







## **REPORT ON COMPLIANCE TESTING**

Zug Island Underfire Combustion Stack

EES Coke Battery, LLC. 1400 Zug Island Road River Rouge, Michigan 48218 Client Reference No. 4701748329

CleanAir Project No. 14968 A2LA ISO 17025 Certificate No. 4342.01 A2LA / STAC Certificate No. 4342.02 Revision 0, Final Report October 26, 2023

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## 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 Submittal:

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10/26/2023

Date

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 Review:

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10/26/2023

Date

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## REPORT REVISION HISTORY

Revision	Date	Pages	Comments
D0a	10/24/2023	All	Draft version of original document.
0	10/26/2023	All	Final version of original document.
	D0a	D0a 10/24/2023	D0a 10/24/2023 All

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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)/  $\leq$  (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) lb/lb-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))

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ml (milliliter(s)) MMBtu (million British thermal units) 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

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 was to perform total particulate matter (TPM), non-sulfate filterable particulate matter (NSFPM), and volatile organic compound (VOC) testing to demonstrate compliance with Michigan Permit to Install (MI-PTI) No. 51-08C. Emissions were sampled while the process operated at  $\geq$  90% operating capacity.

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:

Source		Average	
Constituent	Sampling Method	Emission	Permit Limit
Underfire Combustion Stack			
NSFPM (lb/hr)	EPA 5F (Modified)	5.4	25.7
NSFPM (gr/dscf)	EPA 5F (Modified)	0.004	0.012
FPM (lb/1000 lb exhaust gas @50% EA)	EPA 5	0.026	0.095
TPM, as PM <sub>10</sub> (lb/hr)	EPA 5/202	22.9	73.3
TPM, as PM <sub>2.5</sub> (lb/hr)	EPA 5/202	22.9	73.0
VOC (lb/hr)	EPA 25A	36.1	43.1
VOC (Ib/MMBtu, heat input)	EPA 25A	0.0710	0.0956

<sup>1</sup>Permit limits obtained from MI-PTI No. 51-08C.

## TEST PROGRAM DETAILS

### PARAMETERS

The test program included the following measurements:

- total particulate matter (TPM), filterable and condensable particulate matter (FPM and CPM), report as:
  - o particulate matter less than 10 microns in diameter (PM10)
  - particulate matter less than 2.5 microns in diameter (PM<sub>2.5</sub>)
- non-sulfate acid filterable particulate matter (NSFPM)
- volatile organic compounds (VOC), measured as non-methane hydrocarbons (NMHC)
- 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

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## SCHEDULE

Testing was performed on September 12, 2023. Table 1-2 outlines the on-site schedule followed during the test program.

#### Table 1-2: Test Schedule

Run					Start	End
Number	Location	Method	Analyte	Date	Time	Time
1	Underfire Combustion Stack	USEPA Method 3A, 25A	O2, CO2, VOC	09/12/23	10:56	11:56
2	Underfire Combustion Stack	USEPA Method 3A, 25A	O2, CO2, VOC	09/12/23	14:04	15:04
3	Underfire Combustion Stack	USEPA Method 3A, 25A	O2, CO2, VOC	09/12/23	17:02	18:02
1	Underfire Combustion Stack	USEPA Method 5F	Nonsulfate FPM	09/12/23	10:56	13:18
2	Underfire Combustion Stack	USEPA Method 5F	Nonsulfate FPM	09/12/23	14:04	16:26
3	Underfire Combustion Stack	USEPA Method 5F	Nonsulfate FPM	09/12/23	17:02	19:18
1	Underfire Combustion Stack	USEPA Method 5/202	FPM/CPM	09/12/23	10:56	13:18
2	Underfire Combustion Stack	USEPA Method 5/202	FPM/CPM	09/12/23	14:04	16:26
3	Underfire Combustion Stack	USEPA Method 5/202	FPM/CPM	09/12/23	17:02	19:18

## DISCUSSION

## PM10/PM2.5

Appendix A of MI-PTI No. 51-08C states that testing for PM<sub>10</sub> and PM<sub>2.5</sub> 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 EGLE Air Quality Division (AQD) District Supervisor.

### Modifications to PM10/PM2.5 Testing

The test ports at the sample location are not an adequate size to accommodate the Method 201A PM<sub>10</sub>/PM<sub>2.5</sub> cyclone head. Numerous issues with broken glass due to the narrow and long test ports occurred during the 2015 test campaign. CleanAir used EPA Method 5 in lieu of Method 201A. CleanAir has performed a results comparison between Method 201A/202 versus Method 5/202. Test data from the 2015 compliance program highlights 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.

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 was instead used to determine  $PM_{10}$  and  $PM_{2.5}$  emissions. The use of Method 5 rather than Method 201A was approved during the 2017, 2019,2021 and 2023 test programs.

In addition, this location experiences high winds that increase the likelihood of broken glassware during port changes. CleanAir requested approval to use stainless steel-lined probes and nozzles in lieu of borosilicate glass or quartz liners during the 2019 compliance campaign. This was approved during the 2017, 2019, 2021 and 2023 test program.

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### 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.

### Modifications to NSFPM Testing

CleanAir sampled particulate matter isokinetically and collected on a filter maintained at a temperature in the range of  $320 \pm 25$ °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, 2019, 2021 and 2023 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 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 analytical step:
  - a. The acetone was evaporated in a tared FEP beaker liner while the filter was digested.
  - b. The acetone residue was combined with the filter digestate and brought to volume in a 500 mL flask.
  - c. The flask was settled, and an aliquot was removed for sulfate determinations.
  - d. The solution was re-evaporated in the original tared FEP beaker liner and the normal analytical steps, as outlined in Method 5F, were followed.

In addition, CleanAir requested approval to use stainless steel-lined probes and nozzles in lieu of borosilicate glass or quartz liners during the 2019 compliance campaign as mentioned in the  $PM_{10}/PM_{2.5}$  discussion above. This was approved during the 2017, 2019, 2021 and 2023 test program.

### VOC

VOC emission rates from the Underfire Combustion Stack were measured following EPA Method 25A. A total of three 60-minute tests were performed at a single point following a stratification check performed during Run 1. Results of the stratification check are in Appendix D. VOC results were reported on a propane-basis.

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CleanAir directly measured the NMHC using a Thermo Model 55i Non-Methane Hydrocarbon Analyzer. The 55i analyzer utilizes a back-flush GC/FID system to cut the methane (GC) and measure non-methane hydrocarbons (FID) directly. It has lower detection limits of 20 ppb methane and 50 ppb NMHC. The proprietary column design is unaffected by the oxygen content of the sample and provides complete recovery of low volatility compounds while achieving absolute separation of methane from all carbon (C<sub>2</sub>) compounds. Each measurement cycle takes approximately 70 seconds.

The NMHC measurement is reported as VOC. The NMHC measurements were made on a wet volumetric basis and corrected to a dry basis using concurrent flue gas moisture measurements. Mass emission rates were calculated using the heat value of the fuel in conjunction with relative EPA Method 19 calculations.

End of Section

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# 2. RESULTS

This section summarizes the test program results. Additional results are available in the report appendices.

