

MI-ROP-A4043-2019a and MON Testing  
§63.2493(a)(3)(ii)

Determination of Operating Limits to Comply with Renewable Operating  
Permit Requirements

Emissions Performance Test Plan

Ethylene Oxide Outlet Concentration

Thermal Heat-Recovery Oxidation Unit (THROX)

Dow Silicones Corporation (DSC), Midland, MI

Project Number: 60699646-193788

March 03, 2023

### Quality information

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Dow Silicones Corporation (DSC)  
Ethylene Oxide Compliance Test Report  
2512 THROX 60699646-193788

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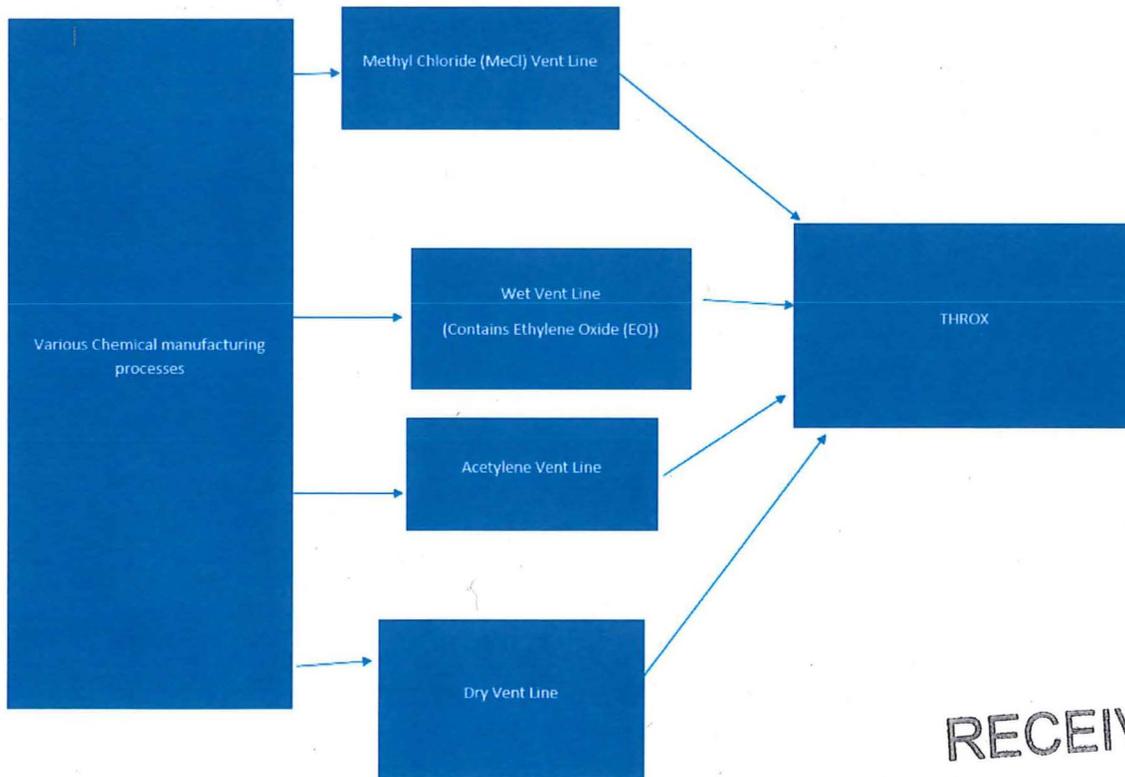
# 1. Introduction

AECOM Technical Services, Inc (AECOM) was contracted by Dow Silicones Corporation (DSC), Midland, MI to conduct an Ethylene Oxide (EO) emissions compliance test to measure total EO from thermal heat recovery oxidation unit located at the 2512 building, used to control emissions from multiple chemical production facilities at the site as shown in Figure 1.

AECOM of Midland, MI conducted the compliance emissions testing to meet the facility's MI-ROP-A4043-2019a and MON Testing §63.2493(a)(3)(ii) requirement to verify the EO emission from the 2512 thermal heat recovery oxidation unit (THROX).

