation from extraction through analysis. Temperature set points were 250°F ± 25°F.

CleanAir used the Thermo 55i analyzer for the measurements. This model used two individual detectors and two individual signal amplifiers. The sample was introduced into one FID for THC readings. The gas sample then ran through a non-methane cutter which eliminated all hydrocarbons except methane before being analyzed by the second FID. The analyzer subtracted the two values to provide a VOC (excluding methane) reading.

End of Section