

REPORT ON COMPLIANCE
TESTING

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Detroit Refinery
Crude/Vacuum Heater Stack

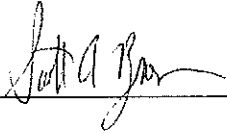
Marathon Petroleum Company LP
1300 South Fort Street
Detroit, MI 48217
Client Reference No. 4101379616

CleanAir Project No. 13714-3
A2LA ISO 17025 Certificate No. 4342.01
A2LA / STAC Certificate No. 4342.02
Revision 0, Final Report
February 13, 2019

COMMITMENT TO QUALITY

To the best of our knowledge, the data presented in this report are accurate, complete, error free and representative of the actual emissions during the test program. Clean Air Engineering operates in conformance with the requirements of ASTM D7036-04 Standard Practice for Competence of Air Emission Testing Bodies.

Report Writer:



February 13, 2019

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I hereby certify that the information contained within the final test report has been reviewed and, to the best of my ability, verified as accurate.

Independent Report and Appendix Reviewer:



February 13, 2019

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REPORT REVISION HISTORY

Version	Revision	Date	Pages	Comments
Draft	D0a	02/12/19	All	Draft version of original document.
Final	0	02/13/19	All	Final version of original document.

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ACRONYMS & ABBREVIATIONS

AAS (atomic absorption spectrometry)	ft ³ (cubic feet)	MW (megawatt(s))
acfm (actual cubic feet per minute)	ft/sec (feet per second)	NCASI (National Council for Air and Stream Improvement)
ACI (activated carbon injection)	FTIR (Fourier Transform Infrared Spectroscopy)	ND (non-detect)
ADL (above detection limit)	FTRB (field train reagent blank)	NDIR (non-dispersive infrared)
AIG (ammonia injection grid)	g (gram(s))	NDO (natural draft opening)
APC (air pollution control)	GC (gas chromatography)	NESHAP (National Emission Standards for Hazardous Air Pollutants)
AQCS (air quality control system(s))	GFAAS (graphite furnace atomic absorption spectroscopy)	ng (nanogram(s))
ASME (American Society of Mechanical Engineers)	GFC (gas filter correlation)	Nm ³ (Normal cubic meter)
ASTM (American Society for Testing and Materials)	gr/dscf (grains per dry standard cubic feet)	% (percent)
BDL (below detection limit)	> (greater than)/ ≥ (greater than or equal to)	PEMS (predictive emissions monitoring systems)
Btu (British thermal units)	g/s (grams per second)	PFGC (pneumatic focusing gas chromatography)
CAM (compliance assurance monitoring)	H ₂ O (water)	pg (picogram(s))
CARB (California Air Resources Board)	HAP(s) (hazardous air pollutant(s))	PJFF (pulse jet fabric filter)
CCM (Controlled Condensation Method)	HI (heat input)	ppb (parts per billion)
CE (capture efficiency)	hr (hour(s))	PPE (personal protective equipment)
°C (degrees Celsius)	HR GC/MS (high-resolution gas chromatography and mass spectrometry)	ppm (parts per million)
CEMS (continuous emissions monitoring system(s))	HRVOC (highly reactive volatile organic compounds)	ppmdv (parts per million, dry volume)
CFB (circulating fluidized bed)	HSRG(s) (heat recovery steam generator(s))	ppmwv (parts per million, wet volume)
CFR (Code of Federal Regulations)	HVT (high velocity thermocouple)	PSD (particle size distribution)
cm (centimeter(s))	IC (ion chromatography)	psi (pound(s) per square inch)
COMS (continuous opacity monitoring system(s))	IC/PCR (ion chromatography with post column reactor)	PTE (permanent total enclosure)
CT (combustion turbine)	ICP/MS (inductively coupled argon plasma mass spectroscopy)	PTFE (polytetrafluoroethylene)
CTI (Cooling Technology Institute)	ID (induced draft)	QA/QC (quality assurance/quality control)
CTM (Conditional Test Method)	in. (inch(es))	QI (qualified individual)
CVAAS (cold vapor atomic absorption spectroscopy)	in. H ₂ O (inches water)	QSTI (qualified source testing individual)
CVAFS (cold vapor atomic fluorescence spectrometry)	in. Hg (inches mercury)	QSTO (qualified source testing observer)
DI H ₂ O (de-ionized water)	IPA (isopropyl alcohol)	RA (relative accuracy)
%dv (percent, dry volume)	ISE (ion-specific electrode)	RATA (relative accuracy test audit)
DLL (detection level limited)	kg (kilogram(s))	RB (reagent blank)
DE (destruction efficiency)	kg/hr (kilogram(s) per hour)	RE (removal or reduction efficiency)
DCI (dry carbon injection)	< (less than)/ ≤ (less than or equal to)	RM (reference method)
DGM (dry gas meter)	L (liter(s))	scf (standard cubic feet)
dscf (dry standard cubic feet)	lb (pound(s))	scfm (standard cubic feet per minute)
dscfm (dry standard cubic feet per minute)	lb/hr (pound per hour)	SCR (selective catalytic reduction)
dscm (dry standard cubic meter)	lb/MMBtu (pound per million British thermal units)	SDA (spray dryer absorber)
ESP (electrostatic precipitator)	lb/TBtu (pound per trillion British thermal units)	SNCR (selective non-catalytic reduction)
FAMS (flue gas adsorbent mercury speciation)	lb/lb-mole (pound per pound mole)	STD (standard)
°F (degrees Fahrenheit)	LR GC/MS (low-resolution gas chromatography and mass spectrometry)	STMS (sorbet trap monitoring system)
FB (field blank)	m (meter)	TBtu (trillion British thermal units)
FCC (fluidized catalytic cracking)	m ³ (cubic meter)	TEOM (Tapered Element Oscillating Microbalance)
FCCU (fluidized catalytic cracking unit)	MACT (maximum achievable control technology)	TEQ (toxic equivalency quotient)
FEGT (furnace exit gas temperatures)	MASS [®] (Multi-Point Automated Sampling System)	ton/hr (ton per hour)
FF (fabric filter)	MATS (Mercury and Air Toxics Standards)	ton/yr (ton per year)
FGD (flue gas desulfurization)	MDL (method detection limit)	TSS (third stage separator)
FIA (flame ionization analyzer)	μg (microgram(s))	USEPA or EPA (United States Environmental Protection Agency)
FID (flame ionization detector)	min. (minute(s))	UVA (ultraviolet absorption)
FPD (flame photometric detection)	mg (milligram(s))	WFGD (wet flue gas desulfurization)
FRB (field reagent blank)	ml (milliliter(s))	%wv (percent, wet volume)
FSTM (flue gas sorbet total mercury)	MMBtu (million British thermal units)	
ft (feet or foot)		
ft ² (square feet)		

