



RECEIVED

JAN 16 2019

AIR QUALITY DIVISION

# Vapor Recovery Unit Emissions Test Report

*Prepared for:*

**Allnex USA Inc.**

Kalamazoo, Michigan

2715 Miller Road  
Kalamazoo, Michigan

Project No. 049AS-376666  
January 3, 2019

BT Environmental Consulting, Inc.  
4949 Fernlee Avenue  
Royal Oak, Michigan 48073  
(248) 548-8070

**EXECUTIVE SUMMARY**

BT Environmental Consulting, Inc. (BTEC) was retained by Allnex USA Inc. (Allnex) to conduct a methanol (MeOH), formaldehyde (HCOH), methylal, and methyl formate emissions test program at the Allnex facility in Kalamazoo, Michigan. The purpose of this document is to present the test report for this emissions test program.

The purpose of the emissions test program was to measure MeOH, HCOH, methylal, and methyl formate emission rates at the inlet and outlet of the Vapor Recovery Unit (VRU) for engineering evaluation purposes.

Testing of the VRU inlet and outlet consisted of triplicate test runs, each for the duration of the batch cycle. The testing was completed on August 29, 2018 with a cryogenic condenser set point temperature of -35°C while running fresh methanol through the system. The results of the VRU emissions test program are summarized by Table I.

**Table I  
VRU System Test Results**

Hazardous Air Pollutant	Run 1 10:30-11:30		Run 2 12:10-13:10		Run 3 13:30-14:30	
	Emission Rate (lbs/hr)		Emission Rate (lbs/hr)		Emission Rate (lbs/hr)	
	Inlet	Outlet	Inlet	Outlet	Inlet	Outlet
Formaldehyde	<0.001	<0.001	<0.001	<0.001	<0.001	<0.001
Methanol	25.91	0.375	12.75	0.029	13.36	0.298
Methylal	87.48	1.133	36.23	0.310	98.89	3.916
Methyl Formate	0.403	0.034	0.245	0.005	0.430	0.039

\*Note: Due to equipment calibrations and data acquisition downloads, data is not available for the following intervals during the test program: Run 1 – 11:14:02 to 11:14:22 and Run 2 – 12:23:02 to 12:24:05

The results listed in Table I yield a 3-test average hazardous air pollutant (HAP) removal efficiency of 98.7%.

**RECEIVED**  
JAN 16 2019  
AIR QUALITY DIVISION

**TABLE OF CONTENTS**

**A. INTRODUCTION ..... 1**

    A.I EMISSIONS TEST RESULTS..... 1

    A.II PROCESS AND CONTROL EQUIPMENT DATA RELATED TO CALCULATING EMISSION RATES . 1

    A.III TEST ERRORS DISCUSSION ..... 2

    A.IV DEVIATIONS FROM REFERENCE TEST METHODS ..... 2

    A.V PRODUCTION DATA ..... 2

**B. FACILITY OPERATIONS..... 2**

    B.I PROCESS DESCRIPTION..... 2

    B.II EMISSIONS CONTROL OPERATING PARAMETERS ..... 3

    B.III FACILITY OPERATING PARAMETERS ..... 3

**C. SAMPLING AND ANALYTICAL PROCEDURES..... 3**

    C.I SAMPLING PORTS..... 3

    C.II SAMPLING POINT DESCRIPTION..... 3

    C.III SAMPLING PROCEDURE DESCRIPTION ..... 4

    C.IV METHOD DEVIATIONS ..... 4

    C.V ANALYTICAL PROCEDURES ..... 6

**D APPENDICES ..... 6**

    D.I RESULTS AND EXAMPLE CALCULATIONS..... 6

    D.II RAW FIELD DATA ..... 6

    D.III LABORATORY REPORT ..... 6

    D.IV CALIBRATION ..... 6

    D.V PROCESS AND CONTROL EQUIPMENT DATA ..... 6

    D.VI TEST LOG ..... 6

    D.VII PROJECT PERSONNEL ..... 6

    D.VIII RELATED CORRESPONDENCE..... 6

**FIGURES**

- Figure 1 – Simplified Process Flow Diagram
- Figure 2 – VRU Inlet Sampling Location Diagram
- Figure 3 – VRU Outlet Sampling Location Diagram

