

**QUALITY ASSURANCE PROJECT PLAN**  
**ETHYLENE OXIDE IN AMBIENT AIR ANALYSIS USING**  
**US EPA METHOD 327 – MODIFIED (ENTHALPY STANDARD OPERATING**  
**PROCEDURE NO. TM327, REVISION 1) AND COLLECTED USING**  
**SOP – METHOD 327 CANISTER SAMPLING (MAQS, APPENDIX B)**  
**AT THE INSTITUTE, WEST VIRGINIA SITE**

January 30, 2025

Prepared for:

**THE DOW CHEMICAL COMPANY**  
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Institute, WV 25112

Prepared by:

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Project No. MAQ - QAPP Preparation

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## List of Abbreviations and Acronyms

<	less than
≤	less than or equal to
>	greater than
≥	greater than or equal to
%D	percent difference
% R	percent recovery
%RSD	percent relative standard deviation
CCV	continuing calibration verification
COC	Chain-of-Custody
DAQ	Division of Air Quality
DL	detection limit
DQO	data quality objective
EDD	electronic data deliverable
EtO	ethylene oxide
FSP	Field Sampling Plan
GC/MS SIM	gas chromatography/mass spectrometry with select ion monitoring
ICAL	initial calibration
ICV	initial calibration verification
LCS	laboratory control sample
MAQS	Montrose Air Quality Services, LLC
MDL	method detection limit
MPC	measurement performance criteria
MS	matrix spike
MSD	matrix spike duplicate
NA	not applicable
oz	ounce
PARCC	precision, accuracy, representativeness, completeness, and comparability
pptv	parts per trillion volume
QA	quality assurance
QC	quality control
QL	quantitation limit
QAPP	Quality Assurance Project Plan
RL	reporting limit
RPD	relative percent difference
RSD	relative standard deviation
SOP	standard operating procedure
TBD	to be determined
UCC	United Carbide Corporation
US EPA	U.S. Environmental Protection Agency
WV DEP	West Virginia Department of Environmental Protection

## 1.0 INTRODUCTION

Environmental Standards, Inc. (Environmental Standards) has prepared this Quality Assurance Project Plan (QAPP) to describe the quality assurance/quality control (QA/QC) aspects of the sampling and analysis of ambient air to assess the performance of a modified Method 327, to the extent current technology allows, to assess ethylene oxide concentration consistency between multiple canisters at the same location and to compare the concentrations found on the site perimeter of a known ethylene oxide-emitting source *versus* a rural background location with no known ethylene oxide sources. The data generated will be used by Dow Chemical Company (Dow), the West Virginia Department of Environmental Protection (WV DEP), and/or the public for the purpose of commenting on any related future rulemaking. Specifically, sample collection, processing, and shipping activities to be conducted for this study are detailed herein. In addition, laboratory analytical procedures and data quality review requirements are described in this QAPP.

### Background

Dow previously contracted Montrose Air Quality Services, LLC (MAQS) to conduct fenceline fugitive emissions testing in 2022 in response to a US EPA Information Collection Request (ICR) at facilities located in South Charleston and Institute, West Virginia. The testing method included US EPA Method TO-15A to collect and measure fenceline samples for ethylene oxide. The Collaborative Agreement between Dow and WV DEP requires fenceline monitoring, such as will be conducted per this QAPP. As a direct result of the past sampling effort, Dow and the West Virginia Department of Environmental Protection (WV DEP) has commissioned MAQS and Enthalpy Analytical, LLC (Enthalpy) to generate data to meet the goal of obtaining data for United Carbide Corporation (UCC), Altivia Ketones and Additives LLC of Houston (Altivia), WV DEP, and the public to make informed comments on the assumed inclusion of Method 327 in the revised 40 CFR Part 63 Subpart Polyether Polyols Production (PEPO) Maximum Achievable Control Technology (MACT). UCC (Dow) and Altivia agreed to undertake a voluntary project to test for ethylene oxide at known emitting sources in furtherance of this goal.

MAQS and its affiliates will provide the trained staff and the necessary equipment to measure ambient air as outlined in this QAPP.

## 2.0 REFERENCES

Quality Assurance Procedure Plan Fenceline TO-15 Sampling Dow Union Carbide Corporation South Charleston & Institute, West Virginia. GC025AS-018372-PP-304R1. Montrose Air Quality Services, LLC. Deer Park, TX, August 2022.

Intergovernmental Data Quality Task Force. Uniform Federal Policy for Implementing Environmental Quality Systems Evaluating, Assessing, and Documenting Environmental Data Collection/Use and Technology Programs. Intergovernmental Task Force, March 2012.

US EPA Guidance on Systematic Planning Using the Data Quality Objectives Process. US EPA QA/G-4. Office of Environmental Information. Washington, DC, February 2006.

US EPA (US Environmental Protection Agency). EPA Requirements for Quality Assurance Project Plans. EPA QA/R-5. Office of Environmental Information. Washington, DC, March 2001.

US EPA. EPA Guidance for Quality Assurance Project Plans. EPA QA/G-5. Office of Environmental Information. Washington, DC, December 2002.

US EPA. Guide to Writing Quality Assurance Project Plans for Ambient Air Monitoring Networks. EPA-454/B-18-006. Office of Air Quality Planning and Standards. Research Triangle Park, NC, August 2018.

US EPA. Best Practices for Review and Validation of Ambient Air Monitoring Data. EPA-454/B-21-007. Office of Air Quality Planning and Standards. Research Triangle Park, NC, August 2021.

US EPA. US EPA Guidance on Environmental Data Verification and Data Validation. US EPA QA/G-8. Washington, DC, November 2002.

US EPA. US EPA Guidance for Data Quality Assessment, Practice Methods for Data Analysis. US EPA QA/G-9. Washington, DC, July 2000.

US EPA. Data Quality Assessment: A Reviewer's Guide. US EPA QA/G-9R. Washington, DC, February 2006.

US EPA. Data Quality Assessment: Statistical Methods for Practitioners. US EPA QA/G-9S. Washington, DC, February 2006.

US EPA. Method 327 – Fugitive and Area Source Measurement of Selected Volatile Organic Hazardous Air Pollutants Using Specially Prepared Canisters. Emissions Measurement Center, March 2023.

US EPA. National Functional Guidelines for Organic Superfund Methods Data Review. EPA 540-R-20-005. Office of Superfund Remediation and Technology Innovation, November 2020.

## QAPP Worksheet #1 and #2: Title and Approval Page

(UFP-QAPP Manual Section 2.1)  
(USEPA 2106-G-05 Section 2.2.1)

**Site Name/Project Name:** Ethylene Oxide in Ambient Air Analysis Using US EPA Method 327 – Modified (Enthalpy Standard Operating Procedure No. TM327, Revision 1) and Collected Using SOP – Method 327 Canister Sampling (MAQS, Appendix B) at the Institute, WV Site

**Site Location:** Institute, WV and Buffalo, WV

**Site Number/Code:** Location 6 and Background

**Contractor Name:** Environmental Standards, Inc. (Environmental Standards)

**Contractor Number:** NA

**Work Assignment Number:** NA

**Lead Organization:** MAQS

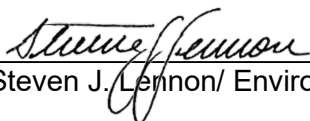
### MAQS Project Manager:



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Jenna Granstra/ MAQS

### Project Quality Assurance Officer:



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Steven J. Lennon/ Environmental Standards

### Stakeholder:

Russell A. Wozniak

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Russell A Wozniak/ Dow



### **Identify guidance used to prepare Quality Assurance Project Plan (QAPP):**

- Uniform Federal Policy for Implementing Environmental Quality Systems Evaluating, Assessing, and Documenting Environmental Data Collection/Use and Technology Programs. Intergovernmental Task Force, March 2012.
- US EPA Requirements for Quality Assurance Project Plans. EPA QA/R-5. Office of Environmental Information. Washington, DC, March 2001.
- US EPA Guidance for Quality Assurance Project Plans. EPA QA/G-5. Office of Environmental Information. Washington, DC, December 2002.
- US EPA Guidance on Systematic Planning Using the Data Quality Objectives Process. US EPA QA/G-4. Office of Environmental Information. Washington, DC, February 2006.
- US EPA Guidance on Environmental Data Verification and Data Validation. US EPA QA/G-8. Washington, DC, November 2002.
- US EPA Guidance for Data Quality Assessment, Practice Methods for Data Analysis. US EPA QA/G-9. Washington, DC, July 2000.
- US EPA Data Quality Assessment: A Reviewer's Guide. US EPA QA/G-9R. Washington, DC, February 2006.

**Identify regulatory program:** NA, voluntary implementation.

**QAPP is project-specific:** Yes.

**Identify approval entity:** WV DEP will review and comment.

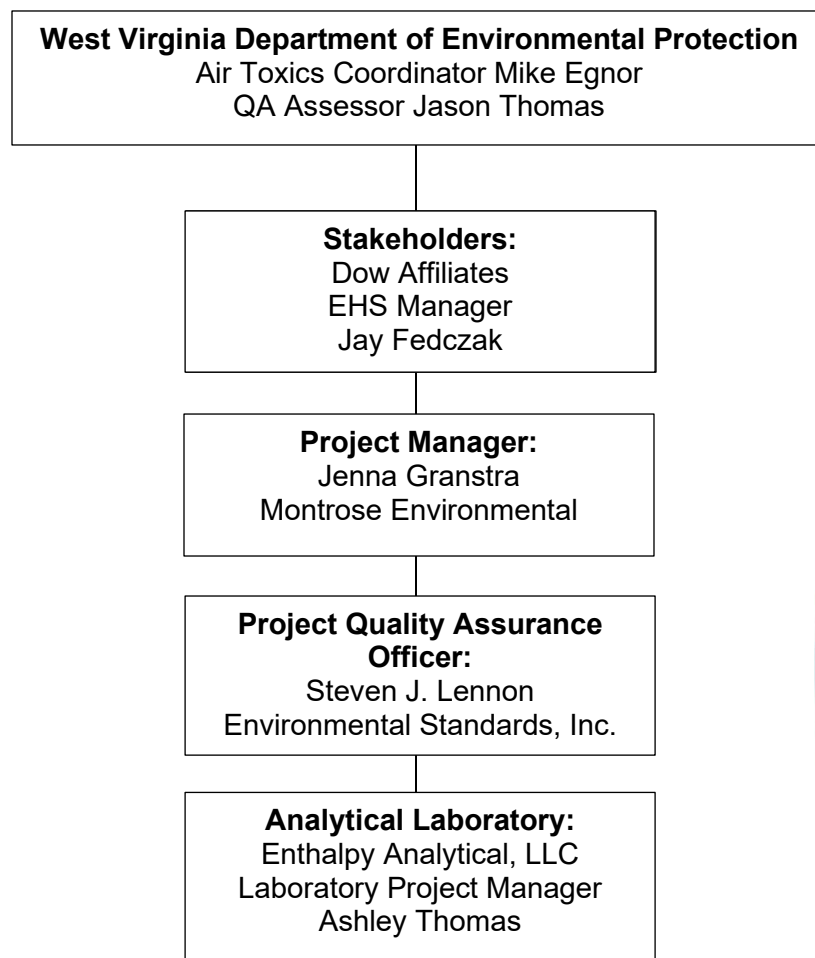
**List organization partners (stakeholders) and connection with lead organization:**

Pilot Study participants:

1. WV DEP
2. Union Carbide Corporation (UCC, Dow Affiliates)
3. Altivia Ketones and Additives LLC of Houston (Altivia)

## QAPP Worksheet #3 and #5: Current Project Organization and QAPP Distribution

(UFP-QAPP Manual Section 2.3 and 2.4)  
(USEPA 2106-G-05 Section 2.2.3 and 2.2.4)





The following people will receive a copy of the final QAPP, subsequent QAPP revisions, addenda, and amendments.

<b>QAPP Recipients</b>	<b>Title/Team</b>	<b>Organization</b>	<b>Telephone Number</b>	<b>E-mail Address</b>
Mr. Mike Egnor, P.E.	Air Toxics Coordinator	WV DEP DAQ	(304) 414-1255	michael.egnor@wv.gov
Mr. Jason Thomas	QA Assessor	WV DEP DAQ	(304) 414-1275	jason.thomas@wv.gov
Mr. Russell Wozniak	US Air Advocacy Leader	Union Carbide Corporation (Dow Affiliates)	(361) 571-5420 (M)	wozniara@dow.com
Ms. Jenna Granstra	Gulf Coast Operations Manager	MAQS	(507) 822-5661	jgranstra@montrose-env.com
Steven J. Lennon	Project Quality Assurance Officer	Environmental Standards, Inc.	(484) 808-2759 extension 112325 (O) (717) 538-2423 (M)	slennon@envstd.com
Ashley Thomas	Laboratory Project Manager	Enthalpy Analytical	(919) 850-4392 Extension 12202	ashley.thomas@enthalpy.com

(O) – Office

(M) – Mobile

## QAPP Worksheet #4, #7, and #8: Personnel Qualifications and Sign-Off Sheet

(UFP-QAPP Manual Sections 2.3.2 – 2.3.4)  
(USEPA 2106-G-05 Sections 2.2.1 and 2.2.7)

**Organization:** MAQS Environmental

Name	Project Title/Role	Education/Experience	Specialized Training/Certifications	Signature/Date
Jenna Granstra	Lead Organization Project Manager	<p>B.S. Meteorology, Minor GIS M.S. Geography</p> <p>Ms. Granstra has over 10 years of experience in ambient air monitoring, specifically fenceline air monitoring. Ms. Granstra has led research projects and regulatory compliance projects over the years using various technologies.</p>	None	

**Organization:** Environmental Standards, Inc.

Name	Project Title/Role	Education/Experience	Specialized Training/Certifications	Signature/Date
Steven J. Lennon	Project Quality Assurance Officer/Project Quality Assurance Chemist III	<p>B.S. Environmental Biology/Marine Biology</p> <p>Mr. Lennon has over 28 years of QA/QC experience. His experience includes the planning, development, and execution of environmental sampling and analytical programs. He has extensive experience with US EPA organic and inorganic analytical methodology and analytical data validation and has overseen the validation efforts for dozens of projects. He has evaluated air sampling trains, taken ambient air samples in canisters, and evaluated air sampling data a multitude of times.</p>	None	
Various	Quality Assurance Chemists/Data Validators	Various	Internal Training	NA

**Organization:** Enthalpy Analytical, LLC

Name	Project Title/Role	Education/Experience	Specialized Training/Certifications	Signature/Date
Ashley Thomas	Laboratory Project Manager  The Laboratory Project Manager acts as the primary point of contact at Enthalpy Analytical, LLC Deer Park, TX facility for the Project QA Coordinator to communicate and resolve sampling, receipt, analysis, and storage issues.	B.S. Biology B.S. Environmental Science  Mrs. Thomas is a Senior Project Manager based at Enthalpy Analytical's Durham, North Carolina laboratory. She has over 20 years' experience in various laboratory settings, with over 10 years serving in the client facing, Project management role.	Active member of Air and Waste Management Association. (2016-current)	
Various	Laboratory Chemists	Various	Internal training program with yearly demonstration of capability	NA

## QAPP Worksheet #6: Communication Pathways

(UFP-QAPP Manual Section 2.4.2)

(USEPA 2106-G-05 Section 2.2.4)

Communication Drivers	Responsible Entity	Name	Contact Information	Procedure (Timing, Pathways, etc.)
Communications concerning the timing of sample canister setups and pickups to be communicated to the landowner of the background sampling site.	WV DEP DAQ	Mike Egnor	(304) 414-1255 <a href="mailto:michael.egnor@wv.gov">michael.egnor@wv.gov</a>	Communicate the sampling setup and pickup dates and times with the background sampling location landowner via phone or email.
Decisions requiring client input or direction; information to be communicated to WV DEP and Dow Affiliates	Lead Organization Project Manager	Jenna Granstra	(507) 822-5661 <a href="mailto:jgranstra@montrose-env.com">jgranstra@montrose-env.com</a>	Maintain project updates as needed with the QA and stakeholders via phone or email. Communicate and adjust project schedules, QAPP and project deviations/addenda, and potential impacts to project DQOs.
Communication with WV DEP and Dow Affiliates	QA Officer	Steven J. Lennon	(484) 808-2759 x110235 <a href="mailto:slennon@envstd.com">slennon@envstd.com</a>	Communicate project updates as needed with the client via phone or email. Communicate project schedules, QAPP and Field Sampling Plan (FSP) deviations/addenda, and potential impacts to project DQOs.
Issues with sample submission coordination, schedule, data deliverable issues, technical issues; and minor QAPP deviations	QA Officer	Steven J. Lennon	(484) 808-2759 x110235 <a href="mailto:slennon@envstd.com">slennon@envstd.com</a>	Communicate with Project Manager and Laboratory Project Manager as needed via phone or email. Communicate minor QAPP deviations to the Project Manager.
Analytical data validation issues	Data Validation Chemist	To be assigned	To be assigned	Communicate with Project QA Officer as needed via phone or email.

Communication Drivers	Responsible Entity	Name	Contact Information	Procedure (Timing, Pathways, etc.)
Laboratory data quality issues, data deliverable issues, reporting issues, and QAPP deviations	Laboratory Project Manager	Ms. Ashley Thomas	(919) 850-4392 x12202 <a href="mailto:ashley.thomas@enthalpy.com">ashley.thomas@enthalpy.com</a>	Communicate with the QA Officer as needed via phone or email.

## QAPP Worksheet #9: Project Planning Session Summary

(UFP-QAPP Manual Section 2.5.1 and Figures 9 to 12)  
(USEPA 2106-G-05 Section 2.2.5)

### Planning sessions:

Dates	Activity/Purpose	Summary/Action Items	Location
December 6, 2024 December 30, 2024	Conference calls and emails between Jenna Granstra, Ashley Thomas and Environmental Standards, Inc.	Summary of Conference Calls: <ul style="list-style-type: none"> <li>Discussed project background, analyses, and objectives.</li> <li>Discussed quantities and types of samples.</li> <li>Discussed sample collection and logistics.</li> <li>Discussed promulgated analytical method <i>versus</i> laboratory analytical method.</li> <li>Discussed QC sample types and frequency.</li> <li>Discussed sample identification and anonymity.</li> <li>Discussed sample handling, shipping, and archiving.</li> <li>Discussed canister sampling SOP.</li> <li>Discussed laboratory login SOP.</li> </ul>	Conference bridge.
January 8, 2025 January 24, 2025	Conference calls and emails between Jenna Granstra, Ashley Thomas, and Environmental Standards, Inc.	Summary of Conference Calls: <ul style="list-style-type: none"> <li>Discussed changes in sample collection and logistics.</li> <li>Discussed Laboratory canister cleanliness certification SOP.</li> <li>Discussed changes to laboratory procedures and equipment.</li> <li>Discussed proposed changes from the WVDEP.</li> <li>Discussed deviations from analytical method and the laboratory analytical SOP.</li> <li>Discussed proposed sampling event dates.</li> <li>Discussed QAPP title.</li> </ul>	Conference bridge.
January 29, 2025	Conference calls and emails between Ashley Thomas, Russ Wozniak and Environmental Standards, Inc.	Summary of Conference Calls: <ul style="list-style-type: none"> <li>Discussed all aspects of the QAPP.</li> <li>Discussed changes to laboratory deviations table and the addition of a deviation.</li> </ul>	Conference bridge.