Run No		1	2	3	Average
Date (2	023)	Sep 12	Sep 12	Sep 12	
Start Tir	ne (approx.)	10:56	14:04	17:02	
Stop Tir	ne (approx.)	13:18	16:26	19:18	
Proces	s Conditions				
P <sub>1</sub>	No. of ovens charged per run	11	9	10	
P <sub>2</sub>	Coal charged (dry tons/run)	358	290	322	323
P <sub>3</sub>	COG used for Underfire combustion (kscf/run)	2,289	2,304	2,227	2,273
Cap	Capacity factor (hours/year)	8,760	8,760	8,760	8,760
Gas Co	nditions				
02	Oxygen (dry volume %)	10.4	10.5	10.5	10.5
CO <sub>2</sub>	Carbon dioxide (dry volume %)	5.2	5.2	5.1	5.1
Ts	Stack temperature (°F)	511	510	511	511
Bw	Actual water vapor in gas (% by volume)	14.9	14.8	15.1	14.9
Gas Flo	w Rate				
Qa	Volumetric flow rate, actual (acfm)	320,000	319,000	328,000	322,000
Qs	Volumetric flow rate, standard (scfm)	169,000	169,000	173,000	170,000
Q <sub>std</sub>	Volumetric flow rate, dry standard (dscfm)	144,000	144,000	147,000	145,000
Sampli	ng Data				
Vmstd	Volume metered, standard (dscf)	67.45	67.27	69.22	67.98
%1	Isokinetic sampling (%)	100.7	100.5	101.3	100.8
Labora	tory Data				
mn	Total NSFPM (g)	0.01508	0.02895	0.01323	
NSFPM	Results				
Csd	Particulate Concentration (lb/dscf)	4.93E-07	9.49E-07	4.21E-07	6.21E-07
C <sub>sd</sub>	Particulate Concentration (gr/dscf)	0.0034	0.0066	0.0029	0.0043
E <sub>lb/hr</sub>	Particulate Rate (Ib/hr)	4.25	8.19	3.71	5.38
ETAr	Particulate Rate (Ton/yr)	18.63	35.86	16.26	23.58

Average includes 3 runs.

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### Table 2-2:

## Underfire Combustion Stack – TPM, Method 5/202

Run No		1	2	3	Average
Date (2	023)	Sep 12	Sep 12	Sep 12	
Start Tir	ne (approx.)	10:56	14:04	17:02	
Stop Tir	ne (approx.)	13:18	16:26	19:18	
Proces	s Conditions				
EA	Excess Air (%)	88	89	90	89
P <sub>1</sub>	No. of ovens per run	11	9	10	
P2	Coal charged (dry tons/run)	358	290	322	323
P <sub>3</sub>	COG used for Underfire Combustion (kscf/run)	2,289	2,304	2,227	2,273
Cap	Capacity factor (hours/year)	8,760	8,760	8,760	8,760
Gas Co	nditions				
02	Oxygen (dry volume %)	10.4	10.5	10.5	10.5
CO2	Carbon dioxide (dry volume %)	5.2	5.2	5.1	5.1
Ts	Stack temperature (°F)	509	505	509	508
$B_{w}$	Actual water vapor in gas (% by volume)	15.0	14.4	13.8	14.4
Gas Flo	w Rate				
Qa	Volumetric flow rate, actual (acfm)	338,000	329,000	344,000	337,000
Qs	Volumetric flow rate, standard (scfm)	179,000	175,000	182,000	179,000
Qstd	Volumetric flow rate, dry standard (dscfm)	152,000	150,000	157,000	153,000
Samplin	ng Data				
Vmstd	Volume metered, standard (dscf)	70.30	69.47	71.06	70.28
%1	Isokinetic sampling (%)	97.5	98.0	95.4	97.0
Laborat	ory Data				
m	Total FPM (g)	0.12794	0.01876	0.02049	
m <sub>CPM</sub>	Total CPM (g)	0.02691	0.02134	0.02420	
m <sub>Part</sub>	Total particulate matter (g)	0.15485	0.04010	0.04469	
FPM Re	sults (Method 5) = PM				
Csd	Particulate Concentration (Ib/dscf)	4.01E-06	5.95E-07	6.36E-07	1.75E-06
Csd	Particulate Concentration (gr/dscf)	0.0281	0.00417	0.00445	0.0122
E <sub>lb/hr</sub>	Particulate Rate (lb/hr)	36.6	5.35	6.00	16.0
ETAT	Particulate Rate (Ton/yr)	160	23.4	26.3	70.0
EEA50%	Particulate Rate - Production-based (Ib/1000 Ib exhaust gas at 50% EA)	0.0595	0.0090	0.0096	0.0261
CPM Re	sults (Method 202)				
Csd	Particulate Concentration (Ib/dscf)	8.442E-07	6.775E-07	7.510E-07	7.576E-07
Csd	Particulate Concentration (gr/dscf)	0.0059	0.0047	0.0053	0.0053
Elb/hr	Particulate Rate (lb/hr)	7.7	6.1	7.1	7.0
ETAr	Particulate Rate (Ton/yr)	33.7	26.6	31.0	30.5
Total Pa	rticulate Matter Results (Method 5/202) = PM <sub>10</sub> = PM <sub>25</sub>				
Csd	Particulate Concentration (Ib/dscf)	4.857E-06	1.273E-06	1.387E-06	2.506E-06
Csd	Particulate Concentration (gr/dscf)	0.0340	0.0089	0.0097	0.0175
Elb/hr	Particulate Rate (lb/hr)	44.3	11.4	13.1	22.9
ETAR	Particulate Rate (Ton/yr)	194	50	57	101

Average includes 3 runs.

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# Table 2-3:

Run No	).	1	2	3	Average
Date (2	(023)	Sep 12	Sep 12	Sep 12	
Start Ti	me (approx.)	10:56	14:04	17:02	
	me (approx)	11:56	15:04	18:02	
Proces	s Conditions				
P <sub>1</sub>	No. of ovens charged per run	5	4	4	
P <sub>2</sub>	Coal charged (dry tons/run)	162	129	130	140
P <sub>3</sub>	COG used for Underfire combustion (ksfc/run)	976	976	977	976
Fd	Oxygen-based F-factor (dscf/MMBtu)	7,908	7,908	7,908	7,908
Hi	Actual heat input (MMBtu/hr)	511	511	512	512
Gas Co	onditions				
02	Oxygen (dry volume %)	10.4	10.5	10.5	10.5
CO <sub>2</sub>	Carbon dioxide (dry volume %)	5.2	5.2	5.1	5.1
Ts	Sample temperature (°F)	509	505	509	508
Bw	Actual water vapor in gas (% by volume)	15.0	14.4	13.8	14.4
Gas Flo	ow Rate				
Q <sub>std</sub>	Volumetric flow rate, dry standard (dscfm)	152,059	148,985	157,186	152,743
Volatile	e Organic Compunds (VOC), measured as NMHC				
	Concentration (ppmwv)	30.45	30.30	27.71	29.49
	Concentration (ppmdv)	35.84	35.39	32.14	34.46
	Mass Rate (lb/hr)	37.42	36.20	34.69	36.11
	Mass Rate (Ib/MMBtu) - Heat Input	0.073	0.071	0.068	0.071

Average includes 3 runs.

Sample temperature, moisture, and gas flow rate obtained from EPA Method 5/202 testing.

End of Section

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# 3. DESCRIPTION OF INSTALLATION

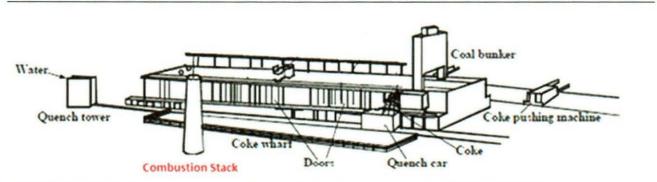
## PROCESS DESCRIPTION

EES Coke Battery, LLC is a facility located on Zug Island in River Rouge, Michigan. The testing described in this document was 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 Sack is located on the other side of the battery.

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## **TEST LOCATION**

EPA Methods 1 and 7E specifications determined the sample point locations. Table 3-1 presents the sampling information for the test location. The figures shown represent the layout of the test location.