The purpose of the compliance test was to demonstrate compliance with the MON Rule, specifically for Ethylene Oxide §63.2493(a)(3)(ii), and to demonstrate the EO concentration from the THROX exhaust stack is less than 1 ppm EO corrected to 3% O<sub>2</sub> while also reestablishing the minimum firebox temperature and maximum stack gas flow rate. The 2512 THROX is reestablishing the minimum firebox temperature and maximum stack gas flow rate due a new process vent, known as the 322 Acetylene line, connecting to the 2512 THROX. Although this new process vent does not contain EO, it does impact the operation of the THROX. Additionally, in the days leading up to the compliance test it was determined that the 321 process vent is no longer in EO service, however the 2504 process vents remain in EO service. Both the 321 process and 2504 process vents, which are connected to the wet vent header, were included in the initial EO compliance test, which was completed on August 24, 2022 and August 25, 2022

**Figure 1.** Michigan Operations Process vent connections to the 2512 THROX



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This report presents the results of the emissions compliance test. Testing of EO was accomplished by measuring on a continuous basis and was performed using US EPA Method 320, which involves a Fourier Transform Infrared (FTIR) analyzer and continuous measurements system optimized for the analysis of the requested compound along with supporting gas analyzers and manual exhaust gas flow rate measurements. The sampling system provided near real-time measurements.

The compliance test was conducted on February 21, 2023. Process operating data is presented in Appendix A. The compliance test established the method detection limit (MDL) for EO at the exhaust stack. The results indicate that the EO concentration from the THROX exhaust stack was less than 1 ppm EO corrected to 3% O<sub>2</sub>. Results are summarized in Table 2-1 both as wet emission concentrations (i.e., ppmvw) and corrected concentrations (i.e., ppmvd).

FTIR instrumentation was used to determine the gas stream characteristics for parameter measurements of analyte concentration and moisture. O<sub>2</sub> data (dry basis) was obtained from the plant process monitors. The use of the FTIR instrument for determination of moisture is an acceptable alternative to US EPA Method 4 gravimetric determinations. Field data and calculations are presented in **Appendix B**.

AECOM recorded EO using FTIR instrumentation. Emission rates for EO from the 2512 THROX exhaust stack were above quantitation limits most of the time and therefore for the few intermittent 1-minute measurement readings that were below detection limits, values were quantified based on the associated MDL. These are developed in the FTIR analytical report, attached as **Appendix C**. The total emission rates of EO, as calculated assuming the MDL was at the applicable detection limit, are reported as 263, 313, and 206 ppbww (i.e., 0.26, 0.31, and 0.21 ppmvw) for test runs 1, 2, and 3, respectively, from the 2512 THROX exhaust stack.

## 1.1 Responsible Parties

AECOM personnel from the Midland, Michigan and Austin, Texas 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 three one-hour test runs on the 2512 THROX exhaust stack.

### AECOM Personnel:

- 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. Mr. Edmister also coordinated with plant operations for the success of the field test effort.
- Quincy Crawford was the onsite representative from AECOM at the Midland facility and served as the Stack Testing Technical Lead for the field team.
- Ignacio Gallardo was the FTIR Senior Scientist and was responsible for the FTIR preparation, performing and overseeing FTIR sampling, FTIR data analysis, and report generation.
- Wayne Washburn provided senior technical review of the test data and report.

### Dow Plant Contacts:

- Brandon Krieger is the Process Focal Point of the Dow, MI THROX Facility. In this role, he had oversight for all aspects of the project and was kept informed of all activities and project milestones as the emissions test progressed. Mr. Krieger had final authority for all decisions concerning test program execution. His contact telephone number is (989)-948-0777.
- Becky Meyerholt is the Environmental Focal Point of the Dow Midland, MI THROX Facility and provided support as the Environmental, Health & Safety Focal Point for this test. The EH&S Focal Point was responsible for ensuring that all regulatory requirements and citations were reviewed and considered for the emissions test. All communication regarding EH&S concerns was completed through this role. Her contact telephone number is (989)-325-6820.

