1. PROJECT OVERVIEW

Test Program Summary

EES Coke Battery, LLC contracted CleanAir Engineering (CleanAir) to complete compliance testing on the Underfire Combustion Stack at the Zug Island facility located in River Rouge, Michigan.

The test program objective is to perform total particulate matter (TPM), non-sulfate filterable particulate matter, and volatile organic compound (VOC) testing to demonstrate compliance with Michigan Permit to Install (MI-PTI) No. 51-08C.

A summary of the test program limits is presented below. Section 2 Results provides a more detailed account of the test conditions and data analysis.

Table 1-1:
Summary of Permit Limits

Source Constituent	Sampling Method	Average Emission	Permit Limit[1]
Underfire Combustion Stack			
PM (lb/hr)[2]	EPA 5F (Modified)	xx.x	25.7
PM (gr/dscf)[2]	EPA 5F (Modified)	x.xxx	0.012
PM (lb/1000 lb exhaust gas @50% EA)	EPA 5	x.xxx	0.095
PM_{10} (lb/hr)[3]	EPA 5/202	xx.x	73.3
$PM_{2.5}$ (lb/hr)[3]	EPA 5/202	xx.x	73.0
VOC (lb/hr)[4]	EPA 25A	xx.x	43.1
VOC (lb/MMBtu, heat input)[4]	EPA 25A	x.xxx	0.0956

[1] Permit limits obtained from MI-PTI No. 51-08C.

[2] Excludes sulfates.

[3] TPM from Method 5/202 will be compared to PM_{10} and $PM_{2.5}$ limits.

[4] Excludes methane concentrations.

Test Program Details

PARAMETERS

The test program will include the following measurements:

- total particulate matter (TPM), filterable and condensable particulate matter (FPM and CPM), reported as:
  - particulate matter less than 10 microns in diameter (PM_{10})
  - particulate matter less than 2.5 microns in diameter ($PM_{2.5}$)
- non-sulfate filterable particulate matter (NSFPM)
- volatile organic compounds (VOC), excluding methane (CH_4), measured as total hydrocarbons (THC)
- flue gas composition (e.g., O_2, CO_2, H_2O)
- flue gas temperature
- flue gas flow rate

SCHEDULE

The test program is tentatively scheduled for the week of September 13, 2021. Table 1-2 outlines the proposed timetable.

Table 1-2:
Test Schedule

Day	Activity	Test Method	Number of Test Runs	Duration of Each Test Run
1	Travel to Project Site Equipment Set-up			
2	NSFPM	EPA 5F (Modified)	2	120 min.
	TPM (PM_{10}/$PM_{2.5}$)	EPA 5/202	2	120 min.
	VOC/NMOC	EPA 25A	3	60 min.
3	NSFPM	EPA 5F (Modified)	1	120 min.
	TPM (PM_{10}/$PM_{2.5}$)	EPA 5/202	1	120 min.
4	Demobilize			

DISCUSSION

$PM_{10}/PM_{2.5}$

Appendix A of MI-PTI No. 51-08C states that testing for PM_{10} and $PM_{2.5}$ follow EPA Methods 201A and 202. The test duration is listed as 120 minutes, with a minimum sample volume requirement of 60 dscf, respectively. The appendix states that any changes to the test methodology must be approved by the Michigan Department of Environment, Great Lakes & Energy (EGLE) Air Quality Division (AQD) District Supervisor.

Proposed Modifications to $PM_{10}/PM_{2.5}$ Testing

The test ports at the sample location are not an adequate size to accommodate the Method 201A $PM_{10}/PM_{2.5}$ cyclone head. Numerous issues with broken glass due to the narrow and long test ports occurred during the 2015 test campaign. CleanAir proposes the use of EPA Method 5 in lieu of Method 201A. This follows the 2019 test program.

CleanAir performed a results comparison between Method 201A/202 versus Method 5/202. 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 Method 201A/202 results. For example, the three-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 2019 test report for CleanAir Project 13938 also included a comparison of results from 2017 to 2019 to ensure results were similar.