**APPENDICES**

- Appendix D.i Results and Example Calculations
- Appendix D.ii Raw Field Data
- Appendix D.iii Laboratory Report
- Appendix D.iv Equipment Calibration Documents
- Appendix D.v Process and Control Equipment Data
- Appendix D.vi Test Log Information
- Appendix D.vii Project Participants and Titles
- Appendix D.viii Related Correspondence

**A. Introduction**

BT Environmental Consulting, Inc. (BTEC) was retained by Allnex USA Inc. (Allnex) to conduct a methanol (MeOH), formaldehyde (HCOH), methylal, and methyl formate emissions test program at the Allnex facility in Kalamazoo, Michigan. The purpose of this document is to present the test report for this emissions test program.

The purpose of the emissions test program was to measure MeOH, HCOH, methylal, and methyl formate emission rates at the inlet and outlet of the Vapor Recovery Unit (VRU) for engineering evaluation purposes while running fresh methanol through the VRU system.

**A.i Emissions Test Results**

Testing of the VRU inlet and outlet consisted of triplicate 60-minute test runs. The testing was completed on August 29, 2018 with cryogenic condenser set point temperature of -35°C while running fresh methanol through the system. The results of the VRU emissions test program are summarized by Table 1.

**Table 1  
VRU System Test Results**

Hazardous Air Pollutant	Run 1 10:30-11:30		Run 2 12:10-13:10		Run 3 13:30-14:30	
	Emission Rate (lbs/hr)		Emission Rate (lbs/hr)		Emission Rate (lbs/hr)	
	Inlet	Outlet	Inlet	Outlet	Inlet	Outlet
Formaldehyde	<0.001	<0.001	<0.001	<0.001	<0.001	<0.001
Methanol	25.91	0.375	12.75	0.029	13.36	0.298
Methylal	87.48	1.133	36.23	0.310	98.89	3.916
Methyl Formate	0.403	0.034	0.245	0.005	0.430	0.039

\*Note: Due to equipment calibrations and data acquisition downloads, data is not available for the following intervals during the test program: Run 1 – 11:14:02 to 11:14:22 and Run 2 – 12:23:02 to 12:24:05

The results listed in Table I yield a 3-test average hazardous air pollutant (HAP) removal efficiency of 98.7%.

**A.ii Process and Control Equipment Data Related to Calculating Emission Rates**

During the testing period, the following process and control equipment data relevant for the calculation of emissions rates was monitored: the stack gas velocity; concentrations of methanol, formaldehyde, methylal, and methyl formate; stack gas temperature; and moisture content. This data is included in Appendix D.i.

### **A.iii Test Errors Discussion**

No errors were observed during the emissions test program.

### **A.iv Deviations from Reference Test Methods**

Testing was conducted using Methods 1, 2, 3, 4, and 320, with specific deviations as set forth in previous test protocols that had been approved by U.S. EPA. There were no deviations from the previously approved test protocols during the testing except that there were several short periods of time when data is not available due to equipment calibrations and data acquisition downloads. Specific test methodology is described more fully in Sections C.iii and C.iv.

### **A.v Production Data**

The feed rate to LUWA 1 was set at 34 gallons per minute for the stack test and is representative of normal operating conditions. This rate did not vary significantly from the set point over the course of the testing. Production data recorded during the emissions test program is provided in Appendix D.v.

## **B. Facility Operations**

Sections B.i through B.iii provide a description of facility operations.

### **B.i Process Description**

The Methylated Resins Plant at Kalamazoo is permitted to produce 76.6 million pounds per year of Cymel® 303LF. Cymel® 303LF is a clear, liquid product produced by reacting melamine with HCOH and MeOH. Figure 1 provides a simplified process flow diagram for the Methylated Resins process.

