



Vapor Recovery Unit and Water Scrubber Emissions Test Report

Prepared for:

Allnex USA Inc.

Kalamazoo, Michigan

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AIR QUALITY DIV.

2715 Miller Road
Kalamazoo, Michigan

Project No. 17-5008.00
July 10, 2017

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Royal Oak, Michigan 48073
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EXECUTIVE SUMMARY

BT Environmental Consulting, Inc. (BTEC) was retained by Allnex USA Inc. (Allnex) to conduct a methanol (MeOH) and formaldehyde (HCOH) 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 and HCOH emission rates at the inlet and outlet of the Vapor Recovery Unit (VRU) as well as at the inlet and outlet of the water scrubber as required by the U.S. EPA Administrative Consent Order (ACO) dated January 25, 2017. The emissions test program included performance of the compliance demonstration procedures for large control devices for the vapor recovery unit (VRU), which consists of the methanol scrubber – Equipment ID 631-509, and cryogenic condenser – Equipment ID 631-516. In addition, as required by the ACO, the emissions test program included performance of the compliance demonstration procedures for large control devices for the water scrubber – Equipment ID 631-501.

Testing of the VRU inlet and outlet consisted of triplicate 60-minute test runs completed on May 16, 2017 with a cryogenic condenser set point temperature of -50°C (Test Runs 1, 2, and 3) and triplicate 60-minute test runs completed on May 17, 2017 with a cryogenic condenser set point temperature of -30°C (Test Runs 4, 5, and 6). The results of the VRU emissions test program are summarized by Table I.

**Table I
VRU System Test Results**

Test Run	Test Run Time	Inlet Formaldehyde Emission Rate (lbs/hr)	Inlet Methanol Emission Rate (lbs/hr)	Outlet Formaldehyde Emission Rate (lbs/hr)	Outlet Methanol Emission Rate (lbs/hr)
1	10:04 - 11:04	0.006	58.025	0.0003	0.5987
2	11:50 - 12:50	0.006	51.512	0.0003	0.3254
3	13:35 - 14:35	0.007	56.402	0.0003	0.3882
3-Test Averages:		0.006	55.313	0.0003	0.4374
4	9:39 - 10:39	0.007	60.194	0.0004	1.8094
5	11:24 - 12:24	0.006	53.823	0.0005	1.6068
6	13:45 - 14:45	0.016	104.60	0.0005	1.3616
3-Test Averages:		0.010	72.871	0.0005	1.5926

The results listed in Table I yield a 3-test average hazardous air pollutant (HAP) removal efficiency of 99.2% for Test Runs 1, 2, and 3 (-50°C) and 97.6% for Test Runs 4, 5, and 6 (-30°C).

Testing of the water scrubber inlet and outlet consisted of triplicate 60-minute test runs completed on May 17, 2017 with a water scrubber water flowrate set point of 35 gallons

per minute (gpm), triplicate 60-minute test runs completed on May 18, 2017 with a water scrubber water flowrate set point of 25 gpm, and triplicate 60-minute test runs completed on May 18, 2017 with a water scrubber water flowrate set point of 15 gpm. The results of the water scrubber emissions test program are summarized by Table II.

**Table II
Water Scrubber Test Results**

Test Run	Test Run Time	Inlet Formaldehyde Emission Rate (lbs/hr)	Inlet Methanol Emission Rate (lbs/hr)	Outlet Formaldehyde Emission Rate (lbs/hr)	Outlet Methanol Emission Rate (lbs/hr)
1	16:00 - 17:00	1.13	1.92	0.01	0.01
2	17:08 - 18:08	1.20	1.95	0.01	0.01
3	18:15 - 19:15	1.82	2.64	0.01	0.01
3-Test Averages:		1.39	2.17	0.01	0.01
4	9:45 - 10:45	1.43	6.41	0.01	0.01
5	10:55 - 11:55	0.90	2.11	0.01	0.01
6	12:02 - 13:02	0.90	1.83	0.01	0.01
3-Test Averages:		1.08	3.45	0.01	0.01
7	13:09 - 14:09	1.25	2.12	0.01	0.01
8	14:16 - 15:16	1.02	2.35	0.01	0.02
9	15:23 - 16:23	1.49	2.31	0.01	0.04
3-Test Averages:		1.25	2.26	0.01	0.02

The results listed in Table II yield a 3-test average hazardous air pollutant (HAP) removal efficiency of 99.5% for Test Runs 1, 2, and 3 (35 gpm) and 99.5% for Test Runs 4, 5, and 6 (25 gpm) and 99.2% for Test Runs 7, 8, and 9 (15 gpm).