TPM is defined as the sum of filterable and condensable particulate matter. Method 5/202 does not provide unique values for PM_{10} and $PM_{2.5}$ and TPM will instead be used to determine PM_{10} and $PM_{2.5}$ emissions. The use of Method 5 rather than Method 201A was allowed during the 2019 test program. This approach will conservatively report PM_{10} and $PM_{2.5}$ results high.

In addition, this location experiences high winds that increase the likelihood of broken glassware during port changes. CleanAir is requesting approval to use stainless steel-lined probes and nozzles in lieu of borosilicate glass or quartz liners, which was approved for the 2017 and 2019 compliance campaigns.

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."*

NSFPM

Appendix A of MI-PTI No. 51-08C states that requirements for particulate matter determinations (excluding sulfates) must be conducted per EPA Method 5, corrected for sulfate. The permit also requires a sample time of 60 minutes, with a minimum sample volume of 30 dscf. Any changes to the testing methods must be approved by the AQD District Supervisor.

Proposed Modifications to NSFPM Testing

CleanAir is proposing particulate matter be withdrawn isokinetically and collected on a filter maintained at a temperature in the range of 320 ±25°F, with a minimum of 60 dscf of sample gas collected over a 120-minute test period for each run. The modification was followed during compliance testing in 2015, 2017 and 2019 based on the conversation documented below.

A conference call between EES, EGLE, 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 filterable particulate matter measurements. This method is contained in Appendix A of 40 CFR 60.

Concern was raised by EGLE 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 will be performed on-site:

1. The sample train nozzle, probe liner, and front-half filter holder will be 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 will be taken by the CleanAir Analytical laboratory, located in Palatine Illinois, during the first analytical step:
    a. The acetone will be evaporated in a tared FEP beaker liner while the filter is being digested.
    b. The acetone residue will be combined with the filter digestate and brought to volume in a 500 mL flask.
    c. The flask will be allowed to settle, and an aliquot will be removed for sulfate determinations.
    d. The solution will be re-evaporated in the original tared FEP beaker liner and the normal analytical steps, as outlined in Method 5F, will be followed.

In addition, CleanAir is requesting approval to use stainless steel-lined probes and nozzles in lieu of borosilicate glass or quartz liners during the 2021 compliance campaign as mentioned in the $PM_{10}/PM_{2.5}$ discussion above. This was approved during the previous test programs.

VOC

VOC emission rates from the Underfire Combustion Stack will be completed following EPA Method 25A. A total of three 60-minute tests will be performed at a single point pending the results of a stratification check. VOC results will be supplied on a propane-basis. The Methodology section of this protocol provides additional information on the approach to VOC determination.

End of Section

2. RESULTS

The example tables summarize how data will be presented in the test report.

Table 2-1:
Underfire Combustion Stack – NSFPM, Method 5F, Modified (Example)

Run No.		1	2	3	Average
Date (2021)		MMDD	MMDD	MMDD	
Start Time (approx.)		hh:mm	hh:mm	hh:mm	
Stop Time (approx.)		hh:mm	hh:mm	hh:mm	
Process Conditions					
Cap	Capacity factor (hours/year)	8,760	8,760	8,760	**8,760**
Gas Conditions					
O_2	Oxygen (dry volume %)	xx.x	xx.x	xx.x	**xx.x**
CO_2	Carbon dioxide (dry volume %)	x.x	x.x	x.x	**x.x**
T_s	Sample temperature (°F)	xxx	xxx	xxx	**xxx**
B_w	Actual water vapor in gas (% by volume)	xx.x	xx.x	xx.x	**xx.x**
Gas Flow Rate					
Q_a	Volumetric flow rate, actual (acfm)	xxx,xxx	xxx,xxx	xxx,xxx	**xxx,xxx**
Q_s	Volumetric flow rate, standard (scfm)	xxx,xxx	xxx,xxx	xxx,xxx	**xxx,xxx**
Q_{std}	Volumetric flow rate, dry standard (dscfm)	xxx,xxx	xxx,xxx	xxx,xxx	**xxx,xxx**
Laboratory Data					
m_n	Total NSFPM (g)	x.xxxxx	x.xxxxx	x.xxxxx	
NSFPM Results					
C_{sd}	Particulate Concentration (lb/dscf)	x.xxE-xx	x.xxE-xx	x.xxE-xx	**x.xxE-xx**
C_{sd}	Particulate Concentration (gr/dscf)	x.xxxx	x.xxxx	x.xxxx	**x.xxxx**
$E_{lb/hr}$	Particulate Rate (lb/hr)	xx.x	xx.x	xx.x	**xx.x**
$E_{T/yr}$	Particulate Rate (Ton/yr)	xx.x	xx.x	xx.x	**xx.x**

Average includes 3 runs.

