

Pre-Extractive Column Vent Primary Condenser Outlet Emissions Test Report

Prepared for:

Solutia Inc.

Trenton, Michigan

Trenton Plant 5100 West Jefferson Avenue Trenton, Michigan 48183

> Project No. 049AS-641117 January 20, 2020

Montrose Air Quality Services, LLC 4949 Fernlee Avenue Royal Oak, Michigan 48073 (248) 548-8070

EXECUTIVE SUMMARY

Montrose Air Quality Services, LLC (Montrose) was retained by Solutia Inc. (Solutia) to evaluate emission rates of acetaldehyde (AcH), vinyl acetate (VAc), ethanol (EtOH), and ethyl acetate (EtOAc) from the pre-extractive column vent primary condenser (i.e., between the primary condenser and the vent condenser during production of LT Bead Resin. The purpose of the emissions test program was to evaluate exhaust gas flow volumes, AcH, VAc, EtOH, and EtOAc concentrations, and corresponding AcH, VAc, EtOH, and EtOAc mass rates. The emission rates measured during the emissions test program will be used by Solutia to demonstrate compliance with the requirements of the Standards of Performance for Volatile Organic Compounds (VOC) Emissions from Synthetic Organic Chemical Manufacturing Industry (SOCMI) Distillation Operations codified at Title 40, Part 60, Subpart NNN of the Code of Federal Regulations (40 CFR 60, Subpart NNN).

The emissions test program was conducted consistent with the emissions test plan and corresponding alternative test methods approval request dated October 21, 2019 (see Appendix G). The emissions test plan and corresponding alternative test methods approval request were approved by AQD in a letter dated and November 13, 2019 (also in Appendix G). No variations from the approved emissions test protocol occurred during the testing with the following exceptions:

- (1) Because the exhaust gas flowrate was extremely low, it could not be measured simultaneously with FTIR concentration measurements. Consequently, with the on-site approval of AQD, exhaust gas flowrate was measured following each concentration measurement test run and used to determine average emission rates.
- (2) Due to instrument issues, FTIR data was compromised during Run 2. Consequently, Run 2 was aborted and not included in the emissions test results.

Overall emissions test results are summarized by Table E-I.

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 Table E-I

 Ethanol, Ethyl Acetate, Acetaldehyde, and Vinyl Acetate Emission Rates

 Pre-Extractive Column Vent Primary Condenser Outlet

 Solutia Inc.

 Trenton Michigan

 Test Date: November 26, 2019

						Average														verage
	Total	Meter			Flow	Exhaust	Average	Average		Average		Average	Average	Average		Average		Average		TOC
	Flow	Barometric	Meter	Total Flow	Measurement	Gas	Ethanol	Ethyl Acetate	Ace	taldehyde	Vi	inyl Acetate	Ethanol	Ethyl Acetate	Ac	etaldehyde	Vi	nyl Acetate	E	mission
	Volume	Pressure	Temperature	Volume	Duration	Flowrate	Concentration	Concentration	Con	centration	Co	oncentration	Emission Rate	Emission Rate	Em	ission Rate	En	nission Rate		Rate
Test Run	(acf)	(in. Hg)	(oF)	(scf)	(min)	(scfm)	(vol%)	(vol%)		(ppmv)		(ppmv)	(lbs/hr)	(lbs/hr)		(lbs/hr)		(lbs/hr)		lbs/hr)
1	0.10	29.23	56	0.10	87	0.001	14.9	15.0	<	250	<	250	0.001	0.002	<	2.0E-06	<	3.8E-06	<	0.004
3	0.21	29.23	55	0.21	60	0.004	11.0	14.3	<	250	<	250	0.003	0.007	<	6.0E-06	<	1.2E-05	<	0.010
4	0.25	29.23	52	0.25	60	0.004	12.3	15.2	<	250	<	250	0.004	0.009	<	7.2E-06	<	1.4E-05	<	0.012
					Averages:	0.003	12.7	14.8	<	250	<	250	0.003	0.006	<	5.1E-06	<	9.9E-06	<	0.009

Molecular Weights								
Compound	M.W.							
Ethanol	46.07							
Ethyl Acetate	88.11							
Acetaldehyde	44.05							
Vinyl Acetate	86.09							