1. PROJECT OVERVIEW

Test Program Summary

Marathon Petroleum Company LP (MPC) contracted CleanAir Engineering (CleanAir) to complete testing on the Crude/Vacuum Heater (EU05-CRUDEHTR-S1 & EU04-VACHTR-S-1) at the Detroit Refinery. The test program included the following objective:

- Perform particulate matter (PM) and sulfuric acid mist (H₂SO₄) quarterly testing to demonstrate compliance with the Michigan Department of Environmental Quality (DEQ) Permit No. MI-ROP-A9831-2012c.

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.

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

Source Constituent	Sampling Method	Average Emission	Permit Limit ¹
<u>Crude/Vacuum Heater</u>			
FPM (lb/MMBtu)	EPA M5	0.0004	NA
H ₂ SO ₄ (lb/MMBtu)	Draft ASTM CCM	0.0003	NA
PM (lb/MMBtu) ^{2,3}	EPA M5 / Draft ASTM CCM	0.0001	0.0019
NSFPM (lb/MMBtu) ⁴	EPA M5B	0.0003	0.0019

¹ Permit limits obtained from MDEQ Renewable Operating Permit No. MI-ROP-A9831-2012c.

² PM assumed equivalent to FPM minus H₂SO₄. The letter from the MDEQ referenced in Appendix K further outlines the correction of particulate emissions for H₂SO₄ bias.

³ Expressed as the average of the three (3) highest valid runs.

⁴ NSFPM measured for supplemental purposes.