In general, the Cymel® 303LF resin production process consists of various liquid and solid material storage vessels, and other containers from which raw materials are charged to a batch reactor. After numerous batch reactor process steps, an intermediate resin is transferred to intermediate storage tanks for subsequent transfer to the continuous portion of the process, which includes a first strip, second reaction, second strip and solids separation. During the stripping steps, volatiles are separated from the product. Following solids separation, the Cymel® 303LF product is then transferred through filtration to product bulk storage tanks.

Dilute exhaust streams from the Methylated Resins manufacturing process (e.g., elephant trunk exhausts and other area ventilation) are exhausted to a water scrubber. Concentrated exhaust streams are vented to the methanol seal pot and then through a methanol scrubber and cryogenic condenser operating in series. From the cryogenic condenser, exhaust gases are discharged to a single rooftop exhaust stack.

## **B.ii Emissions Control Operating Parameters**

VRU operating data that are continuously recorded include:

- Cryogenic Condenser exit gas temperature
- Feed rate to First Stripper
- Methanol feed rate to the methanol scrubber
- Methanol Recirculation Rate

Data recorded during the emissions test program is provided in Appendix D.v.

## **B.iii Facility Operating Parameters**

The LUWA 1 set point of 34 gpm is the operating parameter representative of operation. As demonstrated by the data provided in Appendix D.v, the LUWA 1 set point during the test was 34 gpm, and the feed rate did not vary significantly from this level. Testing further involved three one-hour runs over the course of two batches and included all portions of the entire batch cycle.

For the three VRU test runs with a cryogenic condenser operating temperature of -35°C and fresh methanol being fed into the system (on August 29, 2018), the first batch started at 9:50AM and ended at 12:15PM. The second batch started at 12:16PM and ended at 14:30PM.

Process operating conditions for each test run are summarized in Appendix D.i.

## **C. Sampling and Analytical Procedures**

Sections C.i through C.v summarize the emissions test program Sampling and Analytical Procedures.

### **C.i Sampling Ports**

The sampling locations are illustrated by Figures 2 and 3. The exhaust gas pipe at both VRU sampling locations is six inches in diameter.

### **C.ii Sampling Point Description**

The VRU sampling locations were designated the inlet and outlet sampling locations. Exhaust gas velocity was measured at the center of the pipe at both inlet and outlet and exhaust gas samples were extracted from the center of the pipe.

### **C.iii Sampling Procedure Description**

Sampling and analysis procedures followed the requirements codified at Title 40, Part 60, Appendix A of the Code of Federal Regulations (40 CFR 60, Appendix A) and 40 CFR 63, Appendix A:

- Method 1 - *“Sample and Velocity Traverses for Stationary Sources”* was used to determine the sampling locations.
- Method 2 - *“Determination of Stack Gas Velocity and Volumetric Flowrate”* was used to measure exhaust gas velocity.
- Method 3 - *“Gas Analysis for the Determination of Dry Molecular Weight (Fyrite Analysis)”* was used to determine exhaust gas molecular weight.
- Method 4 - *“Determination of Moisture Content in Stack Gases”* was used to determine exhaust gas moisture content.
- Method 320 - *“Measurement of Vapor Phase Organic and Inorganic Emissions by Extractive Fourier Transform Infrared Spectroscopy”* was used to measure exhaust gas MeOH, HCOH, methylal, and methyl formate concentrations.

### **C.iv Method Deviations**

There were no deviations from test protocols that had been approved for previous VRU testing. With respect to the methods listed in Section C.iii, the following method variations were used during the VRU test program:

- Because MeOH concentrations were very elevated at the VRU inlet sampling location and because inlet and outlet MeOH concentrations were variable as well as the exhaust gas flowrate being variable, MeOH concentrations were determined using Method 320 instead of Methods 18 or 308.
- Because the exhaust gas flowrate through VRU system is variable and because the VRU inlet and outlet sampling locations are only 6 inches in diameter, exhaust gas flowrate was measured using stationary pitot tubes fixed in position at the center of the stack. Figures 2 and 3 illustrate the locations of the velocity pressure sampling locations. Figures 2 and 3 also illustrate the Method 320 sampling locations.
- Because of the configuration of the stack test ports, BTEC used a small S-type pitot tube rather than a standard pitot tube.
- Velocity pressure was measured using differential pressure transmitters with a range of 0 to 1 inch of water and the velocity pressure was datalogged at one

second intervals. The specified accuracy of the differential pressure transmitters was +/- 0.0025 inches of water.