## 1.2 Test Chronology

Table 1-1. Summary of Sample Collection Times

Emissions Source	Test Run	Date/Time		
		Date	Run Start	Run End
2512 THROX	Run 1	02/21/2023	12:00	13:00
	Run 2		14:05	15:05
	Run 3		16:15	17:15

## 2. Results Summary

The results of the emissions compliance test are summarized in **Tables 2-1** below.

**Table 2-1. 2512 THROX – Emissions Results Summary**

Run Identification	Test R1	Test R2	Test R3	Average
Run Date	2/21/23	2/21/23	2/21/23	
Run Time	12:00-13:00	14:05-15:05	16:15-17:05	
<u>Exhaust Gas Conditions</u>				
Oxygen (% dry)	13.09	13.28	13.15	13.17
Flue Gas Moisture (%)	11.35	11.78	10.88	11.34
<u>Ethylene Oxide</u>				
Concentration, measured (ppmvw)	0.263	0.313	0.206	0.261
Concentration (ppmvd)	0.297	0.355	0.231	0.294
Concentration (ppmvd @3% Oxygen)	0.680	0.833	0.534	0.682

Process data for operation during the emissions compliance test are summarized in **Tables 2-2** below.

**Table 2-2. 2512 THROX – Process Data Summary**

Run Identification	Test R1	Test R2	Test R3	Average
Run Date	2/21/23	2/21/23	2/21/23	
Run Time	12:00-13:00	14:05-15:05	16:15-17:05	
<u>THROX Operating Conditions</u>				
Natural Gas Input (MBtu/hr)	35.34	35.28	35.01	35.21
CEMS O2 (%)	13.09	13.28	13.15	13.17
Exhaust Flow (SCFM)	18,970	19,088	18,704	18,921
Temp 1 (deg. F)	1,803	1,803	1,803	1,803
Temp 2 (ceg. F)	1,793	1,792	1,793	1,793
<u>Process Inputs (lb/hr)</u>				
2504 Flow	111.78	100.67	93.24	101.90
322 Flow	42.44	42.55	42.59	42.53
Dry Vent Flow	590.48	584.79	604.45	593.24
Wet Vent Flow	544.93	548.38	550.70	548.00
MeCl Vent Flow	152.38	92.56	87.29	110.74

## 3. Sampling and Analytical Procedures

### 3.1 Sample Time

The duration of each test run was approximately 60 minutes for a total of three (3) test runs (180 minutes) for the 2512 THROX exhaust stack.

### 3.2 FTIR Sample System Description

Exhaust gas stream samples were withdrawn from the source and transported to the AECOM mobile instrumental measurements system laboratory located at ground level. A stainless-steel sampling probe was inserted into each exhaust duct and used to collect sample gas. A heated Teflon sample line transported the sample gas from the sampling probe to the gas instrumental analyzers. The instrumental analyzers were kept at a stable temperature inside the AECOM mobile laboratory. At the mobile laboratory, the untreated (i.e., hot/wet) sample gas was routed to the FTIR instrument for analysis on a wet basis.

The FTIR extractive system was comprised of:

- stainless steel probe (~4 foot);
- stainless steel spiking "T";
- 100-ft heated (150°C) PFA-grade Teflon line; and
- MAX-IR, TOM and MKS 2030 FTIR spectrometers.

The source was measured with a Max Analytical FTIR analyzer (Model: MAX-IR with MCT detector and StarBoost Long pass filter, SN: 00495), equipped with a Thermal Oxidizer (TOM, used only for baseline collection, Model: MXH-TOM-LTO and SN: 00409) and a MKS2030 FTIR analyzer (Model 2030DBG2EKVS13T SN: 016102211) used only for measuring the tracer gas of the calibration standards. Both FTIR analyzers had a heated (150°C) fixed path sample cell, a flow regulating valve, a rotameter, and a sample pump. All connections between the analyzers above were heated at 150°C to match the spectral references used in both FTIR analyzers. A schematic of the FTIR sampling system is depicted in **Figure 3-1**.