Test Program Details

Parameters

The test program included the following measurements:

- filterable particulate matter (FPM)
- particulate matter (PM) – defined as FPM minus sulfuric acid (H₂SO₄)
- nonsulfuric acid filterable particulate matter (NSFPM)
- H₂SO₄ – conducted concurrently with FPM measurements
- flue gas composition (e.g., O₂, CO₂, H₂O)
- flue gas temperature
- flue gas flow rate

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Schedule

Testing was performed on January 9 and 10, 2019. 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	Crude/Vacuum Heater	USEPA Method 5	FPM	01/09/19	10:25	13:13
2	Crude/Vacuum Heater	USEPA Method 5	FPM	01/09/19	14:42	16:50
3	Crude/Vacuum Heater	USEPA Method 5	FPM	01/10/19	08:36	10:47
4	Crude/Vacuum Heater	USEPA Method 5	FPM	01/10/19	11:50	13:58
1	Crude/Vacuum Heater	Draft ASTM CCM	H ₂ SO ₄	01/09/19	10:25	13:13
2	Crude/Vacuum Heater	Draft ASTM CCM	H ₂ SO ₄	01/09/19	14:42	16:50
3	Crude/Vacuum Heater	Draft ASTM CCM	H ₂ SO ₄	01/10/19	08:36	10:47
4	Crude/Vacuum Heater	Draft ASTM CCM	H ₂ SO ₄	01/10/19	11:50	13:58
1	Crude/Vacuum Heater	USEPA Method 5B	NSFPM	01/09/19	10:25	13:13
2	Crude/Vacuum Heater	USEPA Method 5B	NSFPM	01/09/19	14:42	16:50
3	Crude/Vacuum Heater	USEPA Method 5B	NSFPM	01/10/19	08:36	10:47
4	Crude/Vacuum Heater	USEPA Method 5B	NSFPM	01/10/19	11:50	13:58

Discussion

Project Synopsis

PM Testing

A total of four (4) 120-minute EPA Method 5 test runs were performed. PM emission results were calculated in units of pounds per million Btu (lb/MMBtu). All runs were deemed valid.

PM is assumed equivalent to the difference of FPM and H₂SO₄ emissions. H₂SO₄ emissions were determined concurrently with FPM emissions, converted to units of lb/MMBtu, and subtracted from total FPM emissions from each respective run. The final result was expressed as the average of the three (3) highest valid runs.

H₂SO₄ Testing – Draft ASTM Controlled Condensation Method

H₂SO₄ emissions were determined referencing the Draft ASTM Controlled Condensation Method (CCM). Four (4) 120-minute Draft ASTM CCM test runs were performed concurrently with all Method 5 runs. H₂SO₄ emission results were calculated in units of lb/MMBtu. The H₂SO₄ final results were expressed as the average of four (4) valid runs.

Run 1 exhibited an elevated oxygen concentration; however, the moisture content and H₂SO₄ concentration were consistent with subsequent runs. There is no overt explanation for this occurrence.

On January 8, prior to performing official test runs, a 60-minute sample conditioning run (Run 0) was performed in order to minimize the absorption capacity of the front-half components of the sample train (upstream of the H₂SO₄-collecting portion of the sample train). The conditioning run was recovered in the same manner as the official test runs. Samples from Run 0 are archived.

NSFPM Testing – USEPA Method 5B

A total of four (4) 120-minute EPA Method 5B test runs were performed concurrently with Method 5 and Draft ASTM CCM test runs. NSFPM emission results were calculated in units of lb/MMBtu. The NSFPM final results were expressed as the average of four (4) valid runs. NSFPM emissions were determined for supplemental purposes.

Fuel Analysis

Emission results in units of dry volume-based concentration (lb/dscf, ppm_{dv}) were converted into units of lb/MMBtu by calculating an oxygen-based fuel factor (F_d) for refinery gas per EPA Method 19 specifications. The heat content and F_d factor were calculated from percent volume composition analytical data provided by MPC and tabulated heating values for each of the measured constituents.

Test Conditions

The unit was operated at the maximum normal operating capacity during each of the emissions compliance test runs and RATA test runs. MPC was responsible for logging any relevant process-related data and providing it to CleanAir for inclusion in the test report.