## **QAPP Worksheet #10: Conceptual Site Model**

**(UFP-QAPP Manual Section 2.5.2)**

**(USEPA 2106-G-05 Section 2.2.5)**

### **The problem to be addressed by the project is as follows:**

The objectives of this study are to collect ambient air samples and analyze them for ethylene oxide by Method 327 to the extent current technology allows to assess ethylene oxide concentration consistency between multiple canisters at the same location and to compare the ethylene oxide concentrations found on the site perimeter of a known ethylene oxide-emitting source vs. a rural background location with no known ethylene oxide sources. The data generated will be used for the purpose of commenting on any related future rulemaking.

### **The proposed sampling consists of the following:**

Silonite™ coated spherical canister samples of ambient air will be collected by MAQS company personnel or contract personnel as follows:

- Six canisters of ambient air will be collected for each sampling event at Location #6\* (from the ICR Section 114 work) at the Institute, WV site to evaluate the variability of the analytical method among the six canisters. These canisters will be located side-by-side with approximately 2 feet distance between each canister. (\* - Please note, the WV DEP has identified this location as #15 in their documentation).
- Two canisters of ambient air will be collected for each sampling event at a background location, which is about 15 miles north of the site. This location is approximately 3 miles east of Buffalo, West Virginia. The canisters collected at the background site for each sampling round will be designated as a Remote Location sample and a Field Duplicate sample.
- Two unopened canisters for each sampling event will travel to the Location #6 sampling location at Institute, West Virginia site and be designated as a Field Blank and a Field Spike.
- Three rounds of sampling will be conducted tentatively January 31 - February 1; February 3 - February 4; and February 6 - February 7, 2025, pending QAPP approval.
- To reduce the number of components which could affect the results, no automatic timers will be used in the study.
- Three rounds of samples, 3 days apart, eight canisters at Location #6 and two canisters at a remote location.

A total of 30 unique samples of ambient air will be collected for analysis of ethylene oxide by a modified version of US EPA Method 327 as on the table shown in Worksheet #18.

## QAPP Worksheet #11: Project/Data Quality Objectives

(UFP-QAPP Manual Section 2.6.1)

(USEPA 2106-G-05 Section 2.2.6)

Data quality objectives (DQOs) are qualitative and quantitative statements developed to specify the quality of data from data collection activities to support the project. The DQOs describe what data are needed, why the data are needed, and how the data will be used to address the problem being investigated. DQOs also establish numeric limits for the data to allow the data user (or reviewers) to determine whether data collected are of sufficient quality for use in their intended application.

This QAPP supports tasks associated with the following activities:

- Ambient air sampling.
- Analytical analysis of the ambient samples.

### Who will use the data?

The data generated will be used by Dow for the purpose of commenting on any related future rulemaking for ethylene oxide emissions and US EPA Method 327.

### Intended use of the data:

The project objectives are to collect sufficient quality and quantity of data to assess ethylene oxide concentration consistency between multiple canisters at the same location and to compare the concentrations found on the site perimeter of a known ethylene oxide-emitting source *versus* a rural background location with no known ethylene oxide sources. The data generated will be used by Dow, WV DEP, and/or the public for the purpose of commenting on any related future rulemaking.

### Data use data quality objectives:

The DQOs are intended to provide a known level of confidence to utilize the generated analytical data to support its use for evaluation of presence of absence of ethylene oxide and other evaluations to be performed with the analytical data. Analyte-specific methods are used for the generation of the data. Worksheets #12 and #28 and the laboratory standard operating procedures (SOPs) in Attachment A provide DQOs for analytical methods.

The overall DQO is to generate greater than 90% usable analytical data and to collect greater than 90% of the planned samples.

**Type of data that are needed (matrix, target analytes, analytical groups, field screening, on-site analytical, off-site laboratory techniques, sampling techniques):**

Ambient air is anticipated to be collected during the sampling event. No additional matrices are anticipated for the investigative samples. The ambient air samples will be collected using uniform and consistent procedures identified in Worksheet #21 of this QAPP. The field collection procedures d

The field collection procedures deviate from US EPA Method 327 and the WV DEP DAQ EtO Passive Sampling SOP collection procedures in the following manners:

Description of planned field collection procedure deviation	Method Deviation (Y/N)	WVDEP DAQ Deviation (Y/N)
The Method 327, Section 8.7.3.1 requires all sampling locations to initiate sampling within 60 minutes of each other. Due to project sampling logistics such as available field sampling personnel, the number of canisters being deployed at each location, and the distance between the two sampling locations, this requirement may not be achieved by the field sampling team.	Y	N
The WVDEP DAQ SOP Collection of Ethylene Oxide Samples Using Passive Sampling Technique, Revision 1.0, Section 6.2.2.2 requires when ambient sampling temperatures are below 0° Celsius that the canister bellows valve is closed fully, and the canister assembly (canister with vacuum controller attached) is moved indoors to let stand for at least 2 hours at room temperature. Then the final canister pressure is recorded, and the canister is disconnected from the sampling system. The field sampling team will endeavor to schedule sample collection on days with forecasted temperatures above 0° Celsius; however, temperatures may drop below this. When temperatures drop below 0° Celsius, the suggested step will not be performed immediately since it is not practical due to the number of canisters being deployed for each sampling event, the locations where they are being deployed. However, once the samples have been received by the laboratory, the samples will be allowed to reach room temperature for the suggested 2 hours.	N	Y

The ambient air samples will be shipped to off-site laboratories for chemical analysis.

Anticipated analytical methods for samples consist of:

- Ethylene Oxide by US EPA Method 327 - Modified

The laboratory is performing a modified version of the method and deviates from US EPA Method 327 and from laboratory SOP EADP-01 in the following ways:

Description of planned laboratory deviation	Method Deviation (Y/N)	Lab SOP Deviation (Y/N)
The Laboratory is not using Method 327 certified canisters as required by SOP TM327, Section 5.4 and Method 327, Section 8.3. The laboratory does not currently perform the canister zero air verification check and the canister know-standard challenge as required every 18 months. The laboratory performs an annual 48hr leak check and a cleaning blank check on every canister after cleaning.	Y	Y
The laboratory is not using Method 327 certified Mechanical Flow Control Devices (MFCs) as required by SOP TM327, Section 5.5 and Method 327, Section 8.1. The laboratory does not currently perform the MCFD flow control verification test, the sampling device zero air verification check and the sampling device know-standard challenge as required every 12 months. The laboratory performs a flow control flow check prior to and after field deployment and a sampling device leak check immediately before sampling.	Y	Y
The laboratory may use working standards beyond 7 days of preparation. SOP TM327, Section 6.3.4 states that <i>"Expiration dates for working standards are assigned as no longer than 7 days after the date of preparation."</i> Method 327 Section 10.4 states that <i>"Standards prepared in canisters at ambient laboratory conditions must be stored in locations that are free of potential contaminants for up to 7 days."</i>	Y	Y
The laboratory will prepare all working standards using gastight syringes to dispense stock standards and diluent gas addition will be measured barometrically as in Method 327, Section 10.3.2. The method does not explicitly allow or disallow for use of syringes to add standards to a canister.	Y	N

The laboratory may not use a calibrated hygrometer to verify that the relative humidity (RH) of the purge gas is >50%. Method 327, Section 8.4.1.2. specifically states <i>“Humidify the purge gas to &gt;50% RH and measure the humidity by placing a calibrated hygrometer probe in the humidified gas stream.”</i>	Y	N
It is possible that not all sampling canisters used were pressurized with humidified hydrocarbon-free (HCF) zero air prior to cleanliness analysis, as required in Method 327, Section 8.5.1. Canisters that were not pressurized with humidified HCF zero air were pressurized with humidified ultra-high purity (UHP) nitrogen. Current laboratory documentation is insufficient to determine which sampling canisters were or were not pressurized with the required humidified HCF zero air.	Y	N

The samples will be shipped or sent via courier service to the off-site analytical laboratory for analysis. Note that the work will not include the generation of field analytical data.

The target analyte list is provided in QAPP Worksheet #15. QAPP Worksheet #15 also includes the Project Laboratory quantitation limits (QLs) and method detection limits (MDLs), as applicable. Project laboratory analytical SOPs are listed in QAPP Worksheet #23 and provided in Attachment A.

#### **Quality of the data required:**

Data of high quality that meet the requirements presented in this QAPP will be needed to support technically sound and defensible assessments of the presence or absence of ethylene oxide in ambient air. Laboratory analyses will be conducted in accordance with the analytical method and Project Laboratory SOPs. QAPP Worksheets #34 through #37 describe data verification, validation, and usability assessment.

#### **Quantity of data required:**

The number of samples needed are described in this QAPP. The frequency of QC samples to be collected is provided in QAPP Worksheet #20. The anticipated sample types, matrices, analytical groups, and methods is provided in QAPP Worksheet #18.

### **Where, when, and how should the data be collected/generated?**

Refer to Worksheets #16 and #27.

Data will be collected tentatively on January 31 - February 1; February 3 - February 4; and February 6 - February 7, 2025, at the Institute, WV plant facilities. Refer to Worksheet #18 for the sampling locations.

The Field Sampling SOP is listed on Worksheet #21 and provided in Attachment B. The laboratory analytical SOP for the Project Laboratory is listed in QAPP Worksheet #23 and provided in Attachment A.

### **Who will collect/generate the data?**

Samples of ambient air will be collected by MAQS personnel or contract personnel. Enthalpy Analytical, LLC in Deer Park, Texas, will be used as the analytical laboratory for this project.

### **Data reporting:**

The Project Laboratory will provide complete data packages and electronic data deliverables (EDDs) to MAQS and Environmental Standards. Once the data have been validated, MAQS will provide results of sampling tests to the Dow Project Manager and the WV DEP. Data management and reporting are described in QAPP Worksheet #14.



## QAPP Worksheet #12: Measurement Performance Criteria

(UFP-QAPP Manual Section 2.6.2)  
(US EPA 2106-G-05 Section 2.2.6)

The measurement performance criteria (MPC), or acceptance criteria, refer to the degree of uncertainty of analytical measurements concerning precision, accuracy, representativeness, completeness, and comparability (PARCC). Specific objectives for each criterion are typically established to evaluate if the data are acceptable for use in making project decisions. These criteria include the use of laboratory duplicates to assess precision; isotope spikes, laboratory control samples and calibration results to assess accuracy; and blank samples to determine representativeness. The MPC for the project are listed below in this QAPP Worksheet #12.

### Precision

Precision is a measure of the agreement between concentrations of samples collected at the same time from the same location. Precision is measured by performing duplicate measurements in the field or laboratory. Precision is expressed in terms of relative percent difference (RPD) using the following equation:

$$RPD = \frac{|C1 - C2|}{((C1 + C2)/2)} \times 100$$

Where:

C1 = Concentration of one analysis.

C2 = Concentration of other analysis.

Acceptable levels of precision will vary according to the sample matrix, the specific analytical methods, and the analyte concentration relative to the MDL. QA objectives for precision will be met using written laboratory SOPs in which data acceptance criteria will be outlined.

### Accuracy

Accuracy is the degree of agreement of a measurement with an accepted reference or true value. The difference between the values is generally expressed as a percentage or ratio. Through QC checks for accuracy, potential bias of reported sample concentrations is identified.

The accuracy of laboratory analytical procedures is measured through a review of calibration, isotope and cleanup spike and laboratory control sample (LCS) results.

Continuing calibration accuracy checks are assessed by comparing the true value against the reported concentration. The percent difference (%D) between the results is calculated as follows:

Accuracy may be expressed as %D calculated by the following equation:

$$\%D = \frac{V_m - V_t}{V_t} \times 100$$

Where:

$V_t$  = the true or real value expected.

$V_m$  = the measured or observed value.

The degree of accuracy demonstrated for laboratory control samples is expressed as a percent recovery (%R). The %R indicates the amount of known concentration of an analyte that has been detected by the associated instrumentation. The %R is calculated as follows:

$$\%R = \frac{(SSR - SR)}{SA} \times 100$$

Where:

SSR = the spiked sample result

SR = the unspiked sample result

SA = the value of the spike added

The objective for accuracy of laboratory determinations is to demonstrate that the analytical instrumentation provides consistent measurements, which are within US EPA and statistically derived method-specific accuracy criteria.

### Comparability

Comparability is a measure of the degree of confidence with which one set of data can be compared to a related set of data. Comparability is a qualitative objective, which indicates the extent to which comparisons among different measurements of the same quantity will yield valid conclusions.

### Completeness

Completeness is a measure (percentage) of the amount of valid data obtained from a measurement system relative to the total amount that would be expected to be obtained under correct, normal conditions. Valid data is defined by the successful attainment of the DQOs as specified in this QAPP.

$$\text{Completeness \%} = \frac{\text{Number of valid values reported}}{\text{Number of total values reported}} \times 100$$

The QA objective for completeness can be optimized by employing and evaluating frequent QC checks throughout the analytical process so that sample data can be assessed for validity of results and to allow for reanalysis within the hold time when problems are indicated by the QC results. The goal for analytical completeness is > 90% overall usable data.

## Sensitivity

Sensitivity is the ability of the method or instrument to detect the constituent of concern and other target analytes at the levels of interest. Method and instrument sensitivity may be evaluated through instrument DL studies, MDL studies, and analysis of low-level calibration standards. A laboratory control sample is a blank matrix that is spiked at approximately the midpoint of the calibration with the analytes of interest. Sensitivity may be measured by calculating the %R of the analytes at the QL.

QLs or reporting limits (RLs) represent the minimum concentration that can be routinely identified and reliably quantified above the MDL by the laboratory. Sample QLs will be calculated and reported for all parameters and will include the effect of dilutions and sample aliquot size and final concentrated volume.



## MEASUREMENT PERFORMANCE CRITERIA TABLE – ETHYLENE OXIDE (MODIFIED US EPA METHOD 327)

**Matrix:** Ambient Air  
**Analytical Group or Method:** Ethylene Oxide  
**Concentration Level:** All  
**Sampling Procedure<sup>(1)</sup>:** EADP-01  
**Analytical Method<sup>(2)</sup>:** US EPA Method 327 – Modified

Data Quality Indicators (DQIs)	Measurement Performance Criteria	QC Sample and/or Activity Used to Assess Measurement Performance	Activity Assessed by Results
Precision	RPD $\leq$ 25%, if both results are $> 5 \times$ QL Difference $\leq$ QL, if any results are $\leq 5 \times$ QL	Laboratory Duplicate	Analysis
Precision	RPD $\leq$ 30%, if both results are $> 5 \times$ QL Difference $\leq$ QL, if any results are $\leq 5 \times$ QL	Field Duplicate	Analysis
Accuracy/Bias	70% -130%	Laboratory Control Sample (LCS)	Analysis
Accuracy/Bias	$\pm 30\%$ D of most recent continuing calibration verification (CCV); Retention Times within 2 Seconds of CCV	Internal Standards	Analysis
Sensitivity	See QAPP Worksheet #15	MDLs/QLs meet requirements.	Analysis
Data Completeness	$> 90\%$ Overall usable data	Data Completeness Check	Sampling and Analysis

(1) Reference number from QAPP Worksheet #21.

(2) Analytical method reference number from Worksheet #11.

## QAPP Worksheet #13: Secondary Data Uses and Limitations

(UFP-QAPP Manual Section 2.7)  
(USEPA 2106-G-05 Chapter 3: QAPP Elements for Evaluating Existing Data)

<b>Data Type</b>	<b>Source</b>	<b>Data Uses Relative to Current Project</b>	<b>Factors Affecting the Reliability of Data and Limitations on Data Use</b>
No secondary data for this project.			

## QAPP Worksheet #14 and #16: Project Tasks and Schedule

(UFP-QAPP Manual Sections 2.8.1 and 2.8.2)  
(USEPA 2106-G-05 Section 2.2.4)

Ambient Air Sampling Events:

Activity	Responsible Party	Planned Start Date	Planned Completion Date	Deliverable(s)	Deliverable Due Date
Mobilization logistics	MAQS and Environmental Standards.	Within 10 days of approval of QAPP by WV DEP.	15 days from start date.	None	None
Sample collection	MAQS company personnel.	Within ≤30 days from completion of Mobilization logistics.	30 days from start date.	Field notes	14 days from completion of activity
Analysis	Enthalpy Analytical, LLC	Upon sample receipt.	10 business days from sample receipt.	Analytical results, data packages and EDDs	10 business days from sample receipt
Data validation	Environmental Standards	Upon data package receipt.	10 business days from data package receipt or before the end of the proposed comment period; whichever is earlier.	Data validation report	10 business days from data package receipt or before the end of the proposed comment period; whichever is earlier.
Summarize data	MAQS	Upon completion of data validation.	10 days from receipt of comments from WV DEP on draft report.	Text narrative report and data summary spreadsheet.	10 days from receipt of comments from WV DEP on draft report.



## QAPP Worksheet #15: Laboratory-Specific Detection/Quantitation Limits

(UFP-QAPP Manual Section 2.6.2.3 and Figure 15)  
(US EPA 2106-G-05 Section 2.2.6)

Ethylene Oxide in Ambient Air or Air Field-blank					
CAS Number	Analyte	Method/SOP	Laboratory-Specific Quantitation Limit (pptv)	Laboratory-Specific MDL (pptv)	Laboratory Conducting Analysis
75-21-8	Ethylene Oxide	US EPA Method 327 Modified/TM327	20	~10	Enthalpy Analytical, LLC – Deer Park, TX

## **QAPP Worksheet #17 – Sampling Design and Rationale**

**(UFP-QAPP Manual Section 3.1.1)  
(USEPA 2106-G-05 Section 2.3.1)**

### Sampling

- Ambient air samples will be collected over a 24-hour sampling period ( $\pm$  1 hour) during scheduled sampling dates agreed upon by MAQS and Dow at the Institute, WV plant facilities.
- Samples will be collected to represent local ambient air.
- Matrix: Ambient air
- Analytical Group: Ethylene Oxide.
- Refer to Worksheet #20 for number of field samples and QC samples; refer to Worksheet #18 for sampling locations.

### **Sampling Supply Inspection and Acceptance Procedures:**

It will be the responsibility of the MAQS personnel to inspect supplies to be used as part of the field sampling before use. MAQS will coordinate and arrange with the project laboratory to have all supplies necessary to collect samples sent in a sampling kit to the sampling personnel. Supplies to be inspected consist of passive stainless-steel, Silonite coated 6-liter spherical canisters, mechanical flow control devices (MFCD) for all canisters plus one extra, a flow meter, a pressure/vacuum gauge, appropriate wrenches for the sampling system, a tripod (provided by WV DEP for the background site) or mounting system to place the sampling system at head-height, a field data sheet, field logbook, and return shipping label.

If the sampling personnel encounter problems with supplies and/or sample containers and is uncertain how to proceed, the samplers should consult the Project Manager and Quality Assurance Officer for instructions. The Quality Assurance Officer will instruct the samplers of corrective actions that should be implemented.

