

Compliance Emissions Test Report

WWTU Performance Demonstration - Development of
Emission Estimates for Michigan State and Local
Emissions Inventory System (SLEIS) and EPCRA TRI
reporting

Bioreactor R722 and Enclosed Flare (with New V729
Tank)

Corteva Agriscience, LLC
Harbor Beach, Michigan 48441
Renewable Operating Permit (ROP)
MI-ROP-B4942-2020a

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Project Number: 60731664

Quality information

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1. Introduction

1.1 Summary of Test Program

Corteva Agriscience, LLC of Harbor Beach, Michigan (SRN B4942) contracted AECOM to conduct compliance testing the at the Wastewater Treatment Unit (WWTU) located in Harbor Beach, Michigan facility. The testing was completed to assess and update compliance with MI-ROP-B4942-2020a emission limits following the installation of the new V729 tank and to support emission factors used to develop emission estimates used in the Michigan State and Local Emissions Inventory System (SLEIS) and EPCRA TRI reporting. Testing occurred on September 9, 2024.

Table 1-1 summarizes the pertinent data for this compliance event.

Table 1-1. Compliance Summary

Responsible Groups	• Corteva Agriscience, LLC • Michigan Department of Environment, Great Lakes, and Energy (EGLE)
Applicable Regulations	• MI-ROP-B4942-2020a • 40 CFR Part 63, Subparts A and MMM (PAI MACT) • Michigan State and Local Emissions Inventory System (SLEIS) • EPCRA TRI reporting
Plant Location	• 305 N Huron Ave. Harbor Beach, MI 48441
Sampling Contractor	• AECOM 3700 James Savage Rd, 1602 Building Midland, MI 48642
Air Pollution Control Equipment	• Enclosed Flare
Emission Points	• R-722 Condensate • Enclosed Flare Inlet
Pollutants/Diluent Measured	<u>R-722 Condensate:</u> • Methanol • Xylenes (m/o/p isomers) • Total Xylenes (mixed isomer) • 1,2,4- Trimethylbenzene • Ammonia (NH₃) • Formaldehyde • Formic Acid • Total Sulfate Anion • Ammonium Cation • Sodium Cation <u>Unassisted Enclosed Flare:</u> • Flow Rate (Inlet) • BTU Content (Inlet) • TOC Concentration (Inlet) • Visible Emissions (Outlet)
Test Dates	• September 9, 2024

1.2 Overview of the Test Program

WWTU Flare and Bioreactor R-722 measurements were collected while the process unit is operating at capacity (>90% maximum capacity) which equates to the worst-case emission profile for methanol. Corteva states methanol is the only hazardous air pollutant with an emission profile of any significance. Plant personnel are responsible for establishing capacity operations prior to sample collection and collecting all applicable unit-operating data. Data collected includes production rate, bioreactor vent gas flow rate, flare flame temperature verification, and any other process data, as needed.

Bioreactor treatment efficiency sampling was initially accomplished in 2004. In June 2013, Corteva performed sampling to confirm ongoing compliance with the fraction removal requirements. Condensate samples were collected during the July 2021 test event, to again confirm ongoing compliance with fraction removal requirements. Refer to the flowchart above. Samples will be collected at Point 1 (M9 Tank), Point 5 (V-728 Tank), and Point 15 (V-729 Tank).

1.2.1 WWTU / Bioreactor R-722 Overview

The goal of the test program for the Wastewater Treatment Unit (WWTU) was to collect condensate samples at “inlet” and “outlet” liquid streams. Liquid samples were collected into zero-headspace 40mL VOA vials concurrently at three sample points and analyzed for various constituents. The condensate samples were to be shipped to Enthalpy Analytical in Durham, NC for laboratory analysis. The analysis consisted of the following:

- GC/FID analysis for methanol, o-xylene, m-xylene, p-xylene, 1,2,4-trimethylbenzene, and total xylenes
- HPLC/IC analysis for ammonia, formic acid, sulfate, ammonium, and sodium
- Generic spectrophotometric analysis for formaldehyde

The sample points utilized were:

- Point 1 (M9 Tank) (“inlet”)
- Point 5 (V728) (“outlet”)
- Point 15 (V-729) (“outlet”)

Test methodologies, sampling procedures, process descriptions, operating parameters, and quality assurance procedures are described later in this report.

1.2.2 Unassisted Enclosed Flare Overview

The goal of the test program for the enclosed flare was to measure the following:

- EPA Ait-080/073 for Enclosed Flare Inlet Vent Flow Rate
- EPA Method 18 for Enclosed Flare Inlet Total Organic Compounds (TOC)
- ASTM D1946-90 for Enclosed Flare Inlet Net Heating Value
- EPA Method 22 for Enclosed Flare Outlet Visible Emissions

1.3 Key Personnel

The contact for the source and test report is:

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Names and affiliations of personnel, including their roles in the test program, are summarized in the following table.

Table 1-2. Key Personnel

Role	Name	Affiliation	Address	Contact Information
Process and Environmental Focal Point	James McGee	Corteva Agriscience, LLC	305 N Huron Ave, Harbor Beach, MI 48441	james.mcgee@corteva.com 989-479-5283
Sample Project Leader	James Edmister	AECOM	3700 James Savage Rd, 1602 Building, Midland, MI	James.Edmister@aecom.com (585) 721-9128
Field Team Leader	Peter Becker	AECOM	3700 James Savage Rd, 1602 Building, Midland, MI	Peter.Becker@aecom.com (330) 285-6884
Technical Reviewer	Wayne Washburn	AECOM	13640 Briarwick Dr, Suite 200, Austin, TX	Wayne.Washburn@aecom.com (315) 447-0143
Technical Reviewer	Rob Sava	AECOM	564 White Pond Drive, Akron, OH	rob.sava@aecom.com 234-425-8440

1.4 Executive Summary

Tables 1-3 through 1-4 display the WWTU removal efficiencies of the various compounds listed in Table 1-1 above. Efficiencies are displayed as percent (%) removal. To the extent practical, removal efficiencies are displayed for the majority of the compounds. In the case where certain compounds were not present in significant concentrations at the inlet sample location, removal efficiencies are not purposeful (see discussion of results in Section 2).

Tables 1-5 through 1-7 display the WWTU concentrations of each of the various compounds listed in Table 1-1 for each individual liquid sample location. In the cases where the analyses resulted in non-detectable quantities, the analytical detection limit is displayed and is utilized in the removal efficiency calculations. In the cases where the analyses resulted in concentrations above the analytical detection limit but below the laboratory limit of quantification (LOQ), the analytical results are displayed (see discussion of results in Section 2).

Table 1-8 displays the enclosed Flare inlet flow Rate, inlet net heating Value, TOC results, and visible emissions results. Table 1-9 displays the process operating conditions during the testing.

The remainder of this document is organized as follows. **Section 2** of this document provides a summary and discussion of results for the test program, **Section 3** describes the sample locations, process descriptions, and the facility process information, **Section 4** describes the test procedures that were followed, **Section 5** describes the Quality Assurance/Quality control measures for the test program, and **Section 6** describes the data reduction.

Test program participants included: James Edmister and Peter Becker from AECOM, as well as James McGee from Corteva Agriscience, LLC.

Additional information is contained in the Appendices as follows:

Appendix A provides field data sheets from AECOM's test activities during the test program. **Appendix B** contains Facility Data and supporting documentation. **Appendix C** contains the laboratory analytical data. **Appendix D** contains the Test Protocol.