End of Section

2. RESULTS

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

**Table 2-1:
Crude/Vacuum Heater – FPM Emissions**

Run No.		1	2	3	4	Average
Date (2019)		Jan 9	Jan 9	Jan 10	Jan 10	
Start Time (approx.)		10:25	14:42	08:36	11:50	
Stop Time (approx.)		13:13	16:50	10:47	13:58	
Process Conditions						
P ₁	Charge Rate (BPD)	129,336	130,048	129,868	130,176	129,857
F _d	Oxygen-based F-factor (dscf/MMBtu)	8,348	8,348	8,282	8,282	8,315
H _i	Actual heat input (MMBtu/hr)	276	275	282	285	280
Gas Conditions						
O ₂	Oxygen (dry volume %)	7.6	7.5	7.9	7.9	7.7
CO ₂	Carbon dioxide (dry volume %)	7.9	8.0	7.6	7.5	7.8
T _s	Sample temperature (°F)	287	289	289	290	289
B _w	Actual water vapor in gas (% by volume)	13.4	12.6	12.5	12.1	12.7
Gas Flow Rate						
Q _a	Volumetric flow rate, actual (acfm)	111,000	121,000	123,000	120,000	119,000
Q _s	Volumetric flow rate, standard (scfm)	76,300	82,600	85,900	83,200	82,000
Q _{std}	Volumetric flow rate, dry standard (dscfm)	66,100	72,200	75,100	73,100	71,600
Sampling Data						
V _{mstd}	Volume metered, standard (dscf)	79.43	89.88	94.73	91.62	88.91
%I	Isokinetic sampling (%)	97.8	101.4	102.7	102.0	101.0
Laboratory Data						
m _{filter}	Matter collected on filter(s) (g)	0.00053	0.00083	0.00058	0.00056	
m _s	Matter collected in solvent rinse(s) (g)	0.00069	0.00068	0.00092	0.00051	
m _n	Total FPM (g)	0.00122	0.00151	0.00150	0.00107	
FPM Results						
C _{sd}	Particulate Concentration (lb/dscf)	3.39E-08	3.70E-08	3.49E-08	2.58E-08	3.29E-08
E _{lb/hr}	Particulate Rate (lb/hr)	0.134	0.160	0.157	0.113	0.141
E _{Fd}	Particulate Rate - F _d -based (lb/MMBtu)	0.000444	0.000482	0.000465	0.000343	0.000434

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**Table 2-2:
 Crude/Vacuum Heater – H₂SO₄ Emissions**

Run No.	1	2	3	4	Average
Date (2019)	Jan 9	Jan 9	Jan 10	Jan 10	
Start Time (approx.)	10:25	14:42	08:36	11:50	
Stop Time (approx.)	13:13	16:50	10:47	13:58	
Process Conditions					
P ₁ Charge rate (BPD)	129,336	130,048	129,868	130,176	130,176
F _d Oxygen-based F-factor (dscf/MMBtu)	8,348	8,348	8,282	8,282	8,282
H _i Actual heat input (MMBtu/hr)	276	275	282	285	285
Gas Conditions					
O ₂ Oxygen (dry volume %)	10.2	7.7	8.1	8.1	8.5
CO ₂ Carbon dioxide (dry volume %)	6.6	7.9	7.6	7.5	7.4
T _s Sample temperature (°F)	287	286	287	287	287
B _w Actual water vapor in gas (% by volume)	12.9	13.4	13.0	12.8	13.0
Gas Flow Rate					
Q _a Volumetric flow rate, actual (acfm)	111,000	121,000	123,000	120,000	119,000
Q _s Volumetric flow rate, standard (scfm)	76,300	82,600	85,900	83,200	82,000
Q _{std} Volumetric flow rate, dry standard (dscfm)	66,100	72,200	75,100	73,100	71,600
Sampling Data					
V _{mstd} Volume metered, standard (dscf)	55.01	55.32	56.51	56.24	55.77
Laboratory Data (Ion Chromatography)					
m _n Total H ₂ SO ₄ collected (mg)	0.5673	0.6509	0.5100	0.5212	
Sulfuric Acid Vapor (H₂SO₄) Results					
C _{sd} H ₂ SO ₄ Concentration (lb/dscf)	2.274E-08	2.595E-08	1.990E-08	2.044E-08	2.226E-08
C _{sd} H ₂ SO ₄ Concentration (ppmdv)	0.0894	0.102	0.0782	0.0803	0.0875
E _{Fd} H ₂ SO ₄ Rate - Fd-based (lb/MMBtu)	0.000371	0.000343	0.000269	0.000276	0.000315