## QAPP Worksheet #18 – Sampling Locations and Methods/SOP Requirements Table

(UFP-QAPP Manual Sections 3.1.1 and 3.1.2)  
(USEPA 2106-G-05 Sections 2.3.1 and 2.3.2)

Sampling Location/ Designation	Matrix	Sample Type	Analytical Group	Number of Samples*	Number of QC Samples	Sampling SOP Reference	Rationale for Sampling Location
Location #6	Ambient Air	24-Hour	Ethylene Oxide	24	See Worksheet #20	See Worksheet #21	Known ethylene oxide-emitting source.
Buffalo, WV	Ambient Air	24-Hour	Ethylene Oxide	6	See Worksheet #20	See Worksheet #21	No known ethylene oxide-emitting sources nearby.

## QAPP Worksheet #19 and #30 – Sample Containers, Preservation and Holding Times

(UFP-QAPP Manual Sections 3.1.2.2)  
(USEPA 2106-G-05 Sections 2.3.2)

<b>Matrix</b>	<b>Analytical Group<sup>1</sup></b>	<b>Concentration Level</b>	<b>Analytical and Preparation Method Reference<sup>2</sup></b>	<b>Sample Size/ Volume</b>	<b>Containers</b> (number, size, and type)	<b>Preservation Requirements</b> (chemical, temperature, light protected)	<b>Maximum Holding Time<sup>3</sup></b> (preparation/ analysis)
Ambient Air; canister field blank; and field spike	Ethylene Oxide	All	US EPA Method 327 Modified	6 Liters	Silonite coated spherical canisters (manufactured by Entech Instruments)	Store at room temperature.	Canister samples have a holding time of 8 days from collection to analysis.

NA = not applicable

<sup>1</sup> Target analyte lists are provided in QAPP Worksheet #15.

<sup>2</sup> Laboratory SOP referenced in QAPP Worksheet #23.

<sup>3</sup> Technical holding times are calculated from the date and time of sample collection.

## QAPP Worksheet #20 – Field Quality Control Sample Summary Table

(UFP-QAPP Manual Section 3.1.1 and 3.1.2)  
(USEPA 2106-G-05 Section 2.3.5)

Field QC samples will be collected at the frequency summarized below on the field QC sample summary table:

Matrix	Analytical Group <sup>2</sup>	Analytical and Preparation SOP Reference	No. of Field Samples	No. of Field Duplicate Pairs	No. of Trip Blanks	No. of Field Blanks <sup>1</sup>	No. of Laboratory QC Samples <sup>3</sup>	Total Number of Samples
Ambient Air	Ethylene Oxide	EADP-01; EADP-02	21	3_	_	3	3	30

1 Field blanks are canisters filled to approximately 22 psia (7.3 psig) with humidified diluent gas in the laboratory and sent into the field with the other sample canisters as part of the sampling event. In this case, the field blank canisters are being sent to the Location #6 sampling location in Institute, WV, to capture any possible influence of ambient background. Collect one field blank for each sampling event.

2 Refer to QAPP Worksheet #15 for analyte list.

3 Laboratory QC samples include Field Spike Sample analysis for ethylene dioxide. Field Spike samples are canisters filled to 7.5 psia with a humidified, known standard, at approximately 1000 pptv in the laboratory, and sent into the field with the other sample canisters as part of the sampling event. In this case, the field spike canisters are being sent to the Location #6 sampling location in Institute, WV, to capture any possible influence of ambient background. Collect one Field Spike for each sampling event.

## QAPP Worksheet #21: Field Standard Operating Procedures

(UFP-QAPP Manual Section 3.1.2)

(USEPA 2106-G-05 Section 2.3.2)

The Field SOP is included in the Field Sampling Plan (FSP) or as an Appendix to the FSP.

Reference Number	Title, Revision Date and/or Number	Originating Organization	Equipment Type	Modified for Project Work? (Yes/No)
MAQS-01	Method 327 Canister Sampling; MAQS Appendix B, Revision 1 April 24, 2024.	MAQS	<ul style="list-style-type: none"><li>• Ballpoint black ink pens or permanent markers</li><li>• Chain-of-Custody (COC) forms</li><li>• Waterproof bags for COCs</li><li>• Tamper seal/Custody seals</li><li>• Field Data Sheet</li><li>• Field Logbook</li><li>• Laboratory-provided sample canisters</li><li>• NIST-certified pressure/vacuum gauge</li><li>• Laboratory-provided mechanical flow control devices (MFCD) – enough to cover all samples plus one extra</li><li>• NIST-certified flow meter</li><li>• Sample labels</li><li>• Return shipping labels, overnight courier air bills or shipping forms</li><li>• Chains and locks for all sampling canisters that are to remain at the Location #6 and Background sites</li><li>• Camera (optional)</li></ul>	Yes, Project Specific



## QAPP Worksheet #22: Field Equipment Calibration, Maintenance, Testing, and Inspection

(UFP-QAPP Manual Section 3.1.2.4)  
(USEPA 2106-G-05 Section 2.3.6)

Equipment	Calibration Activity	Maintenance Activity	Testing Activity	Inspection Activity	Frequency	Acceptance Criteria	Corrective Action	Person Responsible for Corrective Action	SOP Reference*
Pressure Gauge	Canister Pressure accuracy	NA	NA	NA	Yearly	NA	Send for NIST certification.	Laboratory Personnel/ Outside Vendor	MAQS-01; Section 3.0
Flow Meter	Flow Rate Accuracy	NA	NA	NA	Yearly	NA	Send for NIST certification.	Laboratory Personnel/ Outside Vendor	MAQS-01; Section 3.0
Field Sampling Device (Flow Rate Controller)	NA	NA	Flow Rate	NA	Before and after sample collection <sup>1</sup> .	Flow rates should not deviate from each other by more than 0.2 ccm on average for a minimum of three flow rate measurements and should be within $\pm 10\%$ of the referenced flow rate	Adjust the flow controller based on manufacturer's manual and repeat flow rate check. If the flow controller cannot be adjusted to the point where the flow rate is within the desired range, the flow controller should be replaced, and the flow rate check repeated.	Field Sampling Personnel	MAQS-01; Sections 5.3 and 5.6.2
Field Sampling Device (Flow Rate Controller)	NA	NA	NA	Leak Check	Before and after sample collection <sup>1</sup> .	No observable decrease in vacuum for a 2-minute period.	If a decrease in vacuum is observed ensure all fittings are tight and repeat the process. If a leak is still present replace the canister and flow controller.	Field Sampling Personnel	MAQS-01; Sections 5.4 and 5.6.2
6 Liter Canister	NA	NA	NA	Canister Pressure	Before and after sample collection.	Initial canister pressure should not exceed -29 inHg. Final canister vacuum should be no greater than -3.0 inHg.	If the initial canister pressure is out of specification, replace the canister and flow controller. If the final canister pressure is out of specification, contact Client to determine the path forward (i.e., resampling)	Field Sampling Personnel	MAQS-01; Sections 5.2.7 and 5.6.1

Equipment	Calibration Activity	Maintenance Activity	Testing Activity	Inspection Activity	Frequency	Acceptance Criteria	Corrective Action	Person Responsible for Corrective Action	SOP Reference*
6 Liter Canister	NA	Canister Cleanliness Certification	NA	NA	Before sample collection.	Initial canister should be certified clean (< 20 pptv).	If the canister cleanliness certification is not provided, the canister should not be used and should be replaced with a certified-clean canister for the sampling event.	Laboratory Personnel	EADP-03; Appendix 1

1 Each sample shipment will include a canister field spike and canister field blank for each sampling event. Perform the same leak check and flow verification as routine samples. The only exception is that the canister valve is not opened once they are placed in the field.

## QAPP Worksheet #23 – Analytical Sop References Table

(UFP-QAPP Manual Section 3.2.1)

(USEPA 2106-G-05 Section 2.3.4)

Copies of the listed Project Laboratory's SOPs and the Project Field Sampling SOP are provided in Attachment A and Attachment B, respectively.

Reference Number	Title, Revision Date, and/or Number	Definitive or Screening Data	Analytical Group	Instrument	Organization Performing Analysis	Modified for Project Work? (Y/N)
MAQS-01	Method 327 Canister Sampling; MAQS Appendix B, Revision 1 April 24, 2024.	Definitive	Ethylene Oxide	Sample Collection	MAQS – Austin, TX	Y
EADP-01	Standard Operating Procedure for the Measurement of Organic Hazardous Air Pollutants (oHAPs) by US EPA Method 327; Enthalpy SOP No. TM327, Revision 1 September 9, 2024.	Definitive	Ethylene Oxide	GCMS-SIM	Enthalpy Analytical – Deer Park, TX	Y
EADP-02	Standard Operating Procedure for Sample Receiving; Enthalpy SOP No. WP009, Revision 1 August 2, 2024	NA	NA	Computer	Enthalpy Analytical – Deer Park, TX	N
EADP-03	Standard Operating Procedure for Sampling Equipment Cleaning and Certification; Enthalpy SOP No. WP005, Revision 1 January 26, 2023	NA	NA	Computer	Enthalpy Analytical – Deer Park, TX	N

## QAPP Worksheet #24: Analytical Instrument Calibration

(UFP-QAPP Manual Section 3.2.2)

(USEPA 2106-G-05 Section 2.3.6)

Instrument	Calibration Procedure	Calibration Range	Frequency of Calibration	Acceptance Criteria	Corrective Action (CA)	Person Responsible for CA	SOP Reference
GCMS-SIM	1-Bromo-4-fluorobenzene tune, initial and continuing calibration as required in SOP.	See SOP	Initial calibration (ICAL) after instrument set up, after major instrument changes, and when continuing calibration criteria are not met.	<ul style="list-style-type: none"><li>ICAL RRF %RSD <math>\leq</math> 30% for target analytes;</li><li>Initial calibration verification (ICV) %D <math>\pm</math> 30% for target analytes;</li><li>Opening CCV <math>\pm</math> 30% (quantified vs ICAL) and <math>\pm</math> 30% (quantified vs opening CCV) for subsequent CCVs.</li><li>Internal standards <math>\pm</math> 30% of average ICAL response.</li></ul>	Inspect system, correct problem, rerun calibration and affected samples.	Analyst	EADP-01

## QAPP Worksheet #25: Analytical Instrument and Equipment Maintenance, Testing, and Inspection

(UFP-QAPP Manual Section 3.2.2)

(USEPA 2106-G-05 Section 2.3.6)

Instrument/ Equipment	Maintenance Activity	Testing Activity	Inspection Activity	Frequency	Acceptance Criteria	Corrective Action	Responsible Person	SOP Reference
GCMS-SIM	Pre-concentrator maintenance; clean sources and quadruple rods; maintain vacuum pumps.	Tuning and calibration verification	Instrument performance and sensitivity	Daily as needed	ICAL or CCV passes criteria	Recalibrate after maintenance activities if needed - See SOP.	Analyst/ Supervisor	EADP-01

## QAPP Worksheet #26 and #27: Sample Handling, Custody, and Disposal

(UFP-QAPP Manual Section 3.3)  
(USEPA 2106-G-05 Section 2.3.3)

### Sampling Collection Organization:

MAQS  
2415 Kramer Lane, Suite 18D  
Austin, TX 78758  
507-822-5661

### Laboratory:

Enthalpy Analytical, LLC  
931 Seaco Ct  
Deer Park, TX 77536  
(281) 984-7021

**Method of sample delivery (shipping/carrier):** laboratory courier and/or commercial shipping.

**Number of days from reporting until sample disposal:** Laboratory will archive any remaining samples at room temperature until given authority to discard.

Activity	Organization and Title or Position of Person Responsible for the Activity	SOP Reference
Sample labeling	MAQS personnel or subcontract personnel	MAQS-01
COC form completion	MAQS personnel or subcontract personnel	MAQS-01
Packaging	MAQS personnel or subcontract personnel	MAQS-01
Shipping coordination	MAQS	MAQS-01
Sample receipt, inspection, and log-in	Receiving laboratory staff	EADP-02
Sample custody and storage	Receiving laboratory staff	EADP-02
Sample disposal	Receiving laboratory staff	EADP-02

## QAPP Worksheet #28: Quality Control and Corrective Action

(UFP-QAPP Manual Section 3.4 and Tables 4, 5, and 6)  
(US EPA 2106-G-05 section 2.3.5)

<b>Matrix:</b>		Ambient Air and Air Field-blank			
<b>Analytical Group or Method:</b>		Ethylene Oxide			
<b>Concentration Level:</b>		All			
<b>Sampling Procedure:</b>		MAQS-01			
<b>Analytical Method:</b>		US EPA Method 327 - Modified			
QC Sample	Frequency/ Number	Method/SOP QC Acceptance Limits	Corrective Action (CA)	Person(s) Responsible for CA	Measurement Performance Criteria
Method Blank (MB)	1 per batch or for each 24-hr analytical window; whichever is more frequent.	< 20 pptv	Rerun. If second MB fails re-prepare the MB and reanalyze. If the re- preparation/reanalysis fails, analyze instrument blank to attempt to identify source of contamination and eliminate it. Reanalyze samples with positive results < 10× the method blank concentration.	Laboratory Personnel	Target analyte concentration < QL
Field Blank	1 per sampling event	< 20 pptv	Evaluate impacts on data on a case-by-case basis.	Data Validator	Target analyte concentration < QL
Laboratory Control Sample (LCS)	1 per batch	70% -130%	Reanalyze associated samples. Qualify data as needed.	Laboratory Personnel	Same as QC acceptance criteria
Laboratory Duplicate	1 per batch	RPD ≤ 25%, if both results are > 5× MDL Difference ≤ 2× MDL, if results are ≤ 5× MDL	Qualify data as needed.	Laboratory Personnel	Same as QC acceptance criteria
Field Duplicate	1 per sampling event	RPD ≤ 30%, if both results are > 5× MDL Difference ≤ 2× MDL, if results are ≤ 5× MDL	Qualify data as needed.	Laboratory Personnel	Same as QC acceptance criteria



<b>Matrix:</b> Ambient Air and Air Field-blank <b>Analytical Group or Method:</b> Ethylene Oxide <b>Concentration Level:</b> All <b>Sampling Procedure:</b> MAQS-01 <b>Analytical Method:</b> US EPA Method 327 - Modified					
<b>QC Sample</b>	<b>Frequency/ Number</b>	<b>Method/SOP QC Acceptance Limits</b>	<b>Corrective Action (CA)</b>	<b>Person(s) Responsible for CA</b>	<b>Measurement Performance Criteria</b>
Internal Standards	Every analysis	Area response for each IS compound must be within $\pm 30\%$ of the average response as determined from the most recent ICAL (for Opening CCVs) or the most recent opening CCV (for subsequent CCVs).	Evaluate calculation and instrument performance for error. Reanalyze associated samples as needed. Qualify the data as needed.	Laboratory Personnel	Same as QC acceptance criteria

## QAPP Worksheet #29: Project Documents and Records

(UFP-QAPP Manual Section 3.5.1)

(US EPA 2106-G-05 Section 2.2.8)

Sample Collection Documents and Records	On-site Analysis Documents and Records*	Off-site Analysis Documents and Records	Data Assessment Documents and Records	Other
<ul style="list-style-type: none"> <li>Field Logbooks</li> <li>COCs</li> <li>Site photographs, if taken</li> </ul>	<ul style="list-style-type: none"> <li>NA</li> </ul>	<ul style="list-style-type: none"> <li>Sample Receipt, Custody and Tracking Records</li> <li>Standard Traceability Logs</li> <li>Equipment Calibration Logs</li> <li>Sample Prep Logs</li> <li>Run Logs</li> <li>Corrective Action Forms</li> <li>Reports on Field Sample Results</li> <li>Reported Results for Standards, QC Checks, and QC Samples</li> <li>Instrument Printouts (raw data) for Field Samples, Standards, QC Checks and QC Samples</li> <li>Case Narrative</li> </ul>	<ul style="list-style-type: none"> <li>Analytical Data Packages</li> <li>Electronic Analytical Data Files</li> <li>Data Package Completeness Checklists</li> <li>Data validation worksheets</li> </ul>	<ul style="list-style-type: none"> <li>none</li> </ul>

All files will be maintained at the office of Environmental Standards.

## QAPP Worksheet #31, #32, & #33: Assessments and Corrective Action

(UFP-QAPP Manual Sections 4.1.1, 4.1.2, and 4.2)  
(US EPA 2106-G-05 Sections 2.4 and 2.5.5)

### Assessments:

Assessment Type	Responsible Party & Organization	Number/Frequency	Estimated Dates	Assessment Deliverable	Deliverable Due Date
Review of field sampling procedures	MAQS sample collection personnel	Once/Prior to sample collection	At least one week prior to sampling	Documentation of review	Prior to sample collection event
Review of field documentation	MAQS sample collection personnel	After every day of collection	As warranted	Verbal/Documentation of review	Ongoing
COC Record review	MAQS sample collection personnel	Prior to submission of samples	As warranted	Verbal/Documentation of review	Ongoing
Review of laboratory sample receipt document	Project QA Officer	24 hours from receipt of documentation	As warranted	Documentation of review	Ongoing
Internal laboratory performance audits	Laboratory QA Officer/ Laboratory	Per laboratory QA manual; at least annually	As warranted	Documentation as required by laboratory QA manual	As required by the laboratory QA manual
Sample collection program review	Project QA Officer/ Environmental Standards	Ongoing during sample collection program	As warranted	Documentation of review	Ongoing
Review/validation of analytical reports	Data Validator/ Environmental Standards	Each data package	Within 21 days of receipt	Documentation of review	Ongoing

### Assessment Response and Corrective Action:

<b>Assessment Type</b>	<b>Responsibility for responding to assessment findings</b>	<b>Assessment Response Documentation</b>	<b>Timeframe for Response</b>	<b>Responsibility for implementing Corrective Action</b>	<b>Responsible for monitoring Corrective Action implementation</b>
Review of field sampling procedures	MAQS management	Memorandum	30 days	Sample collection personnel	Lead Organization Project Manager
Review of field documentation	MAQS management	Note in field logbook and retained in correspondence	Within 7 days of identification	Sample collection personnel	Lead Organization Project Manager
COC Record review	MAQS management	Correction of documentation	Prior to submission of samples	Sample collection personnel	Lead Organization Project Manager
Review of laboratory sample receipt document	Laboratory Personnel	Revision of sample information	24 hours from receipt of documentation	Laboratory Personnel	Project QA Officer
Internal laboratory performance audits	Laboratory Personnel	As required by the laboratory QA manual	As required by the laboratory QA manual	Laboratory QA Officer	Project QA Officer
Sample collection program review	Lead Organization Project Manager	Memorandum	Within 7 days of identification	Lead Organization Project Manager	Lead Organization Project Manager
Review/validation of analytical reports	Laboratory Manager	Revision of analytical report, as needed	Within 7 days of identification	Laboratory Manager	Project QA Officer

## QAPP Worksheet #34: Data Verification and Validation Inputs

(UFP-QAPP Manual Section 5.2.1 and Table 9)  
(US EPA 2106-G-05 Section 2.5.1)

Item	Description	Verification (Completeness)	Validation (Conformance to specifications)
<b>Planning Documents/Records</b>			
1	Approved QAPP	X	
2	Field SOPs	X	
3	Laboratory SOPs	X	
4	Field logbooks	X	X
5	COC Records	X	X
6	Sample diagrams	X	X
7	Relevant correspondence (progress reports)	X	X
8	Field audit reports	X	X
9	Field corrective action reports	X	X
10	Photographs	X	X
11	Field program review	X	X
12	Cover sheet (laboratory identifying information)	X	X
13	Case Narrative	X	X
14	Sample receipt records	X	X
15	Field COC Records		
16	Sample chronology ( <i>i.e.</i> , dates and times of receipt, preparation, and analysis)	X	X
17	Communication records	X	X
18	MDL/RL establishment and verification	X	X
19	Standards traceability	X	X
20	Instrument calibration records	X	X
21	Definition of laboratory qualifiers	X	X
22	Results reporting forms	X	X
23	QC sample results	X	X
24	Corrective action reports	X	X
25	Raw data, including instrument outputs	X	X (Stage 4 only)
26	Sample preparation records	X	X
27	Electronic data deliverable	X	X

### QAPP Worksheet #35: Data Verification Procedures

(UFP-QAPP Manual Section 5.2.2)

(US EPA 2106-G-05 Section 2.5.1)

Records Reviewed	Requirement Document	Process Description	Responsible for Person
Field logbook	QAPP, Field Sampling SOP	Verify that records are present and complete for each day of field activities. Verify that all planned samples including field QC samples were collected and that sample collection locations are documented. Verify that changes/exceptions are documented and were reported in accordance with requirements. Verify that any required field monitoring was performed, and results are documented.	Daily – sample collection personnel At conclusion of field activities – Project Manager
COC Records	QAPP, Field Sampling SOP	Verify the completeness of COC Records. Examine entries for consistency with the field logbook. Check that appropriate methods and sample preservation have been recorded. Verify that the required volume of sample has been collected and that enough sample volume is available for QC samples (e.g., laboratory duplicates). Verify that all required signatures and dates are present. Check for transcription errors.	Daily – sample collection personnel At conclusion of field activities – Data Validator
Laboratory Deliverable	QAPP	Verify that the laboratory deliverable contains all records specified in the QAPP. Check sample receipt records to ensure sample condition upon receipt was noted, and any missing/broken sample containers were noted and reported according to plan. Compare the data package with the COCs to verify that results were provided for all collected samples. Review the narrative to ensure all QC exceptions are described. Check for evidence that any required notifications were provided to project personnel as specified in the QAPP. Verify that necessary signatures and dates are present.	Before reporting – Laboratory Project Manager Upon release – Project QA Officer, or Data Validator
Corrective Action Reports	QAPP	For any deficiencies noted, verify that corrective action was implemented according to plan.	Project QA Officer

## QAPP Worksheet #36: Data Validation Procedures

(UFP-QAPP Manual Section 5.2.2)

(USEPA 2106-G-05 Section 2.5.1)

Data validation will be performed on all laboratory analytical data.

