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Marathon Petroleum Company LP Detroit Refinery Report on Compliance Testing CleanAir Project No. 13714-2 Revision 0, Final Report Page ii

# 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:

January 31, 2019

Date

Ken Sullivan Project Manager ksullivan@cleanair.com (800) 627-0033 ext. 4527

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:

Scott Brown, QSTI Senior Project Manager / Quality Director sbrown@cleanair.com (800) 627-0033 ext. 4544 January 31, 2019

Date

# **REPORT REVISION HISTORY**

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Final	0	01/31/19	All	Final version of original document.

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# ACRONYMS & ABBREVIATIONS

AAS (atomic absorption spectrometry) acfm (actual cubic feet per minute) ACI (activated carbon injection) ADL (above detection limit) AIG (ammonia injection grid) APC (air pollution control) AQCS (air quality control system(s)) ASME (American Society of Mechanical Engineers) ASTM (American Society for Testing and Materials) BDL (below detection limit) Btu (British thermal units) CAM (compliance assurance monitoring) CARB (California Air Resources Board) CCM (Controlled Condensation Method) CE (capture efficiency) °C (degrees Celsius) CEMS (continuous emissions monitoring system(s)) CFB (circulating fluidized bed) CFR (Code of Federal Regulations) cm (centimeter(s)) COMS (continuous opacity monitoring system(s)) CT (combustion turbine) CTI (Cooling Technology Institute) CTM (Conditional Test Method) CVAAS (cold vapor atomic absorption spectroscopy) CVAFS (cold vapor atomic fluorescence spectrometry) DI H<sub>2</sub>O (de-ionized water) %dv (percent, dry volume) DLL (detection level limited) DE (destruction efficiency) DCI (dry carbon injection) DGM (dry gas meter) dscf (dry standard cubic feet) dscfm (dry standard cubic feet per minute) dscm (dry standard cubic meter) ESP (electrostatic precipitator) FAMS (flue gas adsorbent mercury speciation) °F (degrees Fahrenheit) FB (field blank) FCC (fluidized catalytic cracking) FCCU (fluidized catalytic cracking unit) FEGT (furnace exit gas temperatures) FF (fabric filter) FGD (flue gas desulfurization) FIA (flame ionization analyzer) FID (flame ionization detector) FPD (flame photometric detection) FRB (field reagent blank) FSTM (flue gas sorbent total mercury) ft (feet or foot) ft<sup>2</sup> (square feet)

ft<sup>3</sup> (cubic feet) ft/sec (feet per second) FTIR (Fourier Transform Infrared Spectroscopy) FTRB (field train reagent blank) g (gram(s)) GC (gas chromatography) GFAAS (graphite furnace atomic absorption spectroscopy) GFC (gas filter correlation) gr/dscf (grains per dry standard cubic feet) > (greater than)/ $\geq$  (greater than or equal to) g/s (grams per second) H<sub>2</sub>O (water) HAP(s) (hazardous air pollutant(s)) HI (heat input) hr (hour(s)) HR GC/MS (high-resolution gas chromatography and mass spectrometry) HRVOC (highly reactive volatile organic compounds) HSRG(s) (heat recovery steam generator(s)) HVT (high velocity thermocouple) IC (ion chromatography) IC/PCR (ion chromatography with post column reactor) ICP/MS (inductively coupled argon plasma mass spectroscopy) ID (induced draft) in. (inch(es)) in. H<sub>2</sub>O (inches water) in. Hg (inches mercury) IPA (isopropyl alcohol) ISE (ion-specific electrode) kg (kilogram(s)) kg/hr (kilogram(s) per hour) < (less than)/ ≤ (less than or equal to) L (liter(s)) lb (pound(s)) lb/hr (pound per hour) lb/MMBtu (pound per million British thermal units) lb/TBtu (pound per trillion British thermal units) Ib/Ib-mole (pound per pound mole) LR GC/MS (low-resolution gas chromatography and mass spectrometry) m (meter) m<sup>3</sup> (cubic meter) MACT (maximum achievable control technology) MASS® (Multi-Point Automated Sampling System) MATS (Mercury and Air Toxics Standards) MDL (method detection limit) µg (microgram(s)) min. (minute(s)) mg (milligram(s)) ml (milliliter(s))