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1. Introduction

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Montrose Air Quality Services, LLC (Montrose) was retained by Solutia Inc. (Solutia) to evaluate emission rates of acetaldehyde (AcH), vinyl acetate (VAc), ethanol (EtOH), and ethyl acetate (EtOAc) from the pre-extractive column vent primary condenser (i.e., between the primary condenser and the vent condenser during production of LT Bead Resin. The purpose of the emissions test program was to evaluate exhaust gas flow volumes, AcH, VAc, EtOH, and EtOAc concentrations, and corresponding AcH, VAc, EtOH, and EtOAc mass rates. The emission rates measured during the emissions test program will be used by Solutia to demonstrate compliance with the requirements of the Standards of Performance for Volatile Organic Compounds (VOC) Emissions from Synthetic Organic Chemical Manufacturing Industry (SOCMI) Distillation Operations codified at Title 40, Part 60, Subpart NNN of the Code of Federal Regulations (40 CFR 60, Subpart NNN).

The emissions test program was conducted consistent with the emissions test plan and corresponding alternative test methods approval request dated October 21, 2019. The purpose of this report is to document the results of the test program. AQD has published a guidance document entitled "Format for Submittal of Source Emission Test Plans and Reports" (March 2018). This document is provided as Appendix A. The following is a summary of the emissions test program and results in the format suggested by the aforementioned document.

1.a Identification, Location, and Dates of Test

Sampling and analysis for the emission test program was conducted on November 26, 2019, at the Solutia facility located in Trenton, Michigan.

1.b Purpose of Testing

The purpose of the emissions test program was to evaluate exhaust gas flow volumes, AcH, VAc, EtOH, and EtOAc concentrations, and corresponding AcH, VAc, EtOH, and EtOAc mass rates. The emission rates measured during the emissions test program will be used by Solutia to (1) demonstrate compliance with the requirements of the Standards of Performance for VOC Emissions from SOCMI Distillation Operations codified at 40 CFR 60, Subpart NNN, and (2) demonstrate compliance with the 2.9 lbs/hr VOC emission limit for EUPREEXTCOL in AQD Permit No. 93-19.

As specified by 40 CFR 60.660(c)(6), "Each affected facility operated with a vent stream flow rate less than 0.008 scm/min is exempt from all provisions of this subpart except for the test method and procedure and the recordkeeping and reporting requirements in § 60.664(g) and paragraphs (i), (l)(5), and (o) of § 60.665." (As per 40 CFR 60.665(i), 0.008 scm/min is equivalent to 0.3 scf/min.) Consequently, an additional purpose of the testing was to measure the vent stream flow rate for comparison to the 40 CFR 60, Subpart NNN cutoff flow rate limit of 0.3 scfm.

1.c Source Description

The Trenton Resins Plant produces polyvinyl butyral resin and in the process, ethyl acetate is formed as a co-product. A recovery system, comprised of multiple distillation columns is used to recover ethyl acetate for sale and ethanol for reuse. Three distillation columns are operated continuously in series to recover ethyl acetate – the pre-extractive column, the extractive column, and the drying column, in that order. The ethyl acetate stream from the drying column is sufficiently purified to be sold as a commodity chemical to customers.

This test involves the first of the ethyl acetate distillation columns, namely the preextractive column. Ethyl acetate is concentrated in the pre-extractive column overheads stream with the use of a primary condenser and a secondary vent condenser. Condensed material from the condensers is sent to the next distillation column, the extractive column, for further concentration of ethyl acetate.

1.d Test Program Contacts

The contact for the source and test report is:

Mr. Charles E. Anderson Environmental Specialist Soultia Inc. 5100 West Jefferson Avenue Trenton, Michigan 48183 (734) 672-7895

Names and affiliations for personnel who were present during the testing program are summarized by Table 1.

2. Summary of Results

Sections 2.a through 2.d summarize the results of the emissions compliance test program.

2.a Operating Data

Process data to be collected during the emissions test program included:

- Cooling tower water temperature
- A-crude feed rate
- Primary condenser exhaust gas temperature

Process data monitored during the emissions test program is provided in Appendix B.