Table 2-2:
Underfire Combustion Stack – TPM, Method 5/202 (Example)

Run No.		1	2	3	Average
Date (2021)		MMDD	MMDD	MMDD	
Start Time (approx.)		hh:mm	hh:mm	hh:mm	
Stop Time (approx.)		hh:mm	hh:mm	hh:mm	
Process Conditions					
EA	Excess Air (%)	xx.xx	xx.xx	xx.xx	**xx.xx**
Cap	Capacity factor (hours/year)	8,760	8,760	8,760	**8,760**
Gas Conditions					
O_2	Oxygen (dry volume %)	xx.x	xx.x	xx.x	**xx.x**
CO_2	Carbon dioxide (dry volume %)	x.x	x.x	x.x	**x.x**
T_s	Sample temperature (°F)	xxx	xxx	xxx	**xxx**
B_w	Actual water vapor in gas (% by volume)	xx.x	xx.x	xx.x	**xx.x**
Gas Flow Rate					
Q_a	Volumetric flow rate, actual (acfm)	xxx,xxx	xxx,xxx	xxx,xxx	**xxx,xxx**
Q_s	Volumetric flow rate, standard (scfm)	xxx,xxx	xxx,xxx	xxx,xxx	**xxx,xxx**
Q_{std}	Volumetric flow rate, dry standard (dscfm)	xxx,xxx	xxx,xxx	xxx,xxx	**xxx,xxx**
Sampling Data					
V_{mstd}	Volume metered, standard (dscf)	xx.xx	xx.xx	xx.xx	**xx.xx**
%I	Isokinetic sampling (%)	xx.x	xx.x	xx.x	**xx.x**
Laboratory Data					
m_n	Total FPM (g)	x.xxxxx	x.xxxxx	x.xxxxx	
m_{CPM}	Total CPM (g)	x.xxxxx	x.xxxxx	x.xxxxx	
m_{Part}	Total particulate matter (g)	x.xxxxx	x.xxxxx	x.xxxxx	
FPM Results (Method 5) = PM					
C_{sd}	Particulate Concentration (lb/dscf)	x.xxE-xx	x.xxE-xx	x.xxE-xx	**x.xxE-xx**
C_{sd}	Particulate Concentration (gr/dscf)	x.xxxx	x.xxxx	x.xxxx	**x.xxxx**
$E_{lb/hr}$	Particulate Rate (lb/hr)	xx.x	xx.x	xx.x	**xx.x**
$E_{T/yr}$	Particulate Rate (Ton/yr)	xxx	xxx	xxx	**xxx**
CPM Results (Method 202)					
C_{sd}	Particulate Concentration (lb/dscf)	x.xxE-xx	x.xxE-xx	x.xxE-xx	**x.xxE-xx**
C_{sd}	Particulate Concentration (gr/dscf)	x.xxxx	x.xxxx	x.xxxx	**x.xxxx**
$E_{lb/hr}$	Particulate Rate (lb/hr)	xx.x	xx.x	xx.x	**xx.x**
$E_{T/yr}$	Particulate Rate (Ton/yr)	xx.x	xx.x	xx.x	**xx.x**
Total Particulate Matter Results (Method 5/202) = PM_{10} = $PM_{2.5}$					
C_{sd}	Particulate Concentration (lb/dscf)	x.xxE-xx	x.xxE-xx	x.xxE-xx	**x.xxE-xx**
C_{sd}	Particulate Concentration (gr/dscf)	x.xxxx	x.xxxx	x.xxxx	**x.xxxx**
$E_{lb/hr}$	Particulate Rate (lb/hr)	xx.x	xx.x	xx.x	**xx.x**
$E_{T/yr}$	Particulate Rate (Ton/yr)	xxx	xxx	xxx	**xxx**
$E_{EA50\%}$	Particulate Rate (lb per 1000lb exhaust gas at 50% EA)	x.xxxx	x.xxxx	x.xxxx	**x.xxxx**

Average includes 3 runs.