Table 1-3. Removal Efficiency - Point 1 (M9 Tank - Inlet) to Point 5 (V728 - Outlet)

Parameter	Average Point 1 (M9 Tank) (ug)	Average Point 5 (V728) (ug)	Removal (%)
Methanol	55793	10861	80.5
m-Xylene	ND	<2.49	N/A
o-Xylene	Below LOQ	<2.53	N/A
p-Xylene	24.8	<2.48	>90.0
Total Xylenes (mixed isomer)	29.3	<2.53	>91.4
1,2,4- Trimethylbenzene	73.9	<4.18	>94.3
Ammonia	404	<1.62	>99.6
Formaldehyde	Below LOQ	17.5	N/A
Formic Acid	4447	<673.1	>84.9
Total Sulfate Anion	116	81.5	29.7
Ammonium Cation	428	<1.72	>99.6
Sodium Cation	22829	25684	0.0
" < " indicates a result that is below the analytical detection limit.			
"Below LOQ" indicates a result that is between the detection limit and the laboratory limit of quantification (LOQ). The laboratory can positively identify the analyte of interest as present, but the value should be considered an estimate.			
" > " indicates a result that includes an analytical result that is either below the detection limit or is a laboratory estimate (see notes " < " and "J" in tables below).			
"ND" indicates a result that is below the laboratory detection limit .			

Table 1-4. Removal Efficiency - Point 1 (M9 Tank - Inlet) to Point 15 (V729 - Outlet)

Parameter	Average Point 1 (M9 Tank) (ug)	Average Point 15 (V729) (ug)	System Removal (%)
Methanol	55793	9839	82.4
m-Xylene	ND	<2.73	N/A
o-Xylene	Below LOQ	<2.77	N/A
p-Xylene	24.8	<2.71	>89.1
Total Xylenes (mixed isomer)	29.3	<2.77	>90.5
1,2,4- Trimethylbenzene	73.9	<4.57	>93.8
Ammonia	404	<1.62	>99.6
Formaldehyde	Below LOQ	16.9	N/A
Formic Acid	4447	249	94.4
Total Sulfate Anion	116	336	0.0
Ammonium Cation	428	<1.72	>99.6
Sodium Cation	22829	25350	0.0
"<" indicates a result that is below the analytical detection limit.			
"Below LOQ" indicates a result that is between the detection limit and the laboratory limit of quantification (LOQ). The laboratory can positively identify the analyte of interest as present, but the value should be considered an estimate.			
">" indicates a result that includes an analytical result that is either below the detection limit or is a laboratory estimate (see notes "<" and "J" in tables below).			
"ND" indicates a result that is below the laboratory detection limit .			

Table 1-5. Liquid Analysis Summary - Point 1 (M9 Tank - Inlet)

Parameter	Run 1 (ug)	Run 2 (ug)	Run 3 (ug)	Average (ug)
Methanol	57194	57657	52529	55793
m-Xylene	< 1.78	< 1.78	< 1.78	< 1.78
o-Xylene	J 8.18	J 2.26	J 2.87	J 4.44
p-Xylene	38.1	18.5	17.9	24.8
Total Xylenes (mixed isomer)	46.3	20.8	20.8	29.3
1,2,4- Trimethylbenzene	126.0	41.4	54.3	73.9
Ammonia	477	380	356	404
Formaldehyde	J 4.34	J 5.38	J 5.45	J 5.06
Formic Acid	4130	4608	4604	4447
Total Sulfate Anion	112	109	127	116.00
Ammonium Cation	505	403	377	428
Sodium Cation	23311	21767	23408	22829
*"<" indicates a result that is below the analytical detection limit.				
*J" indicates a result that is between the detection limit and the laboratory limit of quantification (LOQ). The laboratory can positively identify the analyte of interest as present, but the value should be considered an estimate.				

Table 1-6. Liquid Analysis Summary - Point 5 (V728 - Outlet)

Parameter	Run 1 (ug)	Run 2 (ug)	Run 3 (ug)	Average (ug)
Methanol	11312	9781	11491	10861
m-Xylene	< 2.49	< 2.49	< 2.49	< 2.49
o-Xylene	< 2.53	< 2.53	< 2.53	< 2.53
p-Xylene	< 2.48	< 2.48	< 2.48	< 2.48
Total Xylenes (mixed isomer)	< 2.53	< 2.53	< 2.53	< 2.53
1,2,4- Trimethylbenzene	< 4.18	< 4.18	< 4.18	< 4.18
Ammonia	< 1.62	< 1.62	< 1.62	< 1.62
Formaldehyde	J 20.9	J 10.1	J 21.5	J 17.5
Formic Acid	< 21.4	295	1703	< 673.1
Total Sulfate Anion	189	32.1	23.4	81.5
Ammonium Cation	< 1.72	< 1.72	< 1.72	< 1.72
Sodium Cation	24926	25782	26345	25684
*"<" indicates a result that is below the analytical detection limit.				
*J" indicates a result that is between the detection limit and the laboratory limit of quantification (LOQ). The laboratory can positively identify the analyte of interest as present, but the value should be considered an estimate.				

Table 1-7. Liquid Analysis - Point 15 (V729 - Outlet)

Parameter	Run 1 (ug)	Run 2 (ug)	Run 3 (ug)	Average (ug)
Methanol	9413	10241	9862	9839
m-Xylene	< 2.85	< 2.85	< 2.49	< 2.73
o-Xylene	< 2.89	< 2.89	< 2.53	< 2.77
p-Xylene	< 2.83	< 2.83	< 2.48	< 2.71
Total Xylenes (mixed isomer)	< 2.89	< 2.89	< 2.53	< 2.77
1,2,4- Trimethylbenzene	< 4.77	< 4.77	< 4.18	< 4.57
Ammonia	< 1.62	< 1.62	< 1.62	< 1.62
Formaldehyde	22.2	J 13.5	J 15.0	J 16.9
Formic Acid	J 199	J 192	356	J 249
Total Sulfate Anion	330	344	334	336
Ammonium Cation	< 1.72	< 1.72	< 1.72	< 1.72
Sodium Cation	25096	24951	26004	25350
*"<" indicates a result that is below the analytical detection limit.				
*J" indicates a result that is between the detection limit and the laboratory limit of quantification (LOQ). The laboratory can positively identify the analyte of interest as present, but the value should be considered an estimate.				

Table 1-8. Unassisted Enclosed Flare Results

Parameter	Run 1	Run 2	Run 3	Average
Test Time	10:35 - 11:35	11:37 - 12:37	12:39 - 13:39	-
Flare Flow (lb/hr)	57.7	68.8	67.2	64.6
Inlet Net Heating Value (BTU/scf)	505	630	673	603
Outlet Visible Emissions (minutes)	0	0	0	0
Inlet TOC Concentration (%v as carbon)	72.21	70.31	77.37	73.30
Inlet Nonmethane TOC Concentration (ppmv as carbon)	<101.9	<68.3	<684.5	<284.9
Inlet Nonmethane/Nonethane TOC Concentration (ppmv as carbon)	<96.8	<65.2	<676.8	<279.6

Table 1-9. Process Operating Conditions

Parameter	Run 1	Run 2	Run 3	Average
Test Date	09/09/24	09/09/24	09/09/24	-
Test Time	10:35 - 11:35	11:37 - 12:37	12:39 - 13:39	-
Bed Temp [C] (TC-R722.PV)	34.8	34.9	35.0	34.9
Feed [gpm] (FC-R722-FD.PV)	76.2	80.1	80.0	78.8
pH out (AC-R722-EFF.PV)	7.37	7.37	7.37	7.37
pH In (AC-R722-PH.PV)	7.00	7.00	7.00	7.00
Flare Flow [lb/hr] (FI-R722.PV)	57.7	68.8	67.2	64.6
Flare Temp 2 [F] (BPCS-TT82802.PV)	385	393	387	388
Flare Temp 1 [F] (BPCS-TT82801.PV)	369	376	367	371

2. Summary and Discussion of Results

The purpose of this testing was to assess and update compliance with MI-ROP-B4942-2020a emission limits following the installation of the new V729 tank and to support emission factors used to develop emission estimates used in the Michigan State and Local Emissions Inventory System (SLEIS) and EPCRA TRI reporting. The specific objectives were to:

- Determine the WWTU concentrations of the compounds listed in Table 1-1 above at sample point 1 (M9 Tank – “inlet”), sample point 5 (V728 – “outlet”), and sample point 15 (V729 – “outlet”)
- Utilizing the above concentrations, determine the removal efficiencies (as applicable)
- Determine the Inlet Flow Rate, Inlet Net Heating Value, Inlet TOC Concentration, and Outlet Visible Emissions at the Unassisted Enclosed Flare

WWTU Flare and Bioreactor R-722 measurements were collected while the process unit is operating at capacity (>90% maximum capacity) which equates to the worst-case emission profile for methanol. Corteva states methanol is the only hazardous air pollutant with an emission profile of any significance. Plant personnel were responsible for establishing capacity operations prior to sample collection and collecting all applicable unit-operating data. Data collected includes production rate, bioreactor vent gas flow rate, flare flame temperature verification, and any other process data, as applicable.