Data Validator: Environmental Standards, Inc.

<b>Analytical Group/Method</b>	The data validated will be the data generated by the fixed based laboratory and will not include field generated data.
<b>Data deliverable requirements:</b>	Full Level 4 data package (fully documented)
<b>Analytical specifications:</b>	Will be defined by the project and specified in the QAPP
<b>Measurement of performance criteria:</b>	Worksheets #12 and #28
<b>Percent of data packages to be validated:</b>	100% (1 event - Stage 4 DV Report; 2 events - Stage 2B DV Reports)
<b>Percent of raw data to be validated:</b>	Minimum of 10% in the Stage 4 DV Report
<b>Percent of results to be recalculated:</b>	Minimum of 10% in the Stage 4 DV Report
<b>Validation Procedure:</b>	National Functional Guidelines (most recent version)
<b>Electronic validation program/version:</b>	None

The following data qualifiers will be applied during data validation. Potential impacts on project-specific DQOs will be discussed in the data validation report.

<b>Qualifier</b>	<b>Description</b>
U	This result should be considered “not-detected” because it was detected in a laboratory and/or field-generated blank at a similar level.
R	The data are unusable (note: The analyte may or may not be present in the sample).
J	The associated value is an estimated quantity. (no direction of bias assigned)
UJ	This analyte was analyzed for, but was not detected. The associated reporting limit is an estimate and may be inaccurate or imprecise.
J+	The analyte is present. The reported value may be biased high. The actual value is expected to be lower than reported.
J-	The analyte is present. The reported value may be biased low. The actual value is expected to be higher than reported.
N	The analyte has been “tentatively identified” or “presumptively” as present.



## QAPP Worksheet #37: Usability Assessment

(UFP-QAPP Manual Section 5.2.3)  
(USEPA 2106-G-05 Sections 2.5.2, 2.5.3, and 2.5.4)

The personnel responsible for participating in the data usability assessment are as follows (the specific personnel will vary based on the use of the data):

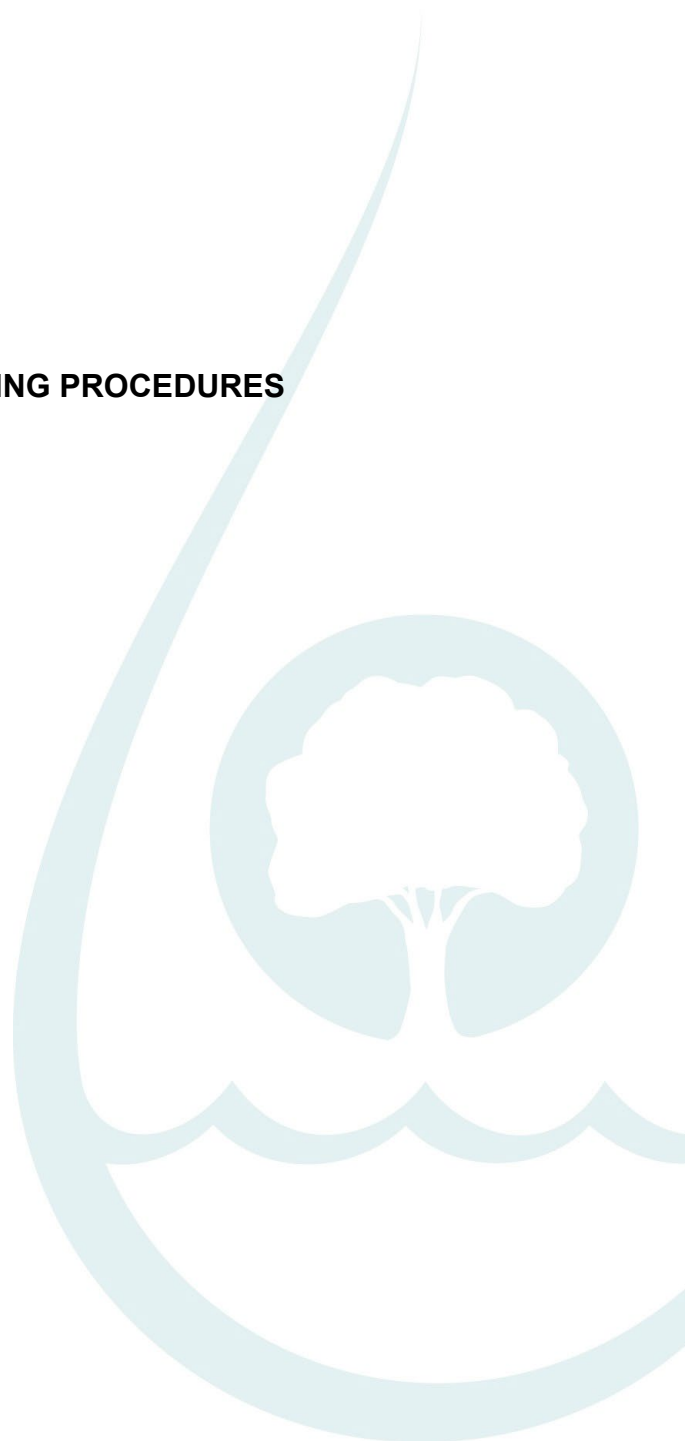
- Lead Organization Project Manager, Jenna Granstra – MAQS
- Project QA Officer, Steven Lennon – Environmental Standards

The data usability assessment process includes the following steps:

Step 1	<b>Review the project's objectives and sampling design:</b> Review the key outputs defined during planning (e.g., DQOs) to make sure they are still applicable. Review the sampling design for consistency with the stated objectives.
Step 2	<b>Review the data verification and data validation outputs:</b> Review available QA reports, including the data verification and data validation reports. Perform basic calculations and summarize the data (e.g., graphs, maps, or tables). Review deviations from planned activities (e.g., location of samples, holding time exceedances, or method deviations) and determine their impacts on the data usability. Evaluate implications of unacceptable QC sample results.
Step 3	<b>Verify the assumptions of the selected statistical method:</b> Statistical evaluation, if any, is expected to be limited for this project. The firms utilizing the data generated will be responsible for Step 3. Verify whether underlying assumptions for selected statistical method are applicable for the project data set. Items to review are the assumptions including the distributional form of the data, independence of the data, dispersion characteristics, homogeneity, and other factors. If serious deviations from assumptions are discovered, then another statistical method will need to be selected.
Step 4	<b>Implement the statistical method:</b> The firms utilizing the data generated will be responsible for Step 4. The firms will utilize statistical procedures or methods tailored to their specific data usages and assessments.
Step 5	<b>Document data usability and draw conclusions:</b> The firms utilizing the data generated will be responsible for Step 5. Determine if the data can be used as intended, considering implications of deviations and corrective actions. Discuss data quality indicators. Assess the performance of the sampling design and Identify limitations on data use. Update the conceptual Site model and document conclusions. Prepare the data usability summary report which can be in the form of text and/or a table.

**ATTACHMENT A**

**ANALYTICAL STANDARD OPERATING PROCEDURES**

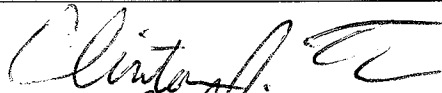


**Enthalpy Analytical  
Standard Operating Procedure**

**Measurement of Organic Hazardous Air Pollutants (oHAPs)  
by EPA Method 327**

Enthalpy SOP #	TM327	Revision #	2024-01
Author	CJT / EDP / JRH	Date Authored	September 9, 2024
Revised by	-	Date Revised	-

Technical Director/designee Approval:



Approval Date: 09-10-24

Quality Assurance Director/designee Review:



Review Date: 09/10/24

Effective Date: 09/10/24

### 1.0 Scope and Application

This SOP describes the EPA Method 327 (M327) analysis of selected volatile organic hazardous air pollutants (oHAPs) using a gas chromatograph equipped with a mass spectrometer (GC/MS).

This document is limited to laboratory preparation and analysis.

### 2.0 Summary of Method

M327 samples of fugitive emissions, area sources, and/or ambient air are collected by clients using specially-prepared evacuated canisters equipped with flow controllers.

The samples are analyzed using a GC/MS with a cryogenic pre-concentrator. A known volume of sample gas is cooled, trapped, desorbed, and refocused for enhanced chromatographic resolution. Samples are typically analyzed for ethylene oxide and vinyl chloride; additional targets may be analyzed, depending on calibration standard availability and client needs.

MS-SIM mode is used to achieve the sensitivity required for M327. The RL is typically 20 pptv, and the MDL is typically 5 to 10 pptv.

### 3.0 Definitions

- 3.1 Reporting Limit (RL): The lowest concentration that can be accurately measured with the consideration for practical limitations such as sample size and matrix interferences.
- 3.2 Method Detection Limit (MDL): The lowest concentrations that can be measured and reported with 99% confidence that the analyte concentration is greater than zero. For M327, the MDL must be at least 20% of the lowest concentration level in the ICAL.
- 3.3 Calibration Standards: A series of known concentration standards used for the calibration of the instrument. These are prepared by diluting a stock or source standard with diluent gas (zero air or nitrogen) to produce working standards, which cover the working range of the instrument. One calibration standard must be at or below the reporting limit for the method. All calibration standards must be humidified to 40-

50% RH using either humidified diluent gas or by the addition of high purity liquid water to the evacuated canister prior to the addition of diluent gas and calibration source gas. If adding water, refer to calculations to determine the appropriate amount of liquid water to add.

- 3.4 Diluent Gas: Hydrocarbon-free (HCF) synthetic “zero” air. In some explicitly stated cases, nitrogen may be used in place of zero air.
- 3.5 Initial Calibration Verification (ICV) / Second Source Calibration Verification (SSCV): A standard used to confirm the accuracy of the instrument calibration, prepared in the lower third of the calibration curve. This is prepared from a different stock (a different vendor or lot number) than was used to prepare the calibration standards, and is run after the initial calibration. Zero air must be used as the diluent gas for the ICV.
- 3.6 Continuing Calibration Verification (CCV): A standard that periodically confirms that the instrument response has not changed significantly from the initial calibration. It also confirms accurate analyte quantitation for the previous samples analyzed. This should be prepared from the same stock that was used to prepare the calibration standards, if available, and prepared in the same manner as the calibration standard. The CCV concentration should be one of the middle levels of the calibration curve. A CCV is run before the first sample, after every 10 field samples, and at the end of the analytical sequence.
  - 3.6.1 Opening CCV: The opening CCV is used to adjust M327 samples results for instrument drift. This is accomplished by replacing the ICAL average RRF for all target compounds with the RRF of the opening CCV. The opening CCV must be within 30% of its tag value for all target compounds when analyzed using the ICAL.
  - 3.6.2 Mid/Closing CCV: Mid and Closing CCVs are analyzed for a maximum of 10 field samples and after the last sample of an analytical batch. Target compounds in these CCVs must be within 30% of their tag values when analyzed using the opening CCV RRFs. These CCVs may also serve as LCS/BS samples.
- 3.7 Calibration Blank (CB): The CB is prepared before each set of standard canisters used for ICAL by filling a certified clean canister filled with humidified (40% to 50% RH) clean diluent gas sourced through the dilution system that will be employed to prepare calibration standards. If calibration standards are not prepared through a dynamic or automated static dilution system, then the CB is a humidified canister of the gas used to dilute the calibration standard. The purpose of the CB is to demonstrate that the diluent gas and dilution apparatus (if employed) is sufficiently clean such that little or no positive bias is imparted to the calibration. The CB is run with the ICAL standards using a volume typically used for sample analysis (nominal load), and is quantified with the ICAL with the same method. The CB functions as a Method Blank.
- 3.8 Method Blank (MB): A certified clean canister filled with diluent gas from the dilution system employed to prepare/pressurize/dilute field samples. The MB is used to assess contamination resulting from the entire analytical process. The injected aliquot of the

method blank must contain the same amount of internal standards that are added to each sample.

- 3.9 Laboratory Control Sample (LCS): The LCS is an analysis of a calibration standard prepared in the same matrix (including sample container type) and analyzed at a concentration that falls within the range of the initial calibration. Any Mid or Closing CCV may serve as an LCS. The LCS may also be called a blank spike (BS).
- 3.10 Laboratory Control Sample Duplicate (LCSD): A second analysis of an LCS. The LCSD is optional for M327. The second analysis of a Mid/Closing CCV may serve as an LCSD. The LCSD may also be called a blank spike duplicate (BSD).
- 3.11 Replicate Sample (SDUP): A randomly-selected or client-assigned field sample analyzed in duplicate following lab's entire analytical procedure. Analysis of the sample duplicate evaluates precision associated with the analytical system.
- 3.12 Field Duplicate Sample: A sample collected in the field at the same physical location (co-located) as the "primary" sample. The field duplicate sample and its associated primary sample must exhibit <25% RPD.
- 3.13 Field Blank (FB) Sample: A canister filled to approximately 22psia (7.3psig) with humidified diluent gas in the laboratory, and sent into the field as part of a Method 327 sampling event. The FB must exhibit <20ppt for all target compounds and associated sample results must be flagged if it does not meet this criteria.
- 3.14 Field Spike Sample: A canister filled to 7.5psia with humidified known standard, at approximately 1000ppt in the laboratory, and sent into the field as part of a M327 sampling event. The field spike sample is pressurized and analyzed as part of the associated set of M327 samples and must exhibit 70-130% recovery. Associated samples must be flagged if the field spike fails to meet these criteria.
- 3.15 Certified Sampling Equipment: Canisters and MFCD (mechanical flow controlling device) equipment must be certified according to the procedures in Method 327 in order to be used for Method 327 sampling. Detailed procedures on how to certify canisters and MFCDs can be found in their associated SOPs.
- 3.16 Certified Sample Introduction Equipment: The sample preconcentrator and autosampler must be certified in accordance with Method 327, following the procedures in this SOP.
- 3.17 MS-SCAN: Mass spectrometric mode of operation in which the gas chromatograph (GC) is coupled to a mass spectrometer (MS) programmed to SCAN all ions repeatedly over a specified mass range. Due to the sample detection limits required by M327, MS-SCAN mode is not currently viable for use with M327.
- 3.18 MS-SIM: Mass spectrometric mode of operation in which the GC is coupled to a MS that is programmed to scan a selected number of ions repeatedly [i.e., selected ion monitoring (SIM) mode].
- 3.19 Nominal Concentration: A requested, target, or named concentration that approximates the true, reference, or certified concentration. For example, a nominal 200 parts per trillion by volume (pptv) standard may have an actual certified concentration of 206 pptv.

- 3.20 Nominal Load: When analyzing using a pre-concentrator, the nominal load is the load volume which results in an instrument dilution factor of 1.0. For example, if the nominal load is 100 mL and a 1 ppbv standard is used:
- 3.20.1 Analysis of a 100 mL load results in a concentration of 1 ppbv (DF = 1x)
  - 3.20.2 Analysis of a 50 mL load results in a concentration of 0.5 ppbv (DF = 2x)
  - 3.20.3 Analysis of a 200 mL load results in a concentration of 2 ppbv (DF = 0.5x)
- 3.21 Tag value (or just “tag”): The concentration of the certified standard as it appears on the certificate of analysis provided by the manufacturer.

#### **4.0 Safety**

Appropriate personal protective equipment (including a lab coat, gloves and safety glasses) should be worn when performing this procedure as deemed necessary. GC ovens, injector ports, valve oven heaters, etc. are all potential sources of burns. Allow the instrument to cool before performing maintenance. Assume all samples contain hazardous and/ or potentially toxic materials; lab coat, gloves, and safety glasses should be worn at all times while handling samples.

#### **5.0 Equipment and Supplies**

- 5.1 Gas chromatograph / mass spectrometer / data system:
- 5.1.1 Agilent 8890 GC equipped with 5977B Mass Selective Detector (MSD), or equivalent
  - 5.1.2 Column: Rxi-624Sil MS; 60m x 0.32mmID x 1.8µm; Cat#13872, or equivalent
  - 5.1.3 Entech 7200A Pre-concentrator, or equivalent. The pre-concentrator must demonstrate <20ppt of the target compounds when analyzing blank diluent gas, and must demonstrate recovery within 15% of the theoretical value when analyzing a known standard (i.e. CCV).
  - 5.1.4 Entech 7016D Autosampler, or equivalent
  - 5.1.5 Agilent Masshunter software
  - 5.1.6 Example instrument parameters can be found in Appendix 3.
- 5.2 Entech 4700 Precision Diluter: Diluent gas (HCF zero air) must be used to prepare all standards. The standards may be humidified using ASTM Type I water injected directly into the canister or the diluent gas may be humidified prior to being added to the standard canister.
- 5.3 Digital pressure gauge, Omega DPG7010-VAC/15 or equivalent; capable of measuring 0-30 psia, at 0.10% full-scale accuracy
- 5.4 Certified 6L canisters: See associated SOP for M327 canister certification procedures. Canisters must be certified every 18 months.