2.b Applicable Permit

The pre-extractive column is included in Renewable Operating Permit No. MI-ROP-B2155-2009a as well as Permit No. 93-19. The pre-extractive column vent is included in flexible groupings FGRES68COL and FGRESTOAC in AQD Permit No. MI-ROP-B2155-2009a. These flexible groupings include operational and monitoring requirements but do not include emission limitations. In addition, Permit No. 93-19 includes an emission limitation of 2.9 lbs/hr VOC for the pre-extractive column.

The pre-extractive column vent is affected by the requirements of 40 CFR 60, Subpart NNN. Specifically, pursuant to 40 CFR 60.662(c), the affected facility must maintain a TRE Index Value greater than 1.0 without use of emission control devices.

As specified by 40 CFR 60.664(e), "For purposes of complying with §60.662(c) the owner or operator of a facility affected by this subpart shall calculate the TRE index value of the vent stream using the equation for incineration in paragraph (e)(1) of this section for halogenated vent streams. The owner or operator of an affected facility with a nonhalogenated vent stream shall determine the TRE index value by calculating values using both the incinerator equation in (e)(1) and the flare equation in (e)(2) of this section and selecting the lower of the two values."

The purpose of the emissions test program was to provide exhaust gas flowrate and individual component concentrations at the sampling location necessary to determine TRE Index Value using the equations presented in 40 CFR 60.664(e)(1) and (e)(2).

As specified by 40 CFR 60.660(c)(6), "Each affected facility operated with a vent stream flow rate less than 0.008 scm/min is exempt from all provisions of this subpart except for the test method and procedure and the recordkeeping and reporting requirements in § 60.664(g) and paragraphs (i), (l)(5), and (o) of § 60.665." (As per 40 CFR 60.665(i), 0.008 scm/min is equivalent to 0.3 scf/min.) Consequently, an additional purpose of the testing was to measure the vent stream flow rate for comparison to the 40 CFR 60, Subpart NNN cutoff flow rate limit of 0.3 scfm.

2.c Results

The overall results of the emission test program are summarized by Table 2.

3. Source Description

Sections 3.a through 3.e provide a detailed description of the process.

3.a Process Description

The Trenton Resins Plant produces polyvinyl butyral resin and in the process, ethyl acetate is formed as a co-product. A recovery system, comprised of multiple distillation columns is used to recover ethyl acetate for sale and ethanol for reuse. Three distillation columns

are operated continuously in series to recover ethyl acetate – the pre-extractive column, the extractive column, and the drying column, in that order. The ethyl acetate stream from the drying column is sufficiently purified to be sold as a commodity chemical to customers.

This test involves the first of the ethyl acetate distillation columns, namely the preextractive column. Ethyl acetate is concentrated in the pre-extractive column overheads stream with the use of a primary condenser and a secondary vent condenser. Condensed material from the condensers is sent to the next distillation column, the extractive column, for further concentration of ethyl acetate.

3.b Process Flow Diagram

The pre-extractive column is simply used for separation and, consequently, a process flow diagram is not necessary.

3.c Raw and Finished Materials

Process production and operating data is provided in Appendix B.

3.d Process Capacity

The capacity of the pre-extractive column process includes approximate production rates of 25 gal/min average and 27 gal/min maximum.

3.e Process Instrumentation

Relevant process instrumentation for the pre-extractive column includes feed flowrate, primary condenser cooling water flowrate, and primary condenser vent temperature.

4. Sampling and Analytical Procedures

Sections 4.a through 4.d provide a summary of the sampling and analytical procedures used.

4.a Sampling Train and Field Procedures

The sampling train for the emissions test program can be separated into the sampling train for measuring exhaust gas flowrates and the sampling train for measuring exhaust gas AcH, VAc, EtOH, and EtOAc concentrations.

Exhaust gas flowrates were measured by allowing all of the exhaust gas to flow into a single Tedlar bag through a Swagelok fitting. The exhaust gas that flows into the Tedlar bag was exhausted from the bag through a second Swagelok fitting, through a vacuum pump, and through a dry gas meter rig (i.e., Method 5 rig).

Exhaust gas AcH, VAc, EtOH, and EtOAc concentrations were measured using Fourier Transform Infrared Spectroscopy (FTIR). The FTIR sampling train consisted of a short length of stainless steel tubing, a heated sample line, the FTIR instrument, and a sample pump. AcH, VAc, EtOH, and EtOAc concentration data was datalogged at fifteen (15) second intervals throughout each emissions test run. The exhaust from the FTIR was returned to the vent stream through a second heated sample line.