- Because the exhaust flowrates and gas characteristics were variable, prior to the first test run and after the last test run, the sampling locations were checked for cyclonic flow at the center of the duct.
- Prior to and after each test run, pitot tube leak checks were conducted.
- The accuracy of the differential pressure transmitters were verified at BTEC's office in Royal Oak, Michigan before the test program. Tubing was teed to a manometer and the manometer readings checked against the high and low pressure sides of both transmitters. Each transmitter side was checked at four levels (zero, low, mid, and high) three times with values recorded on calibration data sheets.
- The static pressure at the sampling locations was measured once before the beginning of the emissions test program and once at the end of the emissions test program.
- Exhaust gas temperature was measured and recorded at ten minute intervals during each test run.
- Exhaust gas moisture content was measured by Fourier Transform Infrared Spectroscopy (FTIR) used to analyze for exhaust gas HCOH, MeOH, methylal, and methyl formate concentrations.
- Bag grab samples were collected at the exhaust from the FTIR unit and analyzed for O<sub>2</sub> content using a Fyrite analyzer. Exhaust gas molecular weight was determined from the measured O<sub>2</sub> content as well as concentration data for other compounds as measured by the FTIR used to analyze for exhaust gas HCOH, MeOH, methylal, and methyl formate concentrations.
- FTIR data will be recorded at an interval of 8 seconds during the emissions test program.
- Because of the high methanol concentrations at the inlet and outlet sampling locations, the Method 320 MeOH and HCOH analyte spikes were performed in ambient air as opposed to the sample stream. This approach confirmed the FTIR/sampling system to accurately deliver and quantify a known concentration of MeOH.



## **C.v Analytical Procedures**

The emissions test program did not include collected samples. Analytical procedures for the on-site Method 320 analyses is included in the Prism Analytical Technologies report included in Appendix D.iii.

## **D Appendices**

Sections D.i through D.vii provide identification of Appendices for the corresponding information.

### **D.i Results and Example Calculations**

Detailed test results are summarized by Tables 2 and 3 in Appendix D.i. Example calculations are also provided in Appendix D.i.

### **D.ii Raw Field Data**

Raw field data are provided in Appendix D.ii.

### **D.iii Laboratory Report**

The Method 320 FTIR report from Prism Analytical Technologies is included in Appendix D.iii.

### **D.iv Calibration**

Equipment calibration documents for the Method 320 FTIR analysis are included in the Prism Analytical Technologies report included in Appendix D.iii. Exhaust gas flowrate equipment calibration documents are included in Appendix D.iv.

### **D.v Process and Control Equipment Data**

Raw process and control equipment data is provided in Appendix D.v.

### **D.vi Test Log**

The test log is summarized by the field notes and data sheets included in Appendix D.ii.

### **D.vii Project Personnel**

Project personnel are summarized by Table 4 in Appendix D.vii.

### **D.viii Related Correspondence**

Correspondence related to the emissions test program is provided in Appendix D.viii.

