

1. Introduction

AECOM Technical Services, Inc. (AECOM) of Midland, Michigan was retained by DuPont Michigan Operations (DuPont/DDP) to perform emissions testing pursuant to the requirements of condition nos. V.1 through 4 in table FG963THROX of Renewable Operating Permit (ROP) No. MI-ROP-P1027-2020a. The ROP required DuPont to verify the destruction and removal efficiency (DRE) for volatile organic compounds (VOCs) by December 8, 2022. A complete report of the test results must be submitted to the Michigan Department of Environment, Great Lakes, and Energy (EGLE) Air Quality Division (AQD) Technical Programs Unit and District Office within 60 days following the last date of the test.

This test report describes how DuPont conducted the VOC DRE emissions compliance test following procedures outlined in US EPA reference test methods. Pursuant to condition nos. V.1 through 4 in table FG963THROX of the ROP, DuPont conducted testing to demonstrate compliance with a VOC DRE of 99.9% or greater. A VOC DRE of 99.9% is required by sending facility air permits, and it's also required by sending facility Title V monitoring plans. Specifics of the test program are discussed in the following sections.

1.1 Program Summary

AECOM conducted the compliance emissions test in order to demonstrate that FG963THROX was meeting a VOC DRE of 99.9% or greater. This was accomplished by completing emissions sampling at the FG963THROX exhaust stack for gaseous parameters including TOC, and fixed gas concentrations of Oxygen (O₂) and Carbon Dioxide (CO₂). During testing, the exhaust gas concentration of TOC was determined along with the stack gas volumetric flow rate. This data was used for subsequent calculations of mass emission rates at the FG963THROX outlet and for the DRE calculations from inlet and outlet mass emission rates. A summary of the test results is presented in **Table 1-1** at the end of this section. The emissions test data and calculated results are presented in **Appendix A**.

FG963THROX was tested on October 19, 2022. The combustion chamber temperature control setpoint was established, the THROX inlet chloromethane/methyl chloride (MeCl) injection rate was stabilized, and runs 1-3 for determination of the outlet stack TOC emission rate was completed. During testing, THROX outlet TOC (as THC) was measured while the following process conditions were recorded: MeCl inlet injection rate, THROX temperature (including associated scrubber flow rate and pH), sending facility vent flow rates (scfm), and associated process data. This data is presented in **Appendix B**. Subsequently, TOC DRE was determined from the inlet MeCl and outlet TOC mass emission rates.

This test report presents results for TOC emission concentrations as total hydrocarbon (THC) concentrations. THC was measured using a flame ionization analyzer calibrated with methane standards and expressed as ppmv carbon (C1) at actual exhaust gas conditions including moisture, wet (ppmvw C1). MeCl is a molecule having one carbon atom and was measured using the THC analyzer along with the other TOC compounds in the exhaust gas and expressed as ppmv at actual exhaust gas conditions including moisture (ppmvw C1).

Testing was performed at the THROX outlet using a total hydrocarbon (THC) analyzer. The sampling system provided real-time measurements. Representatives from the Michigan Department of Environment, Great Lakes & Energy (EGLE) were present during the test.

1.1.1 Worst Case Test Scenario

Due to the absence of a common vent header or convergence point for each of the sending facility vent lines tied to FG963THROX (i.e., they all vent independently into the burner), the total inlet VOC concentration to FG963THROX could not be determined. Therefore, spiking the inlet stream was the only option. No measurements of VOC, fixed gases, or gas stream flow rate was performed at the inlet of FG963THROX. Instead, DRE was determined by spiking a Principal Organic Hazardous Constituent (POHC) at known mass injection rates with the assumption that the injection rate represented the worst-

case minimum inlet TOC loading to the FG963THROX. The POHC for spiking at the inlet to FG963THROX was chloromethane (a.k.a., methyl chloride, CH₃Cl or MeCl).

Methyl chloride is considered a Class I POHC and has historically been used (and approved by EGLE / US EPA) as an acceptable spiking agent during previous DRE THROX performance tests at Michigan Operations (including FG963THROX). Due to the multiple sending units/companies venting to FG963THROX that are operating batch and/or continuous operations, it is difficult to predict what the HAP/VOC load for each sending facility will be during any particular time. Therefore, spiking with a Class I POHC in addition to normal vent flow/operation represented worst-case DRE results for FG963THROX. Since the ability to measure the VOC concentration on the inlet of each sending facility's vent line currently does not exist, spiking the THROX inlet with a known amount of methyl chloride compared to total VOC on the outlet allowed DuPont/DDP to conservatively calculate DRE.