MMBtu (million British thermal units)

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MW (megawatt(s)) NCASI (National Council for Air and Stream Improvement) ND (non-detect) NDIR (non-dispersive infrared) NDO (natural draft opening) NESHAP (National Emission Standards for Hazardous Air Pollutants) ng (nanogram(s)) Nm<sup>3</sup> (Normal cubic meter) % (percent) PEMS (predictive emissions monitoring systems) PFGC (pneumatic focusing gas chromatography) pg (picogram(s)) PJFF (pulse jet fabric filter) ppb (parts per billion) PPE (personal protective equipment) ppm (parts per million) ppmdv (parts per million, dry volume) ppmwv (parts per million, wet volume) PSD (particle size distribution) psi (pound(s) per square inch) PTE (permanent total enclosure) PTFE (polytetrafluoroethylene) QA/QC (quality assurance/quality control) QI (qualified individual) QSTI (qualified source testing individual) QSTO (qualified source testing observer) RA (relative accuracy) RATA (relative accuracy test audit) **RB** (reagent blank) RE (removal or reduction efficiency) RM (reference method) scf (standard cubic feet) scfm (standard cubic feet per minute) SCR (selective catalytic reduction) SDA (spray dryer absorber) SNCR (selective non-catalytic reduction) STD (standard) STMS (sorbent trap monitoring system) TBtu (trillion British thermal units) **TEOM (Tapered Element Oscillating** Microbalance) TEQ (toxic equivalency quotient) ton/hr (ton per hour) ton/yr (ton per year) TSS (third stage separator) USEPA or EPA (United States Environmental Protection Agency) UVA (ultraviolet absorption) WFGD (wet flue gas desulfurization) %wv (percent, wet volume)

# 1. PROJECT OVERVIEW

# Test Program Summary

Marathon Petroleum Company LP (MPC) contracted CleanAir Engineering (CleanAir) to complete testing on the Coker Heater (EU70-COKERHTR-S1) at the Detroit Refinery.

This test program is a re-test of an invalid test conducted on August 15, 2018. The testing produced only two valid test runs. Of the three test runs conducted, the first run was invalidated due to contamination in the sample. Refer to CleanAir Report No. 13647-3 for further details.

This test program included the following objective:

 Perform filterable particulate matter (FPM), condensable particulate matter (CPM), and sulfuric acid mist (H<sub>2</sub>SO<sub>4</sub>) testing to demonstrate compliance with regard to particulate matter (PM) and total particulate matter less than 10 microns in diameter (PM<sub>10</sub>) regulations outlined in the Michigan Department of Environmental Quality (MDEQ) 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. Test program information, including the test parameters, on-site schedule and a project discussion, begins on page 2.

# Table 1-1: Summary of Compliance Results

Source	Sampling Method	Average	
Constituent	(USEPA)	Emission	Permit Limit <sup>1</sup>
Coker Heater Stack			
FPM (lb/MMBtu)	5	0.0006	N/A
PM <sub>10</sub> (lb/MMBtu)	5/202	0.0023	0.0076
H₂SO₄ (Ib/MMBtu)	ASTM Draft CCM	0.0006	N/A
PM (Ib/MMBtu) <sup>2,3</sup>	5 / ASTM Draft CCM	0.00002	0.0019
NSFPM (lb/MMBtu) <sup>4</sup>	5B	0.0004	N/A

<sup>1</sup> Permit limits obtained from MDEQ Renew able Operating Permit No. MI-ROP-A9831-2012c.

<sup>2</sup> Expressed as the average of the three (3) highest valid runs.

<sup>3</sup> PM assumed equivalent to FPM less H<sub>2</sub>SO<sub>4</sub>. See page 2 for further description.