2.1 Emission Test Results

2.1.1 WWTU / Bioreactor R-722 Overview

Liquid samples were collected into zero-headspace 40mL VOA vials concurrently at sample point 1 (M9 Tank – “inlet”), sample point 5 (V728 – “outlet”), and sample point 15 (V729 – “outlet”) and were analyzed for the compounds listed in Table 1-1. The analyses were completed utilizing GC/FID, HPLC/IC, and generic spectrophotometric procedures. The analytical procedures are detailed in Section 3 of this report.

Please note that removal efficiencies that resulted in a negative value are displayed as zero. Any Point 1 (inlet) sample location analytical results that were below laboratory detection limits (ND) or were below the laboratory limit of quantification (LOQ) are noted in the tables where appropriate. ND or LOQ results for the Point 1 sample location generate removal efficiency values that are not purposeful. In this case, the removal efficiency values are displayed as not applicable. Any Point 5 or Point 15 (“outlets”) sample location results that produced ND or LOQ are noted as “less than” (<) where applicable. These values result in “worst-case” removal efficiency values (noted as “>” where applicable).

2.1.2 Unassisted Enclosed Flare Overview

Inlet flow rate was measured utilizing EPA Alt-080/073. Permanently installed mass flow meters are installed to monitor the gas flow rate. Plant personnel are responsible for recording this data during the testing. TOC was determined utilizing EPA Method 18 on sample canisters that were filled onsite at a constant flow rate. Each of 3 test runs was conducted over the course of one hour. The inlet net heating value was also determined on the canister sample gas utilizing ASTM D1946-90. During the test periods, visible emissions were determined by EPA Method 22. The test procedures are detailed in Section 3 of this report.

AECOM field data is presented in **Appendix A**. Facility process data corresponding to the test run times are presented in **Appendix B**.

3. Facility Process and Sampling Location

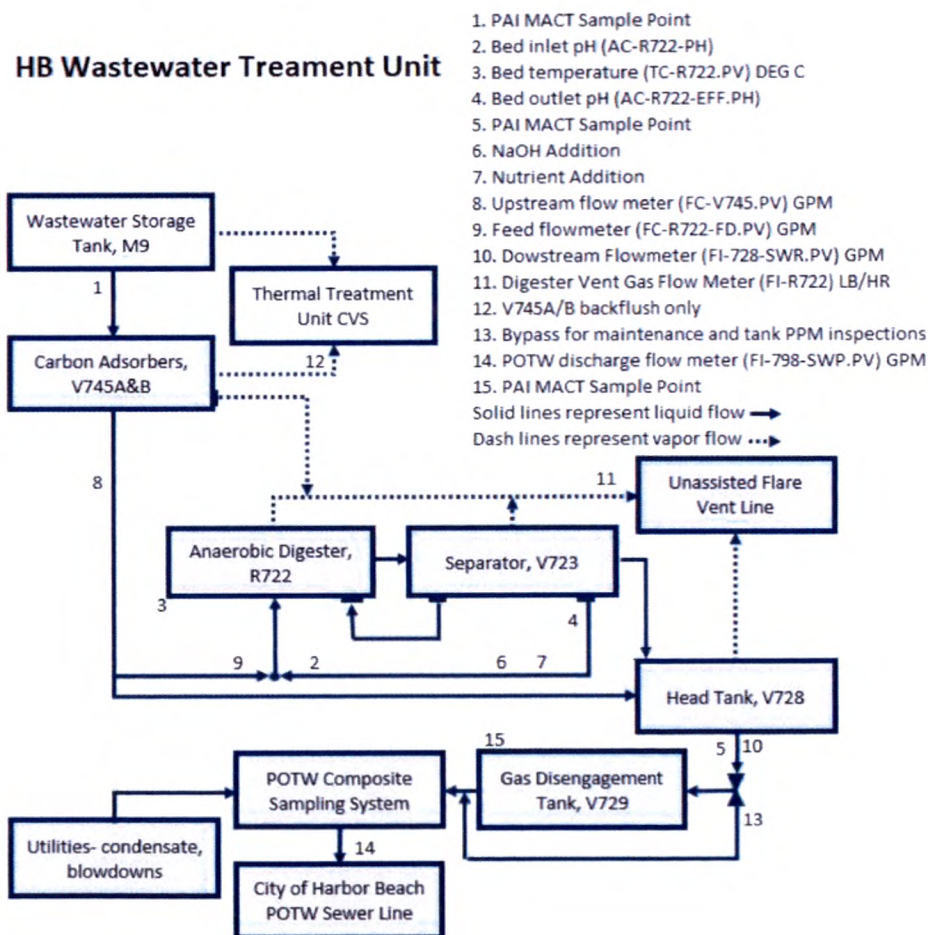
3.1 Process Description

3.1.1 Bioreactor R-722 Wastewater Treatment Process and Unassisted Enclosed Flare

This section briefly describes the WWTU system and pollution controls. The SRN B4942 Pesticide Active Ingredient (PAI) process unit consists of fermentation, product recovery crystallization, filtration, product drying and packaging, and waste broth processing equipment. Wastewater from the PAI process unit is directed to a closed anaerobic biological treatment process (Bioreactor R-722) to reduce the chemical oxygen demand (COD) and remove methanol.

Bioreactor R-722 is a recirculating fluidized bed reactor that utilizes microorganisms in an activated carbon media to anaerobically convert organic material in the presence of water into methane and carbon dioxide. The bioreactor water effluent is discharged to the local publicly owned treatment works (POTW). The exhaust gas from the bioreactor is primarily methane and carbon dioxide with lesser amounts of hydrocarbons and sulfide vapors. The methane gas generated from microorganism activity is vented to an enclosed flare. The flow chart below illustrates the wastewater treatment system.

Figure 3-1. WWTU Process Flow Chart



Gases collect in the headspace of the bioreactor and are exhausted to a non-assisted enclosed flare. Prior to the flare, the bioreactor vent gas is combined with a nitrogen purge stream and sent to a separator vessel for water removal. The bioreactor vent gas and nitrogen purge flow rates are measured prior to being combined using separate permanently installed mass flow meters. Bioreactor vent gas flow is measured using a thermal mass flow meter and automated control valve. The nitrogen flow is maintained using a mass flow meter.