**Table 2-3:
 Crude/Vacuum Heater – PM Emissions**

Run No.	1	2	3	4	Average
Date (2019)	Jan 9	Jan 9	Jan 10	Jan 10	
Start Time (approx.)	10:25	14:42	08:36	11:50	
Stop Time (approx.)	13:13	16:50	10:47	13:58	
Process Conditions					
P ₁ Charge Rate (BPD)	129,336	130,048	129,868	130,176	129,857
F _d Oxygen-based F-factor (dscf/MMBtu)	8,348	8,348	8,282	8,282	8,315
H _i Actual heat input (MMBtu/hr)	276	275	282	285	280
Gas Conditions					
O ₂ Oxygen (dry volume %)	7.6	7.5	7.9	7.9	7.7
CO ₂ Carbon dioxide (dry volume %)	7.9	8.0	7.6	7.5	7.8
T _s Sample temperature (°F)	287	289	289	290	289
B _w Actual water vapor in gas (% by volume)	13.4	12.6	12.5	12.1	12.7
FPM Results					
E _{Fd} Particulate Rate - F _d -based (lb/MMBtu)	0.000444	0.000482	0.000465	0.000343	0.000434
Sulfuric Acid Vapor (H₂SO₄) Results					
E _{Fd} H ₂ SO ₄ Rate - F _d -based (lb/MMBtu)	0.000326	0.000409	0.000319	0.000314	0.000342
Particulate Matter (as PM₁₀) Results¹					
E _{Fd} Particulate Rate - F _d -based (lb/MMBtu)	0.000118	0.000074	0.000146	0.000029	0.000113

¹ Final PM results average of three (3) highest valid runs.

**Table 2-4:
Crude/Vacuum Heater – NSFPM Emissions**

Run No.		1	2	3	4	Average
Date (2019)		Jan 9	Jan 9	Jan 10	Jan 10	
Start Time (approx.)		10:25	14:42	08:36	11:50	
Stop Time (approx.)		13:13	16:50	10:47	13:58	
Process Conditions						
P ₁	Charge Rate (BPD)	129,336	130,048	129,868	130,176	129,857
F _d	Oxygen-based F-factor (dscf/MMBtu)	8,348	8,348	8,282	8,282	8,315
H _i	Actual heat input (MMBtu/hr)	276	275	282	285	280
Gas Conditions						
O ₂	Oxygen (dry volume %)	7.6	7.6	7.9	8.8	8.0
CO ₂	Carbon dioxide (dry volume %)	7.9	8.0	7.6	7.1	7.7
T _s	Sample temperature (°F)	288	287	288	284	287
B _w	Actual water vapor in gas (% by volume)	12.1	12.7	12.7	13.2	12.6
Gas Flow Rate						
Q _a	Volumetric flow rate, actual (acfm)	115,000	111,000	114,000	113,000	113,000
Q _s	Volumetric flow rate, standard (scfm)	78,800	76,100	79,100	79,200	78,300
Q _{std}	Volumetric flow rate, dry standard (dscfm)	69,300	66,400	69,100	68,700	68,400
Sampling Data						
V _{mstd}	Volume metered, standard (dscf)	85.27	79.86	84.66	84.42	83.55
%I	Isokinetic sampling (%)	100.2	97.9	99.8	100.0	99.5
Laboratory Data						
m _{filter}	Matter collected on filter(s) (g)	0.00029	0.00029	0.00055	0.00034	
m _s	Matter collected in solvent rinse(s) (g)	0.00040	0.00056	0.00032	0.00032	
m _n	Total NSFPM (g)	0.00069	0.00085	0.00087	0.00066	
NSFPM Results						
C _{sd}	Particulate Concentration (lb/dscf)	1.78E-08	2.35E-08	2.27E-08	1.72E-08	2.03E-08
E _{lb/hr}	Particulate Rate (lb/hr)	0.0742	0.0936	0.0939	0.0711	0.0832
E _{Fd}	Particulate Rate - F _d -based (lb/MMBtu)	0.000234	0.000308	0.000302	0.000247	0.000273

End of Section

3. DESCRIPTION OF INSTALLATION

Process Description

MPC's facility in Detroit, Michigan, produces refined petroleum products from crude oil. MPC must continue to demonstrate that select process units are in compliance with permitted emission limits.

The Crude Unit (EU05-CRUDE) separates crude oil into various fractions through the use of distillation processes. These fractions are sent to other units in the refinery for further processing. The Crude Unit consists of process vessels (including heat exchangers and fractionation columns), the Alcorn Heater (EU05-CRUDEHTR-S1), tanks, containers, compressors, pumps, piping drains, and various components (pump and compressor seals, process valves, pressure relief valves, flanges, connectors, etc.).