- 5.5 Certified MFCD: See associated SOP for MFCD certification procedures. MFCDs are set to sample a 6L canister for a period of 24 hours and must be recertified every 12 months.
- 5.6 Gas-tight Syringes – various sizes
- 5.7 Calibrated hygrometer

## **6.0 Reagents and Standards**

- 6.1 Reagent Gases
  - 6.1.1 UHP Helium
  - 6.1.2 UHP Nitrogen
  - 6.1.3 Diluent Gas (Zero air): Hydrocarbon-free zero air. The zero air must be verified to contain <20ppt of the target compounds.
  - 6.1.4 Liquid Nitrogen, roughly 50 psig
- 6.2 Source Calibration Standards: NIST certified, where commercially available
  - 6.2.1 All source standards must be logged into LIMS upon receipt and assigned an S#. The LIMS “Standard Definition” is unique to the manufacturer of the standard; if a source standard is obtained from a different manufacturer, a new Definition must be created before the standard is assigned an S#.
  - 6.2.2 Typical expiration dates for gas source standards are assigned as one year from the date of receipt, unless otherwise listed on the Certificate of Analysis (COA).
  - 6.2.3 Label the COA with the S# and the expiration date (if not already listed on the certificate). Scan the entire certificate into LIMS and then verify that the scanned copy is legible.
  - 6.2.4 Label each container with the contents, S#, and expiration date. Store the standards in a secure room temperature environment.
- 6.3 M327 Working Calibration Standards
  - 6.3.1 Typical working standard concentrations should range from 20 to 5000 pptv (0.02 to 5 ppbv), or other appropriate range as necessary.
  - 6.3.2 Document the preparation of all working standards in LIMS, and assign S#s. Each S# must include the appropriate documentation, such as a preparation form recording the reagents, volumes, and equipment used.
  - 6.3.3 Prepare working standards in nitrogen using the Entech 4700A Precision Diluter with a clean canister which has been evacuated to <1 torr, and has been humidified with roughly 12µL of DI water per liter of canister size (e.g. typically 75µL of DI water for a 6L canister, or roughly 180µL of DI water for a 15L canister). Alternatively, gastight syringes may be used to introduce source standard gas to the working standard container, which is then pressurized using nitrogen and an appropriate, calibrated pressure gauge.



- 6.3.4 Expiration dates for working standards are assigned as no longer than 7 days after the date of preparation, and no longer than that of any of the source standards used to prepare it.
- 6.3.5 Label the canisters with the contents, S#, and expiration date. Store the standards at room temperature.
- 6.4 Internal standard(s): Internal standards (IS) are prepared in the same manner as working standards using an appropriate IS source gas. Prepared IS expires in 3 months or when the source gas expires, whichever is sooner. The IS is typically prepared at ~2.5ppb for M327 analysis using MS SIM mode.

## **7.0 Sample Preservation, Storage, and Handling**

- 7.1 Canister samples have a holding time of 8 days.
- 7.2 All samples should be stored at room temperature.
- 7.3 Samples received at an absolute pressure greater than 26.9inHg (-3inHg vacuum) must be flagged.

## **8.0 Quality Control**

The following quality controls are required to be performed in conjunction with this method. See Appendix 1 for a table listing all QC criteria, frequency, and corrective action for out-of-control events. Corrective actions for each QC failure should be followed in the order they are listed for respective QC element.

### **8.1 Instrument Blank (IB)**

- 8.1.1 An IB is an injection of internal standard only, with no sample or standard gas loaded. This serves to confirm that no contamination attributable to the analytical system is present. Target compound concentrations in the IB must be <20ppt.

### **8.2 BFB Tune Check (BFB)**

- 8.2.1 At the start of each 24-hour analytical window, prior to any sample or standard analysis, the tune of the MSD shall be verified by the analysis of <50ng of 1-Bromo-4-fluorobenzene (BFB).
- 8.2.2 The acquired mass spectrum of BFB must meet the relative ion abundance criteria listed in Appendix 2. If the tune verification analysis passes, analysis may proceed.
- 8.2.3 If the instrument tune method is adjusted, ensure that both the SCAN and SIM mode copies of the tune are updated.

### **8.3 Method Blank (MB)**

- 8.3.1 Analyze an MB prior to the OCCV for each batch, and at least once per 24-hour window or for every batch of 20 or fewer samples, whichever is more frequent. The MB should be prepared at least weekly if possible.
- 8.3.2 During ICAL, analyze an MB prior to and following the ICAL.

- 8.3.3 The MB must be <20ppt, and ideally should be less than the MDL. If contamination is identified, re-prepare and re-analyze the MB. If the MB continues to fail, perform troubleshooting on the analytical and/or standard preparation systems. Flag all sample data associated with a failing MB (i.e.  $\geq 20$ ppt).
- 8.4 Continuing Calibration Verification (CCV)
  - 8.4.1 Analyze a CCV after the BFB tune check at the start of each 24-hour analytical window, prior to any samples or other batch QC. This CCV is the Opening CCV (OCCV), and the RRF of the OCCV is used to quantitate all samples and other QC for the analytical batch. The OCCV must be within 70 to 130% of tag when quantified using the ICAL.
  - 8.4.2 For each set of 10 or fewer samples, and after the last sample of the batch, analyze a CCV. These Mid/Closing CCVs must be within 70 to 130% of tag when quantified using the OCCV RRF.
  - 8.4.3 Examine the integration in the data analysis software for every analyte to verify that each peak was correctly integrated. If manual integrations are applied, they must be consistently applied to ICAL, CCVs, and sample integrations.
- 8.5 Laboratory Control Sample (LCS) / LCS Duplicate (LCSD)
  - 8.5.1 Analyze an LCS and during each 24-hour analytical window, or for each batch of 20 or fewer samples, whichever is more frequent. Use the same data acquisition method as for the samples.
  - 8.5.2 Typically, the mid and closing CCVs serve as the LCS/LCSD, respectively. If no mid CCV is analyzed, the closing CCV will serve as the LCS and there will be no LCSD. The LCSD is optional because M327 evaluates analytical precision via the use of a replicate sample analysis (SDUP).
  - 8.5.3 Each LCS must be within 70 to 130% of tag.
- 8.6 Blank Spike (BS) / Blank Spike Duplicate (BSD)
  - 8.6.1 A BS is a mid-level standard prepared and analyzed following the same steps as a sample, and functions as a CCV and/or an LCS.
  - 8.6.2 A BSD is a replicate analysis of the BS, and may function as a CCV, LCS, and/or LCSD. BSD precision may be compared to arbitrary precision criteria, but is not required because M327 evaluates analytical precision via the use of a replicate sample analysis (SDUP).
  - 8.6.3 Sequences often interchangeably use the terms BS, CCV, LCS, BSD, and LCSD.
- 8.7 Replicate Sample (SDUP): The M327 precision requirement is fulfilled by the analysis of a sample and replicate sample (SDUP) in each analytical batch. A client-specified sample may be analyzed as the SDUP, or selection of the sample used for replicate analysis is left to the discretion of the analyst. The sample and SDUP must exhibit no

more than 25% RPD for results that are at least 5x the MDL. If the SDUP fails the criteria, the primary sample and SDUP must be flagged.

- 8.8 The internal standards (bromochloromethane, 1,4-difluorobenzene, and chlorobenzene-d5) are injected with every analytical run.
- 8.9 Field Duplicate (FDUP): The field duplicate sample and its associated primary sample, both determined by the client, must exhibit no more than 30% RPD for results that are at least 5x the MDL. If an FDUP fails the criteria, the primary sample and FDUP must be flagged.
- 8.10 Field Blank (FB): A canister filled to approximately 22psia (7.3psig) with humidified diluent gas in the laboratory, and sent into the field as part of an M327 sampling event. The FB must exhibit <20ppt for all target compounds, or associated sample results must be flagged.
- 8.11 Field Spike (FS): A canister filled to 7.5psia with humidified known standard at a concentration in the lower third of the calibration curve (e.g. approximately 1000ppt for a 20 to 5000 ppt curve), and sent into the field as part of a M327 sampling event. The field spike sample is pressurized and analyzed as part of the associated set of M327 samples and must exhibit 70-130% recovery, or associated samples must be flagged.
- 8.12 Compound Identification Acceptance Criteria
  - 8.12.1 Retention time: The retention time of the target compound must be within 2 seconds of the opening CCV retention time.
  - 8.12.2 Qualifier ions: At least one qualifier ion ratio must be within 30% of the average qualifier ion ratio from the ICAL.
  - 8.12.3 Target compound detections that fail to meet the above criteria must be flagged to denote that compound identification is not positive.
- 8.13 Sample Introduction Equipment Certification
  - 8.13.1 The procedures below must be performed on sample introduction equipment every at least every 12 months, or more frequently if the lab believes that the equipment may be causing biased sample results (e.g. carryover from extremely high concentration samples).
  - 8.13.2 Sample Preconcentrator: The laboratory must prove that the sample preconcentrator is non-biasing. This is done by analyzing a blank, which must be <20ppt for all target compounds, and by analyzing a known standard (less than 1000ppt) for all target compounds, which must exhibit recoveries of 85-115% for all target compounds.
  - 8.13.3 Autosampler: The laboratory must prove that all sample ports on the autosampler are non-biasing. This is done by analyzing a blank, which must be <20ppt for all target compounds, and by analyzing a known standard (less than 1000ppt) for all target compounds, which must exhibit recoveries of 85-115% for all target compounds. This must be done on all autosampler ports.

## 9.0 Calibration

### 9.1 Mass Selective Detector Tuning

- 9.1.1 The MSD should be retuned whenever maintenance is performed (including cleaning the ion source or changing the filament) or if the BFB tune check fails to pass its abundance criteria after repeated attempts.
- 9.1.2 Agilent Technologies MSD can be calibrated with perfluorotributylamine (PFTBA) using the masses of 69, 219 and 502. Use of either Masshunter autotune, BFB target tuning options, or manually adjusting the parameters is acceptable for finding the optimal settings for VOC analysis. All tune files should be printed to pdf and must be stored for historical reference and review. Ensure that both the SCAN and SIM mode copies of the tune are updated.
- 9.1.3 After MSD tuning, a BFB tune check must be analyzed.

### 9.2 Initial Calibration (ICAL)

- 9.2.1 Prepare a humidified CB using the dilution system to be used for ICAL standards.
- 9.2.2 Prepare standards by diluting source and/or working standards into 6L passivated canisters.
- 9.2.3 The typical ICAL range is 0.02 to 5 ppb for SIM mode, composed of at least 8 ICAL levels (minimum of 5). For each compound, the low level must be no more than five times the detection limit, and at least one level must be within 10% of the compound-specific action level.
- 9.2.4 Run an IB prior to running any other ICAL injections. Baking the preconcentrator system is recommended prior to running the IB to remove any lingering contaminants. If target compounds are detected above 20ppt in the IB, it is recommended that the preconcentrator be baked out and another IB be analyzed.
- 9.2.5 Prior to running the calibration curve, a passing BFB tune check must be analyzed.
- 9.2.6 Run a MB prior to ICAL standards, and after all standards are analyzed. Standards should ideally be run in order of increasing concentration (lowest to highest) to reduce the potential for bias created by possible low-level carryover.
- 9.2.7 Prepare a curve of total peak area vs concentration of each constituent of interest.
- 9.2.8 Verify that the ICAL meets the acceptance criterion in Appendix 1. The relative error of the calibration is verified to be acceptable via the calibration meeting the % RSD requirement; ideally, each ICAL level should also be within 30% of tag value to confirm acceptable relative error at each level. Levels outside 30% tag may be used, but should be carefully evaluated.

- 9.2.9 Once run, calibration standards may only be omitted from the curve to improve the RSD if they are removed from the “ends” of the calibration curve. Under no circumstances may a point in the middle of the curve be rejected in order to pass calibration criteria. Calibration levels may be reanalyzed if a poor injection or other anomalous issue is suspected. The reanalysis must occur within 24 hours of the BFB tune used for the ICAL, so long as no samples were analyzed since the last standard or blank.
- 9.2.10 An IB should also be analyzed after the high-level standard to demonstrate that the standard is not carrying over.
- 9.2.11 If the ICAL criteria are met, run an ICV after the IB.
- 9.2.12 An ICAL is established after all the QC checks associated with an ICAL meet the acceptance criteria listed in Appendix 1.

## **10.0 Procedure**

### **10.1 Sample Preparation**

- 10.1.1 Client samples are typically received under negative pressure (i.e. vacuum) and need to be pressurized prior to analysis.
- 10.1.2 Using a clean digital pressure gauge, measure and record the sample canister's incoming pressure. If the pressure varies by more than 0.5psi from the field post-sample pressure reading, the reported result must be flagged.
- 10.1.3 Samples are typically pressurized to approximately 5 psig (19.7 psia), which typically results in a dilution factor of about 1.5. Samples may be pressurized to lower or higher pressures as deemed appropriate by the analyst. If the analyst is unsure of how a sample should be pressurized they should consult their supervisor or the Technical Director.
- 10.1.4 Pressurize the canister to the final pressure using diluent gas (HCF zero air). Measure and record the final pressure reading and the time of the pressurization.
- 10.1.5 Canisters must be allowed to equilibrate for at least 12 hours after pressurization before analysis.

### **10.2 Daily M327 Sequencing**

- 10.2.1 Conduct an air/water check using the Mass Hunter air/water check function. The air/water check passes if the abundance of both nitrogen and water (m/z 28 and 18, respectively) relative to m/z 69 are less than 5%. Print the air/water check to pdf and save it in the mass hunter data folder.
- 10.2.2 Prior to starting the daily sequence, bake out the analytical trap on the preconcentrator and the GC oven at 250°C for 10 minutes.
- 10.2.3 After attaching all samples to the autosampler (quick connect connections are recommended), but before opening the canisters, perform a leak check of all loaded autosampler ports using the leak check function of the Entech 7200 software. Leak rates of less than 0.5psi over 60 seconds are acceptable. If a

leak rate >0.5psi over 60 seconds is observed, repair the leak and repeat the leak check for any failing autosampler ports.

- 10.2.4 Open all canisters once the leak check for each loaded autosampler port has passed.
- 10.2.5 Each sequence must begin with an instrument blank (IB), followed by a BFB tune check, method blank (MB), then an OCCV. Once the tune check, MB, and OCCV have passed acceptance criteria, the remaining QC and client samples may be added to the instrument sequence. All samples (and associated batch QC) must be injected within 24 hours of the associated BFB tune check injection time.
- 10.2.6 The nominal 1X sample volume is 500 mL for the typical instrument configuration. If alternate configurations or instrumentation are used, nominal volumes may need to be adjusted (e.g. for Entech CTS systems, the nominal 1X sample volume is typically 200 mL).
- 10.2.7 If using a portable Dewar for liquid nitrogen supply, verify that the Dewar contains sufficient liquid nitrogen to complete the analytical sequence.
- 10.2.8 An example of a typical sequence is presented below:
  - 10.2.8.1 IB
  - 10.2.8.2 BFB Tune Check (begins the 24-hour clock)
  - 10.2.8.3 MB
  - 10.2.8.4 OCCV/BS
  - 10.2.8.5 Replicate sample
  - 10.2.8.6 <10 samples, including the 3 field QC (FB, FDUP, FSPK) samples and replicate sample (SDUP)
  - 10.2.8.7 CCV/LCS (Mid-CCV)
  - 10.2.8.8 <10 samples
  - 10.2.8.9 BSD (Closing-CCV)
- 10.2.9 For all samples other than those analyzed in the same batch as an ICAL, the average RRF of the ICAL must be replaced with the RRF of the opening CCV.

## **11.0 Interferences, Deviations, and Clarifications**

- 11.1 Method interferences may be caused by impurities in the purge gas, organic compounds out-gassing from the plumbing ahead of the trap, and solvent vapors in the laboratory. The analytical system must be demonstrated to be free from contamination under the conditions of the analysis by running laboratory method and instrument blanks. The use of Polytetrafluoroethylene (PTFE) PTFE thread sealants, or flow controllers with Buna-N rubber components in the purging devices should be avoided wherever possible (exceptions may be granted by the Technical Director if no viable alternative can be found).

- 11.2 Samples can be contaminated by diffusion of volatile organics (particularly fluorocarbons and methylene chloride) through the septum seal into the sample during storage and handling.
- 11.3 Contamination by carryover can occur whenever high level and low-level samples are sequentially analyzed. The trap and other parts of the system are subjected to contamination; therefore, frequent bake-out and purging of the entire system may be required.
- 11.4 The laboratory where volatile analysis is performed should be free of organic solvents.
- 11.5 The GC/MS positively identifies target compounds based on retention times and ion ratios. Thorough data review is critical to ensure that the identifications from the data processing software are verified to be accurate, as interferences from similar compounds due to co-elution or other issues could cause misidentification of compounds or biases in reported results.
  - 11.5.1 The selected quantitation ion (primary ion) for each compound should be the most abundant ion (base peak), unless there is significant spectral interference from a co-eluting or nearby compound. In such cases, the next-highest abundance ion with no or minimal interference should be selected.
  - 11.5.2 Selected qualifier ion(s) (secondary ions) for each compound should be other abundant ions with minimal interference from co-eluting or nearby compounds. At least one qualifier ion per compound is required, though up to five may be selected.
  - 11.5.3 For M327, only a short list of target compounds is usually reported, but non-target compounds must also be considered when selecting ions for quantification and identification. For example, ethylene oxide (target compound) may elute closely to bromomethane (non-target compound), and bromomethane's mass spectrum must be considered when selecting ions for ethylene oxide.

## 12.0 Data Analysis and Calculations

### 12.1 Relative Response Factor (RRF):

$$RRF = \frac{A_x C_{is}}{A_{is} C_x}$$

Where:

$A_x$	=	Area of the primary ion for the target analyte, counts
$A_{is}$	=	Area of the primary ion for the internal standard, counts
$C_x$	=	Concentration of analyte in the calibration standard, ppbv
$C_{is}$	=	Concentration of internal standard spiking mixture, ppbv



## 12.2 Mean RRF

$$\overline{RRF} = \frac{(RRF_1 + RRF_2 + RRF_n)}{n}$$

## 12.3 Percent Relative Standard Deviation

$$\%RSD = \left( \frac{SD_{RRF}}{\overline{RRF}} \right) * 100$$

Where:

$SD_{RRF}$  = Standard deviation of the initial RRF

## 12.4 Relative Retention Time (RRT)

$$RRT = \frac{RT_c}{RT_{is}}$$

Where:

$RT_c$  = Retention time of the target analyte, seconds

$RT_{is}$  = Retention time of the internal standard, seconds

## 12.5 Mean RRT

$$\overline{RRT} = \frac{(RRT_1 + RRT_2 + RRT_n)}{n}$$

## 12.6 Mean Area Response for Internal Standards

$$\bar{Y} = \frac{(Y_1 + Y_2 + Y_n)}{n}$$

## 12.7 Mean RT for Internal Standards

$$\overline{RT} = \frac{(RT_1 + RT_2 + RT_n)}{n}$$

## 12.8 Percent difference for the daily CCV

$$\%D = \left( \frac{RRF_c - \overline{RRF}}{\overline{RRF}} \right) * 100$$

Where:

$RRF_c$  = RRF of a compound in the CCV

$\overline{RRF}$  = Mean RRF of the compound in the current ICAL

## 12.9 Concentration

$$C_x = \frac{A_x C_{is} DF}{A_{is} \overline{RRF}}$$

Where:

- $C_x$  = Compound concentration, ppbv
- $A_x$  = Area of the primary ion for the target analyte, counts
- $A_{is}$  = Area of the primary ion for the internal standard, counts
- $C_{is}$  = Concentration of the internal standard spiking mixture, ppbv
- $\overline{RRF}$  = Mean RRF from the ICAL; replaced by the OCCV RRF for M327
- $DF$  = Dilution factor; if no dilution is performed,  $DF = 1$

## 12.10 LCS Percent Recovery

$$\%Recovery = \frac{\text{Concentration found}}{\text{True concentration}} * 100$$

## 12.11 Relative Percent Difference (RPD)

$$\%RPD = \left( \frac{|LCS\ result - LCSD\ result|}{\frac{LCS + LCSD}{2}} \right) * 100$$

Note: This formula can also be applied to the Sample Duplicate RPD calculation by replacing LCS in the formula with Sample Result and LCSD with Sample Duplicate Result.