Table 2-3:
Underfire Combustion Stack – VOC, Method 25A (Example)

Run No.	1	2	3	Average
Date (2021)	MMDD	MMDD	MMDD	
Start Time	hh:mm	hh:mm	hh:mm	
End Time	hh:mm	hh:mm	hh:mm	
Elapsed Time	hh:mm	hh:mm	hh:mm	
Process Conditions				
Heat Input - Underfire Combustion Stack (MMBtu/hr)	xxx	xxx	xxx	**xxx**
Gas Conditions				
Oxygen (O2) - Underfire Combustion Stack (%dv)	x.x	x.x	x.x	**x.x**
Carbon Dioxide (CO2) - Underfire Combustion Stack (%dv)	x.x	x.x	x.x	**x.x**
Dry Standard Gas Flow Rate - Underfire Combustion Stack (dscfm)	xxx,xxx	xxx,xxx	xxx,xxx	**xxx,xxx**
H2O - Underfire Combustion Stack (%)	xx.x	xx.x	xx.x	**xx.x**
VOC, as Propane - Underfire Combustion Stack				
Concentration (ppmwv)	x.xxx	x.xxx	x.xxx	**x.xxx**
Concentration (ppmdv)	x.xxx	x.xxx	x.xxx	**x.xxx**
Mass Rate (lb/hr)	x.xxx	x.xxx	x.xxx	**x.xxx**
Mass Rate (lb/MMBtu) - Heat Input	x.xxx	x.xxx	x.xxx	**x.xxx**

Notes:
Flow and moisture data will be obtained from particulate testing.

End of Section

3. DESCRIPTION OF INSTALLATION

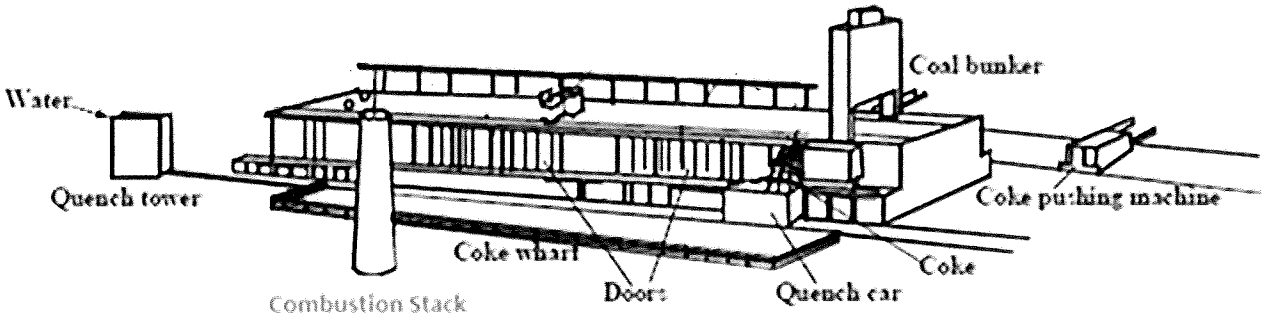
Process Description

EES Coke Battery, LLC is a facility located on Zug Island in River Rouge, Michigan. The testing described in this document will be performed at the Combustion Stack.