3.2 Applicable Regulations and Performance Requirements

Applicable Regulations

- MI-ROP-B4942-2020a
- 40 CFR Part 63, Subparts A and MMM (PAI MACT)
- Michigan State and Local Emissions Inventory System (SLEIS)
- EPCRA TRI

Measurements – WWTU Condensate

- GC/FID analysis for methanol, o-xylene, m-xylene, p-xylene, 1,2,4-trimethylbenzene, and total xylenes
- HPLC/IC analysis for ammonia, formic acid, sulfate, ammonium, and sodium
- Generic spectrophotometric analysis for formaldehyde

Measurements – Unassisted Enclosed Flare

- EPA Ait-080/073 for Enclosed Flare Inlet Vent Flow Rate
- EPA Method 18 for Enclosed Flare Inlet Total Organic Compounds (TOC)
- ASTM D1946-90 for Enclosed Flare Inlet Net Heating Value
- EPA Method 22 for Enclosed Flare Outlet Visible Emissions

3.3 Process Emissions Control Description

Wastewater from the PAI process unit is directed to a closed anaerobic biological treatment process (Bioreactor R-722) to reduce the chemical oxygen demand (COD) and remove methanol. Bioreactor R-722 is a recirculating fluidized bed reactor that utilizes microorganisms in an activated carbon media to aerobically convert organic material in the presence of water into methane and carbon dioxide. The bioreactor water effluent is discharged to the local publicly owned treatment works (POTW). The exhaust gas from the bioreactor is primarily methane and carbon dioxide with lesser amounts of hydrocarbons and sulfide vapors. The methane gas generated from microorganism activity is vented to an enclosed flare.

4. Test Procedures

The following is a description of the testing procedures that were completed at the WWTU and Enclosed Flare sampling locations to fulfill data quality objectives of this compliance sampling project. A typical canister sampling train schematic is shown in Figure 4-1.

4.1 EPA Ait-080

EPA Ait-080: Determination of Flow Rate - Alternative Procedure Utilizing an Ultrasonic Flow Meter

This procedure is an alternative to EPA Method 2 for flow rate measurement. Use an ultrasonic flow meter in lieu of Method 2, 2A, 2C, or 2D to measure the flare exit flow rate. The calibration certification is to be kept in compliance with the manufacturer recommended frequency.

4.2 EPA Ait-073

EPA Ait-073: Determination of Flow Rate - Alternative Procedure Utilizing a Mass Flow Meter

This procedure is an alternative to EPA Method 2 for flow rate measurement. Use a mass flow meter in lieu of Method 2, 2A, 2C, or 2D to measure the flare exit flow rate. The calibration certification is to be kept in compliance with the manufacturer recommended frequency.

4.3 EPA Method 18

EPA Method 18: Measurement of Gaseous Organic Compound Emissions by Gas Chromatography

The major organic components of a gas mixture are separated by gas chromatography (GC) and individually quantified by flame ionization, photoionization, electron capture, or other appropriate detection principles. The retention times of each separated component are compared with those of known compounds under identical conditions. Therefore, the analyst confirms the identity and approximate concentrations of the organic emission components beforehand. With this information, the analyst then prepares or purchases commercially available standard mixtures to calibrate the GC under conditions identical to those of the samples. The analyst also determines the need for sample dilution to avoid detector saturation, gas stream filtration to eliminate particulate matter, and prevention of moisture condensation.

4.4 EPA Method 22

EPA Method 22: Visual Determination of Fugitive Emissions from Material Sources and Smoke Emissions from Flares

This method is applicable for the determination of the frequency of fugitive emissions from stationary sources. This method also is applicable for the determination of the frequency of visible smoke emissions from flares used for combustion of waste process materials. This method determines the amount of time that visible emissions occur during the observation period (the accumulated emission time).

4.5 Spectrophotometric Analysis

Spectrophotometric Analysis: Determination of Compounds by Spectrophotometric Procedure

Different chemical compounds absorb light at specific wavelengths, allowing measurement of the intensity of light absorbed by a sample. This analysis is used to determine the concentration of specific compounds in a sample. Light is passed through a sample solution and the intensity of the transmitted light is measured.

4.6 HPLC/IC analysis

HPLC/IC analysis: High Performance Liquid Chromatography / Ion Chromatography

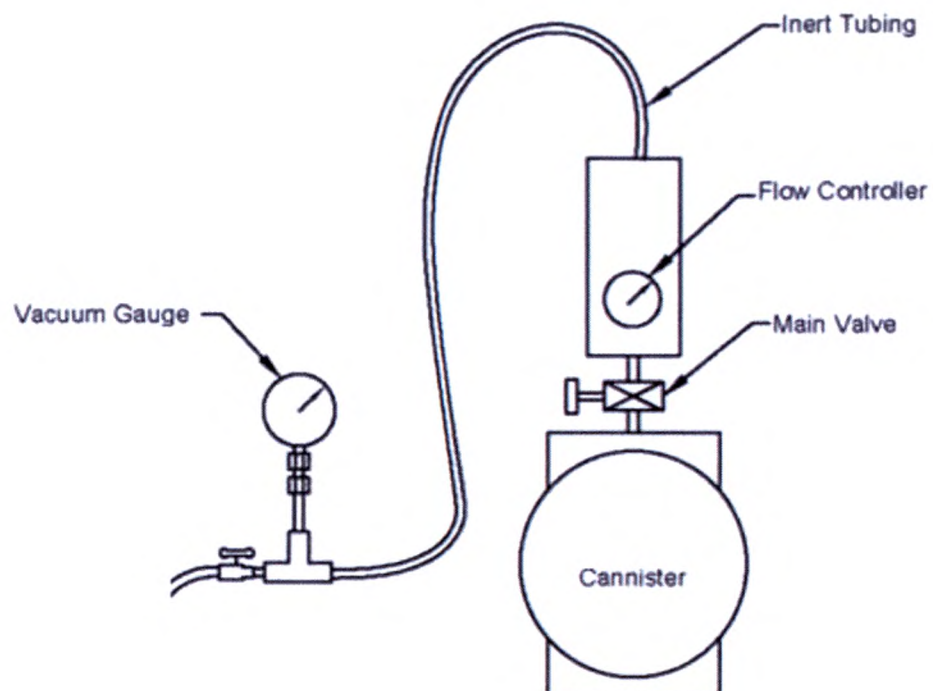
This procedure is used to separate compounds in a mixture and then identify and quantify each compound by high performance liquid chromatography / ion chromatography.

4.7 ASTM D1946-90

ASTM D1946-90: Standard Practice for Analysis of Reformed Gas by Gas Chromatography

This method is used in the determination of the chemical composition of gaseous mixtures containing hydrogen, oxygen, nitrogen, carbon monoxide, carbon dioxide, methane, ethane, and ethylene by gas chromatography.

Figure 4-1. Canister Sampling System Schematic



5. Quality Assurance / Quality Control Measures

5.1 Overview

To ensure accurate and defensible results, strict quality assurance and quality control measures were followed. Testing was performed following standard EPA protocol as outlined in 40 CFR, Part 60, Appendix A. Performance testing was performed while the process was operating at a minimum of 90% of the maximum-rated capacity, or as near thereto as practicable. Process data from the enclosed flare and bioreactor process is compiled in the above report and the following report appendix.

Test criteria were thoroughly documented and checked for completeness. Visible emissions were observed and recorded in accordance with EPA Method 22, as applicable. The flare inlet TOC samples were collected and analyzed in accordance with EPA Method 18 and ASTM Method D1946-90. Method performance specifications were met. Calibration results as well as data sheets and sampling logs produced during testing are documented in the appendix of this report.

Additional laboratory QA/QC measures are documented in the Laboratory Data appendix of this report.

Table 5-1. Manual Method Performance Checks

Activity	EPA Method	Criterion
Sample Train Sampling Rate	18	The sampling rate set at ~0.1 Lpm during each run while sampling into 6L SUMMA canisters.

6. Data Reduction

6.1 Overview

Data have been reduced to fulfill the data quality objective of the test program. Photocopies of the field data sheets and data printouts are also presented in the appendices, as applicable.