Analyte monitoring consisted of continuously drawing a gas sample stream from the sample port through the sample probe, spiking tee, and heated extraction line, into the heated FTIR sample cell and out through the pump and exhaust line. Sample flow was continuous and maintained at approximately eight (8) liters per minute, by a diaphragm pump connected to the inlet of the FTIR cell. Since the pump provided samples slightly above ambient pressure to the FTIR cell, cell pressure was continuously recorded during measurement periods using a pressure sensor calibrated over a range of 0 – 900 Torr. These pressures were then used in the quantification of each spectrum.

#### 3.2.1 Analyte Spiking System

Per US EPA Method 320, analyte spiking must be performed to determine the effectiveness of the FTIR sampling and analytical systems in transporting and quantifying analytes. The aforementioned spiking "T", placed between the probe and the extraction line, enabled injection of the analyte gas standards directly into the extracted sample gas stream.

Method 320 stipulates an analyte spike equal to the native concentration, at no more than 10% of the total flow, be delivered through the entire sampling system. Spikes were performed using one cylinder, with EO as the calibration standard and SF6 as a tracer gas: 10.75 ppm EO with 10.25 ppm SF6. Precise volumes of the gas standard were delivered into the extracted stack gas (system recovery checks). Since the injected standard flow was low compared

to the extracted sample flow (maximum of 10% of total extracted flow), the sample gas matrix (including interferences) was not significantly changed.

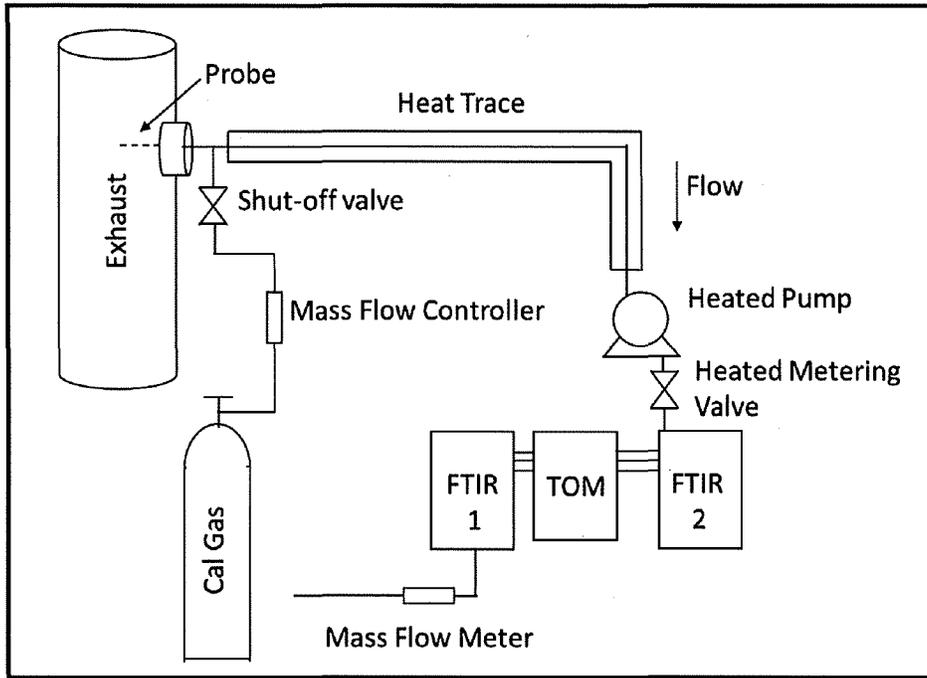


Figure 3-1. FTIR Sample System

Note: This figure shows the configuration for the test. The heated pump was placed between the sample line and the FTIR 2 (MKS) analyzer. The MAX-IR (FTIR 1), TOM and MKS2030 (FTIR 2) were all at 150°C.

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### FTIR Spectrum Analyses Method

An infrared spectrum can be collected and analyzed in approximately one second, but data are typically averaged over one- to five-minute integration periods to produce adequate signal-to-noise and ppb-level detection limits. For this testing, all run data signals were averaged for one minute. Shorter scan durations (10-second) were used for other QA, spikes, and mechanical response tests to better characterize system retention/response times, and interpolations were made to get sub-second estimation or minute averages.