A. Introduction

BT Environmental Consulting, Inc. (BTEC) was retained by Allnex USA Inc. (Allnex) to conduct a methanol (MeOH) and formaldehyde (HCHO) 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 and HCHO emission rates at the inlet and outlet of the Vapor Recovery Unit (VRU) as well as at the inlet and outlet of the water scrubber as required by the U.S. EPA Administrative Consent Order (ACO) dated January 25, 2017. The emissions test program included performance of the compliance demonstration procedures for large control devices for the vapor recovery unit (VRU), which consists of the methanol scrubber – Equipment ID 631-509, and cryogenic condenser – Equipment ID 631-516. In addition, as required by the ACO, the emissions test program included performance of the compliance demonstration procedures for large control devices for the water scrubber – Equipment ID 631-501.

A.i Emissions Test Results

Testing of the VRU inlet and outlet consisted of triplicate 60-minute test runs completed on May 16, 2017 with a cryogenic condenser set point temperature of -50°C (Test Runs 1, 2, and 3) and triplicate 60-minute test runs completed on May 17, 2017 with a cryogenic condenser set point temperature of -30°C (Test Runs 4, 5, and 6). The results of the VRU emissions test program are summarized by Table 1.

**Table 1
VRU System Test Results**

Test Run	Test Run Time	Inlet Formaldehyde Emission Rate (lbs/hr)	Inlet Methanol Emission Rate (lbs/hr)	Outlet Formaldehyde Emission Rate (lbs/hr)	Outlet Methanol Emission Rate (lbs/hr)
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2	11:50 - 12:50	0.006	51.512	0.0003	0.3254
3	13:35 - 14:35	0.007	56.402	0.0003	0.3882
3-Test Averages:		0.006	55.313	0.0003	0.4374
4	9:39 - 10:39	0.007	60.194	0.0004	1.8094
5	11:24 - 12:24	0.006	53.823	0.0005	1.6068
6	13:45 - 14:45	0.016	104.60	0.0005	1.3616
3-Test Averages:		0.010	72.871	0.0005	1.5926

The results listed in Table 1 yield a 3-test average hazardous air pollutant (HAP) removal efficiency of 99.2% for Test Runs 1, 2, and 3 (-50°C) and 97.6% for Test Runs 4, 5, and 6 (-30°C).

Testing of the water scrubber inlet and outlet consisted of triplicate 60-minute test runs completed on May 17, 2017 with a water scrubber water flowrate set point of 35 gallons per minute (gpm), triplicate 60-minute test runs completed on May 18, 2017 with a water scrubber water flowrate set point of 25 gpm, and triplicate 60-minute test runs completed on May 18, 2017 with a water scrubber water flowrate set point of 15gpm. The results of the water scrubber emissions test program are summarized by Table 2.

**Table 2
Water Scrubber Test Results**

Test Run	Test Run Time	Inlet Formaldehyde Emission Rate (lbs/hr)	Inlet Methanol Emission Rate (lbs/hr)	Outlet Formaldehyde Emission Rate (lbs/hr)	Outlet Methanol Emission Rate (lbs/hr)
1	16:00 - 17:00	1.13	1.92	0.01	0.01
2	17:08 - 18:08	1.20	1.95	0.01	0.01
3	18:15 - 19:15	1.82	2.64	0.01	0.01
3-Test Averages:		1.39	2.17	0.01	0.01
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3-Test Averages:		1.08	3.45	0.01	0.01
7	13:09 - 14:09	1.25	2.12	0.01	0.01
8	14:16 - 15:16	1.02	2.35	0.01	0.02
9	15:23 - 16:23	1.49	2.31	0.01	0.04
3-Test Averages:		1.25	2.26	0.01	0.02

The results listed in Table 2 yield a 3-test average hazardous air pollutant (HAP) removal efficiency of 99.5% for Test Runs 1, 2, and 3 (35 gpm) and 99.5% for Test Runs 4, 5, and 6 (25 gpm) and 99.2% for Test Runs 7, 8, and 9 (15 gpm).