1.1.2 THC Analyzer MeCl Response Factor

Subsequent to calibration of the THC analyzer with methane standards, a known standard of MeCl was introduced to the THC analyzer for determination of a response factor. This resulted in a response factor of 1.099, meaning that the MeCl standard was measured by the THC analyzer as greater than the actual certified concentration by a factor of 1.099. Therefore, the measured outlet TOC exhaust gas concentrations are likely overestimated by nearly the same factor since most of the outlet TOC was expected to be composed of MeCl. However, since the response factor was found to be only slightly greater than 1.0, outlet TOC concentrations were not corrected using the response factor to lower the test results but instead were taken as measured, expressed as ppmv carbon (C1), as a worst-case result for outlet TOC emissions. TOC mass emission rates were then determined from the measured TOC concentrations using the molecular weight of MeCl so that outlet mass emission rates (lb/hr) were consistent with inlet MeCl mass feed rates (lb/hr) for subsequent calculation of DRE.

Table 1-1. Summary of TOC Emission Rates and DRE Results ¹

Source	THROX Operating Temperature	Mass Emission Rate (lb/hr)		DRE (%) ²
		Inlet MeCl Feed Rate	Outlet TOC Emission Rate (as MeCl)	
FG963THROX	708.3 C	24.82	0.013	99.95

¹ Results are presented from the average of three (3) test runs.

² DRE is calculated as: (Inlet-Outlet)/Inlet * 100%.

1.2 Responsible Parties

AECOM personnel from the Midland, MI and Austin, TX offices conducted the sampling and analysis during this field effort. The primary responsibility of AECOM personnel was the analysis of the stack effluent for the requested compounds during the one-hour sampling duration test runs.

AECOM CONTACTS:

- James Edmister served as the Project Manager. In this role, he had the overall responsibility for the success and quality of the project. Mr. Edmister had primary authority for all decisions concerning sampling and analysis.
- Wayne Washburn, QSTI was the local representative from AECOM at the DuPont facility and served as the Technical Lead for the field program as well as operating the O₂, CO₂, and THC analyzers. Mr. Washburn also coordinated with plant operations for the success of the test.

- Quincy Crawford served as the stack platform technician for measurements of exhaust gas flow rate and maintaining sampling system integrity throughout the test program.

DUPONT CONTACTS:

- Jennifer Kraut provided support as the Environmental Focal Point for this test. The Environmental Focal Point is responsible for ensuring that all regulatory requirements and citations are reviewed and considered for the testing. All agency communications were completed through this role. Contact information is (989) 264-7009.
- James DeRosier provided support as the Process Focal Point. The Process Focal Point is responsible for coordinating the plant operation during the test and ensuring the unit is operating at the agreed-upon conditions in the test plan. They also serve as the key contact for collecting any process data required and providing all technical support related to process operation.

1.3 Test Chronology

Table 1-2. Summary of Sample Collection Times

Exhaust Stack	Run	Average Operating Temperature (degrees F)	Date/Time		
			Date	Run Start	Run End
FG963THROX	1	708.3 C	19-Oct- 2022	09:00	10:00
	2			11:15	12:15
	3			12:50	13:50

2. Results Summary

The results of the Renewable Operating Permit (MI-ROP-P1027-2020a) VOC DRE Test are summarized in Table 2-1 below.