Compliance Emissions Test Report

WWTU Performance Demonstration - Development of
Emission Estimates for Michigan State and Local
Emissions Inventory System (SLEIS) and EPCRA TRI
reporting

Bioreactor R722 and Enclosed Flare (with New V729
Tank)

Corteva Agriscience, LLC
Harbor Beach, Michigan 48441
Renewable Operating Permit (ROP)
MI-ROP-B4942-2020a

Test Date: September 9, 2024

Project Number: 60731664

Quality information

Prepared by



Rob Sava, QSTI
Env. Engineering V

Approved by



James Edmister
Project Manager

Revision History

Revision	Revision date	Details	Authorized	Name	Position
0	October 7, 2024	Initial draft	RS	Rob Sava	Env. Eng.
1	October 15, 2024	Final draft for review – process data received	RS	Rob Sava	Env. Eng.
2	October 16, 2024	Final Review and Approval	JRE	James Edmister	Project Manager
3	October 16, 2024	Diagram, table, content update	RS	Rob Sava	Env. Eng.
4	October 18, 2024	Test Date and SLEIS update	RS	Rob Sava	Env. Eng.

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- Appendix A Field Data Sheets
- Appendix B Facility Process Data
- Appendix C Laboratory Analysis Data
- Appendix D Test Protocol

1. Introduction

1.1 Summary of Test Program

Corteva Agriscience, LLC of Harbor Beach, Michigan (SRN B4942) contracted AECOM to conduct compliance testing the at the Wastewater Treatment Unit (WWTU) located in Harbor Beach, Michigan facility. The testing was completed to assess and update compliance with MI-ROP-B4942-2020a emission limits following the installation of the new V729 tank and to support emission factors used to develop emission estimates used in the Michigan State and Local Emissions Inventory System (SLEIS) and EPCRA TRI reporting. Testing occurred on September 9, 2024.

Table 1-1 summarizes the pertinent data for this compliance event.

Table 1-1. Compliance Summary

Responsible Groups	- Corteva Agriscience, LLC - Michigan Department of Environment, Great Lakes, and Energy (EGLE)
Applicable Regulations	- MI-ROP-B4942-2020a 40 CFR Part 63, Subparts A and MMM (PAI MACT) - Michigan State and Local Emissions Inventory System (SLEIS) - EPCRA TRI reporting
Plant Location	305 N Huron Ave. Harbor Beach, MI 48441
Sampling Contractor	AECOM 3700 James Savage Rd, 1602 Building Midland, MI 48642
Air Pollution Control Equipment	Enclosed Flare
Emission Points	- R-722 Condensate - Enclosed Flare Inlet
Pollutants/Diluent Measured	<u>R-722 Condensate:</u> - Methanol - Xylenes (m/o/p isomers) - Total Xylenes (mixed isomer) 1,2,4- Trimethylbenzene - Ammonia (NH₃) - Formaldehyde - Formic Acid - Total Sulfate Anion - Ammonium Cation - Sodium Cation <u>Unassisted Enclosed Flare:</u> - Flow Rate (Inlet) - BTU Content (Inlet) - TOC Concentration (Inlet) - Visible Emissions (Outlet)
Test Dates	September 9, 2024

1.2 Overview of the Test Program

WWTU Flare and Bioreactor R-722 measurements were collected while the process unit is operating at capacity (>90% maximum capacity) which equates to the worst-case emission profile for methanol. Corteva states methanol is the only hazardous air pollutant with an emission profile of any significance. Plant personnel are responsible for establishing capacity operations prior to sample collection and collecting all applicable unit-operating data. Data collected includes production rate, bioreactor vent gas flow rate, flare flame temperature verification, and any other process data, as needed.

Bioreactor treatment efficiency sampling was initially accomplished in 2004. In June 2013, Corteva performed sampling to confirm ongoing compliance with the fraction removal requirements. Condensate samples were collected during the July 2021 test event, to again confirm ongoing compliance with fraction removal requirements. Refer to the flowchart above. Samples will be collected at Point 1 (M9 Tank), Point 5 (V-728 Tank), and Point 15 (V-729 Tank).

1.2.1 WWTU / Bioreactor R-722 Overview

The goal of the test program for the Wastewater Treatment Unit (WWTU) was to collect condensate samples at “inlet” and “outlet” liquid streams. Liquid samples were collected into zero-headspace 40mL VOA vials concurrently at three sample points and analyzed for various constituents. The condensate samples were be shipped to Enthalpy Analytical in Durham, NC for laboratory analysis. The analysis consisted of the following:

- GC/FID analysis for methanol, o-xylene, m-xylene, p-xylene, 1,2,4-trimethylbenzene, and total xylenes
- HPLC/IC analysis for ammonia, formic acid, sulfate, ammonium, and sodium
- Generic spectrophotometric analysis for formaldehyde

The sample points utilized were:

- Point 1 (M9 Tank) (“inlet”)
- Point 5 (V728) (“outlet”)
- Point 15 (V-729) (“outlet”)

Test methodologies, sampling procedures, process descriptions, operating parameters, and quality assurance procedures are described later in this report.

1.2.2 Unassisted Enclosed Flare Overview

The goal of the test program for the enclosed flare was to measure the following:

- EPA Alt-080/073 for Enclosed Flare Inlet Vent Flow Rate
- EPA Method 18 for Enclosed Flare Inlet Total Organic Compounds (TOC)
- ASTM D1946-90 for Enclosed Flare Inlet Net Heating Value
- EPA Method 22 for Enclosed Flare Outlet Visible Emissions

1.3 Key Personnel

The contact for the source and test report is:

James McGee
989-479-5283
E: james.mcgee@corteva.com

Names and affiliations of personnel, including their roles in the test program, are summarized in the following table.

Table 1-2. Key Personnel

Role	Name	Affiliation	Address	Contact Information
Process and Environmental Focal Point	James McGee	Corteva Agriscience, LLC	305 N Huron Ave, Harbor Beach, MI 48441	james.mcgee@corteva.com 989-479-5283
Sample Project Leader	James Edmister	AECOM	3700 James Savage Rd, 1602 Building, Midland, MI	James.Edmister@aecom.com (585) 721-9128
Field Team Leader	Peter Becker	AECOM	3700 James Savage Rd, 1602 Building, Midland, MI	Peter.Becker@aecom.com (330) 285-6884
Technical Reviewer	Wayne Washburn	AECOM	13640 Briarwick Dr, Suite 200, Austin, TX	Wayne.Washburn@aecom.com (315) 447-0143
Technical Reviewer	Rob Sava	AECOM	564 White Pond Drive, Akron, OH	rob.sava@aecom.com 234-425-8440

1.4 Executive Summary

Tables 1-3 through 1-4 display the WWTU removal efficiencies of the various compounds listed in Table 1-1 above. Efficiencies are displayed as percent (%) removal. To the extent practical, removal efficiencies are displayed for the majority of the compounds. In the case where certain compounds were not present in significant concentrations at the inlet sample location, removal efficiencies are not purposeful (see discussion of results in Section 2).

Tables 1-5 through 1-7 display the WWTU concentrations of each of the various compounds listed in Table 1-1 for each individual liquid sample location. In the cases where the analyses resulted in non-detectable quantities, the analytical detection limit is displayed and is utilized in the removal efficiency calculations. In the cases where the analyses resulted in concentrations above the analytical detection limit but below the laboratory limit of quantification (LOQ), the analytical results are displayed (see discussion of results in Section 2).

Table 1-8 displays the enclosed Flare inlet flow Rate, inlet net heating Value, TOC results, and visible emissions results. Table 1-9 displays the process operating conditions during the testing.

The remainder of this document is organized as follows. **Section 2** of this document provides a summary and discussion of results for the test program, **Section 3** describes the sample locations, process descriptions, and the facility process information, **Section 4** describes the test procedures that were followed, **Section 5** describes the Quality Assurance/Quality control measures for the test program, and **Section 6** describes the data reduction.

Test program participants included: James Edmister and Peter Becker from AECOM, as well as James McGee from Corteva Agriscience, LLC.

Additional information is contained in the Appendices as follows:

Appendix A provides field data sheets from AECOM's test activities during the test program. **Appendix B** contains Facility Data and supporting documentation. **Appendix C** contains the laboratory analytical data. **Appendix D** contains the Test Protocol.