<sup>4</sup> NSFPM measured for supplemental purposes.



# Test Program Details

#### Parameters

The test program included the following emissions measurements:

- filterable particulate matter (FPM)
- total particulate matter less than 10 microns in diameter (PM<sub>10</sub>), assumed equivalent to the sum of the following constituents:
  - o FPM
  - o condensable particulate matter (CPM)
- sulfuric acid mist (H<sub>2</sub>SO<sub>4</sub>)
- PM assumed equivalent to FPM minus H<sub>2</sub>SO<sub>4</sub>
- nonsulfuric acid particulate matter (NSFPM)
- flue gas composition (e.g., O<sub>2</sub>, CO<sub>2</sub>, H<sub>2</sub>O)
- flue gas temperature
- flue gas flow rate

#### Schedule

Testing was performed on December 4 and 5, 2018. The on-site schedule followed during the test program is outlined in Table 1-2.

#### Table 1-2: Test Schedule

(CSC Scher	2010					
Run Number	Location	Method	Analyte	Date	Start Time	End Time
1	Coker Heater Stack	LISEPA Method 5 / 202	FPM/CPM	12/04/18	13:18	16:46
2	Coker Heater Stack	USEPA Method 5 / 202	FPM/CPM	12/05/18	09:49	11:55
3	Coker Heater Stack	USEPA Method 5 / 202	FPM/CPM	12/05/18	13:08	15:18
4	Coker Heater Stack	USEPA Method 5 / 202	FPM/CPM	12/05/18	16:30	19:05
1	Coker Heater Stack	USEPA Method 5B	NSFPM	12/04/18	13:18	16:46
2	Coker Heater Stack	USEPA Method 5B	NSFPM	12/05/18	09:44	11:55
3	Coker Heater Stack	USEPA Method 5B	NSFPM	12/05/18	13:08	15:18
4	Coker Heater Stack	USEPA Method 5B	NSFPM	12/05/18	16:29	19:05
0	Coker Heater Stack	Draft ASTM CCM	Sulfuric Acid	12/04/18	09:07	10:07
1	Coker Heater Stack	Draft ASTM CCM	Sulfuric Acid	12/04/18	13:18	16:46
2	Coker Heater Stack	Draft ASTM CCM	Sulfuric Acid	12/05/18	09:44	11:55
3	Coker Heater Stack	Draft ASTM CCM	Sulfuric Acid	12/05/18	13:08	15:18
4	Coker Heater Stack	Draft ASTM CCM	Sulfuric Acid	12/05/18	16:30	19:05
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#### Discussion

#### Project Synopsis

#### PM and PM<sub>10</sub> Testing

A total of four (4) 120-minute EPA Method 5/202 test runs were performed. PM and PM<sub>10</sub> 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 H<sub>2</sub>SO<sub>4</sub> emissions. H<sub>2</sub>SO<sub>4</sub> 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.

 $PM_{10}$  is assumed equivalent to the sum of FPM and CPM. The Method 5/202 sample train yields a front-half, FPM result and a back-half, CPM result. The total PM result (FPM plus CPM) from Method 5/202 can be used as a worst-case estimation of total  $PM_{10}$  since Method 5 collects all FPM present in the flue gas (regardless of particle size). The final result was expressed as the average of four (4) valid runs.

#### H<sub>2</sub>SO<sub>4</sub> Testing – Draft ASTM Controlled Condensation Method

H<sub>2</sub>SO<sub>4</sub> 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 5/202 runs. H<sub>2</sub>SO<sub>4</sub> emission results were calculated in units of lb/MMBtu. The H<sub>2</sub>SO<sub>4</sub> final results were expressed as the average of four (4) valid runs.