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 and formaldehyde, 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 the approved test protocol. There were no deviations from the approved test protocol during the testing. Specific test methodology is described more fully in Sections C.iii and C.iv.

A.v Production Data

As approved in the test protocol, 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

The water scrubber water flowrate is also recorded continuously. Data recorded during the emissions test program is provided in Appendix D.v.

B.iii Facility Operating Parameters

The approved test protocol identified the LUWA 1 set point of 34 gpm as the operating parameter representative of operation. The test protocol was designed to capture all portions of the batch cycle, using three one-hour runs over the course of two batches for each VRU operating condition (for a total of six VRU test runs). 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 each test run.

For the three VRU test runs with a cryogenic condenser operating temperature of -50°C (on May 16, 2017), the first batch started at 9:16AM and ended at 11:25AM. The second batch started at 11:47AM and ended at 2:19PM. For the three VRU test runs with a cryogenic condenser operating temperature of -30°C (on May 17, 2017), the first batch started at 9:38AM and ended at 11:57AM. The second batch started at 12:51PM and ended at 3:08PM.

Triplicate test runs were conducted on the water scrubber at each of three water scrubber water flowrate setpoint conditions (35 gpm, 25 gpm, and 15 gpm). 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, 3, 4, and 5. The exhaust gas pipe at both VRU sampling locations is six inches in diameter and the exhaust gas duct 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. The water scrubber sampling locations were designated the inlet and outlet sampling locations. Exhaust gas velocity was measured at Method 1 points 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 and HCOH concentrations.

C.iv Method Deviations

There were no deviations from the approved test protocol during the 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 and MeOH concentrations.
- Bag grab samples were collected at the exhaust from the FTIR unit and analyzed for O₂ content using a Fyrite analyzer. Exhaust gas molecular weight was determined from the measured O₂ content as well as concentration data for other compounds as measured by the FTIR used to analyze for exhaust gas HCOH and MeOH 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.

With respect to the methods listed above, the following method variations were requested and approved for the water scrubber testing:

- MeOH concentrations were determined using Method 320 instead of Methods 18 or 308.
- Because methanol concentrations at the water scrubber inlet were elevated, the Method 320 MeOH and HCOH analyte spikes at the water scrubber inlet were performed in ambient air as opposed to the sample stream. Method 320 spiking was conducted at the water scrubber outlet. This approach confirmed the FTIR/sampling system accurately delivered and quantified 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 3, 4, 5, and 6 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 7 in Appendix D.vii.