# Figures

# Methylated Resins Department

Vapor Recovery And Scrubbing Process

7.2 How Is The Vapor Recovery And Scrubbing Process Operated? – Integrated Flows And Controls

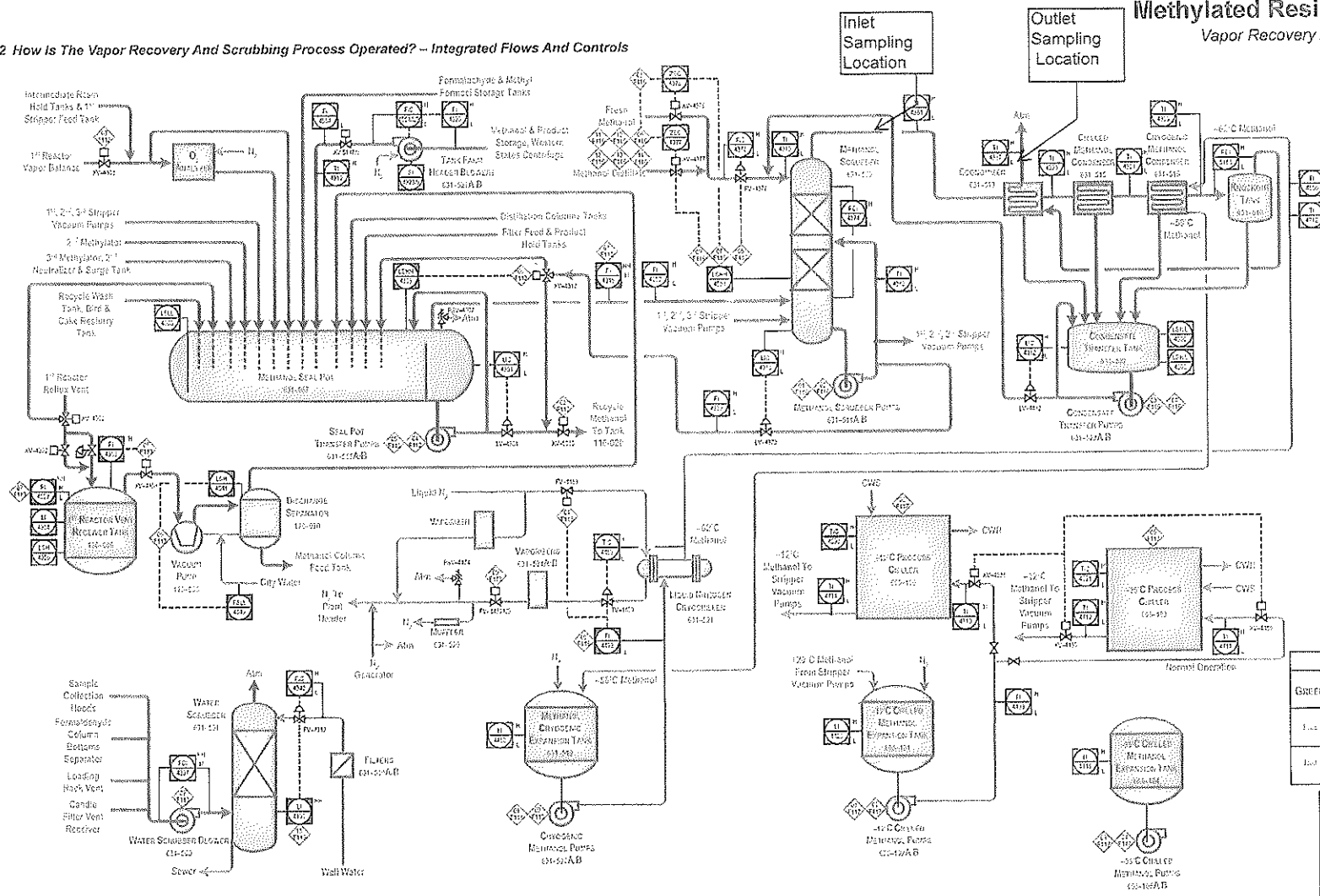
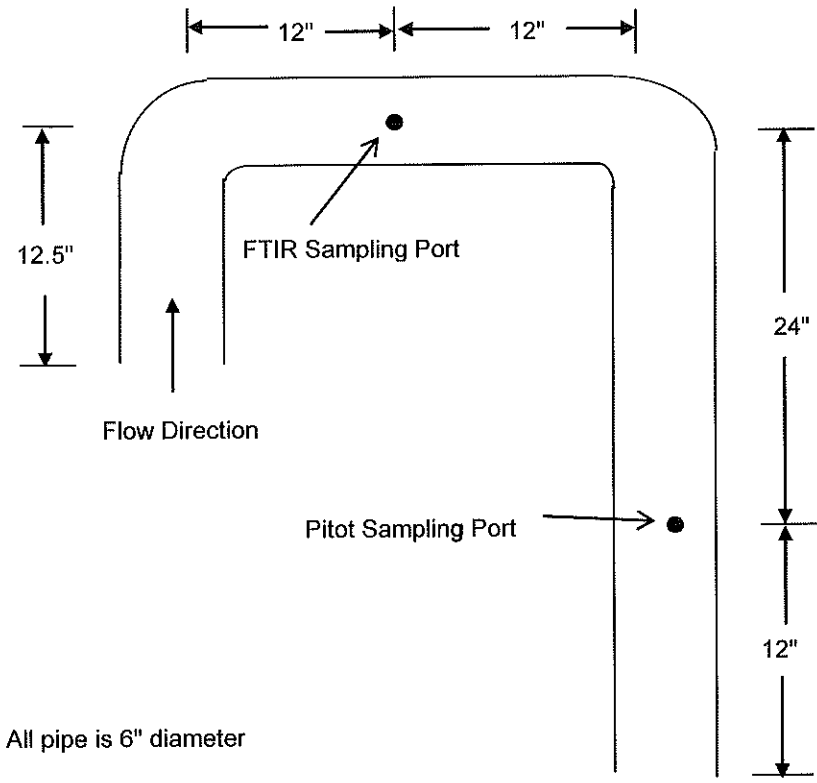