Table 1-3. Removal Efficiency - Point 1 (M9 Tank - Inlet) to Point 5 (V728 - Outlet)

Parameter	Average Point 1 (M9 Tank) (ug)	Average Point 5 (V728) (ug)	Removal (%)
Methanol	55793	10861	80.5
m-Xylene	ND	<2.49	N/A
o-Xylene	Below LOQ	<2.53	N/A
p-Xylene	24.8	<2.48	>90.0
Total Xylenes (mixed isomer)	29.3	<2.53	>91.4
1,2,4- Trimethylbenzene	73.9	<4.18	>94.3
Ammonia	404	<1.62	>99.6
Formaldehyde	Below LOQ	17.5	N/A
Formic Acid	4447	<673.1	>84.9
Total Sulfate Anion	116	81.5	29.7
Ammonium Cation	428	<1.72	>99.6
Sodium Cation	22829	25684	0.0
"<" indicates a result that is below the analytical detection limit.			
"Below LOQ" indicates a result that is between the detection limit and the laboratory limit of quantification (LOQ). The laboratory can positively identify the analyte of interest as present, but the value should be considered an estimate.			
">" indicates a result that includes an analytical result that is either below the detection limit or is a laboratory estimate (see notes "<" and "J" in tables below).			
"ND" indicates a result that is below the laboratory detection limit .			

Table 1-4. Removal Efficiency - Point 1 (M9 Tank - Inlet) to Point 15 (V729 - Outlet)

Parameter	Average Point 1 (M9 Tank) (ug)	Average Point 15 (V729) (ug)	System Removal (%)
Methanol	55793	9839	82.4
m-Xylene	ND	<2.73	N/A
o-Xylene	Below LOQ	<2.77	N/A
p-Xylene	24.8	<2.71	>89.1
Total Xylenes (mixed isomer)	29.3	<2.77	>90.5
1,2,4- Trimethylbenzene	73.9	<4.57	>93.8
Ammonia	404	<1.62	>99.6
Formaldehyde	Below LOQ	16.9	N/A
Formic Acid	4447	249	94.4
Total Sulfate Anion	116	336	0.0
Ammonium Cation	428	<1.72	>99.6
Sodium Cation	22829	25350	0.0
< indicates a result that is below the analytical detection limit.			
Below LOQ indicates a result that is between the detection limit and the laboratory limit of quantification (LOQ). The laboratory can positively identify the analyte of interest as present, but the value should be considered an estimate.			
> indicates a result that includes an analytical result that is either below the detection limit or is a laboratory estimate (see notes "<" and ">" in tables below).			
ND indicates a result that is below the laboratory detection limit .			

Table 1-5. Liquid Analysis Summary - Point 1 (M9 Tank - Inlet)

Parameter	Run 1 (ug)	Run 2 (ug)	Run 3 (ug)	Average (ug)
Methanol	57194	57657	52529	55793
m-Xylene	< 1.78	< 1.78	< 1.78	< 1.78
o-Xylene	J 8.18	J 2.26	J 2.87	J 4.44
p-Xylene	38.1	18.5	17.9	24.8
Total Xylenes (mixed isomer)	46.3	20.8	20.8	29.3
1,2,4- Trimethylbenzene	126.0	41.4	54.3	73.9
Ammonia	477	380	356	404
Formaldehyde	J 4.34	J 5.38	J 5.45	J 5.06
Formic Acid	4130	4608	4604	4447
Total Sulfate Anion	112	109	127	116.00
Ammonium Cation	505	403	377	428
Sodium Cation	23311	21767	23408	22829
"<" indicates a result that is below the analytical detection limit.				
"J" indicates a result that is between the detection limit and the laboratory limit of quantification (LOQ). The laboratory can positively identify the analyte of interest as present, but the value should be considered an estimate.				

Table 1-6. Liquid Analysis Summary - Point 5 (V728 - Outlet)

Parameter	Run 1 (ug)	Run 2 (ug)	Run 3 (ug)	Average (ug)
Methanol	11312	9781	11491	10861
m-Xylene	< 2.49	< 2.49	< 2.49	< 2.49
o-Xylene	< 2.53	< 2.53	< 2.53	< 2.53
p-Xylene	< 2.48	< 2.48	< 2.48	< 2.48
Total Xylenes (mixed isomer)	< 2.53	< 2.53	< 2.53	< 2.53
1,2,4- Trimethylbenzene	< 4.18	< 4.18	< 4.18	< 4.18
Ammonia	< 1.62	< 1.62	< 1.62	< 1.62
Formaldehyde	J 20.9	J 10.1	J 21.5	J 17.5
Formic Acid	< 21.4	295	1703	< 673.1
Total Sulfate Anion	189	32.1	23.4	81.5
Ammonium Cation	< 1.72	< 1.72	< 1.72	< 1.72
Sodium Cation	24926	25782	26345	25684
"<" indicates a result that is below the analytical detection limit.				
"J" indicates a result that is between the detection limit and the laboratory limit of quantification (LOQ). The laboratory can positively identify the analyte of interest as present, but the value should be considered an estimate.				

Table 1-7. Liquid Analysis - Point 15 (V729 - Outlet)

Parameter	Run 1 (ug)	Run 2 (ug)	Run 3 (ug)	Average (ug)
Methanol	9413	10241	9862	9839
m-Xylene	< 2.85	< 2.85	< 2.49	< 2.73
o-Xylene	< 2.89	< 2.89	< 2.53	< 2.77
p-Xylene	< 2.83	< 2.83	< 2.48	< 2.71
Total Xylenes (mixed isomer)	< 2.89	< 2.89	< 2.53	< 2.77
1,2,4- Trimethylbenzene	< 4.77	< 4.77	< 4.18	< 4.57
Ammonia	< 1.62	< 1.62	< 1.62	< 1.62
Formaldehyde	22.2	J 13.5	J 15.0	J 16.9
Formic Acid	J 199	J 192	356	J 249
Total Sulfate Anion	330	344	334	336
Ammonium Cation	< 1.72	< 1.72	< 1.72	< 1.72
Sodium Cation	25096	24951	26004	25350
*"<" indicates a result that is below the analytical detection limit.				
*"J" indicates a result that is between the detection limit and the laboratory limit of quantification (LOQ). The laboratory can positively identify the analyte of interest as present, but the value should be considered an estimate.				

Table 1-8. Unassisted Enclosed Flare Results

Parameter	Run 1	Run 2	Run 3	Average
Test Time	10:35 - 11:35	11:37 - 12:37	12:39 - 13:39	-
Flare Flow (lb/hr)	57.7	68.8	67.2	64.6
Inlet Net Heating Value (BTU/scf)	505	630	673	603
Outlet Visible Emissions (minutes)	0	0	0	0
Inlet TOC Concentration (%v as carbon)	72.21	70.31	77.37	73.30
Inlet Nonmethane TOC Concentration (ppmv as carbon)	<101.9	<68.3	<684.5	<284.9
Inlet Nonmethane/Nonethane TOC Concentration (ppmv as carbon)	<96.8	<65.2	<676.8	<279.6

Table 1-9. Process Operating Conditions

Parameter	Run 1	Run 2	Run 3	Average
Test Date	09/09/24	09/09/24	09/09/24	-
Test Time	10:35 - 11:35	11:37 - 12:37	12:39 - 13:39	-
Bed Temp [C] (TC-R722.PV)	34.8	34.9	35.0	34.9
Feed [gpm] (FC-R722-FD.PV)	76.2	80.1	80.0	78.8
pH out (AC-R722-EFF.PV)	7.37	7.37	7.37	7.37
pH In (AC-R722-PH.PV)	7.00	7.00	7.00	7.00
Flare Flow [lb/hr] (FI-R722.PV)	57.7	68.8	67.2	64.6
Flare Temp 2 [F] (BPCS-TT82802.PV)	385	393	387	388
Flare Temp 1 [F] (BPCS-TT82801.PV)	369	376	367	371