D.viii Related Correspondence

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

Methylated Resins Department
Vapor Recovery And Scrubbing Process

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

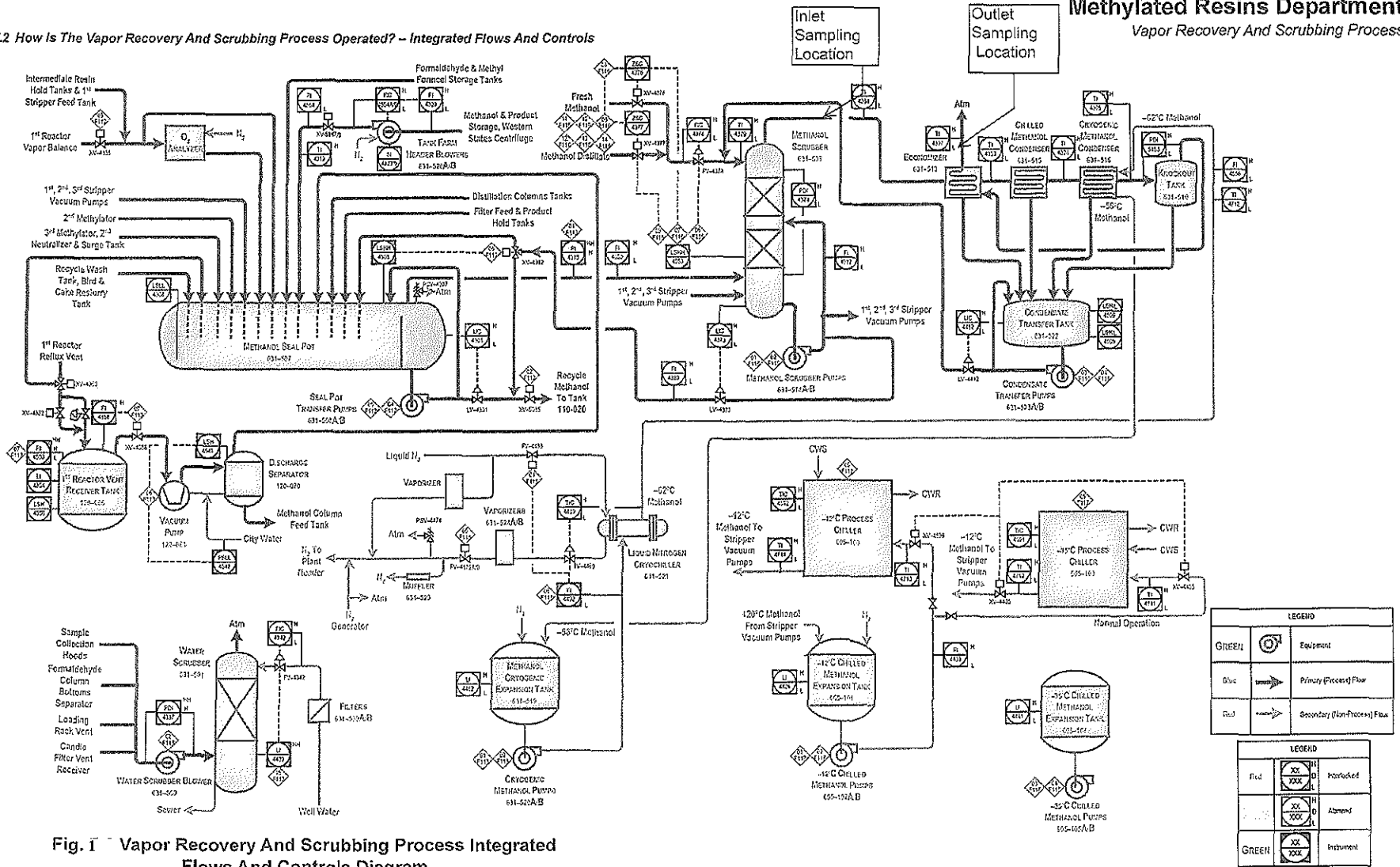
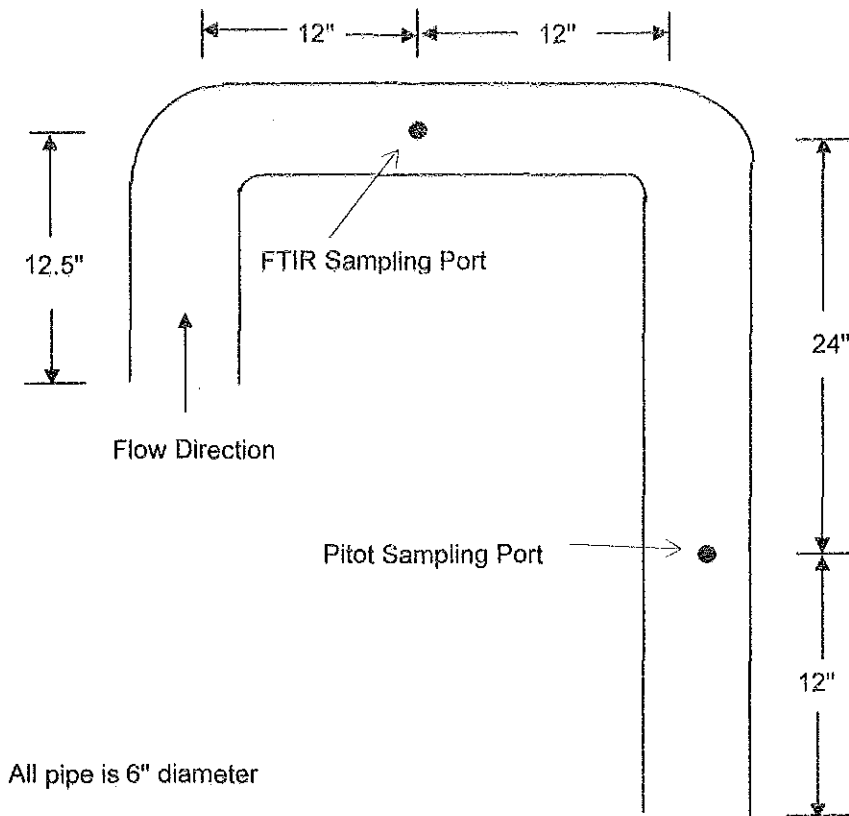