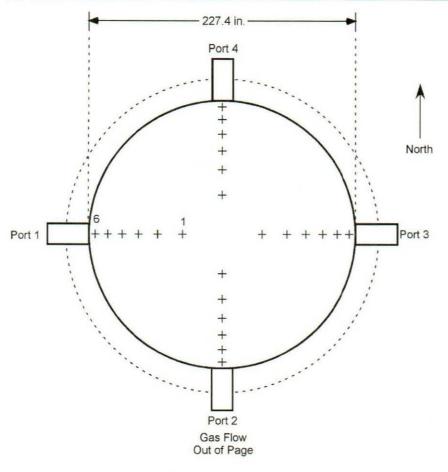
#### Table 3-1: Sampling Information

Source Constituent	Method	Run No.	Ports	Points per Port	Minutes per Point	Total Minutes	Figure
Underfire Combustio	n Stack						
NSFPM	EPA 5F (Modified)	1-4	4	6	5	120	3-2
TPM	EPA 5/202	1-3	4	6	5	120	3-2
VOC <sup>1</sup>	EPA 25A	1	1	3	20	60	3-3
VOC	EPA 25A	2-3	1	1	60	60	NA

<sup>1</sup> A stratification check was conducted during Run 1. The location was unstratified and testing for Runs 2 and 3 were conducted a single point that most closely matched the mean calculated during the stratification check.

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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	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

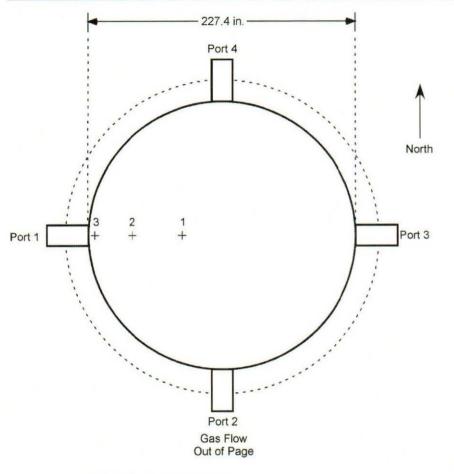
Duct diameters upstream from flow disturbance (A): 10.9	Limit: 0.5
Duct diameters downstream from flow disturbance (B): 2.7	Limit: 2.0

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### Figure 3-3:

Underfire Combustion Stack EPA Method 25A Stratification Check (EPA Method 7E)



Sampling Point	Port to Port Distance (meters)	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): 10.9LimDuct diameters downstream from flow disturbance (B): 2.7Lim

Limit: 0.5 Limit: 2.0

Note: Stratification Check performed during Run 1.

End of Section

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# 4. METHODOLOGY

## PROCEDURES AND REGULATIONS

The test program sampling measurements followed 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. 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 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 19	"Determination of Sulfur Dioxide Removal Efficiency and Particulate Matter, Sulfur Dioxide and Nitrogen Oxide Emission Rates"
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"

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## 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 was not repeated, and results are available in Appendix E of this report.

## 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 four test ports at six 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.

## NON-SULFATE FILTERABLE PARTICULATE MATTER – METHOD 5F (MODIFIED)

Particulate matter was withdrawn isokinetically and collected on a quartz fiber filter maintained at a temperature of  $160^{\circ}C \pm 14^{\circ}C$  ( $320^{\circ}F \pm 25^{\circ}F$ ). 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.

### TOTAL PARTICULATE MATTER DETERMINATION – METHOD 5/202

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

Flue gas exiting the front-half heated filter passed through a coiled condenser and dry impinger system jacketed by water continually circulated at ambient temperature. Moisture was removed from the flue gas without bubbling through the condensed water. Flue gas then passed through a Teflon 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 passed through two additional impingers surrounded by ice in a "cold" section of the impinger bucket. The moisture collected in these impingers was not analyzed for CPM and was only 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) 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) were recovered per Method 202 requirements. The impinger train was purged with N<sub>2</sub> at a rate of 14 liters per minute (lpm) for one hour following each test run and prior to recovery.

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A field train blank was assembled, purged, leak checked, 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 also collected to quantify background contamination. All samples and blanks were returned to CleanAir Analytical Services for gravimetric analysis. Method 202 samples were maintained at a temperature < 85°F during transport to the laboratory.

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

## VOLATILE ORGANIC COMPOUNDS - METHOD 25A

Monitoring of O<sub>2</sub>, CO<sub>2</sub>, and NMHC emissions was performed using a combination of EPA Methods 3A and 25A. A gas sample was continuously extracted and delivered to a series of gas analyzers, which measured the pollutant or diluent concentrations in the gas. The analyzers were calibrated on-site using certified mixtures of EPA Protocol 1 propane calibration gases.

The system utilized a heated stainless-steel probe for gas withdrawal. The heated stainless-steel probe tip was equipped with a sintered stainless-steel filter for particulate removal. The end of the probe was connected to a heated Teflon sample line that delivered 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 was performed during Run 1 as described in 40 CFR 60, Appendix A, Method 7E, §8.1.2. The stack measurement line was 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 differed from the mean concentration for all traverse points by no more than  $\pm$  5.0% of the mean concentration. The gas stream was considered unstratified and a single point that most closely matched the mean was used for Runs 2 and 3.

Calibration error checks were 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 were 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<sub>2</sub>, CO<sub>2</sub>, (dry basis), and THC (wet basis) were collected over a period of 60 minutes for each run. CleanAir used the JUM Model 109A analyzer, or equivalent, for the measurements. This model uses two individual detectors and two individual signal amplifiers. Samples were 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 subtracts the two values to provide a THC (excluding methane) reading.

End of Section

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## 5. APPENDIX

Appendix A: Test Method Specifications Appendix B: Sample Calculations Appendix C: Parameters Appendix D: QA/QC Data Appendix E: Field Data Appendix F: Field Data Printouts Appendix G: Reference Method Monitor Data Appendix H: Laboratory Data Appendix I: Facility Operating Data Appendix J: CleanAir Resumes and Certifications

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## APPENDIX A: TEST METHOD SPECIFICATIONS

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### **Specification Sheet for**

Source Location Name(s) Pollutant(s) to be Determined Other Parameters to be Determined from Train

#### Pollutant Sampling Information

Duration of Run No. of Sample Traverse Points Sample Time per Point Sampling Rate

#### Sampling Probe

Nozzle Material Nozzle Design Probe Liner Material Effective Probe Length Probe Temperature Set-Point

#### Velocity Measuring Equipment

Pitot Tube Design Pitot Tube Coefficient Pitot Tube Calibration by Pitot Tube Attachment

#### Metering System Console

Meter Type Meter Accuracy Meter Resolution Meter Size Meter Calibrated Against Pump Type Temperature Measurements Temperature Resolution ΔP Differential Pressure Gauge ΔH Differential Pressure Gauge Barometer

#### **Filter Description**

Filter Location Filter Holder Material Filter Support Material Cyclone Material Filter Heater Set-Point Filter Material

#### Other Components

Description Location Operating Temperature

### **EPA Method 5F**

Underfire Combustion Stack Non-Sulfate Filterable Particulate Matter (NSFPM) Gas Density, Moisture, Flow Rate

#### Standard Method Specification

N/A N/A N/A Isokinetic (90-110%)

Stainless Steel or Glass Button-Hook or Elbow Borosilicate or Quartz Glass N/A 320'F±25'F

Type S N/A Geometric or Wind Tunnel Attached to Probe

Dry Gas Meter ±2% N/A N/A Wet Test Meter or Standard DGM N/A N/A 5.4°F Inclined Manometer or Equivalent Inclined Manometer or Equivalent Mercury or Aneroid

After Probe Borosilicate Glass Glass Frit N/A 320°F±25°F Glass Fiber

N/A N/A N/A 120 minutes 24 5 minutes Isokinetic (90-110%)

Actual Specification Used

Stainless Steel Button-Hook Stainless Steel 12 feet 320°F±25°F

Type S 0.84 Geometric Attached to Probe

Dry Gas Meter ±1% 0.01 cubic feet 0.1 dcf/revolution Wet Test Meter Rotary Vane Type K Thermocouple/Pyrometer 1.0°F Inclined Manometer Inclined Manometer Digital Barometer calibrated w/Mercury Aneroid

Exit of Probe Borosilicate Glass Teflon None 320°F±25°F Glass Fiber

N/A N/A N/A

### Specification Sheet for

Impinger Train Description Type of Glassware Connections Connection to Probe or Filter by Number of Impingers Impinger Stem Types Impinger 1 Impinger 2 Impinger 3 Impinger 4 Impinger 5 Impinger 6