2. Summary and Discussion of Results

The purpose of this testing was to assess and update compliance with MI-ROP-B4942-2020a emission limits following the installation of the new V729 tank and to support emission factors used to develop emission estimates used in the Michigan State and Local Emissions Inventory System (SLEIS) and EPCRA TRI reporting. The specific objectives were to:

- Determine the WWTU concentrations of the compounds listed in Table 1-1 above at sample point 1 (M9 Tank – “inlet”), sample point 5 (V728 – “outlet”), and sample point 15 (V729 – “outlet”)
- Utilizing the above concentrations, determine the removal efficiencies (as applicable)
- Determine the Inlet Flow Rate, Inlet Net Heating Value, Inlet TOC Concentration, and Outlet Visible Emissions at the Unassisted Enclosed Flare

WWTU Flare and Bioreactor R-722 measurements were collected while the process unit is operating at capacity (>90% maximum capacity) which equates to the worst-case emission profile for methanol. Corteva states methanol is the only hazardous air pollutant with an emission profile of any significance. Plant personnel were responsible for establishing capacity operations prior to sample collection and collecting all applicable unit-operating data. Data collected includes production rate, bioreactor vent gas flow rate, flare flame temperature verification, and any other process data, as applicable.

2.1 Emission Test Results

2.1.1 WWTU / Bioreactor R-722 Overview

Liquid samples were collected into zero-headspace 40mL VOA vials concurrently at sample point 1 (M9 Tank – “inlet”), sample point 5 (V728 – “outlet”), and sample point 15 (V729 – “outlet”) and were analyzed for the compounds listed in Table 1-1. The analyses were completed utilizing GC/FID, HPLC/IC, and generic spectrophotometric procedures. The analytical procedures are detailed in Section 3 of this report.

Please note that removal efficiencies that resulted in a negative value are displayed as zero. Any Point 1 (inlet) sample location analytical results that were below laboratory detection limits (ND) or were below the laboratory limit of quantification (LOQ) are noted in the tables where appropriate. ND or LOQ results for the Point 1 sample location generate removal efficiency values that are not purposeful. In this case, the removal efficiency values are displayed as not applicable. Any Point 5 or Point 15 (“outlets”) sample location results that produced ND or LOQ are noted as “less than” (<) where applicable. These values result in “worst-case” removal efficiency values (noted as “>” where applicable).

2.1.2 Unassisted Enclosed Flare Overview

Inlet flow rate was measured utilizing EPA Alt-080/073. Permanently installed mass flow meters are installed to monitor the gas flow rate. Plant personnel are responsible for recording this data during the testing. TOC was determined utilizing EPA Method 18 on sample canisters that were filled onsite at a constant flow rate. Each of 3 test runs was conducted over the course of one hour. The inlet net heating value was also determined on the canister sample gas utilizing ASTM D1946-90. During the test periods, visible emissions were determined by EPA Method 22. The test procedures are detailed in Section 3 of this report.

AECOM field data is presented in **Appendix A**. Facility process data corresponding to the test run times are presented in **Appendix B**.

3. Facility Process and Sampling Location

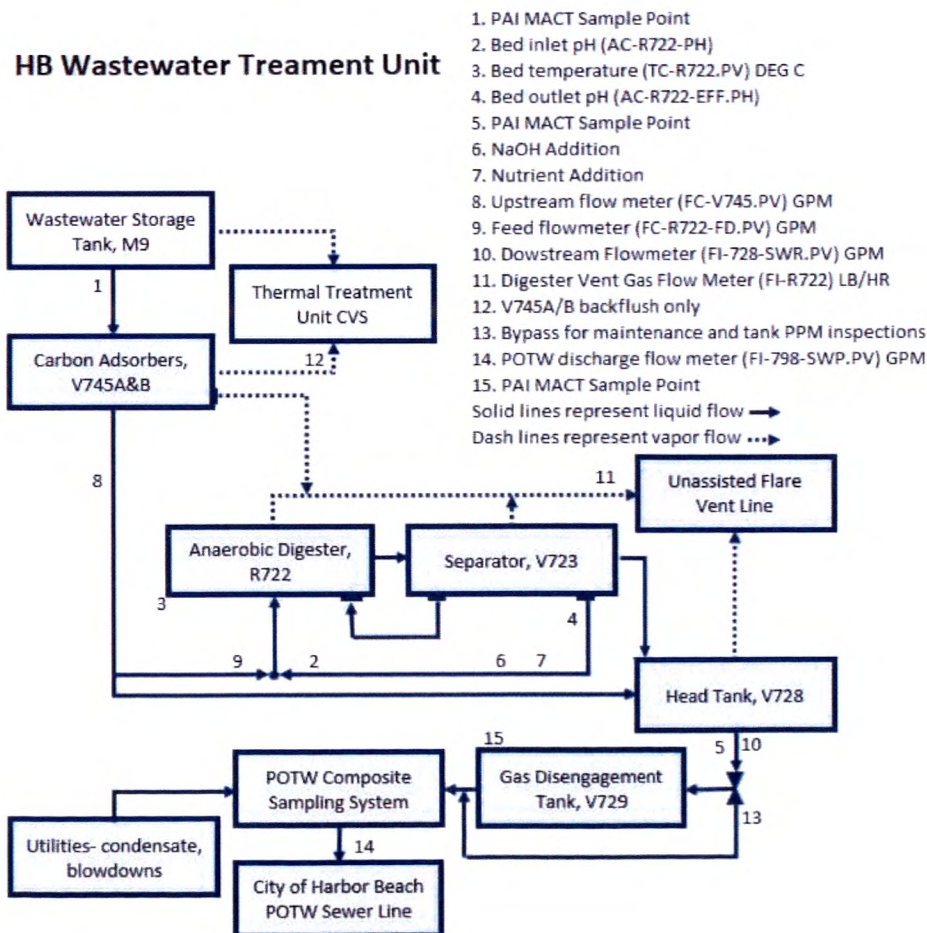
3.1 Process Description

3.1.1 Bioreactor R-722 Wastewater Treatment Process and Unassisted Enclosed Flare

This section briefly describes the WWTU system and pollution controls. The SRN B4942 Pesticide Active Ingredient (PAI) process unit consists of fermentation, product recovery crystallization, filtration, product drying and packaging, and waste broth processing equipment. Wastewater from the PAI process unit is directed to a closed anaerobic biological treatment process (Bioreactor R-722) to reduce the chemical oxygen demand (COD) and remove methanol.

Bioreactor R-722 is a recirculating fluidized bed reactor that utilizes microorganisms in an activated carbon media to anaerobically convert organic material in the presence of water into methane and carbon dioxide. The bioreactor water effluent is discharged to the local publicly owned treatment works (POTW). The exhaust gas from the bioreactor is primarily methane and carbon dioxide with lesser amounts of hydrocarbons and sulfide vapors. The methane gas generated from microorganism activity is vented to an enclosed flare. The flow chart below illustrates the wastewater treatment system.

Figure 3-1. WWTU Process Flow Chart



Gases collect in the headspace of the bioreactor and are exhausted to a non-assisted enclosed flare. Prior to the flare, the bioreactor vent gas is combined with a nitrogen purge stream and sent to a separator vessel for water removal. The bioreactor vent gas and nitrogen purge flow rates are measured prior to being combined using separate permanently installed mass flow meters. Bioreactor vent gas flow is measured using a thermal mass flow meter and automated control valve. The nitrogen flow is maintained using a mass flow meter.