An infrared spectrum analysis is performed by matching the features of an observed spectrum to those of reference standards. If more than one feature is present in the same region, a linear combination of references is used to match the compound features. The standards are scaled to match the observed band intensities; this scaling also matches the unknown concentrations.

The scaled references are added together to produce a composite that represents the best match with the sample. A classical least squares mathematical technique is used to match the reference standards' absorption profiles with those of the observed sample spectrum in specified spectral analysis regions. Compounds of interest and any known compounds expected to present spectral interference (water and carbon dioxide for this data set) are included in the analyte regions. The analysis method for this sampling was optimized for the analyte analysis during sampling and later refined to best fit the interferences within the analytes analysis regions. The method was challenged in the field by spiking the analyte standards close to the detection limits of the instrument.

### 3.2.2 Analyte Measurements

Analyte measurements and spiking were performed in accordance with the US EPA Method 320. To meet these objectives, the stack gas was monitored over one-hour runs. The following paragraphs discuss and present the sampling locations, pre- and post-test QA requirements and collection methods used in the study.

The FTIR measured the listed analytes on a hot wet basis. **Table 3-1** correlates the measured component to its corresponding analysis method.

**Table 3-2. Test Methods**

Compound Monitored	Test Methodology
Ethylene Oxide	EPA Method 320
Moisture	FTIR
O <sub>2</sub>	Dow provided

The FTIR and QA spiking systems are described in previous sections. Gas was continuously extracted from the center of the Stack exhaust pipe and delivered to the FTIR sample cell. A data point was obtained every minute during the runs and reflected the average of 60 individual spectra. Ten-second averages (10 averaged spectra) were collected during QA spiking. EO was not observed above the minimum detection limit (MDL) during the runs. The test results are presented in **Tables 2-1** through **2-3** and the QA spiking results are presented and discussed in Section 4.1.4.

## 4. QAQC

### 4.1 FTIR

As per US EPA Method 320, a significant amount of QA/QC activity had to be performed in order to demonstrate the effectiveness of the FTIR and sampling system to accurately monitor and transport analyte containing gas samples. These pre-test and post-test QC/QA checks verify that the FTIR was capable of monitoring analytes at acceptable (low enough) concentrations, and that the system operated in a stable fashion throughout each run as well as the entire study period. Similarly, EPA Method 3A and the AECOM internal QA/QC procedures call for rigorous checks and calibrations to ensure a high degree of data quality.

The sections below present detailed discussions of the QA/QC activities associated with FTIR sampling and analysis, as well as a data quality assessment. The overall conclusion of the QA/QC assessment is that the results of this test are of high quality and are appropriate for their intended use.

#### 4.1.1 FTIR QA/QC Results

This section describes the US EPA Method 320 QA/QC requirements and presents the results. The Method 320 pre-test QC requirements were done on site immediately before/after the testing and compared to the post-test results to ensure the FTIR system operated in a stable fashion throughout the entire sampling duration. These QA tests demonstrate that the FTIR and extractive system were capable of monitoring and transporting analytes at concentrations below those required to meet the test objectives.

#### 4.1.2 Pre-Test US EPA Method 320 QA/QC Verifications

A series of tests were performed to demonstrate analytes quantification accuracy, system response time, and other QC parameters shown in **Table 4.1** below. The results were obtained using nitrogen, a Calibration Transfer Standard (CTS) and a certified cylinder containing analytes. It should be noted that some of the Method 320 required checks have passing criteria that are user-defined based upon the test objectives (i.e., path length). All the results demonstrate an acceptable performance of the FTIR and sampling system for analytes detection.