Impinger 7 Impinger 8

#### Gas Density Determination

Sample Collection Sample Collection Medium Sample Analysis

#### Sample Recovery Information

Nozzle and Probe Brush Material Nozzle and Probe Rinse Reagent Nozzle and Probe Rinse Wash Bottle Material Nozzle and Probe Rinse Storage Container Filter Recovered? Filter Storage Container Impinger Contents Recovered? Impinger Rinse Reagent Impinger Wash Bottle Impinger Storage Container

#### Analytical Information

Method 4 H<sub>2</sub>O Determination by Filter Preparation Conditions Front-Half Rinse Preparation Back-Half Analysis Additional Analysis

## EPA Method 5F

#### Standard Method Specification

Ground Glass or Equivalent Direct Glass Connection 4

Modified Greenburg-Smith Greenburg-Smith Modified Greenburg-Smith Modified Greenburg-Smith

Multi-point integrated Flexible Gas Bag Orsat or Fyrite Analyzer

Nylon Bristle Deionized Distilled Water Glass or Polyethylene Glass or Polyethylene Yes N/A Provision Deionized Distilled Water Glass or Polyethylene Glass or Polyethylene

Gravimetric

Gravimetric per Analytical Flow Chart Gravimetric per Analytical Flow Chart N/A None

#### Actual Specification Used

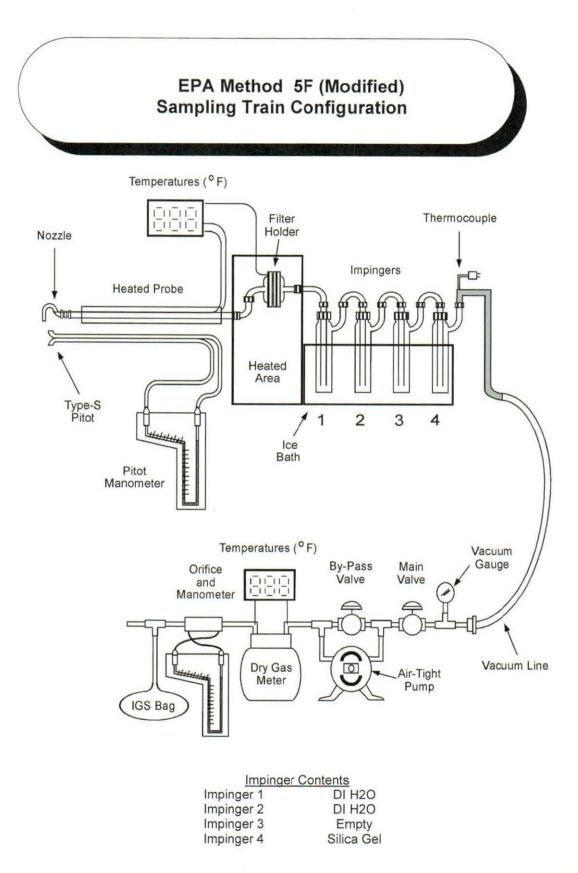
Screw Joint with Silicone Gasket Direct Glass Connection 4

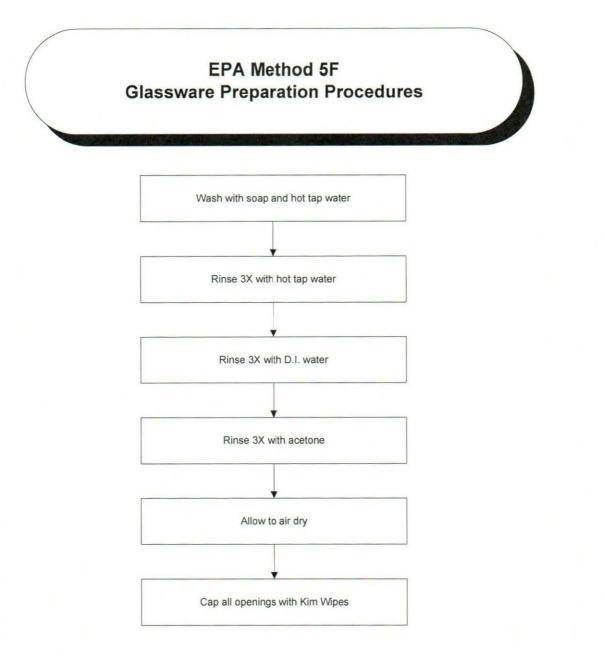
Modified Greenburg-Smith Greenburg-Smith Modified Greenburg-Smith Modified Greenburg-Smith

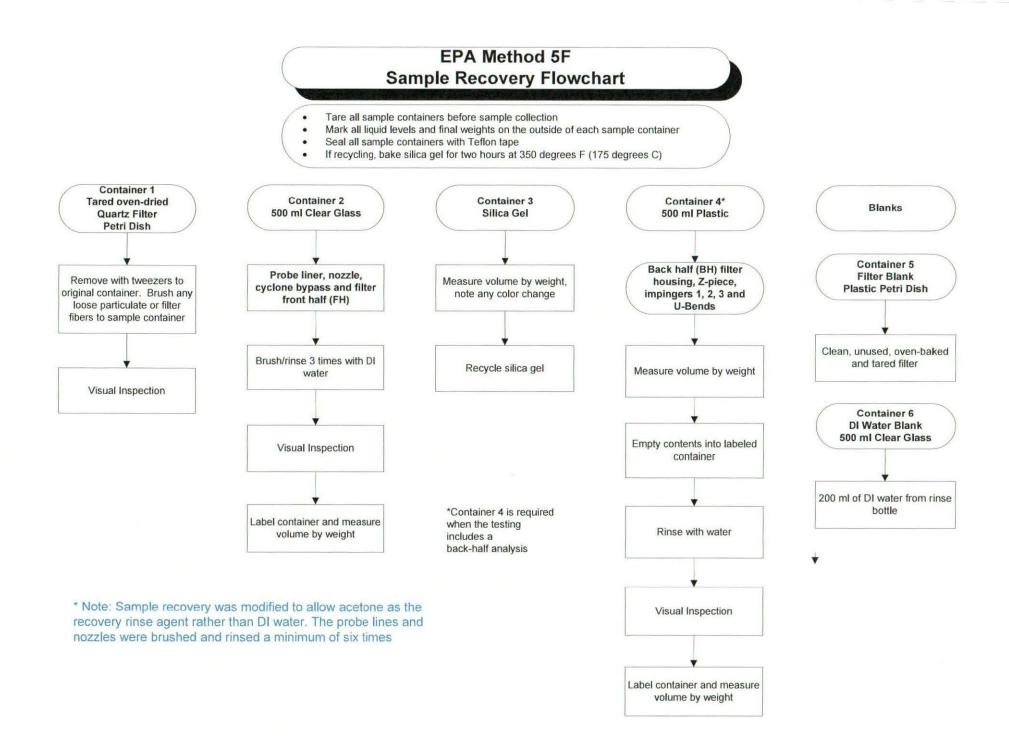
Multi-Point Integrated Vinyl Bag CEM

Nylon Bristle Deionized Distilled Water Polyethylene Yes Polystyrene Archived N/A N/A N/A