Fig. 1 Vapor Recovery And Scrubbing Process Integrated Flows And Controls Diagram



LEGEND	
GREFF	Equipment
1.1.1	Primary (Process) Pipe
1.1.2	Secondary (Dist. Process) Pipe

LEGEND	
1.1.1	129 C Methanol From Stripper Vacuum Pumps
1.1.2	129 C Methanol From Stripper Vacuum Pumps
1.1.3	129 C Methanol From Stripper Vacuum Pumps
GREFF	Equipment



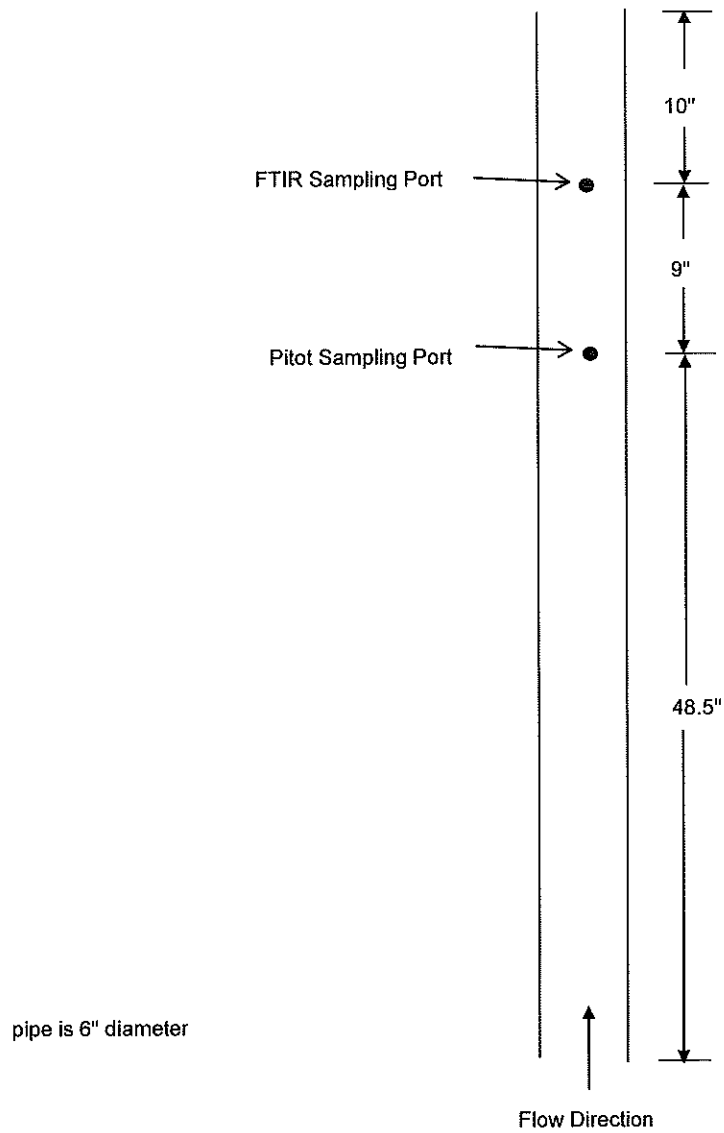
DRAWING IS NOT TO SCALE

Figure 2

Site:  
VRU Inlet Sampling Location  
Allnex USA Inc.  
Kalamazoo, Michigan

Sampling Date:  
August 29, 2018

BT Environmental Consulting, Inc.  
4949 Fernlee Avenue  
Royal Oak, Michigan



DRAWING IS NOT TO SCALE

**Figure 3**

**Site:**  
VRU Outlet Sampling Location  
Allnex USA Inc.  
Kalamazoo, Michigan

**Sampling Date:**  
August 29, 2018

**BT Environmental Consulting, Inc.**  
4949 Fernlee Avenue  
Royal Oak, Michigan

**Appendix D.i**  
**Results and Sample Calculations**

**Table 2**  
**Methanol and Formaldehyde Emission Test Results**  
**Vapor Recovery Unit Inlet Sampling Location**  
**Allnex USA Inc.**  
**Kalamazoo, Michigan**

Test Run	Test Run Time	Average Stack Exhaust Gas Temperature (°F)	Exhaust Stack Gas Static Pressure (in. Hg)	Exhaust Gas Molecular Weight (lbs/lb-mole)	Average Formaldehyde Concentration (ppmv)	Average Methanol Concentration (ppmv)	Average Methylal Concentration (ppmv)	Average Methyl Formate Concentration (ppmv)	Exhaust Gas Flowrate (acfm)	Exhaust Gas Flowrate (scfm)	Formaldehyde Emission Rate (lbs/hr)	Methanol Emission Rate (lbs/hr)	Methylal Emission Rate (lbs/hr)	Methyl Formate Emission Rate (lbs/hr)