## 12.12 Water to add to a canister for a desired RH

$$V_w = D_{sat} * RH_d * V_c * \frac{P_c}{P_s} * \frac{1}{D_w}$$

Where:

- $V_w$  = Volume of water to add, uL
- $D_{sat}$  = Saturation vapor density of water<sup>1</sup>, mg/L
- $RH_d$  = Desired RH, decimal percent (i.e. 50% RH = 0.5)
- $V_c$  = Nominal canister volume, L

$P_c$  = Final absolute canister pressure, kPa<sup>2</sup>  
 $P_s$  = Standard ambient pressure, 101.3 kPa  
 $D_w$  = Density of water, 1 mg/uL

<sup>1</sup>At ambient temperature, see Table 3 in EPA Method 327

<sup>2</sup>101.3 kPa = 1 atm = 760 mmHg = 14.7 psi = 29.92 inHg

### 13.0 Method Performance

Method performance is demonstrated through MDL studies, analysis of second source samples, demonstrations of capability performed by the analyst (IDOCs, DOCs) and participation in PT studies as necessary.

### 14.0 Pollution Prevention and Waste Management

Gas samples and standards in canisters should be vented outdoors using the canister cleaning system, or may be vented in a vent hood if necessary. Calibration standard cylinders should be returned to vendors and are not disposed of by the laboratory.

### 15.0 References

15.1 EPA Method 327, "Fugitive and Area Source Measurement of Selected Volatile Organic Hazardous Air Pollutants Using Specially Prepared Canisters", 07/08/2024

### 16.0 Tables, Diagrams, and Flow Charts

- 16.1 Appendix 1: QC Requirements Summary
- 16.2 Appendix 2: BFB Abundance Criteria
- 16.3 Appendix 3: Example Equipment Parameters
- 16.4 Appendix 4: Target Analyte List

Revision History			
Revision #	Date	Author	Comments
2024-01	09/09/24	CJT / EDP / JRH	New document

### Appendix 1 QC Requirements Summary

QC Element	Frequency	Acceptance Limits	Corrective Action
BFB Tune Check	At the start of each 24-hour analytical batch, prior to any other samples or standards	See Appendix 2 for ion abundance criteria	Adjust MSD tuning parameters either manually or by using the automated Autotune or BFB Target tune programs
CB	When the ICAL is established, and when prepping any new CCV	Must be sufficiently clean such that little or no positive bias is imparted to the standards prepared;	Perform troubleshooting and maintenance, and rerun. ICAL cannot proceed until an acceptable CB is analyzed.

		should be < 20 ppt all targets	
ICAL	When CCVs fail and maintenance does not correct the problem	<ul style="list-style-type: none"> <li>• At least 5 points</li> <li>• RRF %RSD <math>\leq 30</math> for each target compound</li> <li>• RRT <math>\leq 0.06</math> RRT units of ICAL average</li> <li>• Each individual IS area count <math>\pm 30\%</math> of the ICAL mean</li> <li>• Each level should be 70-130% of tag value</li> </ul>	Perform troubleshooting and maintenance, and reanalyze curve. If level(s) outside tag but curve passes all other criteria, discuss with Technical Director to evaluate if curve and/or levels are acceptable.
ICV	After ICAL, prior to sample analysis	%Recovery 70-130%	Perform troubleshooting and maintenance, and rerun; if still out, reanalyze ICAL
OCCV / BS	At the start of each 24-hour analytical window (when not running ICAL); after BFB tune and prior to any sample or other standard analysis	%D $\leq 30\%^*$	Rerun. If 2 <sup>nd</sup> CCV fails - Perform troubleshooting and maintenance, and rerun; if still out, reanalyze ICAL and reanalyze all samples affected since the last acceptable CCV.
Mid/Closing-CCVs / LCS/LCSD / BS/BS	Analyze after a maximum 10 field samples have been analyzed. All samples must be bracketed by CCVs (opening, mid, and/or closing)	%Recovery: 70-130% when quantified using Opening CCV RRF	<ul style="list-style-type: none"> <li>• All samples not bracketed by passing CCVs must be reanalyzed</li> <li>• If rerun fails, reprep standard and reanalyze CCV</li> <li>• If reprepared CCV fails</li> </ul>
MB	One for each 24-hour analytical window, or for each batch of 20 or fewer samples, whichever is more frequent; during ICAL, before and after ICAL	< 20ppt	Rerun. If 2 <sup>nd</sup> MB fails – <ul style="list-style-type: none"> <li>• Reprep the MB and reanalyze.</li> <li>• If reanalysis fails evaluate analytical system by running an IB to confirm that contamination is not from analytical system.</li> <li>• For failing targets, sample results that are ND or &gt; 10x the MB result can be reported.</li> <li>• Rerun other samples if possible. If a passing MB cannot be achieved, report the data with the appropriate flags and narrative note.</li> </ul>
FB	One FB is collected and analyzed for each set of M327 Samples	<20ppt	
SDUP	One SDUP for each batch of 20 or fewer samples	%RPD $\leq 25\%$ for results $\geq 5x$ MDL	Verify calculations, integrations, and system performance; correct as needed. Re-analyze the sample and SDUP. Flag results associated with failing SDUP.
FDUP	One field duplicate is collected per M327 event	%RPD $\leq 30\%$ for results $\geq 5x$ MDL	Verify calculations, integrations, and system performance; correct as needed. Re-analyze the sample and FDUP. Flag results associated with failing FDUP.
Internal Standard(s)	Analyzed with all analytical runs (ICAL,	IS RTs: within 2 seconds of most recent opening CCV	Verify appropriate integrations. Reprep the IS canister

	CCV, QC, and client samples)	For opening CCV: IS areas $\pm$ 30% of the ICAL mean For mid/closing CCV: IS areas $\pm$ 30% of the opening CCV areas	If the failure was due to the analytical system, reanalyze samples that fail to meet IS acceptance criteria. Submit data only from reanalysis within limits. If reanalysis is not possible or fails, flag the data as unacceptable and notify the Project Manager.
RT – Retention time	Applies to all target compounds and internal standards	RTs must be within 2 seconds of most recent opening CCV. See ICAL section of this table for ICAL RT requirements	Reanalyze sample or standard. If persistent RT shifts continue for a given sample, results may be flagged. If persistent RT shifts continue for all samples/standards, perform instrument maintenance and/or recalibrate the instrument.

\*Note: for CCV, LCS/LCSD, MB, and DUP, failures for individual compounds may be ignored if no samples within that analytical sequence require those compounds to be reported.

## Appendix 2

### BFB Abundance Criteria

Mass	Relative Abundance
50	8.0 to 40.0% of mass 95
75	30.0 to 66.0% of mass 95
95	Base Peak, 100% relative abundance
96	5.0 to 9.0% of mass 95
173	less than 2.0% of mass 174
174	50.0 to 120.0% of mass 95
175	4.0 to 9.0% of mass 174
176	93.0 to 101.0% of mass 174
177	5.0 to 9.0% of mass 176



## Entech 7200A Events

CTD Method - Events		
File: Default.7200A.CTD		
<b>Event Durations</b>		
M2->M3 (Focusing)	3.0 min	
Inject Time	1.0 min	
System Bake	16.0 min	
M3 Cool Delay (Step 15)	0.1 min	
More M2 M3	0.0 min	
<b>Preflush Durations</b>		
Internal Std	10 sec	
Calibration Std	10 sec	
Sample	10 sec	
Sweep/Purge Gas	10 sec	
<b>Focuser Bake</b>		
Focuser Bake Mode	7200	
M3 Bake Time (Step 3)	0.0 min	
M3 Bake Time (Step 13)	0.0 min	
Post Injection Delay	30.0 min	

## GC Inlet Paraments

### Split-Splitless Inlet

Select Liner...

A Liner has not been selected.

	Actual	Setpoint
<input checked="" type="checkbox"/> Heater:	150 °C	150 °C
<input checked="" type="checkbox"/> Pressure:	8.431 psi	8.4317 psi
Total Flow:	12 mL/min	12 mL/min
<input checked="" type="checkbox"/> Septum Purge Flow:	3.001 mL/min	3 mL/min
<input type="checkbox"/> Pre-Run Flow Test (Pre-Run Flow Test not enabled - injector not installed.)		
Action on Failure:	Continue	

### ▲ Inlet Mode (Split 5 : 1)

Split



Split Ratio:

5 :1

Split Flow 7.5 mL/min

### ▲ Gas Saver (Off)

☐ On

20 mL/min

After: 2 min

## GC Column

### Columns

#	Selection
1	Front SS Inlet He ---> Agilent 13872: <Not Inventoried> Restek Rxi-624SILMS 1644691 -60 °C—325 °C (350 °C): 60 m x 320 µm x 1.8 µm ---> MSD

#### Control Mode

☒ On

Actual

Setpoint

Flow

1.5 mL/min

1.5 mL/min

Pressure

8.431 psi

8.4317 psi

Average Velocity

31.323 cm/sec

Holdup Time

3.1926 min

(Initial): 0 min  
He @ 35 °C Oven  
Out: MSD  
60 m x 320 µm x 1.8 µm

Constant Flow

## GC Oven

### Oven

Actual

☒ Oven Temp On

35 °C

35 °C

Equilibration Time

0 min

Maximum Oven Temperature

325 °C

☐ Override Column Max: 350 °C

	Rate °C/min	Value °C	Hold Time min	Run Time min
► (Initial)		35	4	4
Ramp 1	10	220	1.5	24
*				

Post Run: 70 °C

Post Run Time: 0 min



## MS SIM

### Tune File

bfb\_MS08\_072324.u

Tune Type EI

Tune EMV 967

CI Gas Valve -----

CI Flow ----- %

Actual Setpoint

MS Source 230 230

MS Quad 150 150

Apply

Run Time 8.13 min

Solvent Delay 4.00 min

Detector Setting

☒ Trace Ion Detection

EM Setting Absolute EMV

Absolute Voltage (V) 967

Applied EM Voltage (V) 967

☐ EM Saver

Limit

Sum Limit 1e8 (Default)

Acquisition Type SIM

### Scan Time Segments

	Time	Start Mass	End Mass	Threshold	Scan Speed (u/s)	Frequency (scans/sec)	Cycle Time (ms)	Step Size (m/z)
	4.00	28.50	300.00	50	1,562 [N=2]	5.1	196.41	0.1

### SIM Time Segments

	Time	Group Name	Number of Ions	Total Dwell Time (ms)	Cycle Time (Hz)	Resolution	Absolute EMV	Calculated EMV
▶	4.00		1	11	550	1.7278	Low	967
	5.50		2	8	400	2.3463	Low	967
	6.50		3	8	480	1.9747	Low	967
	8.20		4	7	280	3.2757	Low	967
	11.00		5	18	720	1.3258	Low	967
	13.70		6	13	520	1.8168	Low	967
	16.20		7	14	840	1.1478	Low	967
	18.70		8	6	330	2.8208	Low	967
	21.50		9	4	200	4.4815	Low	967
*								

## Appendix 4 Target Analyte List

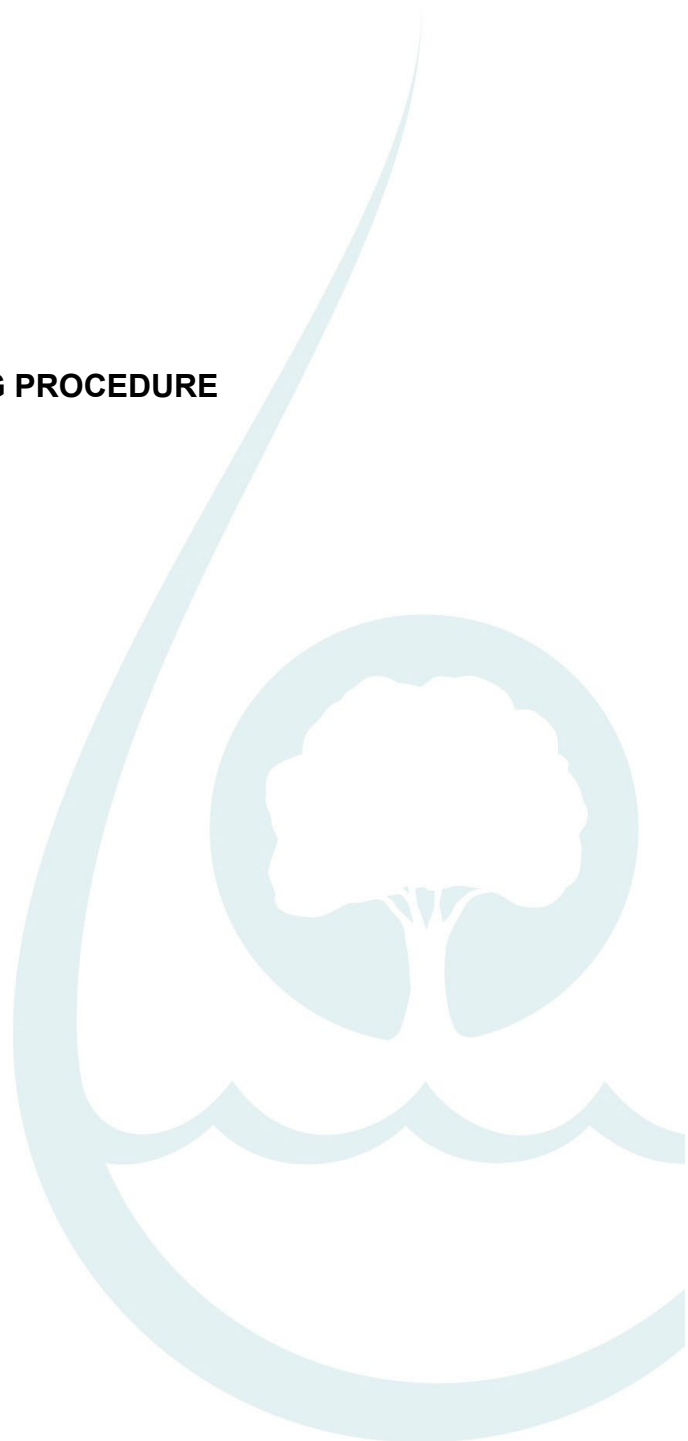
Target analytes vary based on client requests, calibration standard availability, equipment used, and other factors. Non-target analytes may be included in this list for informational, historical, or reference purposes. Target ions for analytes may be adjusted as needed, provided that no/minimal interferences are observed in the selected ions.

Compound	CAS Number	Quant Ion (m/z)	Secondary Ions (m/z)	M327 Action Limit (ug/m <sup>3</sup> )	M327 Action Limit (pptv)
Vinyl chloride	75-01-4	62	64	3	1166
Ethylene oxide	75-21-8	29	43,44	0.2	110
Benzene	71-43-2	78	77	9	2825
1,3-Butadiene	106-99-0	54	53,50	4	1348
1,2-Dichloroethane	107-06-2	62	64	4	982

Internal / Tuning Standards	CAS Number	Quant Ion (m/z)	Secondary Ions (m/z)
Bromochloromethane	74-97-5	128	49,130
1,4-Difluorobenzene	540-36-3	114	88,63
Chlorobenzene-d5	3114-55-4	117	82,119
4-Bromofluorobenzene	460-00-4	174	95,176,75

**ATTACHMENT B**

**FIELD STANDARD OPERATING PROCEDURE**



**STANDARD OPERATING PROCEDURE****SOP Title:** Method 327 Canister Sampling**Implementation Date:** April 24, 2024**Document Number:****SOP Owner (Department):** AQS**Revision Number:** R1**SOP Approval:****Technical Approval:** Darrin Barton/Client Project Manager**Quality Approval:** Name/Title**Signature:** \_\_\_\_\_**Signature:** \_\_\_\_\_**1.0 Purpose**

- 1.1. This SOP describes the procedures for collecting samples of fugitive emissions and area sources in ambient air using passivated canisters for EPA Method 327 (M327) sampling. Field Operators should use this SOP with instructions provided by the manufacturers and refer to the applicable Project Monitoring Plan to ensure successful canister deployment and sample collection for M327 sampling.

**2.0 Scope & Applicability**

- 2.1. The SOP is applicable to M327 passivated canister air sampling through flow controllers/sample regulators calibrated for 24-hour sampling periods.

**3.0 Equipment and Supplies**

- Passive stainless-steel 6L canister with an attached shipping tag containing information associated with the canister (shipped from the analysis laboratory)
- A NIST certified flow controller (yearly certification)
- Alicat or similar NIST certified Flow Meter (yearly certification)
- NIST Certified Pressure/Vacuum Gauge (yearly certification)
- Canister Timer (if applicable)
- 9/16" wrench
- 1/2" wrench
- Tripod stand or Carabiner attached to fence or other rigid structure.
- Field Data Sheet
- Field Logbook
- Return label (shipped from the analysis laboratory)

**4.0 Prior to Field Sampling****4.1. Sampling Device Bias Check**

- 4.1.1. This is conducted by the laboratory and performed at a minimum every twelve months, after cleaning, replacement of sampling system components, or following the collection of potentially contaminating samples.
- 4.1.2. Note the date of the sampling device (canister, particulate filter, flow controller, timer (optional) bias check on the field data sheet.

**4.2. Sampling Device Standard Check**

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**Document Number:**
**SOP Owner (Department):** AQS

**Revision Number:** R1

**SOP Approval:**

- 4.2.1. This procedure is also conducted by the laboratory and performed at a minimum every twelve months, after cleaning, replacement of sampling system components, or following the collection of potentially contaminating samples.

- 4.2.2. Note the date of sampling device standard check on the field data sheet.

### 4.3. Flow Control Verification Test – Performed Every 12 Months

- 4.3.1. The flow control verification test will also be conducted by the laboratory.
- 4.3.2. Refer to Figure 1 for flow verification configuration.
- 4.3.3. Using a tee fitting, connect the vacuum gauge to an evacuated canister.
- 4.3.4. The remaining open end of the tee should be connected to a flow controller. Connect the flow meter to the upstream portion of the flow controller.
- 4.3.5. Ensure the flow controller is set to record values at a minimum every hour.
- 4.3.6. Open the canister to verify the flow meter and the flow controller are sampling at approximately 3ccm. Flow verification is acceptable when the flow rate is constant over a 24-hour period and until at least 75% of the canister volume is collected.
- 4.3.7. Record the 24-hour average flow rate of the flow controller in the field logbook or canister field data sheet template which will be deemed the reference flow rate for that specific flow controller. This value will be utilized as a comparison during field flow rate verifications.