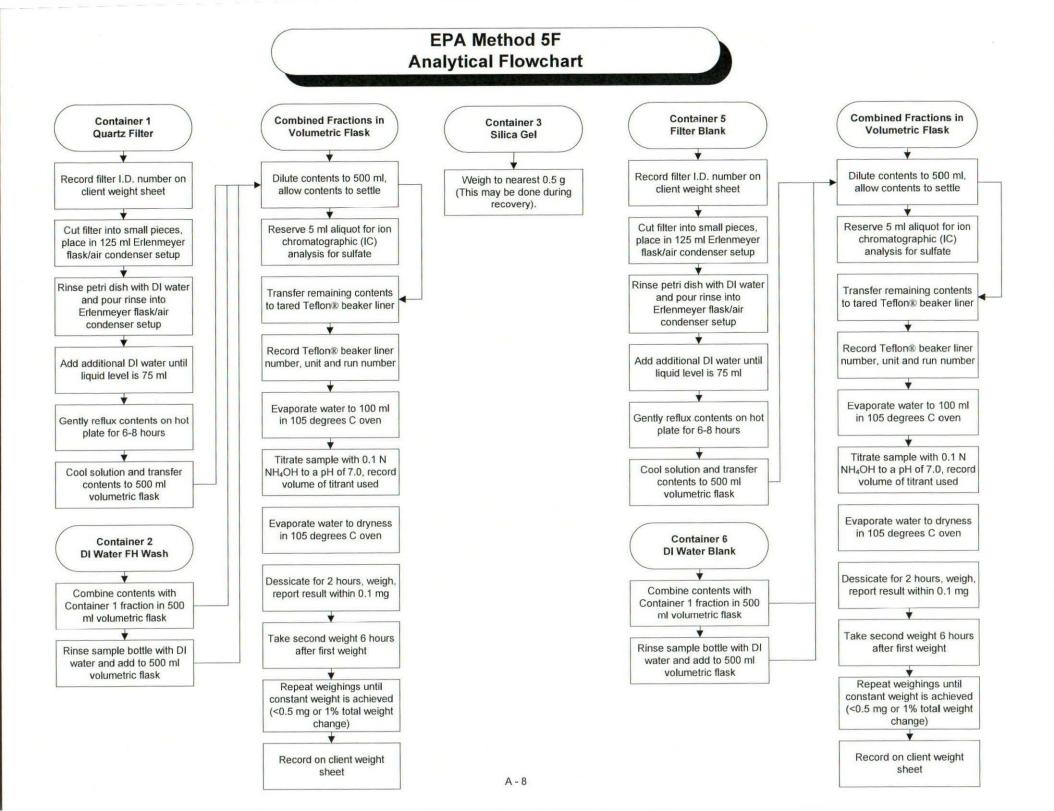
Gravimetric See Analytical Flow Chart See Analytical Flow Chart N/A None







A - 7



### Specification Sheet for

Source Location Name(s) Pollutant(s) to be Determined Other Parameters to be Determined from Train

#### Pollutant Sampling Information

Duration of Run No. of Sample Traverse Points Sample Time per Point Sampling Rate

#### Sampling Probe

Nozzle Material Nozzle Design Probe Liner Material Effective Probe Length Probe Temperature Set-Point

#### Velocity Measuring Equipment

Pitot Tube Design Pitot Tube Coefficient Pitot Tube Calibration by Pitot Tube Attachment

#### Metering System Console

Meter Type Meter Accuracy Meter Resolution Meter Size Meter Calibrated Against Pump Type Temperature Measurements Temperature Resolution ΔP Differential Pressure Gauge Barometer

### **FPM Filter Description**

Filter Location Filter Holder Material Filter Support Material Cyclone Material Filter Heater Set-Point Filter Material

#### Other Components

Description Location Operating Temperature

### EPA Method 5/202

Underfire Combustion Stack Filterable Particulate Matter (FPM) and Condensable Particulate Matter (CPM) Gas Density, Moisture, Flow Rate

#### Standard Method Specification Actual Specification Used N/A 120 minutes N/A 24 N/A 5 minutes Isokinetic (90-110%) Isokinetic (90-110%) Stainless Steel or Glass Stainless Steel Button-Hook or Elbow Button-Hook Glass or Teflon Stainless Steel N/A 12 feet 248°F±25°F 248°F±25°F Type S Type S N/A 0.84 Geometric or Wind Tunnel Geometric Attached to Probe Attached to Probe Dry Gas Meter Dry Gas Meter ±2% ±1% N/A 0.01 cubic feet N/A 0.1 dcf/revolution Wet Test Meter or Standard DGM Wet Test Meter N/A Rotary Vane N/A Type K Thermocouple/Pyrometer 5.4°F 1.0'F Inclined Manometer or Equivalent Inclined Manometer Inclined Manometer or Equivalent Inclined Manometer Mercury or Aneroid Digital Barometer calibrated w/Mercury Aneroid

Exit of Probe Borosilicate Glass Teflon None 248'F±25'F Quartz Fiber

> Condenser Before 1st Impinger ≤85°F

After Probe

Glass Frit

248°F±25°F

Glass Fiber

Condenser

≤85°F

Before Impinger 1

Quartz

N/A

### Specification Sheet for

#### Impinger Train Description

Type of Glassware Connections Connection to Probe or Filter by Number of Impingers Impinger Stem Types Impinger 1 Impinger 2 Impinger 3 Impinger 4 Impinger 5

Impinger 6 Impinger 7

Impinger 8

#### **CPM** Filter Description

Filter Location Filter Holder Material Filter Support Material Cyclone Material Filter Heater Set-Point Filter Material

#### Gas Density Determination

Sample Collection Sample Collection Medium Sample Analysis

#### Sample Recovery Information

Nozzle Brush Material Nozzle Rinse Reagent Nozzle Rinse Wash Bottle Material Nozzle Rinse Storage Container Filter Recovered? Filter Storage Container Impinger Contents Recovered? Impinger Rinse Reagent Impinger Wash Bottle Impinger Storage Container

#### **Analytical Information**

Method 4 H<sub>2</sub>O Determination by Filter Preparation Conditions Front-Half Rinse Preparation Back-Half Analysis Additional Analysis

### EPA Method 5/202

#### Standard Method Specification

Leak-Free Glass Connectors Direct or Flexible Connection 4

Shortened Stem (open tip) Modified Greenburg-Smith Modified Greenburg-Smith Modified Greenburg-Smith

Between 2nd and 3rd Impingers Glass, Stainless Steel or Tefion Tefion None >65°F but ≤85°F Tefion Membrane

Multi-point integrated Flexible Gas Bag Orsat or CEM Analyzer

Nylon Bristle or Teflon Acetone Glass or Polyethylene Glass or Polyethylene Yes FH filter in petri dish, CPM filter in petri dish Yes DI Water/Acetone/Hexane Inorganic in polyethylene, organic in Teflon Inorganic in polyethylene, organic in glass

#### Gravimetric

Dessicate 24 Hours or Filter Extraction Evaporate at ambient temperature and pressure Sonication and Extraction N/A

#### Actual Specification Used

Screw Joint with Silicone Gasket Direct Glass Connection 4

Shortened Stem (open tip) Modified Greenburg-Smith Modified Greenburg-Smith Modified Greenburg-Smith

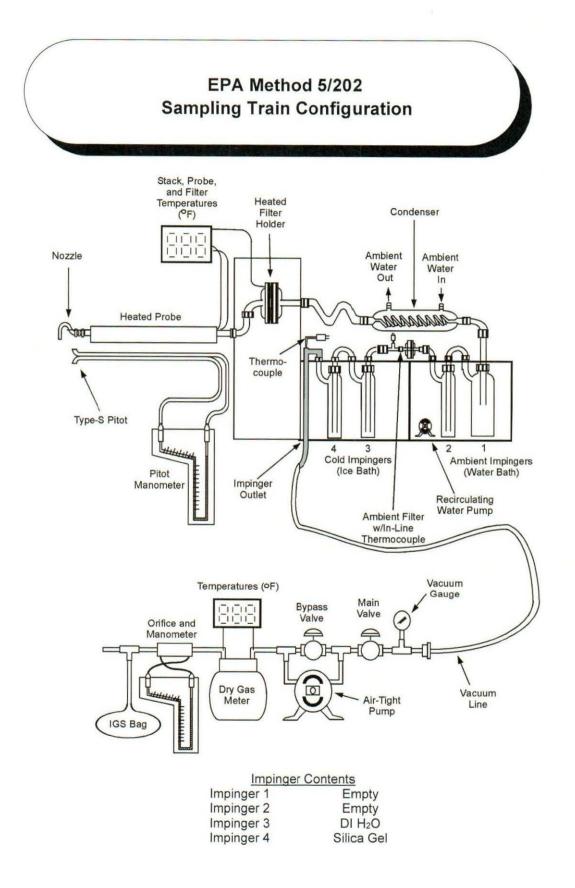
Between 2nd and 3rd Impingers Borosilicate Glass Teflon None >65°F but ≤85°F Teflon Membrane