**Table 4-1. US EPA Method 320 Pre-Test Requirements and Results**

Source	Procedure (Checks)	Result	Criteria	Comments
2512 THROX	Leak Check	<0.200 lpm	None Stated-ASTM D6348-12 specifies a less than 0.200 lpm flow through system while at terminal vacuum.	Evacuated cell and extraction line to terminal pressure (~>300 torr) and monitored residual flow via rotameter between cell and pump. Performed between each run of the 3-run test.
	System Zero	95% of CO <sub>2</sub> is gone after 55 seconds	None Stated-Should be much lower than source levels (~5% CO <sub>2</sub> )	Flow N <sub>2</sub> through sampling system, (keeping pump on) and monitor how fast CO <sub>2</sub> goes to zero.
	Path Length	10.01 of 10.21 ppm	± 5% accuracy	Direct injection of CTS to cell. Concentration w/in 5% demonstrated accurate Path Length
	System Reaction Time	10.2 of 10.7 ppm After 85 seconds	± 5% accuracy and a reasonable response time	Flow EO Standard through sampling system and monitor time to reach 95% of cylinder direct inject.
	System Mechanical Response Time	9.96 of 10.01 ppm After 60 seconds	± 5% accuracy and a reasonable response time.	Flow Ethylene Standard through sampling system and monitor time to reach 95% of CTS.

### 4.1.3 Pre- and Post-Test Data QC Results

The Method 320 requires a set of QC checks to be done prior to testing. A series of daily operational checks as well as more frequent (pre- and post-test) system accuracy and stability checks were performed per Method 320 procedures, thus ensuring high quality data. The following checks were done at a minimum of once per day:

1. A system noise-equivalent-absorbance (NEA) under a nitrogen atmosphere was measured. NEA is a measure of the system noise and a good indicator that the system is properly aligned and operating optimally. NEA is also used to determine a best-case minimum detectable concentration. All the NEA checks were acceptable for quantifying analytes below the regulatory limit.
2. System background spectra were collected by purging the cell with UHP nitrogen (which does not absorb infrared radiation). This profiles the IR detector's response absent of all compound absorption. The background, once generated, is ratioed to all subsequent sample spectra.
3. Resolution Checks. The resolution was checked before the first run and after the 3-run test by measuring the field width at half maximum (FWHM) of a water band when a nitrogen purge was applied before the testing. The resolution check was compared to the lab reference and expected resolution. The spectral resolution was at or near 0.5 cm<sup>-1</sup> throughout the test and the results listed in **Table 4-2** are acceptable for this test.
4. Line Position. Since each reference in this analysis method had been normalized (shifted) to a specific frequency, it was imperative that the sample spectra were also aligned at this frequency and maintained this alignment throughout the 3-run test. This was achieved by monitoring the position of an H<sub>2</sub>O absorption peak which was injected by leaking air and N<sub>2</sub> into the system. This line position was checked before the test, and it was compared to the lab reference and expected line position. The results listed in **Table 4-2** demonstrate acceptable performance.

**Table 4-2. Line Position and Resolution Checks**

Date	Description	Test Line Position (cm <sup>-1</sup> )	Set Line Position (cm <sup>-1</sup> )	Set Resolution (cm <sup>-1</sup> )	Actual Resolution (cm <sup>-1</sup> )
02/13//23	Pre-Test	3133.076	3133.068 ± 0.02	1.04±0.04	1.010
02/21//23	Post-Test	3133.081			1.038

Note: MAX-IR Passing criterion is line position stability within the range of ± 0.02cm<sup>-1</sup>. For resolution, criteria MAX-IR defines a range of ±0.04cm<sup>-1</sup>.

5. System Leak Checks. The FTIR, QA spiking system, and extractive system were leak checked before and after the testing. The probe was removed from the stack and capped. The sampling system was then evacuated to terminal pressure and the flow through the system monitored using a rotameter (0-1 lpm scale) positioned between the pump and the FTIR cell. In each case the rotameter ball went to dead (0 or seated). Typically, a leak rate of 4% of the sample system volume is acceptable. Since the leak rates were monitored as a function of flow, a criterion of < 0.200 lpm was considered passing (per Method 320). The results were all acceptable and are presented in **Table 4-3**.

**Table 4-3. Leak Check Results**

Source	Time	Leak Rate <sup>1</sup> (lpm)
2512 THROX	02/21//23 Pre-Test	<0.200
	02/21//23 Post-Test	<0.200

<sup>1</sup> As read from 1 lpm rotameter on exhaust of sampling system. In both instances the rotameter reading was 0.