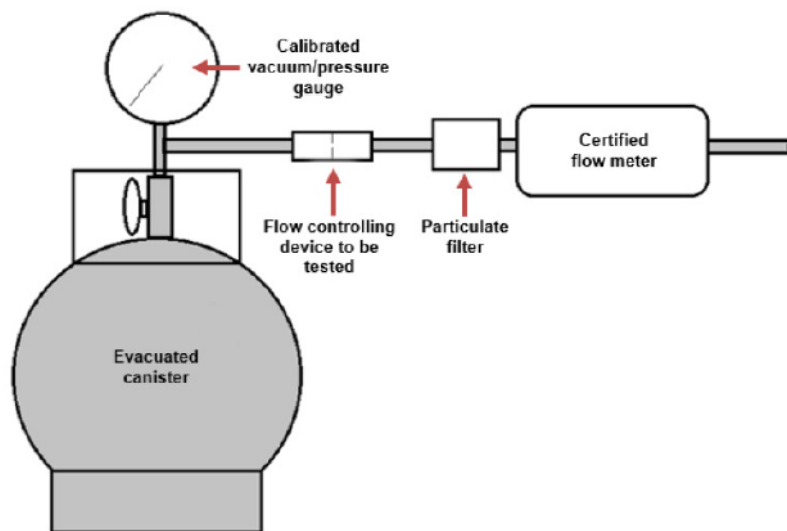


Figure 1. Apparatus for Characterizing the Flow Control Device

### 4.4. Pre Field-Sampling Activities

- 4.4.1. Prior to leaving for the field ensure all equipment and supplies contained in Section 3 are in your vehicle and accounted for.

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- 4.4.2. Confirm the laboratory shipment contains the appropriate number of canisters needed for sampling including blanks, duplicates, and spikes.

## 5.0 Field Sampling

Locate a Field Data Sheet and Chain of Custody form associated with the sampling program in the respective customer subfolder under the folder: *16\_4 Operations AQS Ambient Private\Sensible EDP\_Ops\Customers* (see example of the Field Data Sheet in Section 5 of this SOP).

- 5.1. The following information should be documented on the Field Data Sheet. This information will be entered prior to and after sampling:

- Client Name
- Sample Location
- Date
- Sampling Start Time
- Sampling End Time
- Project Number
- GPS Coordinates accurate within 3 meters. Use decimal degrees to at least five decimal places
- Canister Identification Number
- Flow Controller Identification Number
- Timer Identification Number (if applicable)
- Flow Meter Serial Number
- Flow Controller Reference Value
- Flow Controller Field Verification Value
- Leak Check Acceptance
- Initial Canister Vacuum
- Final Canister Vacuum
- Current Weather Conditions
- Any Applicable Notes (new equipment nearby, idling vehicles, etc.)

- 5.1.1. Prepare equipment and supplies:

**Note:** The canister should be deployed based on the specific sampling schedule and time defined in the Project Monitoring Plan.

**Note:** The best practice is to document all activities and verifications associated with canister sampling in the Field Data Sheet/Chain of Custody form.

**Note:** Before sampling begins, avoid volatile contaminants such as cleaning products, perfumes, gasoline, clothes that have been recently dry cleaned, or organic solvents.

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### 5.2. Sample Setup and Deployment

- 5.2.1. Remove the canister and the flow controller from the shipping box.

**Note:** Save the original packaging materials to return the sample to the laboratory for analysis.

- 5.2.2. Ensure the unique canister and flow controller IDs match the IDs in the Field Data Sheet.

**Note:** At the time of successful deployment, operators must assign a unique sample ID code that includes information providing the date and abbreviated location of sample collection (STE-LOC##-YYYYMMDD, i.e., XYZ-LOC09-20240423). Operators should affix this information to the canister or on a canister tag as a label that is water-resistant or waterproof. This unique ID should also be used as the sample ID on the laboratory chain of custody. In certain instances, the laboratory may place unique sample location labels to canisters and their respective flow controllers as well as timers. In this case canister tags would not be used.

- 5.2.3. Check the plug/brass cup on the inlet of the canister is on tight.

- 5.2.4. Ensure the canister was blanked within the last 30 days.

- 5.2.5. Confirm that the canister valve is completely closed by turning the knob clockwise.

- 5.2.6. Once verifying the above, operators can start assembling the sampling system. Remove the plug/brass cup from the inlet of the canister and reserve it for use after sample collection. If an additional fitting is attached to the inlet, hold the hex fitting below the plug with a 1/2" wrench to remove the plug. Failure to do this may cause the canister to lose pressure.

- 5.2.7. Check the initial canister pressure using the NIST certified pressure gauge. Before sampling, the initial canister pressure should not exceed -29 in Hg.

- 5.2.8. Remove the cap from the bottom of the flow controller and store it in a safe place for use when the sampling procedure is complete.

**Note:** There is a removable graphite/vespel ferrule on this fitting or a stainless-steel fitting and ferrule set – take care and ensure it is not lost when removing. If the ferrule falls out, place the ferrule on the tubing stub of the flow controller with the tapered end facing the connection to be made.

- 5.2.9. Connect the flow controller to the top of the canister and tighten it. First, tighten the nut on the flow controller onto the canister inlet using your fingers; then, with a 9/16" wrench, tighten the nut one-quarter turn past finger-tight. Make sure to hold the hex of the additional fitting (if present) still with a 1/2" wrench to ensure it is snug.

**Note:** Do not overtighten to avoid potential leaks

**STANDARD OPERATING PROCEDURE****SOP Title:** Method 327 Canister Sampling**Implementation Date:** April 24, 2024**Document Number:****SOP Owner (Department):** AQS**Revision Number:** R1**SOP Approval:****5.3. Field Flow Control Check**

**Note:** If a timer will be utilized during sample collection all steps in Sections 5.3 and 5.4 should be followed with the timer placed between the canister and flow controller. Follow the timer manual to actuate the timer valve to the open position.

- 5.3.1. Turn the flow meter on and connect it to the flow controller utilizing the same steps as the flow control verification in Section 4.1.
- 5.3.2. Open the canister, and allow the flow rate to stabilize for at least one minute. Once stabilized flow rates should not deviate from each other by more than 0.2ccm Average a minimum of three flow rate measurements and verify they are within 10% of the reference flow rate.
- 5.3.3. If flow rate is not within 10% of the lab's verification, adjust the flow controller based on manufacture's manual and repeat steps above. Entech flow controllers can be adjusted by removing the hex screw on the back of the device. Once removed adjust the set screw inside the opening to achieve the desired flow rate. If the flow controller cannot be adjusted to the point where the flow rate is within the desired range, the flow controller should be replaced and the steps above repeated.

**5.4. Field Sampling Device Leak Check**

- 5.4.1. Prior to canister deployment ensure sampling system is leak free.
- 5.4.2. Attach flow controller to the canister and ensure cap is tightened securely on the upstream opening. Open the canister valve by turning the knob counterclockwise to generate a vacuum in the flow controller.
- 5.4.3. Verify sufficient vacuum on both the canister and flow controller gauges (which should be no greater than 28" Hg near sea level, or 1" Hg closer to zero per 1,000' of elevation)
- 5.4.4. Close the valve by turning clockwise.
- 5.4.5. Observe the vacuum gauge for a minimum of two minutes.
- 5.4.6. If a decrease in vacuum is observed ensure all fittings are tight and repeat the process. If a leak is still present replace the canister and flow controller.
- 5.4.7. If there is no change observed on the vacuum gauge record this value on the field data sheet.
- 5.4.8. Each sample shipment will include a canister spike and blank for each sampling event. Perform the same leak check and flow verification as routine samples mentioned in the previous steps. The only exception is that the canister valve is not opened once they are placed in the field.

**5.5. Canister Deployment**

**Note:** Ensure all canister locations will start sampling within 60 minutes of each other.



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**Note:** Always pick the canister up by the outer ring and never by the valve or flow controller.

5.5.1. Place the canister in the sampling location by mounting it on a tripod stand, fencing or other structure as outlined in the Project Monitoring Plan. The sample inlet height should be 5.0 to 9.5 feet above ground level. For collocated/duplicate samples, inlets should be placed as close together as possible, generally within 12 in. (both vertically and horizontally).

5.5.2. To begin sampling, remove the nut/plug fitting from the inlet of the flow controller and safekeep it for use after sample collection.

5.5.3. Open the canister valve by turning the knob counterclockwise to collect the sample for the required sampling period per the Project Monitoring Plan.

**Note:** You should not hear a hissing sound after opening the canister valve. If so, close the valve, tighten all fittings an 1/8 of a turn, and repeat the previous steps for flow verification and leak check.

5.5.4. Document the following sampling information on the Field Data Sheet:

- Sample collection start date
- Sample collection start time (the time the canister valve is open, or timer is programmed to start)
- Sample collection initial canister vacuum

5.5.5. Record any unusual activities/events (e.g., vehicle exhaust, mowing activities, road paving, roofing activities, etc.) in the surrounding and nearby areas of the sampling location that may impact sample results. Document this information in the Field Data Sheet and Field Logbook.

**Note:** Include the current conditions from the nearby weather station, where applicable, in the Field Data Sheet.

**5.6. Sample Retrieval**

Once the canister has collected for the specified sampling period of 24 hours  $\pm$  1 hour (at the end of the collection time), close the canister valve and record the following sampling information on the Field Data sheet along with the operator's initials:

- Sample collection end date
- Sample collection end time (the time the canister valve is closed, or the elapsed time indicated on the timer)
- Sample collection final canister pressure
- Post sampling flow controller flow rate

**STANDARD OPERATING PROCEDURE****SOP Title:** Method 327 Canister Sampling**Implementation Date:** April 24, 2024**Document Number:****SOP Owner (Department):** AQS**Revision Number:** R1**SOP Approval:****5.6.1. Final Canister Pressure Verification**

5.6.1.1. Connect the field pressure measurement gauge to the flow controller.

5.6.1.2. Open the canister valve and actuate timer valve if applicable. Record the final canister vacuum value on the field data sheet and any additional sampling information such as unique events occurring near the location or significant weather. Final canister vacuum should be no greater than -3.0inHg.

**5.6.2. Post Sampling Flow Control Check**

5.6.2.1. Follow the steps outlined in Section 5.3 to complete the post sampling flow control check.

5.6.3. Close tight the canister valve by turning the knob clockwise.

5.6.4. Disconnect the flow controller and timer if applicable from the canister.

5.6.5. If the canister does not have quick connect fittings, reattach the nut/plug fitting on top of the canister and tighten one-quarter turn past finger-tight.

5.6.6. IF the flow controller does not have quick connect fittings, reattach the cap to the bottom of the flow controller and tighten one-quarter turn past finger tight.

5.6.7. Replace the nut/plug fitting onto the inlet of the flow controller and tighten one-quarter turn past finger tight.

5.6.8. If provided affix the tamper seal/custody seal with the sampling period, signature, and affix the seal onto the canister valve.

5.6.9. If applicable take a picture(s) of the canister valve affixed with a tamper seal and the manila shipping tag facing up.

5.6.10. Return the canister and the flow controller to the original box.

5.6.11. Canisters should be affixed with the return label for shipment back to the laboratory. Canisters should be shipped the same day they were retrieved. M327 specifies a hold time of 7 days for canisters.

**Note:** The canister shipment to the laboratory must include a completed and signed copy of the Field Data Sheet as well as a Chain of Custody.

**6.0 Revision History**

**STANDARD OPERATING PROCEDURE****SOP Title:** Method 327 Canister Sampling**Implementation Date:** April 24, 2024**Document Number:****SOP Owner (Department):** AQS**Revision Number:** R1**SOP Approval:**

Revision Number	Revision Description
0	Initial document approval/implementation

## STANDARD OPERATING PROCEDURE

**SOP Title:** Method 327 Canister Sampling

**Implementation Date:** April 24, 2024

**Document Number:**
**SOP Owner (Department):** AQS

**Revision Number:** R1

**SOP Approval:**

### 7.0 Example of the Field Data Sheet



#### EPA M327 Field Data Sheet

**General**

Sampling Personnel:		Date:	
Sampling Location ID:			
Dup/Spike/Blank:			

**Sampling Equipment**

Canister ID:		Clean Date:	
Canister Expected Vacuum Reading (mmHg):			
Sampling Device ID:		Timer ID:	
Sampling Device Expected Flow Rate (ccm/min):			

**Sampling Information**

Reference Flow Meter S/N:		Exp. Date:	
Pressure Gauge/Transducer S/N:		Exp. Date:	
Leak Check (Date/Time):		Leak Check (P/F):	
Sampling Device Actual Flow Rate (cc/min) (Before Sampling):			
Start Canister Vacuum/Pressure Reading (mmHg):			
Sampling Start Time (Date/Time):			
Sampling Device Actual Flow Rate (cc/min) (After Sampling):			
End Canister Vacuum/Pressure Reading (mmHg):			
Sampling End Time (Date/Time):			

**Comments/Notes**

TM327-001\_2024-01

**ATTACHMENT C**

**LABORATORY CERTIFICATIONS**







STATE OF LOUISIANA  
DEPARTMENT OF ENVIRONMENTAL QUALITY

Is hereby granting a Louisiana Environmental Laboratory Accreditation to



**Enthalpy Analytical LLC**  
**931 Seaco Ct**  
**Deer Park, Texas 77536**

**Agency Interest No. 83657**  
**Activity No. ACC20240001**

According to the Louisiana Administrative Code, Title 33, Part I, Subpart 3, LABORATORY ACCREDITATION, the State of Louisiana formally recognizes that this laboratory is technically competent to perform the environmental analyses listed on the scope of accreditation detailed in the attachment.

The laboratory agrees to perform all analyses listed on this scope of accreditation according to the Part I, Subpart 3 requirements and agrees to adapt to any changes in the requirements. It also acknowledges that continued accreditation is dependent on successful ongoing compliance with the applicable requirements of Part I and the 2009 TNI Standard by which the laboratory was assessed. Please contact the Department of Environmental Quality, Louisiana Environmental Laboratory Accreditation Program (LELAP) to verify the laboratory's scope of accreditation and accreditation status.

Accreditation by the State of Louisiana is not an endorsement or a guarantee of validity of the data generated by the laboratory. Accreditation of the environmental laboratory does not imply that a product, process, system, or person is approved by LELAP. To be accredited initially and maintain accreditation, the laboratory agrees to participate in two single-blind, single-concentration PT studies, where available, per year for each field of testing for which it seeks accreditation or maintains accreditation as required in LAC 33:I.4711.

Tonya Landry  
Administrator  
Public Participation and Permit Support Services Division

Issued Date: 12/30/2024

Effective on Issue Date  
Expiration Date: **June 30, 2025**  
Certificate Number: **03067**





STATE OF LOUISIANA  
DEPARTMENT OF ENVIRONMENTAL QUALITY

Effective Date: December 30, 2024

931 Seaco Ct, Deer Park, Texas 77536

Certificate Number: 03067

Enthalpy Analytical LLC  
AI Number: 83657  
Activity No. ACC20240001  
Expiration Date: June 30, 2025

## Air Emissions

Analyte	Method Name	Method Code	Type	AB
4938 - 2-Methylbutane (Isopentane)	ASTM D1945	1499	NELAP	LA
4942 - 2-methylpropane (Isobutane)	ASTM D1945	1499	NELAP	LA
100593 - C6+	ASTM D1945	1499	NELAP	LA
3755 - Carbon dioxide	ASTM D1945	1499	NELAP	LA
4747 - Ethane	ASTM D1945	1499	NELAP	LA
4926 - Methane	ASTM D1945	1499	NELAP	LA
1843 - Nitrogen	ASTM D1945	1499	NELAP	LA
5007 - n-Butane	ASTM D1945	1499	NELAP	LA
5028 - n-Pentane	ASTM D1945	1499	NELAP	LA
3915 - Particulates	TCEQ-23	2533	NELAP	TX
4795 - Ethylene oxide	EPA Method 327	9474	NELAP	LA
5235 - Vinyl chloride (Chloroethene)	EPA Method 327	9474	NELAP	LA
3805 - Fine particulates <2.5 um	40 CFR Part 50 Appendix L	10000709	NELAP	TX
9318 - 1,3-Butadiene	EPA Method 18	10246636	NELAP	TX
4836 - 1-Propene	EPA Method 18	10246636	NELAP	TX
4375 - Benzene	EPA Method 18	10246636	NELAP	TX
5142 - Butene (all isomers)	EPA Method 18	10246636	NELAP	TX
4747 - Ethane	EPA Method 18	10246636	NELAP	TX
4752 - Ethene	EPA Method 18	10246636	NELAP	TX
4765 - Ethylbenzene	EPA Method 18	10246636	NELAP	TX
100170 - Gaseous Organic Compound Emissions	EPA Method 18	10246636	NELAP	LA
4926 - Methane	EPA Method 18	10246636	NELAP	TX
5140 - Toluene	EPA Method 18	10246636	NELAP	TX
5260 - Xylene (total)	EPA Method 18	10246636	NELAP	TX
5007 - n-Butane	EPA Method 18	10246636	NELAP	TX
4855 - n-Hexane	EPA Method 18	10246636	NELAP	TX
5028 - n-Pentane	EPA Method 18	10246636	NELAP	TX
5029 - n-Propane	EPA Method 18	10246636	NELAP	TX
3755 - Carbon dioxide	EPA 3C	10247708	NELAP	LA
3775 - Carbon dioxide, oxygen, nitrogen, methane	EPA 3C	10247708	NELAP	LA
3780 - Carbon monoxide	EPA 3C	10247708	NELAP	LA
1772 - Hydrogen	EPA 3C	10247708	NELAP	LA
4926 - Methane	EPA 3C	10247708	NELAP	LA
1843 - Nitrogen	EPA 3C	10247708	NELAP	LA
3895 - Oxygen	EPA 3C	10247708	NELAP	LA
5160 - 1,1,1-Trichloroethane	EPA TO-14A	10248609	NELAP	TX
5185 - 1,1,2-Trichloro-1,2,2-trifluoroethane (Freon 113)	EPA TO-14A	10248609	NELAP	TX
4630 - 1,1-Dichloroethane	EPA TO-14A	10248609	NELAP	TX
4640 - 1,1-Dichloroethylene	EPA TO-14A	10248609	NELAP	TX
4585 - 1,2-Dibromoethane (EDB, Ethylene dibromide)	EPA TO-14A	10248609	NELAP	TX
4610 - 1,2-Dichlorobenzene	EPA TO-14A	10248609	NELAP	TX
4635 - 1,2-Dichloroethane (Ethylene dichloride)	EPA TO-14A	10248609	NELAP	TX
4655 - 1,2-Dichloropropane	EPA TO-14A	10248609	NELAP	TX
5215 - 1,3,5-Trimethylbenzene	EPA TO-14A	10248609	NELAP	TX

Clients and Customers are urged to verify the laboratory's current certification status with the Louisiana Environmental Laboratory Accreditation Program.