Table 2-1. FG963THROX TOC Emissions, Process Data and DRE Test Results Summary

Run Identification	Run 1	Run 2	Run 3	Average
Run Date	10/19/22	10/19/22	10/19/22	
Run Time	09:00-10:00	11:15-12:15	12:50-13:50	
Exhaust Gas Conditions				
Oxygen (% dry)	11.20	11.33	11.29	11.27
Carbon Dioxide (% dry)	6.30	6.10	6.26	6.22
Flue Gas Moisture (%)	2.93	3.10	3.61	3.05
Flue Gas Velocity (ft/sec)	28.72	32.54	26.49	28.94
Flue Gas Flow Rate (acfm)	3,045	3,450	2,809	3,069
Flue Gas Flow Rate (scfm)	2,932	3,314	2,672	2,949
Flue Gas Flow Rate (dscfm)	2,846	3,212	2,575	2,860
Process Data				
THROX exit gas temperature (°C)	711.5	704.1	709.2	708.3
THROX scrubber flow rate (gpm)	81.9	81.9	81.3	81.7
THROX scrubber pH	6.8	7.8	8.3	7.6
THROX quench exit gas temperature (°C)	47.6	49.8	49.3	48.9
Corteva 948 Herbicides [EU03-S1/EU12B-S1] (SCFM)	33.5	33.0	35.0	33.8
Trinseo 743 Latex [EUB1-S2] (SCFM)	21.5	29.3	22.9	24.6
IFF Methocel High BTU [EUB2] (SCFM)	54.6	32.2	53.3	46.7
DuPont Special Resins/CMMC/Fine Mesh [EURule290] (SCFM)	0.0	0.0	0.0	0.0
DuPont Ion Exchange [EUAnion_XCHG/EU88] (SCFM)	150.0	163.9	142.1	152.0
IFF Methocel Low BTU [EUB2] (SCFM)	39.2	47.9	44.1	43.7
Dow 588 VBC [EU82-S1] (SCFM)	6.7	13.3	6.1	8.7
Total Hydrocarbons (as Chloromethane)				
Concentration (ppmvd)	0.72	0.64	0.37	0.58
Emission rate (lb/hr) (as CH ₃ Cl)	0.016	0.016	0.007	0.013
Inlet Loading of CH ₃ Cl (lb/hr)	24.33	24.83	25.29	24.82
Destruction Efficiency of CH ₃ Cl (%)	99.93%	99.94%	99.97%	99.95%

3. Sampling and Analytical Procedures

The emissions testing was performed in accordance US EPA Reference Method (RM) procedures. The RM calibration data, including manual equipment calibration checks and including analyzer initial calibration error tests, pre-run and post-run system bias and drift checks, system response time tests, and NO₂ converter efficiency test data (only for NO_x analyzers) are provided in **Appendix C**. Certificates of analysis for the RM analyzer calibration gases are provided in **Appendix D**. A copy of the emissions test protocol and Michigan EGLE approval letter are presented in **Appendix E**.

3.1 Sample Time

The duration of each test run was approximately sixty (minutes) for a total of 180 minutes sampling from the outlet stack for the THROX at the tested operating temperature setpoint with methyl chloride injected at the THROX inlet to simulate worst-case loading of the THROX for determination of TOC DRE.

3.2 Manual Test Methods - Flow Rate, Gas Composition, and Moisture

Concurrent with the performance of instrumental analyzer test runs, measurements were made to determine stack gas volumetric flow rate from readings of gas velocity and temperature (EPA Method 2), gas molecular weight composition (EPA Method 3A), and gas moisture (EPA Method 4).

3.3 Instrumental Analyzer Test Methods

Effluent gas was withdrawn from the FG963THROX exhaust stack and transported to the AECOM mobile instruments laboratory located at ground level. A stainless-steel sampling probe was inserted into the exhaust stack and used to collect sample gas. A heated Teflon sample line transported the sample gas from the sampling probe to the gas conditioning system serving the instrumental analyzers. The instrumental analyzers were kept at a stable temperature inside the AECOM mobile laboratory. At the mobile laboratory, a portion of the untreated (i.e., hot/wet) sample gas was routed to the THC analyzer for analysis on a wet basis, while the remainder of the sample gas was routed to a moisture condenser and then transported to the analyzers for analysis of O₂ and CO₂ on a dry basis.

The analyzers' electronic output signals were converted to a digital format and stored by AECOM's computerized data acquisition system. The system translated this digital signal into the proper units of measurement (e.g., percent CO₂ by volume on a dry basis) and stored them on a hard drive. The system stores the data as ten-second averages.

The instrumental analyzers were calibrated prior to initiating testing using appropriately certified standards as specified by EPA Methods 3A and 25A. Only EPA Traceability Protocol gases or certified pure zero nitrogen and air gases were used for calibration.

For the O₂ and CO₂ samples, a three-point direct calibration error test was performed on the instrumental analyzers prior to testing. Zero, mid-range, and span gases were introduced directly to the instruments to establish calibration error, or linearity. Then, the zero and mid-range gases were introduced through the entire sample acquisition system as a QC system bias check. The instrument direct response for each of these gases was no more than $\pm 2\%$ of span from the calibration gas value, and the system bias check for each of these gases was no more than $\pm 5\%$ of span from the direct response value.