Diluent concentrations ( $\%O_2$ ,  $\%CO_2$ ) from concurrent Method 5/202 runs were utilized to convert H<sub>2</sub>SO<sub>4</sub> concentrations to units of Ib/MMBtu. There was no diluent concentration data collected during H<sub>2</sub>SO<sub>4</sub> runs because, due to sufficiently low ambient temperature, there was insufficient sample flow to create pressure drop to collect a slip stream of the sample gas. This measure was approved on-site by Tom Gasoli of MDEQ.

Prior to the first official test run, 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 H<sub>2</sub>SO<sub>4</sub>-collecting portion of the sample train). The conditioning run was recovered in the same manner as the official test runs.

#### NSFPM Testing – USEPA Method 5B

A total of four (4) 120-minute EPA Method 5B test runs were performed concurrently with Method 5/202 and Draft ASTM CCM test runs. NSFPM emission results were calculated in units of pounds per million Btu (Ib/MMBtu). The NSFPM final results were expressed as the average of four (4) valid runs. NSFPM emissions were determined for supplemental purposes.

Run 1 moisture content was comparatively low. There was no overt explanation for this occurrence. NSFPM results were consistent with the other runs so the results from Run 1 were included in the final results.

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#### Fuel Analysis

Emission results in units of dry volume-based concentration (lb/dscf, ppmdv) were converted into units of pounds per million Btu (lb/MMBtu) by calculating an oxygen-based fuel factor (F<sub>d</sub>) for refinery gas per EPA Method 19 specifications. The heat content and F<sub>d</sub> 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. MPC was responsible for logging any relevant process-related data and providing it to CleanAir for inclusion in the test report.

# 2. RESULTS

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

#### Table 2-1: Coker Heater – FPM & PM<sub>10</sub> Emissions

Run No.	1	2	3	4	Average
Date (2018)	Dec 4	Dec 5	Dec 5	Dec 5	
Start Time (approx.)	13:18	09:49	13:08	16:30	
Stop Time (approx.)	16:46	11:55	15:18	19:05	
Process Conditions					
P <sub>1</sub> Charge rate (BPD)	42,570	42,600	42,570	42,570	42,600
P <sub>2</sub> Heater duty (MMBtu/hr)	250	247	248	250	249
F <sub>d</sub> Oxygen-based F-factor (dscf/MMBtu)	8,350	8,356	8,356	8,356	
Gas Conditions					
O <sub>2</sub> Oxygen (dry volume %)	7.6	7.9	7.7	7.9	7.8
CO <sub>2</sub> Carbon dioxide (dry volume %)	7.8	7.7	7.5	7.6	7.7
T <sub>s</sub> Sample temperature (°F)	393	395	395	394	394
Sampling Data					
V <sub>mstd</sub> Volume metered, standard (dscf)	86.79	78.56	81.53	79.60	81.62
%I Isokinetic sampling (%)	101.1	101.4	102.8	99.9	101.3
Laboratory Data					
m <sub>n</sub> Total FPM (g)	0.00192	0.00133	0.00167	0.00138	
m <sub>CPM</sub> Total CPM (g)	0.00552	0.00363	0.00509	0.00510	
m <sub>Part</sub> Total particulate matter (as PM <sub>10</sub> ) (g)	0.00744	0.00496	0.00676	0.00648	
FPM Results					
C <sub>sd</sub> Particulate Concentration (lb/dscf)	4.88E-08	3.73E-08	4.52E-08	3.82E-08	4.24E-08
E <sub>lb/w</sub> Particulate Rate (lb/hr)	0.170	0.118	0.146	0.124	0.139
E <sub>Fd</sub> Particulate Rate - F <sub>d</sub> -based (Ib/MMBtu)	0.000640	0.000502	0.000598	0.000514	0.000563
CPM Results					
C <sub>sd</sub> Particulate Concentration (lb/dscf)	1.40E-07	1.02E-07	1.38E-07	1.41E-07	1.30E-07
E <sub>lb/m</sub> Particulate Rate (lb/hr)	0.490	0.321	0.444	0.458	0.428
E <sub>Fd</sub> Particulate Rate - F <sub>d</sub> -based (lb/MMBtu)	0.00184	0.00137	0.00182	0.00190	0.00173
Total Particulate Matter (as PM <sub>10</sub> ) Results					
C <sub>sd</sub> Particulate Concentration (lb/dscf)	1.89E-07	1.39E-07	1.83E-07	1.80E-07	1.73E-07
E <sub>lb/br</sub> Particulate Rate (lb/hr)	0.660	0.439	0.590	0.582	0.568
E <sub>Fd</sub> Particulate Rate - F <sub>d</sub> -based (lb/MMBtu)	0.00248	0.00187	0.00242	0.00241	0.00229