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

Figure 3-1:
Process Schematic



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

Test Location

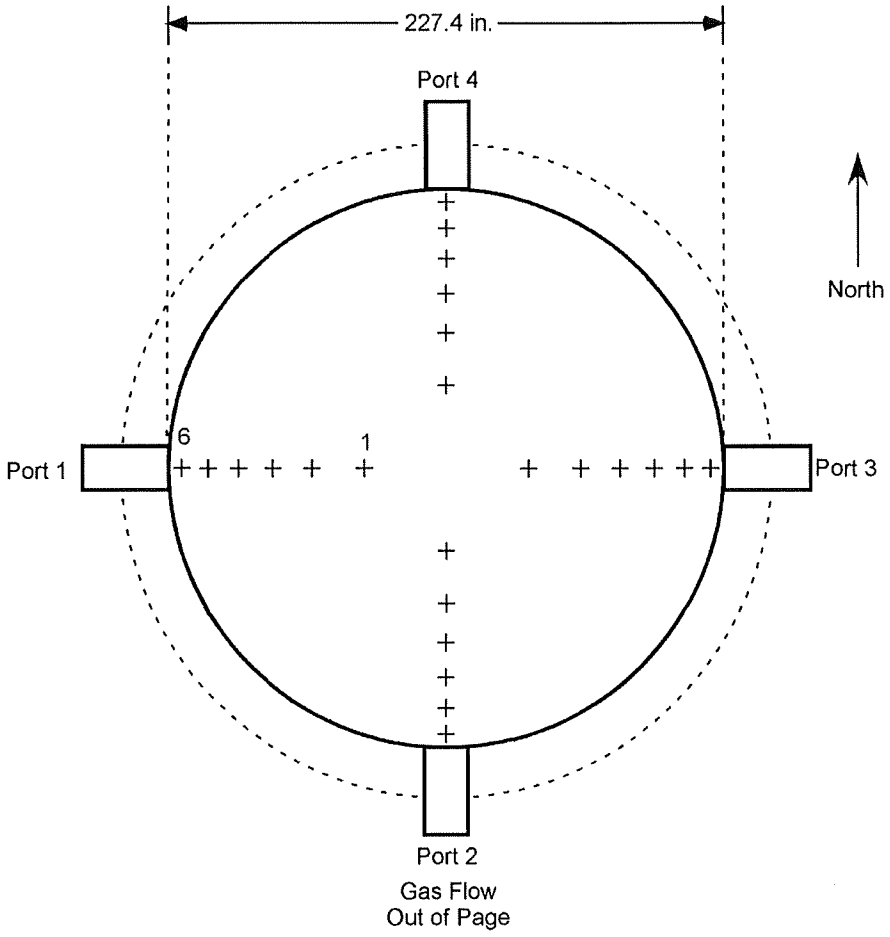
The sample point placement will be determined by EPA Method 1 specifications. Table 3-1 presents the sampling information for the test location. The figure represents the proposed layout of the test location.

Table 3-1:
Sampling Information

Source Constituent	Method	Run No.	Ports	Points per Port	Minutes per Point	Total Minutes	Figure
Underfire Combustion Stack							
NSFPM	EPA 5F (Modified)	1-3	4	6	5	120	3-2
TPM	EPA 5/202	1-3	4	6	5	120	3-2
VOC[1]	EPA 25A	1-3	1	1	60	60	3-3

[1] VOC measurements will be collected from a single point pending results of a stratification check.