Exhaust gas AcH, VAc, EtOH, and EtOAc concentrations (as well as moisture and carbon dioxide concentrations) were evaluated using the procedures of Method 320 with method variations as described in the emissions test plan and approved by AQD. Exhaust gas was extracted from the duct through a short section of stainless steel tubing before it entered a heated sample line. The sample line kept the gas at approximately 300 to 350°F before it entered a heated pump followed by the FTIR analyzer.

4.b Recovery and Analytical Procedures

This test program did not include laboratory samples and, consequently, sample recovery and analysis is not applicable to this test program.

4.c Sampling Ports

Sampling ports are illustrated by Figure 1.

4.d Traverse Points

Because of the small diameter of each sampling location, all measurements consisted of single point sampling.

5. Test Results and Discussion

Sections 5.a through 5.k provide a summary of the test results.

5.a **Results Tabulation**

Overall test program results are summarized by Table 2. Process data monitored during the emissions test program are provided in Appendix B.

5.b Discussion of Results

Table 2 summarizes the results of the emissions test program.

5.c Sampling Procedure Variations

The emissions test program was conducted consistent with the emissions test plan and corresponding alternative test methods approval request dated October 21, 2019 (see Appendix G). The emissions test plan and corresponding alternative test methods approval request were approved by AQD in a letter dated and November 13, 2019 (also in Appendix

G). No variations from the approved emissions test protocol occurred during the testing with the following exceptions:

- (1) Because the exhaust gas flowrate was extremely low, it could not be measured simultaneously with FTIR concentration measurements. Consequently, with the on-site approval of Ms. Regina Angellotti of AQD, exhaust gas flowrate was measured following each concentration measurement test run and used to determine average emission rates.
- (2) Due to instrument issues, FTIR data was compromised during Run 2. Consequently, Run 2 was aborted and not included in the emissions test results.

Because the exhaust gas flowrate was extremely low, AQD requested a brief verification of exhaust gas velocity using a stationary pitot tube positioned at the center of the exhaust pipe and connected to a pressure transmitter. This was conducted for a ten minute-period at the end of the test program with velocity pressure values recorded at one-minute intervals. Each individual velocity pressure measurement was 0.000" H_2O .

5.d Process or Control Device Upsets

No upset conditions occurred during testing.

5.e Control Device Maintenance

There was no major maintenance performed on the air pollution control devices during the three-month period prior to this emissions test.

5.f Re-Test

The emissions test program was not a re-test.

5.g Audit Sample Analyses

No audit samples were collected as part of the test program.

5.h Calibration Sheets

Relevant equipment calibration documents are provided in Appendix C.

5.i Sample Calculations

Sample calculations are provided in Appendix D.

5.j Field Data Sheets

Field documents relevant to the emissions test program are presented in Appendix E.

5.k Laboratory Data

There are no laboratory results for this test program. The Method 320 FTIR emissions test report provided by Prism Analytical Technologies is included in Appendix F.

Name	Affiliation
Charles Anderson	Solutia
Randal Tysar	Montrose Environmental
David Koponen	Montrose Environmental
David Schuberg	Prism Analytical
Regina Agellotti	EGLE – AQD

Table 1Testing Personnel

Table 2 Ethanol, Ethyl Acetate, Acetaldehyde, and Vinyl Acetate Emission Rates Pre-Extractive Column Vent Primary Condenser Outlet Solutia Inc. Trenton Michigan Test Date: November 26, 2019

						Average													A	verage
	Total	Meter			Flow	Exhaust	Average	Average		Average		Average	Average	Average		Average		Average		TOC
	Flow	Barometric	Meter	Total Flow	Measurement	Gas	Ethanol	Ethyl Acetate	Ac	etaldehyde	Vi	inyl Acetate	Ethanol	Ethyl Acetate	Ac	cetaldehyde	Vi	nyl Acetate	E	mission
	Volume	Pressure	Temperature	Volume	Duration	Flowrate	Concentration	Concentration	Cor	ncentration	Co	ncentration	Emission Rate	Emission Rate	En	nission Rate	En	ission Rate		Rate
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Molecular Weights							
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