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#### Table 2-2: Coker Heater – H<sub>2</sub>SO<sub>4</sub> Emissions

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Run No	•	1	2	3	4	Average
Date (2	018)	Dec 4	Dec 5	Dec 5	Dec 5	
Start Ti	me (approx.)	13:18	09:44	13:08	16:30	
Stop Ti	me (approx.)	16:46	11:55	15:18	19:05	
Proces	s Conditions					
P1	Charge rate (BPD)	42,570	42,570	42,570	42,570	42,570
P <sub>2</sub>	Heater duty (MMBtu/hr)	250	247	248	250	249
Fd	Oxygen-based F-factor (dscf/MMBtu)	8,350	8,356	8,356	8,356	
Gas Co	nditions <sup>1</sup>					
O <sub>2</sub>	Oxygen (dry volume %)	7.6	7.9	7.7	7.4	7.7
CO <sub>2</sub>	Carbon dioxide (dry volume %)	7.8	7.7	7.5	8.0	7.8
Ts	Sample temperature (°F)	394	397	396	395	395
$B_{w}$	Actual water vapor in gas (% by volume)	12.9	13.4	13.3	13.1	13.2
Sampli	ing Data					
V <sub>rrstd</sub>	Volume metered, standard (dscf)	54.76	54.67	54.36	53.65	54.36
Labora	tory Data (Ion Chromatography)					
mn	Total H <sub>2</sub> SO <sub>4</sub> collected (mg)	1.1421	0.8781	1.1513	1.2457	
Sulfuri	c Acid Vapor (H₂SO₄) Results					
$C_{sd}$	H <sub>2</sub> SO <sub>4</sub> Concentration (lb/dscf)	4.60E-08	3.54E-08	4.67E-08	5.12E-08	4.48E-08
$C_{sd}$	$H_2SO_4$ Concentration (ppmdv)	0.181	0.139	0.184	0.201	0.176
E <sub>Fd</sub>	H <sub>2</sub> SO <sub>4</sub> Rate - F <sub>d</sub> -based (Ib/MMBtu)	0.000603	0.000476	0.000618	0.000662	0.000590

<sup>1</sup> Diluent concentrations from concurrent EPA Method 5/202 runs.

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#### Table 2-3: Coker Heater – PM Emissions

001.01						
Run No	).	1	2	3	4	Average
Date (2018)		Dec 4	Dec 5	Dec 5	Dec 5	
Start Ti	me (approx.)	13:18	09:49	13:08	16:30	
Stop Ti	me (approx.)	16:46	11:55	15:18	19:05	
Proces	ss Conditions					
P <sub>1</sub>	Charge rate (BPD)	42,570	42,600	42,570	42,570	42,600
$P_2$	Heater duty (MMBtu/hr)	250	247	248	250	249
Fd	Oxygen-based F-factor (dscf/MMBtu)	8,350	8,356	8,356	8,356	
Gas Co	onditions					
O2	Oxygen (dry volume %)	7.6	7.9	7.7	7.9	7.8
CO <sub>2</sub>	Carbon dioxide (dry volume %)	7.8	7.7	7.5	7.6	7.7
T <sub>s</sub>	Sample temperature (°F)	393	395	395	394	394
FPM R	esults					
$E_{Fd}$	Particulate Rate - F <sub>d</sub> -based (Ib/MMBtu)	0.00064	0.00050	0.00060	0.00051	0.00056
Sulfuri	c Acid Vapor (H <sub>2</sub> SO <sub>4</sub> ) Results					
E <sub>Fd</sub>	H <sub>2</sub> SO <sub>4</sub> Rate - F <sub>d</sub> -based (lb/MMBtu)	0.00060	0.00048	0.00062	0.00066	0.00059
Partic	ulate Matter (as PM <sub>10</sub> ) Results <sup>1</sup>					
E <sub>Fd</sub>	Particulate Rate - F <sub>d</sub> -based (lb/MMBtu) <sup>2</sup>	3.66E-05	2.57E-05	-2.03E-05	-1.49E-04	2.08E-05