### 4.1.4 QA System Recovery Spiking

As part of quality assurance procedures of the Method 320, a total of 12 QA spikes of the target analyte must be performed prior and after testing, for each source. These checks challenge the analysis method for accuracy of each analyte quantification while simultaneously verifying that the extractive system and analyzer are unreactive with

analytes. Successful spiking is also an indication of a good analyte direct-inject measurement. The spiking procedure for the system recovery that was done separately for all analysis described in detail in the Method 320 and summarized for Ethylene Oxide (EO) below.

A test, in which the EO gas standard, was introduced directly into the heated sample cell (bypassing the extractive assembly), was performed. In addition to EO, the gas cylinder also contained a spectroscopic tracer (a broad, strong IR absorber which behaves linearly over a broad range of concentrations) to calculate dilution factors. Sulfur hexafluoride (SF6) was the tracer used in the system recovery checks. After the cell was sufficiently purged with the EO/SF6 standard, the analysis method returned values for SF6 and EO that were then compared to the certified cylinder values (SF6<sub>cylinder</sub> & EO<sub>cylinder</sub>). Upon direct injection of the certified analyte / SF6 standard into the FTIR sample cell, the SF6 and the analyte concentrations read from the FTIR compared within the acceptable criterion of 10% for reactive gases to the cylinder certified values as listed in **Table 4-4** below.

**Table 4-4. FTIR QA Analytical Direct Injections**

Analyte	DI Measurement Result (ppm)	Certified Concentration (ppm)	Relative % Difference	Pass/Fail	Cylinder
Ethylene	10.01	10.21	2.0	Pass	CTS
Methane	10.124	9.743	3.9	Pass	
Ethylene Oxide	10.75	10.75	0.0	Pass	Analyte
SF6	10.51	10.25	2.5	Pass	

Note: SF6 is the tracer gas in the Ethylene Oxide Cylinder and was measured with the MKS2030 FTIR

The gas standard was then injected into the spiking "T" downstream of the probe as the stack effluent was drawn through the FTIR system. The EO/ SF6 gas standard injection flow was maintained at a constant rate using a flow controlling needle valve. After the FTIR cell was sufficiently purged with the gas standard/stack effluent mix (stable for ~5 minutes), the analysis method returned a value (SF6<sub>sample</sub>) which represents the concentration of SF6 diluted by the stack effluent. From the SF6 concentrations the dilution factor (DF) can be determined by dividing the SF6<sub>sample</sub> by the SF6<sub>cylinder</sub>.

The expected concentration of EO (EO<sub>Theoretical</sub>) is the sum of diluted cylinder concentration (spiked) and the native stack concentration (also diluted by the injected spike) and was calculated as follows:

$$EO_{Theoretical} = \left( \frac{R22_{sample}}{R22_{cylinder}} \right) (EO_{cylinder}) + \left[ 1 - \left( \frac{R22_{sample}}{R22_{cylinder}} \right) \right] (EO_{stack})$$

Where:

- EO<sub>Theoretical</sub> = Theoretical EO concentration (ppm);
- SF6<sub>sample</sub> = SF6 concentration (ppm) as seen by the FTIR during QA spiking;
- SF6<sub>cylinder</sub> = SF6 concentration observed during the direct inject;
- EO<sub>cylinder</sub> = EO concentration observed during the direct inject; and
- EO<sub>stack</sub> = The native EO concentration (ppm) of the stack during stable conditions.

The criterion for a successful recovery, per the Method 320, is a measured concentration within 0.7-1.3 times the calculated theoretical concentration. This test demonstrated recoveries within the criterion, ranging from 72 – 125%. **Table 4-5** summarizes all the recovery spikes done as part of the test.