The Vacuum Unit (EU04-VACUUM) separates the reduced crude from the crude unit through the use of a vacuum column. The reduced crude is separated into light vacuum gas oil, medium vacuum gas oil, heavy vacuum gas oil and a bottoms product called flux. The various fractions are sent to other units in the refinery for further processing. The vacuum unit consists of process vessels (including heat exchangers and vacuum column), two process heaters, tanks, containers, two cooling towers, flare, compressors, pumps, piping drains, and various components (pumps and compressor seals, process valves, pressure relief valves, flanges, connectors, etc.).

Both the Crude Heater (EU05-CRUDEHTR-S1) and the Vacuum Heater (EU04-VACHTR-S1) are fired by refinery fuel gas. Emissions are vented to the atmosphere via a common stack known as the Crude/Vacuum Heater Stack (SV04-H1-05-H1), where testing was performed.

Test Location

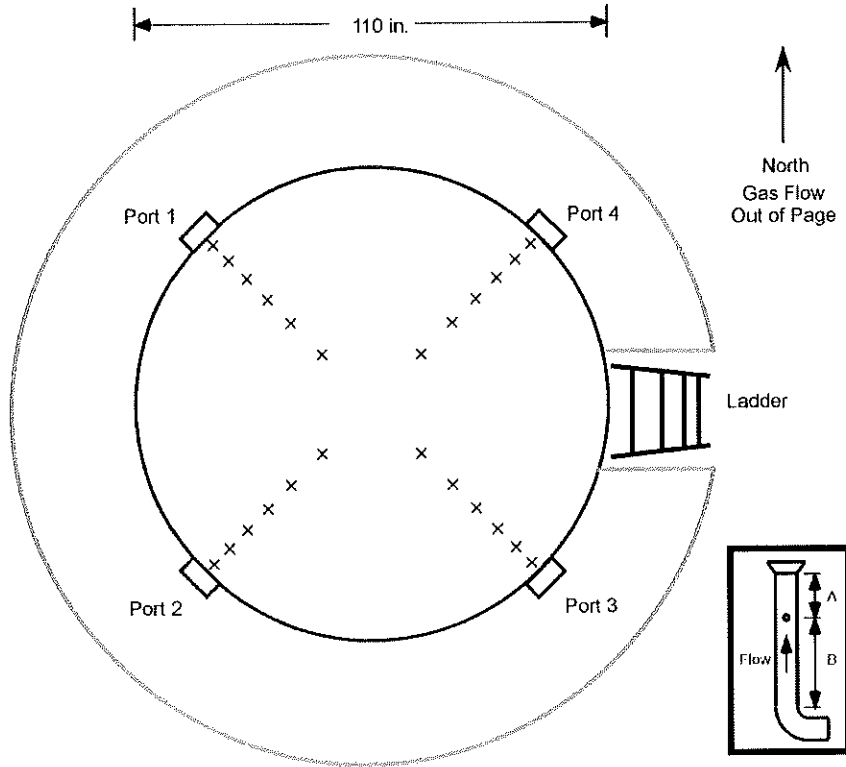
The sample point locations were determined by EPA Method 1. Table 3-1 presents the sampling information for the test location described in this report. The figure shown on page 9 represents the layout of the test location.

**Table 3-1:
Sampling Information**

Source Constituent	Method (USEPA)	Run No.	Ports	Points per Port	Minutes per Point	Total Minutes	Figure
<u>Crude/Vac Heater Stack</u>							
FPM	5	1-4	4	6	5	120	3-1
H ₂ SO ₄	Draft ASTM CCM	1-4	1	1	120	120	N/A ¹
NSFPM	5B	1-4	4	6	5	120	3-1

¹ Draft ASTM CCM sampling occurred at a single point near the center of the duct.

**Figure 3-1:
 Particulate Sample Point Layout**



Sampling Point	% of Stack Diameter	Port to Point Distance (inches)
1	35.6	39.2
2	25.0	27.5
3	17.7	19.5
4	11.8	13.0
5	6.7	7.4
6	2.1	2.3

Duct diameters upstream from flow disturbance (A): 5.3 Limit: 0.5
 Duct diameters downstream from flow disturbance (B): 2.4 Limit: 2.0

4. METHODOLOGY

Procedures and Regulations

The test program sampling measurements followed procedures and regulations outlined by the United States Environmental Protection Agency (USEPA) and the DEQ. 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. Any modifications to standard test methods are explicitly indicated in this appendix. In accordance with ASTM D7036 requirements, CleanAir included a description of any such modifications along with the full context of the objectives and requirements of the test program in the test protocol submitted prior to the measurement portion of this project. Modifications to standard methods are not covered by the ISO 17025 and TNI portions of CleanAir's A2LA accreditation.