Fig. 1 - Vapor Recovery And Scrubbing Process Integrated Flows And Controls Diagram





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Figure 2

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

Sampling Dates:
May 16 and 17, 2017

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

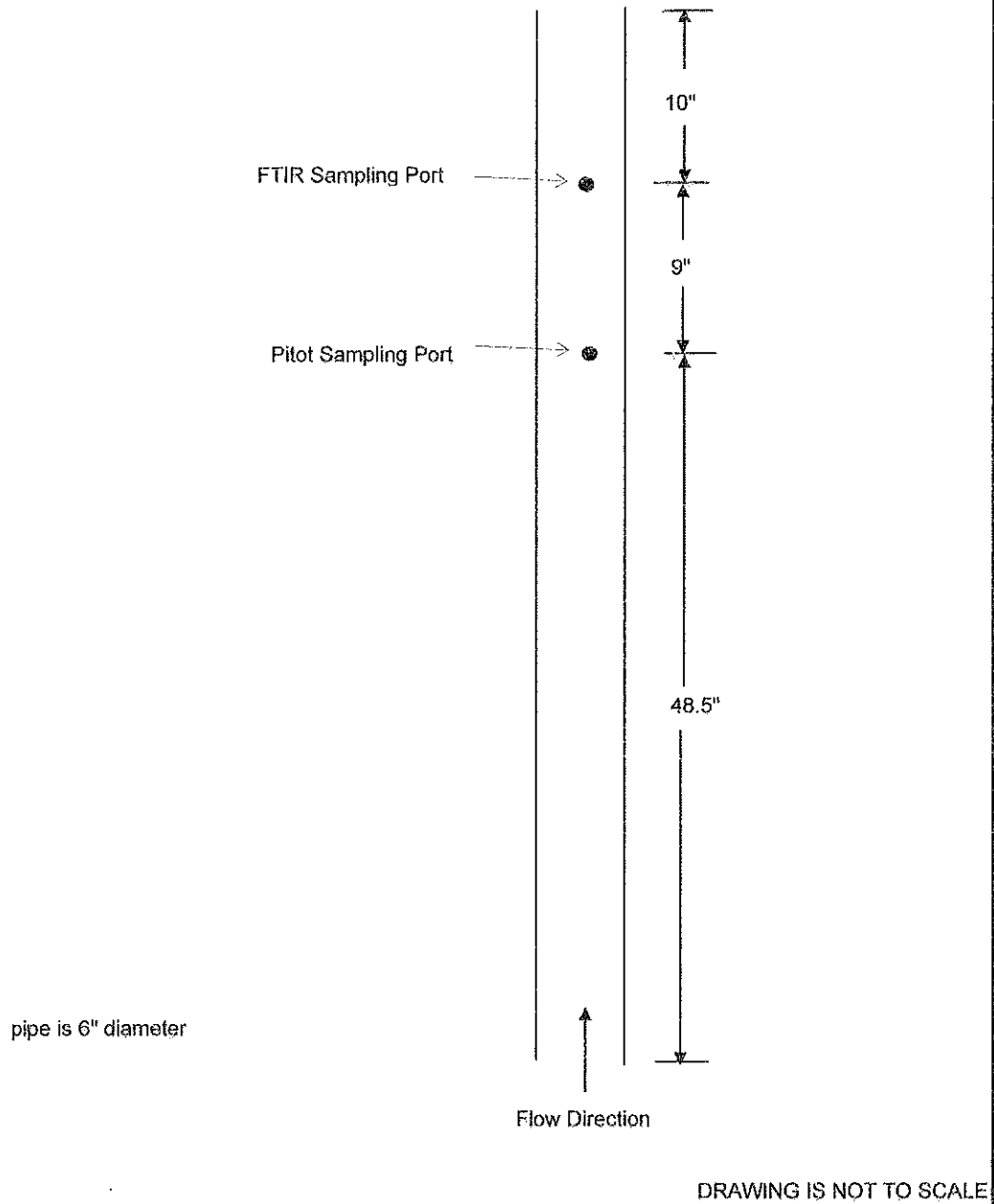


Figure 3

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

Sampling Dates:
May 16 and 17, 2017

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

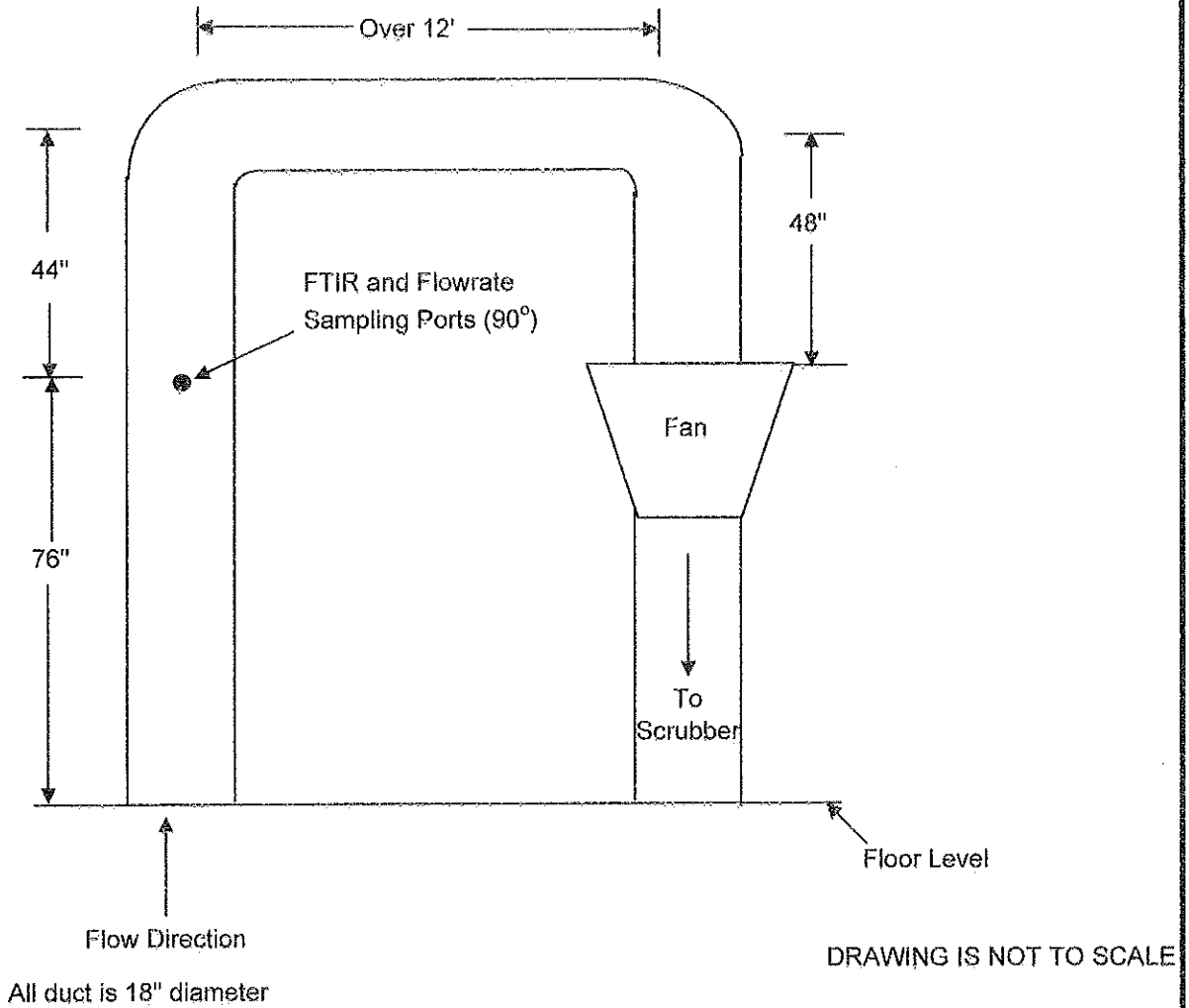
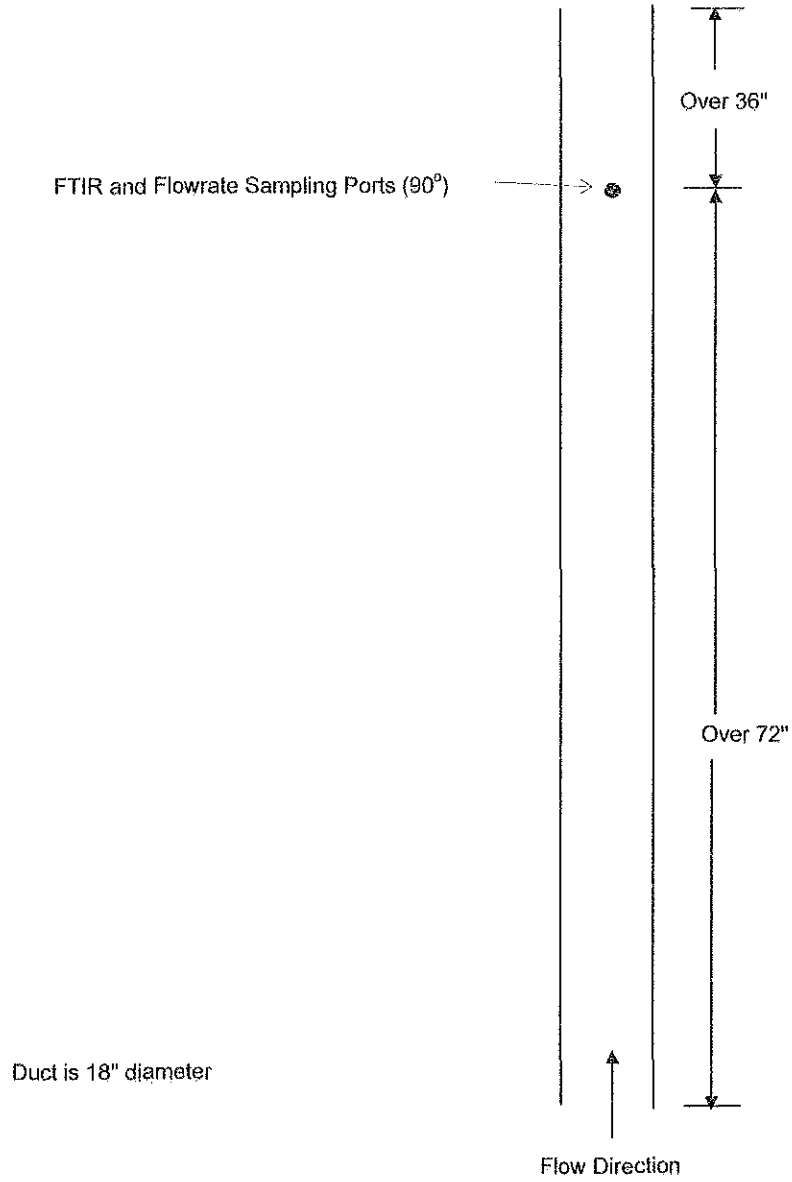


Figure 4

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

Sampling Dates:
May 17 and 18, 2017

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



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Figure 5

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

Sampling Start Date:
May 17 and 18, 2017

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