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

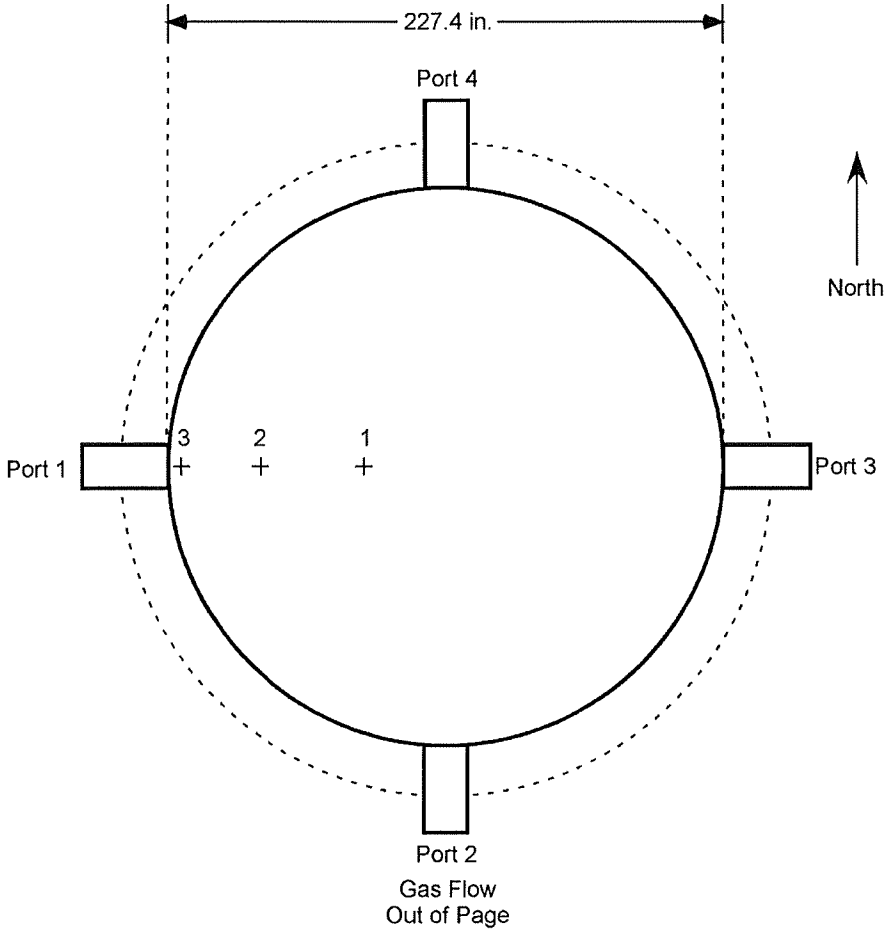


Sampling Point	% of Stack Diameter	Port to Point Distance (inches)
1	35.6	81.0
2	25.0	56.9
3	17.7	40.2
4	11.8	26.8
5	6.7	15.2
6	2.1	4.8

Duct diameters upstream from flow disturbance (A): 10.9 Limit: 0.5

Duct diameters downstream from flow disturbance (B): 2.7 Limit: 2.0

Figure 3-3:
Underfire Combustion Stack EPA Method 25A Stratification Check (EPA Method 7E)



Sampling Point	% of Stack Diameter	Port to Point Distance (inches)
1	2.0	78.7
2	1.2	47.2
3	0.4	15.7

Duct diameters upstream from flow disturbance (A): 10.9 Limit: 0.5

Duct diameters downstream from flow disturbance (B): 2.7 Limit: 2.0

End of Section

4. METHODOLOGY

Procedures and Regulations

The test program sampling measurements will follow procedures and regulations outlined by the USEPA and Michigan Department of Environment, Great Lakes, and Energy (EGLE). 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 will follow 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. Additional QA/QC measures are 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

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

Note: A cyclonic flow check per EPA Method 1, Section 2.4 was completed during the compliance test program in 2015. The results of that test indicated an absence of cyclonic flow. This test will not be repeated, and results will be available in the appendix of the final test report.

DETERMINATION OF FLUE GAS COMPOSITION – METHODS 1-4

CleanAir will measure flow rates using S-type pitot tubes following sampling point requirements of EPA Methods 1 and 2. The testing will occur in 4 test ports at 6 points per port for a total of 24 points. The pitot tube measurements will be used to determine the stack gas velocity and volumetric flow rate. EPA Method 3A will be followed to determine the oxygen and carbon dioxide content of the flue gas. Values will be obtained via

continuous extraction of CleanAir CEMS or via grab samples. EPA Method 4 will be followed to determine the moisture content of the sample.

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

Non-sulfate Filterable Particulate Matter – Method 5F (modified)

Particulate matter will be withdrawn isokinetically from the source and collected on a quartz fiber filter maintained at a temperature of 160°C ± 14°C (320°F ± 25°F). A minimum of 60 dry standard cubic feet of sample gas will be collected over a two-hour test period for each run. Flue gas volumetric flow rate, moisture concentration and flue gas molecular weight are also determined as part of the sample method. The previously agreed upon method of analysis, discussed in Section 1 Modifications to Test Methodology, will be followed.