For the THC sample, a four-point system calibration error test was performed on the instrumental analyzer prior to testing by passing calibration gas through the entire sample acquisition system. Zero and span gases were introduced through the entire sampling system to establish calibration set points. Then, the low and mid-range gases were introduced through the entire system as a QC calibration error check. The instrument system response for each of these gases was no more than $\pm 5\%$ of the respective calibration gas certified value.

The AECOM sampling system response time was checked. The total system, which includes the probe, sample line, sample pump, and condenser, were incorporated into the system response time test.

A system response time test for each parameter was performed and documented.

A schematic of the instrumental sampling system is shown in **Figure 3-1**.

3.3.1 EPA Method 3A (O₂/CO₂)

AECOM used a Servomex 1440 Series dual instrument analyzer to measure O₂ and CO₂ concentrations, on a dry volume basis according to EPA Method 3A. The analyzer employs paramagnetic detection for O₂ and non-dispersive infrared (NDIR) detection for CO₂.

3.3.2 EPA Method 25A (THC)

AECOM used a VIG Industries Model 20/2 analyzer to measure THC concentrations by volume on a wet (actual) basis according to EPA Method 25A. The THC instrument is a flame ionization analyzer (FIA).

3.4 RM Instrumental Analyzer Calibration Procedures

The initial phase of the instrumental analyzer methods (e.g., Methods 3A, 6C, 7E, 10, and 25A) requires initial measurement system performance tests to be performed, including calibration error tests, system bias checks, response-time tests, an NO₂ converter test (for NO_x analyzers), and interference checks, as applicable.

Prior to performing test runs with the dry-measurement analyzers (i.e., Methods 3A, 6C, 7E, and 10 instruments), AECOM conducted direct instrument calibration error tests using zero and two upscale gases each for the O₂ and CO₂ analyzers prior to initiation of testing. Following these direct calibrations, an initial system bias check was performed by sending zero and one upscale gas, from one gas cylinder at a time, up to the sample probe and back down through the components of the sampling system. Following the initial system bias checks, response-time data was obtained for each analyzer. Subsequently, system bias and drift checks were performed both prior to and following each test run using zero and one upscale calibration gas. These system checks allowed for the determination of initial and final system bias, as well as system drift for each test run set.

Prior to performing test runs with the hot-wet measurement analyzers (i.e., Method 25A instruments), AECOM conducted whole-system calibration error tests using zero and three upscale gases for the THC analyzer prior to initiation of testing. The initial system calibration error test was performed by sending zero and each of three upscale gases, from one gas cylinder at a time, up to the sample probe and back down through the components of the sampling system. Following these system calibrations, response-time data was obtained. Subsequently, system drift checks were performed both prior to and following each test run using zero and one upscale calibration gas. These system checks allowed for the determination of system drift for each test run set.

Test runs were performed during a continuous and uninterrupted period of 60 minutes followed by a system bias and drift check. The calibration gases used during this program were prepared in accordance with EPA Protocol G1 procedures as specified by the National Institute of Standards and Technology (NIST). The O₂/CO₂ combination calibration compressed gas standards were contained in individual cylinders having a purified nitrogen gas balance.

Interference check data provided by each instrument's manufacturer is maintained on file to meet the requirements of Method 7E (Subsection 8.2.7) as referenced in Methods 3A, 6C, and 10, as applicable.