1	10:30-11:30	86.0	29.2	35.69	< 6	130,513	127,733	808	50	47	< 0.001	25.9	87.5	0.4
2	12:10-13:10	85.0	29.2	33.32	< 2	95,197	103,608	1,043	28	26	< 0.001	12.8	36.2	0.2
3	13:30-14:30	87.0	29.2	35.83	< 2	65,028	140,307	1,134	46	43	< 0.001	13.4	98.9	0.4
3-Test Averages:		86.0	29.2	34.95	< 3	96,913	123,883	995	41	39	< 0.001	17.3	74.2	0.4

Table 3  
**Methanol and Formaldehyde Emission Test Results**  
**Vapor Recovery Unit Outlet Sampling Location**  
**Allnex USA Inc.**  
**Kalamazoo, Michigan**

Test Date	Test Run	Test Run Time	Average Stack Exhaust Gas Temperature (°F)	Exhaust Stack Gas Static Pressure (in. Hg)	Exhaust Gas Molecular Weight (lbs/lb-mole)	Average Formaldehyde Concentration (ppmv)	Average Methanol Concentration (ppmv)	Average Methylal Concentration (ppmv)	Average Methyl Formate Concentration (ppmv)	Exhaust Gas Flowrate (acfm)	Exhaust Gas Flowrate (scfm)	Formaldehyde Emission Rate (lbs/hr)	Methanol Emission Rate (lbs/hr)	Methylal Emission Rate (lbs/hr)	Methyl Formate Emission Rate (lbs/hr)
August 29, 2018	1	10:30-11:30	76.0	29.2	29.26	< 2	4.722	4.965	252	14	14	< 0.001	0.38	1.13	0.03
	2	12:10-13:10	69.0	29.2	29.80	< 2	4.904	19.538	316	1	1	< 0.001	0.03	0.31	0.005
	3	13:30-14:30	64.0	29.2	30.02	< 2	4.201	23.753	285	14	14	< 0.001	0.30	3.92	0.04
3-Test Averages:			69.7	29.2	29.69	< 2	4.609	16.085	284	10	10	< 0.001	0.23	1.79	0.03

Permit Limits (lbs/hr)	
HCOH	1.3
MeOH	6.1