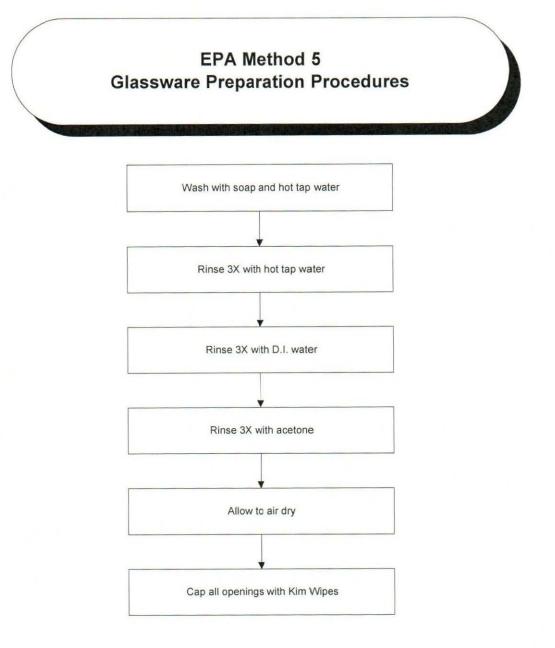
Multi-Point Integrated Vinyl Bag CEM

Nylon Bristle Acetone Inorganic in polyethylene, organic in Teflon Glass Yes FH filter in petri dish, CPM filter in petri dish Yes DI Water/Acetone/Hexane Inorganic in polyethylene, organic in Teflon Inorganic in amber glass, organic in amber glass

#### Gravimetric

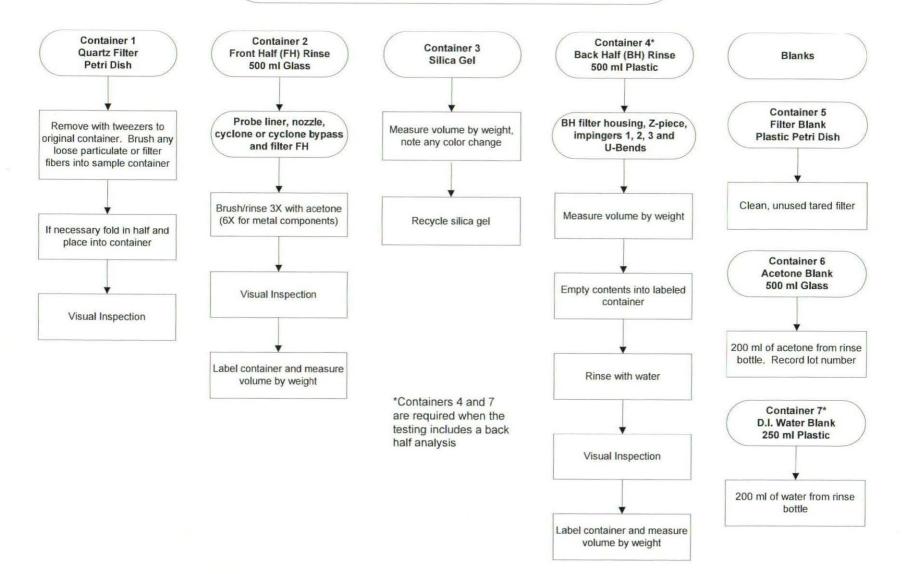
See Analytical Flow Chart Evaporate at ambient temperature and pressure See Analytical Flow Chart None

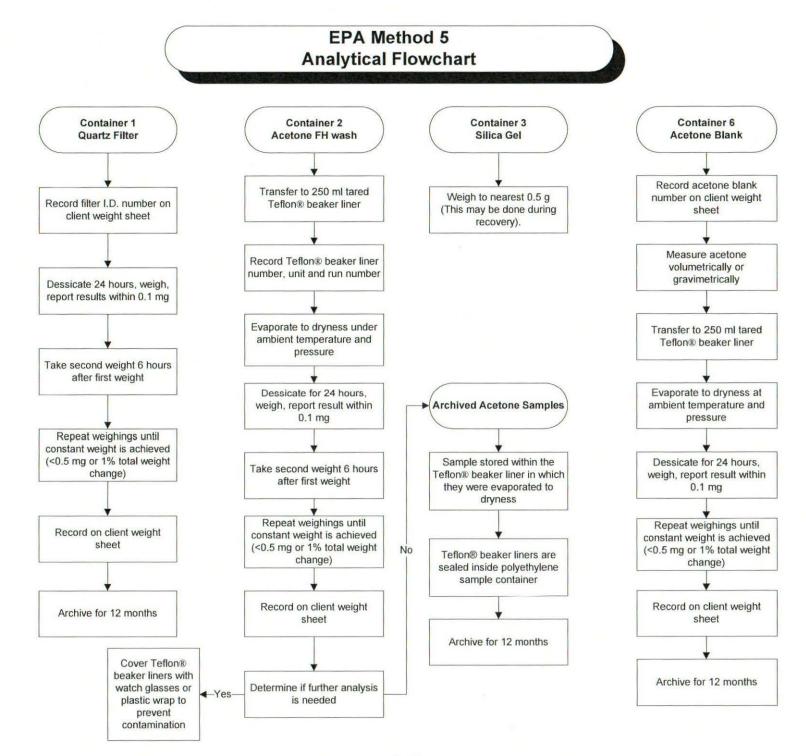


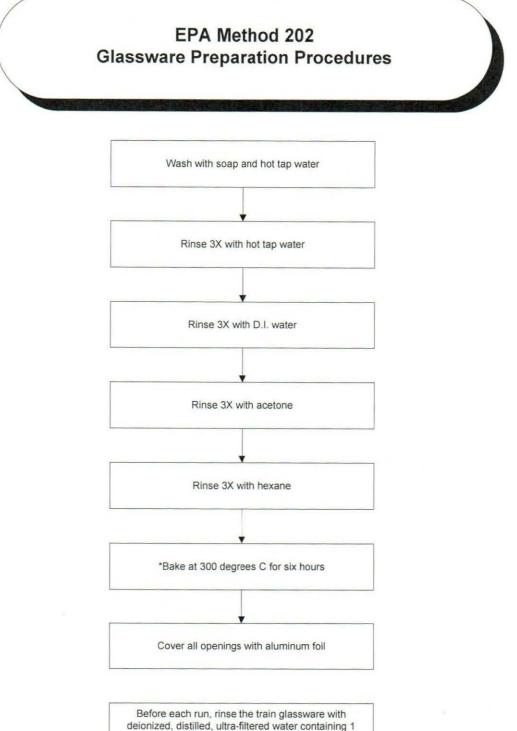


## EPA Method 5 Sample Recovery Flowchart

- Tare all sample containers before sample collection
- · Mark all liquid levels and final weights on the outside of each sample container
- Seal all sample containers with Teflon tape
- If recycling, bake silica gel for two hours at 350 degrees F (175 degrees C)