**Table 4-5. FTIR QA Analytical Spiking Recoveries**

Source	Analyte	Spike #	Native Analyte	Tracer Spike (ppmv)	Analyte Spike (ppmv)	Expected Spike (ppmv)	Percent Recovery (%)	Comment
<b>2512 THROX</b>	<b>Ethylene Oxide Pre-test</b>	#1	0.258	0.358	0.540	0.615	88%	Pass
		#2	0.258	0.433	0.609	0.689	89%	Pass
		#3	0.258	0.434	0.570	0.690	83%	Pass
		#4	0.258	0.395	0.489	0.651	75%	Pass
		#5	0.258	0.187	0.343	0.444	77%	Pass
		#6	0.258	0.310	0.448	0.566	79%	Pass
		#7	0.258	0.127	0.365	0.384	95%	Pass
		#8	0.258	0.122	0.413	0.380	109%	Pass
		#9	0.258	0.311	0.408	0.568	72%	Pass
		#10	0.258	0.409	0.590	0.665	89%	Pass
		#11	0.258	0.467	0.901	0.723	125%	Pass
		#12	0.258	0.590	0.787	0.845	93%	Pass
	<b>Ethylene Oxide Post-test</b>	#1	0.251	0.918	0.929	1.164	80%	Pass
		#2	0.251	0.651	0.667	0.898	74%	Pass
		#3	0.251	0.356	0.550	0.605	91%	Pass
		#4	0.251	0.266	0.430	0.515	84%	Pass
		#5	0.251	0.250	0.407	0.499	82%	Pass
		#6	0.251	0.204	0.407	0.454	90%	Pass
		#7	0.251	0.064	0.288	0.315	91%	Pass
		#8	0.251	0.061	0.253	0.311	81%	Pass
		#9	0.251	0.166	0.316	0.416	76%	Pass
		#10	0.251	0.458	0.802	0.706	114%	Pass
		#11	0.251	0.734	1.230	0.981	125%	Pass
		#12	0.251	0.939	1.368	1.185	115%	Pass

Note: Results are on a wet basis, uncorrected for O<sub>2</sub> concentration.

#### 4.1.4.1 QC System Stability and System Zero

Method 320 requires a system stability check before testing, between each run and at the end of the day or the end of the test per each source. This was accomplished by direct injection of the calibration transfer standard (CTS) into the FTIR cell. The quantified concentrations of the CTS component, (C<sub>2</sub>H<sub>4</sub>) for this test, were directly compared. If the quantified concentration varies by more than ±5% from the certified value, it is indicative of an unstable system. All the CTS stability checks were within ±5% of the certified concentration and are presented in **Table 4-6**.

**Table 4-6. Calibration Transfer Standard Stability Results**

Source	Description	Ethylene Result (ppm)	Direct Inject Ethylene Concentration (ppm)	Relative % Difference	Comments
2512 THROX	Pre-Test	9.92	10.01	0.8	Pass
	Post-Run1	9.71		2.9	Pass
	Post-Run2	9.83		1.7	Pass
	Post-Run3	10.11		1.2	Pass

A system zero analysis was also performed by injecting a high enough flow of N<sub>2</sub> through the calibration line, into the spiking "T" such that it flooded the "T" and probe assembly. The N<sub>2</sub> was then pulled through the system via pump. The time required to purge the system to <5% of native stack concentrations was approximately one minute. Similarly, the time it took to achieve 95% of the native stack concentration levels once the N<sub>2</sub> was turned off was approximately one minute. See **Table 4-2**.

#### 4.1.5 Evaluation of Completeness

Completeness is a measure of the extent to which the results from a measurement effort fulfills objectives for the amount of data required. For this program, completeness is defined in terms of the number of valid sample results collected compared with the number planned. All samples planned and all analyses planned were performed. No results were invalidated based on a data quality assessment.

#### 4.1.6 Deviations from the US EPA Method 320 Procedures

No deviations from reported Methods.

#### 4.1.7 Sample Handling

Individual FTIR sample spectra were electronically stored in interferogram format on the system hard drive and backed-up onto various storage media. Each spectrum is time stamped and has the path length, pressure, and temperature it was collected at stored with it. All support spectra (N<sub>2</sub>, background, QA etc.) were also stored in various formats. Electronic copies of all spectra have been stored on USB flash drives.

#### 4.1.8 Calibration

Calibration of the FTIR and sampling system were completed per the EPA Method 320 requirements and QA/QC procedures. The FTIR references used to build the analysis method were developed by the manufacturer of the FTIR and implemented by AECOM scientists. The FTIR instrument uses the above method to predict and simulate the transmission and emission of light in the atmosphere. This analysis is based on a set of analyte references generated from multiple certified gas cylinders. These analyte references have been used reliably on many occasions.