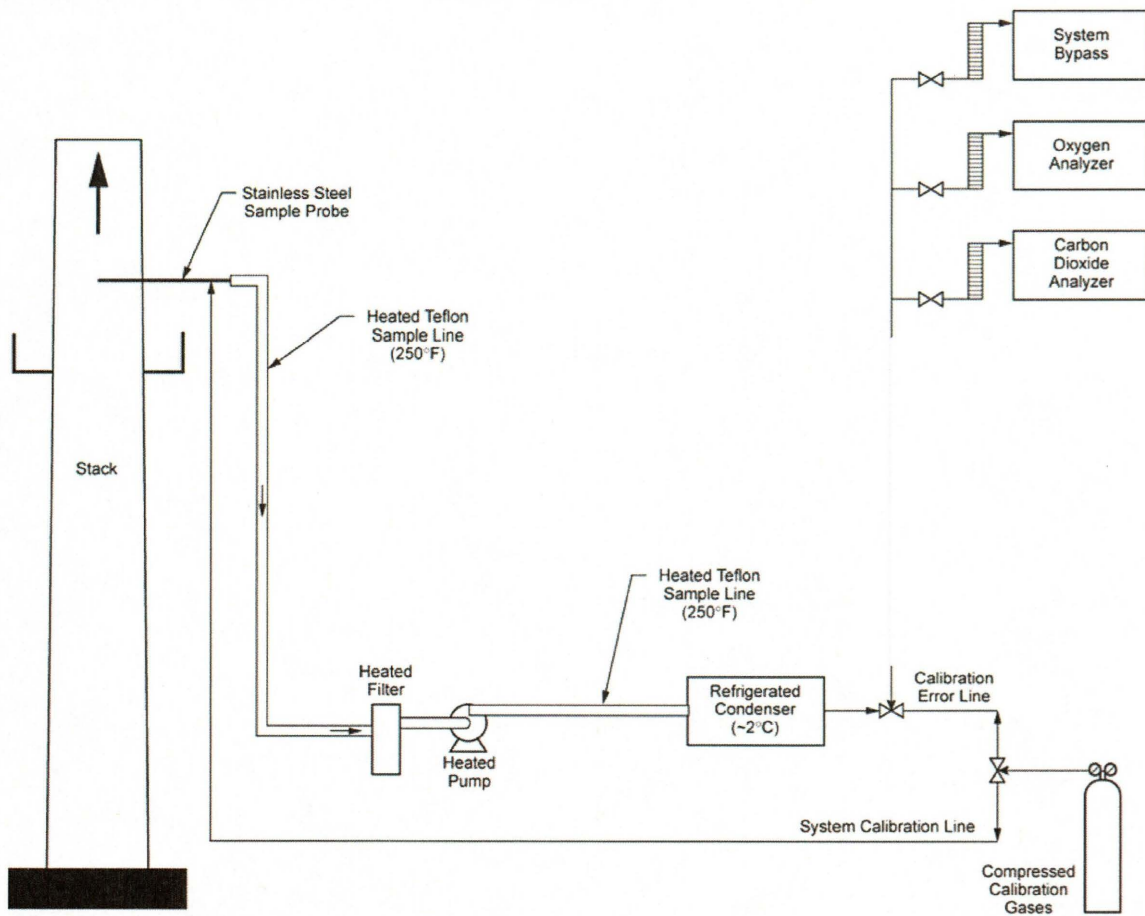
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Figure 3-1. Instrumental Analyzers Sampling System



4. Quality Assurance/Quality Control

4.1 Overview

During the sampling and measurements phase of the program, a strict quality assurance/quality control (QA/QC) program was adhered to. The QA/QC aspects of the program are discussed below.

4.2 Leak Check Procedure

Prior to conducting the instrumental analyzer testing, AECOM's Instrumental Measurements System was leak checked and verified to be leak free. Following the initial leak check, the system bias and drift criteria (as referenced in EPA Method 7E, 40 CFR Part 60, Appendix A) served as a continuous sample integrity check.

4.3 Instrumental Measurements System Calibrations

During the test program, AECOM used EPA instrumental analyzer methods (i.e., 3A, 6C, 7E, and 10, as applicable, in 40 CFR Part 60, Appendix A) for the measurement of O₂/CO₂, NO_x, and CO. The initial phase of instrumental analysis requires calibration of the involved monitors. Prior to performing test runs, AECOM conducted direct instrument calibration error tests using zero and two upscale gases each for the O₂/CO₂ and CO instruments prior to initiation of testing. Following these direct calibrations, an initial system bias check was performed by sending zero and one upscale gas, from one gas cylinder at a time, up to the sample probe and back down through the relevant components of the sampling system. During the initial system bias checks, response-time data was obtained for each analyzer. Subsequently, system bias checks were performed both prior to and following each test run using zero and one upscale calibration gas. These system checks allowed for the determination of initial and final system bias, as well as system drift for each test run. The calibration gases used during this program were prepared to EPA Protocol G1/G2 standards. Certificates of analysis for the calibration gases are presented in **Appendix D**. The measurement system performance criteria in 40 CFR Part 60, Appendix A, Methods 3A and 10 are listed below and were the performance criteria for the reference method instruments during this program.

Procedure	Performance Criterion
Calibration error	<±2% of the calibration span
System bias	<±5% of the calibration span
System drift	<±3% of the calibration span

The instrumental analysis methods also require correction of data for calibration drift and/or bias. The values used for the determination of emission rates were corrected for system drift and bias observed during each test run. System bias and drift as well as response-time data are presented in **Appendix C** of this report.