3.2 Applicable Regulations and Performance Requirements

Applicable Regulations

- MI-ROP-B4942-2020a
- 40 CFR Part 63, Subparts A and MMM (PAI MACT)
- Michigan State and Local Emissions Inventory System (SLEIS)
- EPCRA TRI

Measurements – WWTU Condensate

- GC/FID analysis for methanol, o-xylene, m-xylene, p-xylene, 1,2,4-trimethylbenzene, and total xylenes
- HPLC/IC analysis for ammonia, formic acid, sulfate, ammonium, and sodium
- Generic spectrophotometric analysis for formaldehyde

Measurements – Unassisted Enclosed Flare

- EPA Alt-080/073 for Enclosed Flare Inlet Vent Flow Rate
- EPA Method 18 for Enclosed Flare Inlet Total Organic Compounds (TOC)
- ASTM D1946-90 for Enclosed Flare Inlet Net Heating Value
- EPA Method 22 for Enclosed Flare Outlet Visible Emissions

3.3 Process Emissions Control Description

Wastewater from the PAI process unit is directed to a closed anaerobic biological treatment process (Bioreactor R-722) to reduce the chemical oxygen demand (COD) and remove methanol. Bioreactor R-722 is a recirculating fluidized bed reactor that utilizes microorganisms in an activated carbon media to aerobically convert organic material in the presence of water into methane and carbon dioxide. The bioreactor water effluent is discharged to the local publicly owned treatment works (POTW). The exhaust gas from the bioreactor is primarily methane and carbon dioxide with lesser amounts of hydrocarbons and sulfide vapors. The methane gas generated from microorganism activity is vented to an enclosed flare.

4. Test Procedures

The following is a description of the testing procedures that were completed at the WWTU and Enclosed Flare sampling locations to fulfill data quality objectives of this compliance sampling project. A typical canister sampling train schematic is shown in Figure 4-1.

4.1 EPA Alt-080

EPA Alt-080: Determination of Flow Rate - Alternative Procedure Utilizing an Ultrasonic Flow Meter

This procedure is an alternative to EPA Method 2 for flow rate measurement. Use an ultrasonic flow meter in lieu of Method 2, 2A, 2C, or 2D to measure the flare exit flow rate. The calibration certification is to be kept in compliance with the manufacturer recommended frequency.

4.2 EPA Alt-073

EPA Alt-073: Determination of Flow Rate - Alternative Procedure Utilizing a Mass Flow Meter

This procedure is an alternative to EPA Method 2 for flow rate measurement. Use a mass flow meter in lieu of Method 2, 2A, 2C, or 2D to measure the flare exit flow rate. The calibration certification is to be kept in compliance with the manufacturer recommended frequency.

4.3 EPA Method 18

EPA Method 18: Measurement of Gaseous Organic Compound Emissions by Gas Chromatography

The major organic components of a gas mixture are separated by gas chromatography (GC) and individually quantified by flame ionization, photoionization, electron capture, or other appropriate detection principles. The retention times of each separated component are compared with those of known compounds under identical conditions. Therefore, the analyst confirms the identity and approximate concentrations of the organic emission components beforehand. With this information, the analyst then prepares or purchases commercially available standard mixtures to calibrate the GC under conditions identical to those of the samples. The analyst also determines the need for sample dilution to avoid detector saturation, gas stream filtration to eliminate particulate matter, and prevention of moisture condensation.

4.4 EPA Method 22

EPA Method 22: Visual Determination of Fugitive Emissions from Material Sources and Smoke Emissions from Flares

This method is applicable for the determination of the frequency of fugitive emissions from stationary sources. This method also is applicable for the determination of the frequency of visible smoke emissions from flares used for combustion of waste process materials. This method determines the amount of time that visible emissions occur during the observation period (the accumulated emission time).

4.5 Spectrophotometric Analysis

Spectrophotometric Analysis: Determination of Compounds by Spectrophotometric Procedure

Different chemical compounds absorb light at specific wavelengths, allowing measurement of the intensity of light absorbed by a sample. This analysis is used to determine the concentration of specific compounds in a sample. Light is passed through a sample solution and the intensity of the transmitted light is measured.

4.6 HPLC/IC analysis

HPLC/IC analysis: High Performance Liquid Chromatography / Ion Chromatography

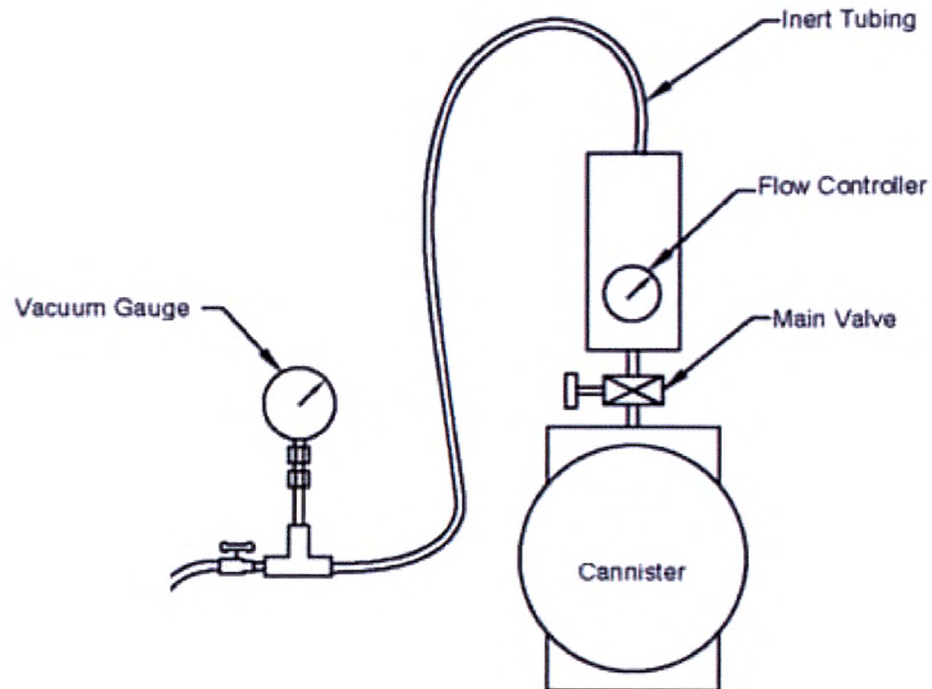
This procedure is used to separate compounds in a mixture and then identify and quantify each compound by high performance liquid chromatography / ion chromatography.

4.7 ASTM D1946-90

ASTM D1946-90: Standard Practice for Analysis of Reformed Gas by Gas Chromatography

This method is used in the determination of the chemical composition of gaseous mixtures containing hydrogen, oxygen, nitrogen, carbon monoxide, carbon dioxide, methane, ethane, and ethylene by gas chromatography.

Figure 4-1. Canister Sampling System Schematic



5. Quality Assurance / Quality Control Measures

5.1 Overview

To ensure accurate and defensible results, strict quality assurance and quality control measures were followed. Testing was performed following standard EPA protocol as outlined in 40 CFR, Part 60, Appendix A. Performance testing was performed while the process was operating at a minimum of 90% of the maximum-rated capacity, or as near thereto as practicable. Process data from the enclosed flare and bioreactor process is compiled in the above report and the following report appendix.

Test criteria were thoroughly documented and checked for completeness. Visible emissions were observed and recorded in accordance with EPA Method 22, as applicable. The flare inlet TOC samples were collected and analyzed in accordance with EPA Method 18 and ASTM Method D1946-90. Method performance specifications were met. Calibration results as well as data sheets and sampling logs produced during testing are documented in the appendix of this report.

Additional laboratory QA/QC measures are documented in the Laboratory Data appendix of this report.

Table 5-1. Manual Method Performance Checks

Activity	EPA Method	Criterion
Sample Train Sampling Rate	18	The sampling rate set at ~0.1 Lpm during each run while sampling into 6L SUMMA canisters.

6. Data Reduction

6.1 Overview

Data have been reduced to fulfill the data quality objective of the test program. Photocopies of the field data sheets and data printouts are also presented in the appendices, as applicable.