Total Particulate Matter Determination – Method 5/202

The front-half (Method 5 portion) of the sampling train will consist of a glass nozzle, glass liner, filter holder heated to 250°F, and a quartz fiber filter. Flue gas samples will be extracted isokinetically, per Method 5 requirements.

Flue gas exiting the front-half heated filter will pass through a coiled condenser and dry impinger system jacketed by water continually circulated at ambient temperature. Moisture will be removed from the flue gas without bubbling through the condensed water. Flue gas will then pass through a Teflon membrane filter at ambient temperature. The temperature of the flue gas at the exit of the filter will be directly measured with an in-line thermocouple and maintained in the temperature range of 65°F to 85°F.

After exiting the ambient filter, the flue gas will pass through two additional impingers surrounded by ice in a "cold" section of the impinger bucket. The moisture collected in these impingers will not be analyzed for CPM and will only be collected to determine the flue gas moisture and thoroughly dry the gas prior to the metering device.

The front-half portion of the sample train (nozzle, probe and heated filter) will be recovered per Method 5 requirements, using acetone as the recovery solvent. The back-half of the sample train (heated filter outlet, condenser, dry impingers and TFE membrane filter) will be recovered per Method 202 requirements. The impinger train will be purged with N_2 at a rate of 14 liters per minute (lpm) for one hour following each test run and prior to recovery.

A field train blank will be assembled, purged, and recovered as if it were an actual test sample. Analysis of the field train blank will be used to blank-correct the test run results. Reagent blanks will also be collected to quantify background contamination. All samples and blanks will be returned to CleanAir Analytical Services for gravimetric analysis. Method 202 samples will be maintained at a temperature < 85°F during transport to the laboratory.

Three 120-minute Method 5/202 test runs will be performed. The results will be expressed as the average of three valid runs.

VOLATILE ORGANIC COMPOUNDS, EXCLUDING METHANE – METHOD 25A

Monitoring of O_2, CO_2, and THC emissions will be performed using a combination of EPA Methods 3A and 25A. A gas sample will be continuously extracted from the source and delivered to a series of gas analyzers, which will measure the pollutant or diluent concentrations in the gas. The analyzers will be calibrated on-site using certified mixtures of EPA Protocol 1 calibration gases.

The system will utilize a heated stainless-steel probe for gas withdrawal. The heated stainless-steel probe tip will be equipped with a sintered stainless-steel filter for particulate removal, if appropriate. The end of the probe will be connected to a heated Teflon sample line that will deliver the sample gases from the stack to the CEM system. The heated sample line is designed to maintain the gas temperature above 250°F, to prevent condensation of stack gas moisture within the line.

A stratification check will be performed prior to or during the first sample run as described in 40 CFR 60, Appendix A, Method 7E, §8.1.2. The stack measurement line will be traversed at 2.0m, 1.2m, and 0.4m of the stack diameter to verify the absence of a stratified flue gas.

The concentration at each traverse point may differ from the mean concentration for all traverse points by no more than ± 5.0% of the mean concentration. The gas stream will be considered unstratified and a single point that most closely matched the mean will be used.

Calibration error checks will be performed by introducing zero nitrogen (N_2), high range and mid-range calibration gases to the inlet of each analyzer during calibration error checks. Bias checks will be performed before and after each sampling run by introducing calibration gas to the inlet of the sampling system's heated filter.

Minute-average data points for O_2, CO_2, (dry basis), and THC (wet basis) will be collected over a period of 60 minutes for each run. CleanAir will use the JUM Model 109A analyzer, or equivalent, for the measurements. This model uses two individual detectors and two individual signal amplifiers. Sample will be introduced into one FID for THC readings. The gas sample will then run through a non-methane cutter which eliminates all hydrocarbons except methane before being analyzed by the second FID. The analyzer subtracts the two values to provide a THC (excluding methane) reading.

CleanAir will use the JUM Model 109A analyzer, or equivalent, for the measurements. This model uses two individual detectors and two individual signal amplifiers. Sample will be introduced into one FID for THC readings. The gas sample will then run through a non-methane cutter which eliminates all hydrocarbons except methane before being analyzed by the second FID. The analyzer subtracts the two values to provide a THC (excluding methane) reading.

End of Section