# Air Emissions

Analyte	Method Name	Method Code	Type	AB
4615 - 1,3-Dichlorobenzene	EPA TO-14A	10248609	NELAP	TX
4620 - 1,4-Dichlorobenzene	EPA TO-14A	10248609	NELAP	TX
4375 - Benzene	EPA TO-14A	10248609	NELAP	TX
5635 - Benzyl chloride	EPA TO-14A	10248609	NELAP	TX
4455 - Carbon tetrachloride	EPA TO-14A	10248609	NELAP	TX
4475 - Chlorobenzene	EPA TO-14A	10248609	NELAP	TX
4485 - Chloroethane (Ethyl chloride)	EPA TO-14A	10248609	NELAP	TX
4505 - Chloroform	EPA TO-14A	10248609	NELAP	TX
4765 - Ethylbenzene	EPA TO-14A	10248609	NELAP	TX
4835 - Hexachlorobutadiene	EPA TO-14A	10248609	NELAP	TX
4950 - Methyl bromide (Bromomethane)	EPA TO-14A	10248609	NELAP	TX
4960 - Methyl chloride (Chloromethane)	EPA TO-14A	10248609	NELAP	TX
4975 - Methylene chloride (Dichloromethane)	EPA TO-14A	10248609	NELAP	TX
5100 - Styrene	EPA TO-14A	10248609	NELAP	TX
5115 - Tetrachloroethylene (Perchloroethylene)	EPA TO-14A	10248609	NELAP	TX
5140 - Toluene	EPA TO-14A	10248609	NELAP	TX
5170 - Trichloroethene (Trichloroethylene)	EPA TO-14A	10248609	NELAP	TX
5235 - Vinyl chloride	EPA TO-14A	10248609	NELAP	TX
5260 - Xylene (total)	EPA TO-14A	10248609	NELAP	TX
4645 - cis-1,2-Dichloroethylene	EPA TO-14A	10248609	NELAP	TX
4680 - cis-1,3-Dichloropropene	EPA TO-14A	10248609	NELAP	TX
5160 - 1,1,1-Trichloroethane	EPA TO-15	10248803	NELAP	TX
5110 - 1,1,2,2-Tetrachloroethane	EPA TO-15	10248803	NELAP	TX
5185 - 1,1,2-Trichloro-1,2,2-trifluoroethane (Freon 113)	EPA TO-15	10248803	NELAP	TX
5165 - 1,1,2-Trichloroethane	EPA TO-15	10248803	NELAP	TX
4630 - 1,1-Dichloroethane	EPA TO-15	10248803	NELAP	TX
4640 - 1,1-Dichloroethylene	EPA TO-15	10248803	NELAP	TX
5155 - 1,2,4-Trichlorobenzene	EPA TO-15	10248803	NELAP	TX
5210 - 1,2,4-Trimethylbenzene	EPA TO-15	10248803	NELAP	TX
4585 - 1,2-Dibromoethane (EDB, Ethylene dibromide)	EPA TO-15	10248803	NELAP	TX
4695 - 1,2-Dichloro-1,1,2,2-tetrafluoroethane (Freon-114)	EPA TO-15	10248803	NELAP	TX
4610 - 1,2-Dichlorobenzene	EPA TO-15	10248803	NELAP	TX
4635 - 1,2-Dichloroethane (Ethylene dichloride)	EPA TO-15	10248803	NELAP	TX
4655 - 1,2-Dichloropropane	EPA TO-15	10248803	NELAP	TX
5215 - 1,3,5-Trimethylbenzene	EPA TO-15	10248803	NELAP	TX
9318 - 1,3-Butadiene	EPA TO-15	10248803	NELAP	TX
4615 - 1,3-Dichlorobenzene	EPA TO-15	10248803	NELAP	TX
4620 - 1,4-Dichlorobenzene	EPA TO-15	10248803	NELAP	TX
4735 - 1,4-Dioxane (1,4-Diethyleneoxide)	EPA TO-15	10248803	NELAP	TX
4836 - 1-Propene	EPA TO-15	10248803	NELAP	TX
4410 - 2-Butanone (Methyl ethyl ketone, MEK)	EPA TO-15	10248803	NELAP	TX
4535 - 2-Chlorotoluene	EPA TO-15	10248803	NELAP	LA
4542 - 4-Ethyltoluene	EPA TO-15	10248803	NELAP	TX
4995 - 4-Methyl-2-pentanone (MIBK)	EPA TO-15	10248803	NELAP	TX
4315 - Acetone	EPA TO-15	10248803	NELAP	LA
4325 - Acrolein (Propenal)	EPA TO-15	10248803	NELAP	LA
4355 - Allyl chloride (3-Chloropropene)	EPA TO-15	10248803	NELAP	LA
4375 - Benzene	EPA TO-15	10248803	NELAP	TX

Enthalpy Analytical LLC

Effective Date: December 30, 2024

Certificate Number: 03067

AI Number: 83657  
Activity No. ACC20240001  
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# Air Emissions

Analyte	Method Name	Method Code	Type	AB
4395 - Bromodichloromethane	EPA TO-15	10248803	NELAP	TX
4400 - Bromoform	EPA TO-15	10248803	NELAP	TX
4450 - Carbon disulfide	EPA TO-15	10248803	NELAP	LA
4455 - Carbon tetrachloride	EPA TO-15	10248803	NELAP	TX
4475 - Chlorobenzene	EPA TO-15	10248803	NELAP	TX
4575 - Chlorodibromomethane (dibromochloromethane)	EPA TO-15	10248803	NELAP	TX
4485 - Chloroethane (Ethyl chloride)	EPA TO-15	10248803	NELAP	TX
4505 - Chloroform	EPA TO-15	10248803	NELAP	TX
4555 - Cyclohexane	EPA TO-15	10248803	NELAP	TX
4625 - Dichlorodifluoromethane (Freon-12)	EPA TO-15	10248803	NELAP	TX
4750 - Ethanol	EPA TO-15	10248803	NELAP	LA
4755 - Ethyl acetate	EPA TO-15	10248803	NELAP	LA
4765 - Ethylbenzene	EPA TO-15	10248803	NELAP	TX
4835 - Hexachlorobutadiene	EPA TO-15	10248803	NELAP	TX
4895 - Isopropyl alcohol (2-Propanol, Isopropanol)	EPA TO-15	10248803	NELAP	LA
4950 - Methyl bromide (Bromomethane)	EPA TO-15	10248803	NELAP	TX
4960 - Methyl chloride (Chloromethane)	EPA TO-15	10248803	NELAP	TX
5000 - Methyl tert-butyl ether (MTBE)	EPA TO-15	10248803	NELAP	TX
4975 - Methylene chloride (Dichloromethane)	EPA TO-15	10248803	NELAP	TX
5005 - Naphthalene	EPA TO-15	10248803	NELAP	LA
5100 - Styrene	EPA TO-15	10248803	NELAP	TX
5115 - Tetrachloroethylene (Perchloroethylene)	EPA TO-15	10248803	NELAP	TX
5140 - Toluene	EPA TO-15	10248803	NELAP	TX
5170 - Trichloroethene (Trichloroethylene)	EPA TO-15	10248803	NELAP	TX
5175 - Trichlorofluoromethane (Fluorotrichloromethane, Freon 11)	EPA TO-15	10248803	NELAP	TX
5225 - Vinyl acetate	EPA TO-15	10248803	NELAP	TX
5235 - Vinyl chloride	EPA TO-15	10248803	NELAP	TX
5260 - Xylene (total)	EPA TO-15	10248803	NELAP	TX
4645 - cis-1,2-Dichloroethylene	EPA TO-15	10248803	NELAP	TX
4680 - cis-1,3-Dichloropropene	EPA TO-15	10248803	NELAP	TX
5240 - m+p-xylene	EPA TO-15	10248803	NELAP	TX
5250 - o-Xylene	EPA TO-15	10248803	NELAP	TX
4930 - Methanol	EPA 308	10274507	NELAP	TX
5110 - 1,1,2,2-Tetrachloroethane	EPA TO-14A, Rev.2	10312002	NELAP	TX
5165 - 1,1,2-Trichloroethane	EPA TO-14A, Rev.2	10312002	NELAP	TX
5182 - 1,2,3-Trimethylbenzene	EPA TO-14A, Rev.2	10312002	NELAP	LA
5155 - 1,2,4-Trichlorobenzene	EPA TO-14A, Rev.2	10312002	NELAP	TX
5210 - 1,2,4-Trimethylbenzene	EPA TO-14A, Rev.2	10312002	NELAP	TX
4695 - 1,2-Dichloro-1,1,2,2-tetrafluoroethane (Freon-114)	EPA TO-14A, Rev.2	10312002	NELAP	TX
9318 - 1,3-Butadiene	EPA TO-14A, Rev.2	10312002	NELAP	LA
4917 - 1-Butene	EPA TO-14A, Rev.2	10312002	NELAP	LA
4832 - 1-Hexene	EPA TO-14A, Rev.2	10312002	NELAP	LA
4833 - 1-Pentene	EPA TO-14A, Rev.2	10312002	NELAP	LA
4836 - 1-Propene	EPA TO-14A, Rev.2	10312002	NELAP	LA
5220 - 2,2,4-Trimethylpentane (Isooctane)	EPA TO-14A, Rev.2	10312002	NELAP	LA
4666 - 2,2-Dimethylbutane	EPA TO-14A, Rev.2	10312002	NELAP	LA
4667 - 2,3,4-Trimethylpentane	EPA TO-14A, Rev.2	10312002	NELAP	LA
4669 - 2,3-Dimethylbutane	EPA TO-14A, Rev.2	10312002	NELAP	LA
4671 - 2,3-Dimethylpentane	EPA TO-14A, Rev.2	10312002	NELAP	LA

Enthalpy Analytical LLC

Effective Date: December 30, 2024

Certificate Number: 03067

AI Number: 83657  
Activity No. ACC20240001  
Expiration Date: June 30, 2025

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## Air Emissions

Analyte	Method Name	Method Code	Type	AB
4672 - 2,4-Dimethylpentane	EPA TO-14A, Rev.2	10312002	NELAP	LA
4538 - 2-Ethyltoluene	EPA TO-14A, Rev.2	10312002	NELAP	LA
4937 - 2-Methylbutadiene (Isoprene)	EPA TO-14A, Rev.2	10312002	NELAP	LA
4938 - 2-Methylbutane (Isopentane)	EPA TO-14A, Rev.2	10312002	NELAP	LA
4939 - 2-Methylheptane	EPA TO-14A, Rev.2	10312002	NELAP	LA
4946 - 2-Methylhexane	EPA TO-14A, Rev.2	10312002	NELAP	LA
4941 - 2-Methylpentane (Isohexane)	EPA TO-14A, Rev.2	10312002	NELAP	LA
4942 - 2-methylpropane (Isobutane)	EPA TO-14A, Rev.2	10312002	NELAP	LA
4531 - 3-Ethyltoluene	EPA TO-14A, Rev.2	10312002	NELAP	LA
4532 - 3-Methylheptane	EPA TO-14A, Rev.2	10312002	NELAP	LA
4533 - 3-Methylhexane	EPA TO-14A, Rev.2	10312002	NELAP	LA
4534 - 3-Methylpentane	EPA TO-14A, Rev.2	10312002	NELAP	LA
4542 - 4-Ethyltoluene	EPA TO-14A, Rev.2	10312002	NELAP	LA
4323 - Acetylene	EPA TO-14A, Rev.2	10312002	NELAP	LA
4555 - Cyclohexane	EPA TO-14A, Rev.2	10312002	NELAP	LA
4562 - Cyclopentane	EPA TO-14A, Rev.2	10312002	NELAP	LA
4625 - Dichlorodifluoromethane (Freon-12)	EPA TO-14A, Rev.2	10312002	NELAP	TX
4747 - Ethane	EPA TO-14A, Rev.2	10312002	NELAP	LA
4752 - Ethene	EPA TO-14A, Rev.2	10312002	NELAP	LA
4900 - Isopropylbenzene (Cumene)	EPA TO-14A, Rev.2	10312002	NELAP	LA
4965 - Methylcyclohexane	EPA TO-14A, Rev.2	10312002	NELAP	LA
4966 - Methylcyclopentane	EPA TO-14A, Rev.2	10312002	NELAP	LA
4602 - cis-2-Butene	EPA TO-14A, Rev.2	10312002	NELAP	LA
4603 - cis-2-pentene	EPA TO-14A, Rev.2	10312002	NELAP	LA
5240 - m+p-xylene	EPA TO-14A, Rev.2	10312002	NELAP	LA
4676 - m-Diethylbenzene (1,3-Diethylbenzene)	EPA TO-14A, Rev.2	10312002	NELAP	LA
5007 - n-Butane	EPA TO-14A, Rev.2	10312002	NELAP	LA
5875 - n-Decane	EPA TO-14A, Rev.2	10312002	NELAP	LA
6235 - n-Dodecane	EPA TO-14A, Rev.2	10312002	NELAP	LA
4825 - n-Heptane	EPA TO-14A, Rev.2	10312002	NELAP	LA
4855 - n-Hexane	EPA TO-14A, Rev.2	10312002	NELAP	LA
5026 - n-Nonane	EPA TO-14A, Rev.2	10312002	NELAP	LA
5027 - n-Octane	EPA TO-14A, Rev.2	10312002	NELAP	LA
5028 - n-Pentane	EPA TO-14A, Rev.2	10312002	NELAP	LA
5029 - n-Propane	EPA TO-14A, Rev.2	10312002	NELAP	LA
5090 - n-Propylbenzene	EPA TO-14A, Rev.2	10312002	NELAP	LA
6747 - n-Undecane	EPA TO-14A, Rev.2	10312002	NELAP	LA
5250 - o-Xylene	EPA TO-14A, Rev.2	10312002	NELAP	LA
5253 - p-Diethylbenzene	EPA TO-14A, Rev.2	10312002	NELAP	LA
4700 - trans-1,2-Dichloroethylene	EPA TO-14A, Rev.2	10312002	NELAP	TX
4685 - trans-1,3-Dichloropropylene	EPA TO-14A, Rev.2	10312002	NELAP	TX
4607 - trans-2-Butene	EPA TO-14A, Rev.2	10312002	NELAP	LA
4608 - trans-2-pentene	EPA TO-14A, Rev.2	10312002	NELAP	LA
3915 - Particulates	EPA 17	10402707	NELAP	TX
4467 - Condensable Particulate Matter	EPA 202	10403006	NELAP	TX
3915 - Particulates	EPA 5	10404305	NELAP	TX
3915 - Particulates	EPA 5A	10404407	NELAP	TX
3915 - Particulates	EPA 5B	10404509	NELAP	TX
3915 - Particulates	EPA Method 5D	10404601	NELAP	TX
3915 - Particulates	EPA 5G	10404907	NELAP	TX
3915 - Particulates	EPA 5H	10405002	NELAP	TX
3915 - Particulates	EPA 5I	10405104	NELAP	TX
1772 - Hydrogen	ASTM D1945	30024443	NELAP	LA
3895 - Oxygen	ASTM D1945	30024443	NELAP	LA

Enthalpy Analytical LLC

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## Air Emissions

Analyte	Method Name	Method Code	Type	AB
4855 - n-Hexane	ASTM D1945	30024443	NELAP	LA
5029 - n-Propane	ASTM D1945	30024443	NELAP	LA
3755 - Carbon dioxide	ASTM D1946-90, Rev.1990	30024465	NELAP	TX
3780 - Carbon monoxide	ASTM D1946-90, Rev.1990	30024465	NELAP	TX
4747 - Ethane	ASTM D1946-90, Rev.1990	30024465	NELAP	TX
100806 - Hydrocarbons and Fixed Gases	ASTM D1946-90, Rev.1990	30024465	NELAP	LA
1772 - Hydrogen	ASTM D1946-90, Rev.1990	30024465	NELAP	TX
4926 - Methane	ASTM D1946-90, Rev.1990	30024465	NELAP	TX
1843 - Nitrogen	ASTM D1946-90, Rev.1990	30024465	NELAP	TX
3895 - Oxygen	ASTM D1946-90, Rev.1990	30024465	NELAP	TX
4842 - 1-Propanethiol	ASTM D5504-12	30032270	NELAP	LA
100872 - 1-Propanethiol (n-Propyl mercaptan)	ASTM D5504-12	30032270	NELAP	LA
6113 - 2,5-Dimethylthiophene	ASTM D5504-12	30032270	NELAP	LA
4843 - 2-Propanethiol	ASTM D5504-12	30032270	NELAP	LA
4544 - 2-Ethylthiophene	ASTM D5504-12	30032270	NELAP	LA
100873 - 2-Methyl-1-propanethiol (Isobutyl mercaptan)	ASTM D5504-12	30032270	NELAP	LA
3725 - 2-Methyl-1-propanethiol (i-Butanethiol)	ASTM D5504-12	30032270	NELAP	LA
100870 - 2-Propanethiol (Isopropyl mercaptan)	ASTM D5504-12	30032270	NELAP	LA
5783 - 3-Methylthiophene	ASTM D5504-12	30032270	NELAP	LA
4450 - Carbon disulfide	ASTM D5504-12	30032270	NELAP	LA
7215 - Carbonyl sulfide	ASTM D5504-12	30032270	NELAP	LA
6078 - Diethyl disulfide	ASTM D5504-12	30032270	NELAP	LA
6081 - Diethyl sulfide	ASTM D5504-12	30032270	NELAP	LA
4728 - Dimethyl disulfide	ASTM D5504-12	30032270	NELAP	LA
6116 - Dimethyl sulfide	ASTM D5504-12	30032270	NELAP	LA
7506 - Ethanethiol	ASTM D5504-12	30032270	NELAP	LA
100869 - Ethanethiol (Ethyl mercaptan)	ASTM D5504-12	30032270	NELAP	LA
3840 - Hydrogen sulfide	ASTM D5504-12	30032270	NELAP	LA
100868 - Methanethiol (Methyl mercaptan)	ASTM D5504-12	30032270	NELAP	LA
7507 - Methyl mercaptan	ASTM D5504-12	30032270	NELAP	LA
9574 - Tetrahydrothiophene	ASTM D5504-12	30032270	NELAP	LA
9578 - Thiophene	ASTM D5504-12	30032270	NELAP	LA
1942 - Total Sulfur	ASTM D5504-12	30032270	NELAP	LA
3970 - Total reduced sulfur	ASTM D5504-12	30032270	NELAP	LA
9508 - n-Butanethiol	ASTM D5504-12	30032270	NELAP	LA
100875 - n-Butanethiol (n-Butyl mercaptan)	ASTM D5504-12	30032270	NELAP	LA
9554 - s-Butanethiol	ASTM D5504-12	30032270	NELAP	LA
100874 - s-Butanethiol (sec-Butyl mercaptan)	ASTM D5504-12	30032270	NELAP	LA
9556 - tert-Butanethiol	ASTM D5504-12	30032270	NELAP	LA
100871 - tert-Butanethiol (t-Butyl mercaptan)	ASTM D5504-12	30032270	NELAP	LA

## Non Potable Water

Analyte	Method Name	Method Code	Type	AB
NONE	NONE	NONE	NONE	NONE

Enthalpy Analytical LLC

Effective Date: December 30, 2024

Certificate Number: 03067

AI Number: 83657  
Activity No. ACC20240001  
Expiration Date: June 30, 2025

Clients and Customers are urged to verify the laboratory's current certification status with the Louisiana Environmental Laboratory Accreditation Program.

## Solid Chemical Materials

Analyte	Method Name	Method Code	Type	AB
NONE	NONE	NONE	NONE	NONE

## Biological Tissue

Analyte	Method Name	Method Code	Type	AB
NONE	NONE	NONE	NONE	NONE