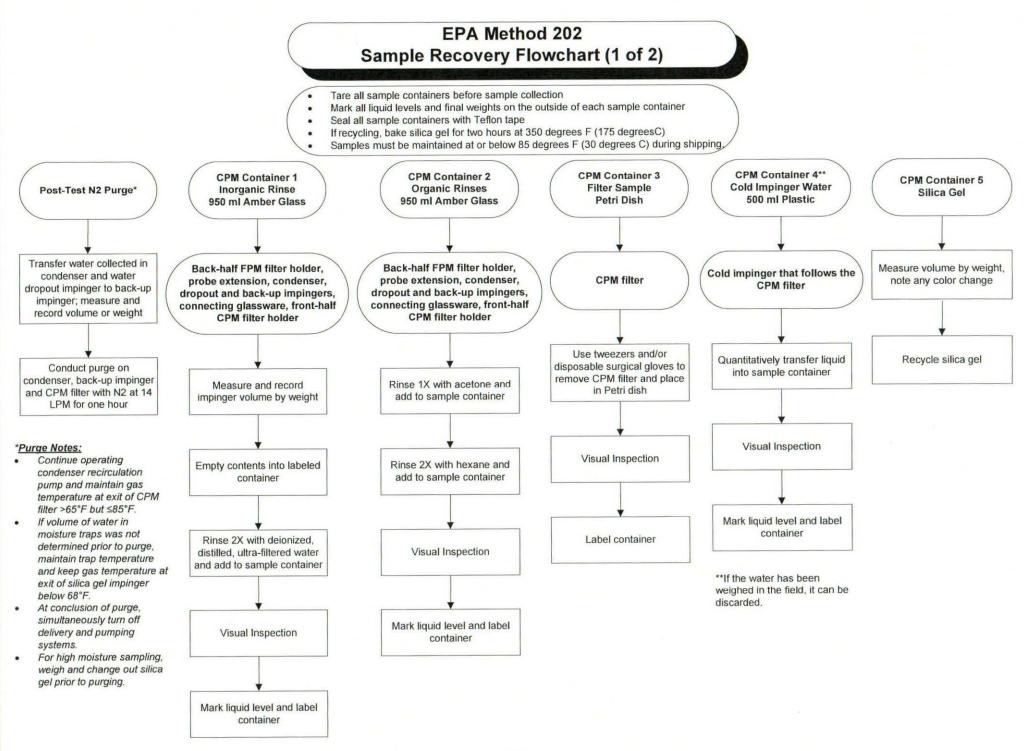






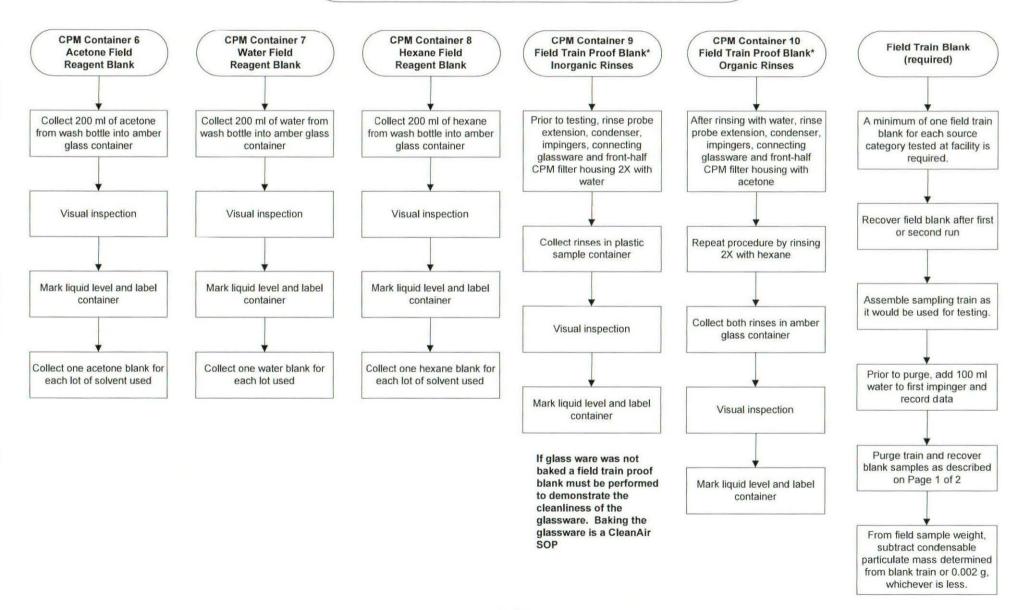
ppmwv residual mass or less.

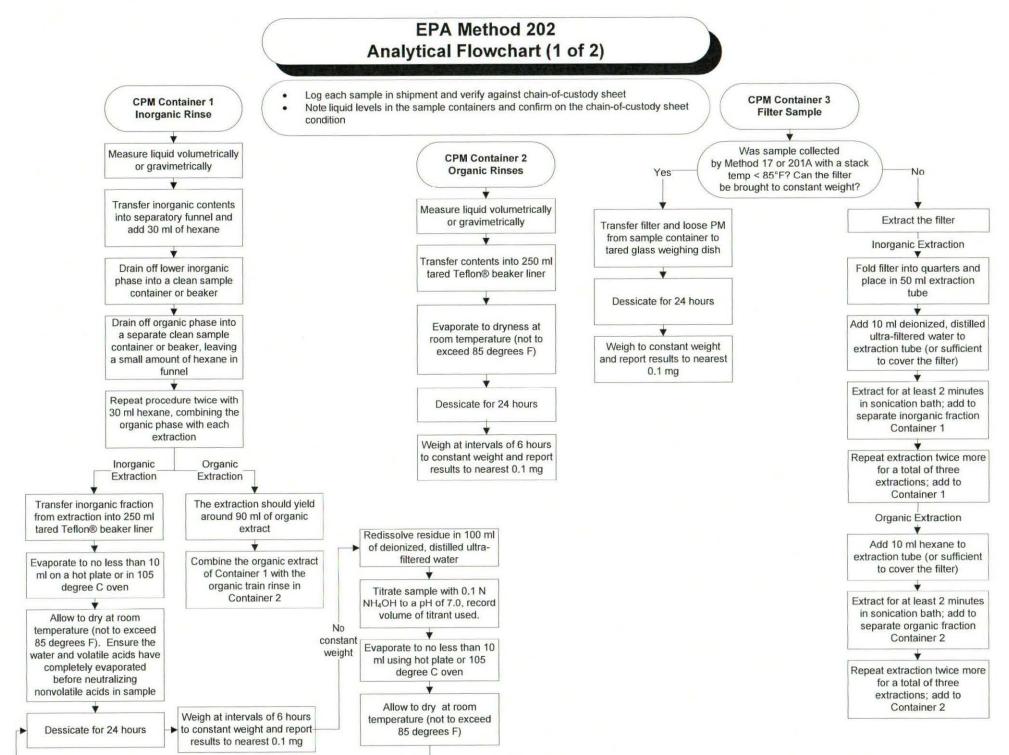
\*As an alternative to baking glassware, a field train proof blank can be performed on the sampling train glassware.

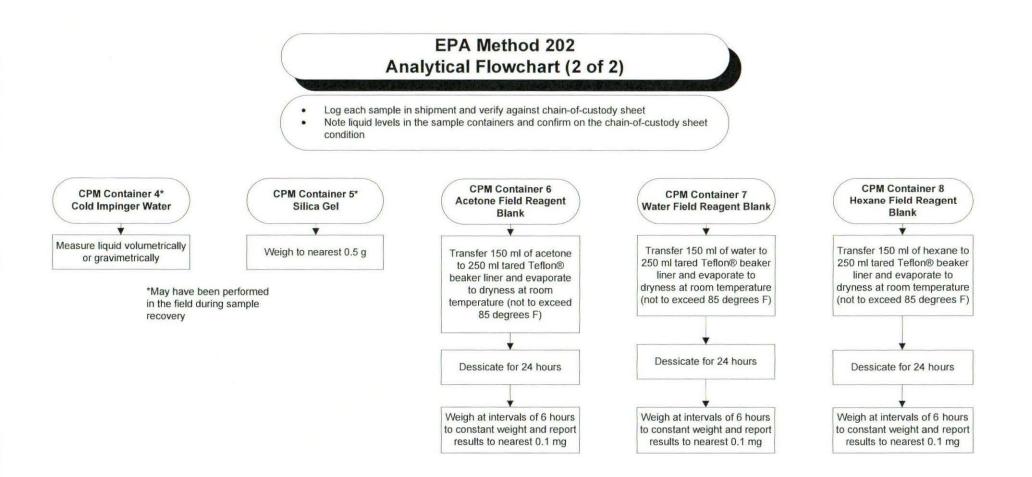


## EPA Method 202 Sample Recovery Flowchart (2 of 2)

- Tare all sample containers before sample collection
- · Mark all liquid levels and final weights on the outside of each sample container
- Seal all sample containers with Teflon tape
- If recycling, bake silica gel for two hours at 350 degrees F (175 degrees C)
  - Samples must be maintained at or below 85 degrees F (30 degrees C) during shipping.