<sup>1</sup> Final PM results average of three (3) highest valid runs.

<sup>2</sup>Negative values considered zero in final average.

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### Table 2-4:

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Coker Heater – NSFPM Emissions	
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Run No.		1	2	3	4	Average
Date (2018)		Dec 4	Dec 5	Dec 5	Dec 5	
Start Tir	ne (approx)	13:18	09:44	13:08	16:29	
Stop Tir	ne (approx)	16:46	11:55	15:18	19:05	
Proces	s Conditions					
P <sub>1</sub>	Charge rate (BPD)	42,570	42,570	42,570	42,570	42,570
$P_2$	Heater duty (MMBtu/hr)	250	247	248	250	248
Fd	Oxygen-based F-factor (dscf/MMBtu)	8,350	8,356	8,356	8,356	
Gas Co	nditions					
$O_2$	Oxygen (dry volume %)	8.0	7.4	7.9	7.8	7.8
$CO_2$	Carbon dioxide (dry volume %)	7.2	8.0	7.6	7.7	7.6
T <sub>s</sub>	Sample temperature (°F)	393	396	397	397	396
B <sub>w</sub>	Actual water vapor in gas (% by volume)	7.7	13.5	13.7	13.7	12.1
Sampli	ng Data					
V <sub>mstd</sub>	Volume metered, standard (dscf)	74.83	76.58	76.57	77.75	76.43
%1	Isokinetic sampling (%)	93.2	100.5	101.0	101.3	99.0
Labora	tory Data					
m <sub>FPM</sub>	Total FPM (g)	0.00120	0.00120	0.00089	0.00109	
NSFPM	Results					
C <sub>sd</sub>	Particulate Concentration (lb/dscf)	3.54E-08	3.46E-08	2.56E-08	3.09E-08	3.16E-08
E <sub>lb/hr</sub>	Particulate Rate (lb/hr)	0.115	0.107	0.079	0.097	0.100
E <sub>Fd</sub>	Particulate Rate - F <sub>d</sub> -based (lb/MMBtu)	0.000478	0.000447	0.000344	0.000412	0.000420

# 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 Coker unit (EU70-COKER) converts Vacuum Resid (Crude Vacuum Tower Bottoms), a product normally sold as asphalt or blended into residual fuel oil, into lighter, more valuable products. The Vacuum Resid feedstock is heated before it enters the main fractionator, where lighter material vaporizes. The fractionator bottoms are routed through a fired heater and then into a coke drum. This emission unit consists of process vessels (fractionators), coke drums, heater (EU70-COKERHTR-S1), cooling tower, compressors, pumps, piping, drains and various components (pumps and compressor seals, process valves, pressure relief valves, flanges, connectors, etc.). This emission group includes the Coke Handling System, which collects, sizes and transports the petroleum coke created during the coking process. The system consists of a coke pit, storage pad, enclosed crusher, enclosed conveyors and surge bins.

The Coker Heater is fired by refinery fuel gas. Emissions are vented to the atmosphere via the Coker Heater Stack (SV70-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 10 represents the layout of the test location.