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 3	"Gas Analysis for the Determination of Dry Molecular Weight"
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 5B	"Determination of Nonsulfuric Acid Particulate Matter Emissions from Stationary Sources"
Method 19	"Determination of Sulfur Dioxide Removal Efficiency and Particulate Matter, Sulfur Dioxide and Nitrogen Oxide Emission Rates"

CTM-013 (Mod.)/Draft ASTM Controlled Condensation Method (Draft ASTM CCM)

"Determination of Sulfur Oxides Including Sulfur Dioxide, Sulfur Trioxide and Sulfuric Acid Vapor and Mist from Stationary Sources Using a Controlled Condensation Sampling Apparatus"

Methodology Discussion

Filterable Particulate Matter – USEPA Methods 5

FPM emissions were determined using EPA Method 5.

The front-half of the sampling train consisted of a glass nozzle, glass liner and filter holder heated to 248°F ± 25°F, and a quartz fiber filter. Flue gas samples were extracted isokinetically per Method 5 requirements.

After exiting the front-half filter, the flue gas passed through a series of knock-out jars. Condensate in the knock-out jars were collected to determine the flue gas moisture and thoroughly dry the gas. The sample gas then flowed into a calibrated dry gas meter where the collected sample gas volume was determined.

The front-half portion of the sample train (nozzle, probe and heated filter) was recovered per Method 5 requirements, using acetone as the recovery solvent.

All samples and blanks were returned to CleanAir Analytical Services in Palatine, Illinois, for gravimetric analysis. Upon receipt, the filters desiccated for 24 hours at ambient temperature. The front-half rinses were evaporated at ambient temperature and pressure. The masses from each fraction were then summed for a total FPM mass.

Nonsulfuric Acid Filterable Particulate Matter – USEPA Method 5B

EPA Method 5B, "Determination of Nonsulfuric Acid Particulate Matter Emissions from Stationary Sources", was utilized for the nonsulfuric filterable particulate matter (NSFPM) measurements. This method is 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°C ± 14°C (320°F ± 25°F). The front-half portion of the sample train (nozzle, probe and heated filter) was recovered per Method 5 requirements, using acetone as the recovery solvent.

The collected samples were prepared according to the method and then heated in an oven at 160°C (320°F) for six hours to volatilize any condensed sulfuric acid that may have been collected. The nonsulfuric acid particulate mass was determined gravimetrically by CleanAir Analytical Services.

H₂SO₄ Testing – Draft ASTM CCM

H₂SO₄ emissions were determined referencing the Draft ASTM CCM.

A gas sample was extracted from the source at a constant flow rate using a quartz-lined probe maintained at a temperature of 650°F ± 25°F and a quartz fiber filter (to remove particulate matter) maintained at the same temperature as the probe. The sample was then passed through a glass coil condenser for collection of sulfuric acid vapor and/or mist. A second quartz fiber filter (referred to as the sulfuric acid mist (SAM) filter) was located at the condenser outlet for the collection of residual SAM not collected by the condenser. The condenser temperature was regulated by a water jacket and the SAM filter was regulated by a closed oven. Both the water jacket and SAM filter oven were maintained at 140°F ± 9°F plus 2°F for each 1% moisture above 16% flue gas moisture (above the water dew point, which eliminates the oxidation of dissolved sulfur dioxide (SO₂) into the H₂SO₄-collecting fraction of the sample train).

After exiting the SAM filter, the sample gas then continued through a series of four (4) glass knock-out jars; two (2) containing water, one (1) empty and one (1) containing silica gel for residual moisture removal. The exit temperature from the knock-out jar set was maintained below 68°F. The sample gas then flowed into a dry gas meter, where the collected sample gas volume was determined by means of a calibrated, dry gas meter or an orifice-based flow meter.

The H₂SO₄-collecting portion of the sample train (condenser and SAM filter) was recovered into a single fraction using DI H₂O as the recovery/extraction solvent; any H₂SO₄ disassociates into sulfate ion (SO₄²⁻) and is stabilized in the H₂O matrix until analysis.

Samples and blanks were returned to CleanAir Analytical Services, for ion chromatography (IC) analysis.

End of Section