4.4 Interference Checks

Interference checks are required for each make and model of instrumental analyzer used for reference method measurements and signed documentation of the results must be included in each test report (as referenced in 40 CFR 60, Appendix A, Method 7E, Subsection 8.2.7). Copies of the instrument specific test results are presented in **Appendix C** of this document.

Appendix A

Emissions Test Data

Emission Summary Table
963 THROX TOC DRE Compliance Test
DuPont MiOps - Midland, MI
Building 963 THROX Stack

Run Identification	Run 1	Run 2	Run 3	Average
Run Date	10/19/22	10/19/22	10/19/22	
Run Time	09:00-10:00	11:15-12:15	12:50-13:50	
Exhaust Gas Conditions				
Oxygen (% dry)	11.20	11.33	11.29	11.27
Carbon Dioxide (% dry)	6.30	6.10	6.26	6.22
Flue Gas Moisture (%)	2.93	3.10	3.61	3.05
Flue Gas Velocity (ft/sec)	28.72	32.54	26.49	28.94
Flue Gas Flow Rate (acfm)	3,045	3,450	2,809	3,069
Flue Gas Flow Rate (scfm)	2,932	3,314	2,672	2,949
Flue Gas Flow Rate (dscfm)	2,846	3,212	2,575	2,860
Total Hydrocarbons (as Chloromethane)				
Concentration (ppmvd)	0.72	0.64	0.37	0.58
Concentration (ppmvd @0% Oxygen)	1.56	1.39	0.80	1.25
Conversion Factor (Cd) (lb/dscf/ppm)	1.31E-07	1.31E-07	1.31E-07	
Concentration (lb/dscf) (as CH ₃ Cl)	9.50E-08	8.35E-08	4.79E-08	7.55E-08
Emission rate (lb/hr) (as CH ₃ Cl)	0.016	0.016	0.007	0.013
Inlet Loading of CH ₃ Cl (lb/hr)	24.33	24.83	25.29	24.82
Destruction Efficiency of CH ₃ Cl (%)	99.93%	99.94%	99.97%	99.95%

Flow Rate and Moisture Summary			
Facility:	DuPont MiOps - Midland, MI		
Source:	Building 963 THROX Stack		
Project ID:	60675780		
Date	19-Oct-22		

Run Number	Flow Run 1	Flow Run 2	Flow Run 3
Moisture Run Number	Moisture 1	Moisture 2	Moisture 3
Date	19-Oct-22	19-Oct-22	19-Oct-22
Time Start	8:36	10:50	12:41
Time Finish	11:12	12:51	14:23
Overall Time	08:36-11:12	10:50-12:51	12:41-14:23
Operator	QC / W-W	QC / W-W	QC / W-W
Duct Diameter (ft)	1.5	1.5	1.5
Stack CrossSectional Area (sq ft)	1.7671	1.7671	1.7671
Pitot Tube Correction Factor (Cp)	0.84	0.84	0.84
Dry Gas Meter Calibration (Yd)	0.964	0.964	0.964
Barometric Pressure Measured ("Hg)	29.10	29.13	29.12
Stack Elevation Relative to Barometer (ft)	0	0	0
Barometric Pressure used in Calculations ("Hg)	29.10	29.13	29.12
Static Pressure ("H2O)	0.23	0.25	0.15
Average Square Root of ΔP	0.504	0.570	0.462
Average Stack Temperature (°F)	73.6	75.5	80.5
% CO2	6.30	6.10	6.26
% O2	11.20	11.33	11.29
% N2	82.50	82.57	82.45
Flue Gas Moisture - Saturation (%)	2.93	3.10	3.61
Flue Gas Moisture for Calculations (%)	2.93	3.10	3.61
Gas Molecular Weight (Wet) (g/g-mole)	29.12	29.08	29.04
Absolute Stack Pressure ("Hg)	29.12	29.15	29.13
Absolute Stack Temperature (°R)	533.6	535.5	540.5
Average Gas Velocity (ft/sec)	28.72	32.54	26.49
Avg Flow Rate (acfm)	3,045	3,450	2,809
Avg Flow Rate (scfm)	2,932	3,314	2,672
Avg Flow Rate (dscfm)	2,846	3,212	2,575