#### Table 3-1: Sampling Point Information

Source				Points per	Minutes	Total	
Constituent	Method (USEPA)	Run No.	Ports	Port	per Point	Minutes	Figure
Coker Heater Stack	en de la constante de la consta						
FPM/CPM	5 / 202	1-4	4	3	10	120	3-1
H₂SO₄	Draft ASTM CCM	1-4	1	1	120	120	N/A <sup>1</sup>
NSFPM	5B	1-4	4	3	10	120	3-1

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

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#### Figure 3-1:



Sampling Point	% of Stack Diameter	Port to Point Distance (inches)
1	29.6	31.8
2	14.6	15.5
3	4.4	4.7

Duct diameters upstream from flow disturbance (A): 5.2	Limit: 0.5
Duct diameters downstream from flow disturbance (B): 8.3	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 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"
Title 40 CF	R Part 51. Appendix M

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

# 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"

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# Methodology Discussion

#### PM & PM<sub>10</sub> Testing – USEPA Method 5/202

PM and PM<sub>10</sub> emissions were determined using EPA Method 5/202.

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.

The back-half (Method 202 portion) of the sampling train is designed to mimic ambient conditions and collect only the particles that would truly form CPM in the atmosphere by minimizing the sulfur dioxide ( $SO_2$ ) and nitrogen oxide ( $NO_X$ ) interferences observed with earlier versions of the method, in which flue gas was bubbled through cold water, and  $SO_2$  and  $NO_X$  were absorbed and partially oxidized before they could be purged out with nitrogen ( $N_2$ ).

Flue gas exiting the front-half heated filter passes through a coiled condenser and dry impinger system jacketed by water continually circulated at ambient temperature. Moisture is removed from the flue gas without bubbling through the condensed water. Flue gas then passes through a tetrafluoromethane (TFE) membrane filter at ambient temperature. The temperature of the flue gas at the exit of the filter was 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 passes through two (2) 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 is only collected to determine the flue gas moisture and to thoroughly dry the gas. The sample gas then flows into a calibrated dry gas meter where the collected sample gas volume is 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. The back-half of the sample train (heated filter outlet, condenser, dry impingers and TFE membrane filter) was recovered per Method 202 requirements. The impinger train was purged with  $N_2$  at a rate of 14 liters per minute (lpm) for one (1) hour following each test run and prior to recovery.

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

All samples and blanks were returned to CleanAir Analytical Services in Palatine, Illinois, for gravimetric analysis. Upon receipt, the filters dessicated 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.

#### H<sub>2</sub>SO<sub>4</sub> Testing – Draft ASTM CCM

H<sub>2</sub>SO<sub>4</sub> emissions were determined referencing the Draft ASTM CCM.

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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  $\pm$  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) is located at the condenser outlet for the collection of residual SAM not collected by the condenser. The condenser temperature is regulated by a water jacket and the SAM filter is regulated by a closed oven. Both the water jacket and SAM filter oven were maintained at 140°F  $\pm$  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 (SO<sub>2</sub>) into the H<sub>2</sub>SO<sub>4</sub>-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 is 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 H<sub>2</sub>SO<sub>4</sub>-collecting portion of the sample train (condenser and SAM filter) was recovered into a single fraction using DI H<sub>2</sub>O as the recovery/extraction solvent; any H<sub>2</sub>SO<sub>4</sub> disassociates into sulfate ion (SO<sub>4</sub><sup>2-</sup>) and is stabilized in the H<sub>2</sub>O matrix until analysis.

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

#### NSFPM Testing – USEPA Method 5B

NSFPM emissions were determined using EPA Method 5B.

The front-half of the sampling train consisted of a glass nozzle, glass liner and filter holder heated to  $320^{\circ}F \pm 25^{\circ}F$  and a quartz fiber filter. Flue gas samples were extracted isokinetically per Method 5B requirements. The front-half portion of the sample train (nozzle, probe and heated filter) was recovered per Method 5B requirements, using acetone as the recovery solvent.

All samples and blanks were returned to CleanAir Analytical Services in Palatine, Illinois, for gravimetric analysis.