### Specification Sheet for

Source Location Name(s) Pollutant(s) to be Determined Other Parameters to be Determined from Train

#### Pollutant Sampling Information

Duration of Run No. of Sample Traverse Points Sample Time per Point Sampling Rate

#### Sampling Probe

Nozzle Material Nozzle Design Probe Liner Material Effective Probe Length Probe Temperature Set-Point

#### **Particulate Filter**

In-Stack Filter In-Stack Filter Material External Filter External Filter Material External Filter Set-Point

#### Sample Delivery System

Heated Sample Line Material Heated Sample Line Set-Point Heated Sample Line Connections Moisture Removal System Sample Pump Type Sample Pump Material Sample Flow Control Non-Heated Sample Line Material Non-Heated Sample Line Connections Additional Filters Additional Filter Type Additional Filter Location Filter Material

#### Analyzer Description

Oxygen (O<sub>2</sub>) Carbon Dioxide (CO<sub>2</sub>) Sulfur Dioxide (SO<sub>2</sub>) Nitrogen Oxides (NO<sub>x</sub>) Carbon Monoxide (CO) Total Hydrocarbon (THC) Hydrogen Chloride (HCI) Ammonia (NH<sub>3</sub>)

### **EPA Method 25A**

Underfire Combustion Stack Total Gaseous Organic Concentration Diluents (O2 and CO2) - USEPA Method 3A

#### Standard Method Specification

N/A N/A N/A Constant Rate

#### N/A

N/A Stainless Steel or Equivalent Sufficient to Traverse Points Prevent Condensation (>230°F)

Optional N/A Yes Glass Fiber Mat Prevent Condensation (>230°F)

Stainless Steel or Teflon Prevent Condensation (>230°F) Probe Exit to Analyzer N/A N/A Non-reactive to sample gases Constant Rate N/A N/A Optional N/A Optional N/A

EPA Method 3A (Paramagnetic) EPA Method 3A (NDIR) N/A N/A EPA Method 25A (Flame Ionization Detection) N/A N/A

# 60 minutes

Actual Specification Used

- 1 1 minutes Constant Rate
- None N/A Stainless Steel 11 feet 248°F±25°F

Yes Fritted Stainless Steel Yes Borosilicate Glass Fiber Mat 248"F±25"F

Teflon 248°F±25°F Probe to Moisture System and THC Analyzer Refrigerator-type condenser Diaphragm Teflon Constant Rate (±10%) Teflon Moisture Removal to Diluent Analyzers Yes Particulate Removal Before Analyzers Glass Fiber

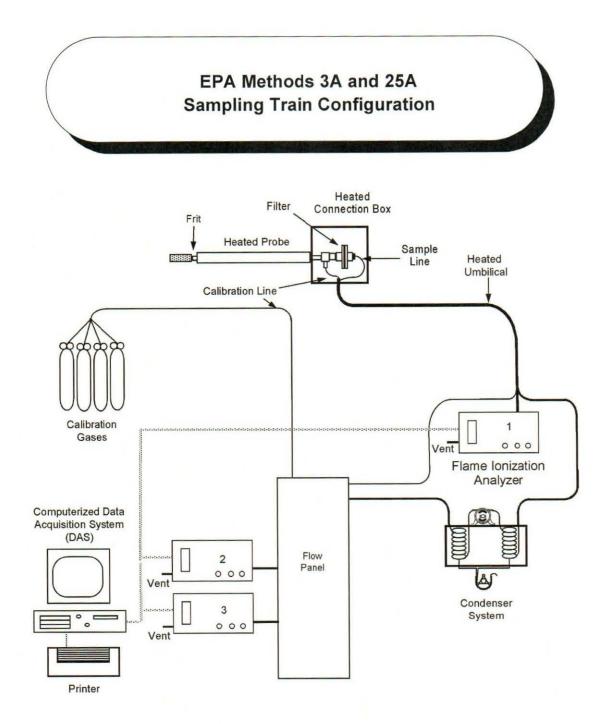
EPA Method 3A (Paramagnetic) EPA Method 3A (NDIR)

EPA Method 25A (Flame Ionization Detection)

## **Specification Sheet for**

### **EPA Method 25A**

	Standard Method Specification	Actual Specification Used
Instrument Span Range		
Oxygen (O <sub>2</sub> )	1.33 x Expected Maximum	0-20.2%
Carbon Dioxide (CO <sub>2</sub> )	1.33 x Expected Maximum	0-10.29%
Sulfur Dioxide (SO <sub>2</sub> )	N/A	N/A
Nitrogen Oxides (NO <sub>x</sub> )	N/A	N/A
Carbon Monoxide (CO)	N/A	N/A
Total Hydrocarbon (THC)	1.5 to 2.5 x Expected Maximum	0-82.15 ppm
Hydrogen Chloride (HCI)	N/A	N/A
Ammonia (NH <sub>3</sub> )	N/A	N/A
Data Acquisition		
Data Recorder	Strip chart, Analog Computer or Digital Recorder	Analog Computer
Recorder Resolution	0.5 Percent of Span	0.1 Percent of Span
Data Storage	Manually or Automatic	Manually
Measurement Freq. <60 min. Sample Time	1-min. intervals or 30 measurements (less restrictive)	One reading per second
Recording Freq. <60 min. Sample Time	1-min. intervals or 30 measurements (less restrictive)	One Minute Average (60, 1 second readings)
Measurement Freq. >60 min. Sample Time	2-min. intervals or 96 measurements (less restrictive)	N/A
Recording Freq. >60 min. Sample Time	2-min. intervals or 96 measurements (less restrictive)	N/A
Calibration Gas Specifications		
Oxygen (O <sub>2</sub> )	EPA Protocol 1	EPA Protocol 1
Carbon Dioxide (CO <sub>2</sub> )	EPA Protocol 1	EPA Protocol 1
Sulfur Dioxide (SO <sub>2</sub> )	N/A	
Nitrogen Oxides (NOx)	N/A	
Carbon Monoxide (CO)	N/A	
Total Hydrocarbon (THC)	EPA Protocol 1	EPA Protocol 1
Hydrogen Chloride (HCI)	N/A	
Ammonia (NH <sub>3</sub> )	N/A	





## SPEC SHEET

## Servomex 1420C O2 Analyzer

## **Rental and Application Notes**

- · Shipping Weight: 28 lbs.
- The analyzer measures the partial pressure of oxygen in the sample gas. Therefore, any change in sample pressure at the measuring cell will have an effect, which is proportional to the change in absolute pressure from the time of calibration.
- The Servomex 1420C/1415C can be plumbed together in a 19" rack mount. The combined weight is 44 lbs.
- These units are compatible with the older 1400B series



	Specifications
Weight	12 lbs
Dimensions	19" x 7" x 14"
Power	120VAC
Output	0-1v non-isolated or 4-20mA
Range	02 0-25%, 0-100%
Response Time	<3 seconds
Accuracy	+/- 0.1%
Flow Rate	1 - 6 L/min
Inlet Pressure	1 - 10 psig
Vent Pressure	11.8-15.9 psia
Linearity	+/- 0.1%
Repeatability	+/- 0.1%
Zero Drift	< + 0.002% O2/hour
Span Drift	< + 0.002% O2/hour
Relative Humidity	0 - 90% non-condensing
Storage Temperature	-4° F to 158° F