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40 CFR 63 Subpart NNNNN
National Emission Standards for Hazardous Air Pollutants Emissions from Hydrochloric Acid Production
Compliance Test Plan

Dual Pressure Distillation (DPD) Process
Scrubber 24388 (EU356-01)
356 Process Area

Dow Silicones Corporation
Michigan Operations
Midland, Michigan

**** Please note the process unit is the final copy holder and owner of this document. A temporary electronic copy will be retained by test team for a short period of time.***

Compliance Sample Report

I certify that I have personally examined and am familiar with the information submitted herein, and based on my inquiries of those individuals immediately responsible for obtaining the information; I believe the submitted information is true, accurate, and complete.

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TABLE OF CONTENTS

1.0 INTRODUCTION....2

2.0 PLANT AND SAMPLING LOCATION DESCRIPTIONS....6

3.0 SUMMARY AND DISCUSSION OF TEST RESULTS....9

4.0 SAMPLING AND ANALYTICAL PROCEDURES....12

5.0 CALCULATIONS....15

6.0 RAW FIELD DATA....19

7.0 ANALYTICAL DATA....35

8.0 CALIBRATIONS....168

9.0 RESUMES....175

10.0 PROCESS DATA....181

1.0 INTRODUCTION

1.1 Summary of Test Program

This compliance test report describes the test procedures performed on the packed scrubber, Scrubber 24388 (EU356-01), located in the 356 Area as part of the Dual Pressure Distillation Process (DPD) owned and operated by Dow Silicones Corporation (DSC) in Midland, Michigan. This test demonstrated compliance with the requirements of 40 CFR 63 Subpart NNNNN.

The testing was conducted by internal stack testing team (AECOM Inc) personnel. The HCl and Cl2 analysis was performed by a contract lab Enthalpy. Pollutants measured were: HCl, Cl2.

This compliance test report also contains the results of the CMS performance evaluation, as required by 40 CFR 63 Subpart A §63.8(e)(5)(i) and §63.10(e)(2)(i). On September 18, 2018 a performance evaluation was conducted on the Continuous Monitoring Systems (CMS) as required by 40 CFR 63 Subpart A, §63.8(e), as modified by §63.9005(d) and Table 7 of 40 CFR 63 Subpart NNNNN. As specified in the CMS Site-Specific Monitoring Plan, the FT28651B flow meter on Scrubber 24388 is calibrated with an acceptance criteria of ± 1%. The max span for the FT28651B flow meter on Scrubber 24388 is 50 gpm (±1% is ±0.5 gpm). During the instrument calibration on September 18, 2018, Flow Transmitter FT28651B was accurate to ± 0.5 GPM and no adjustments were needed.

The vent testing was completed on October 30th, 2018. The test consisted of a minimum of three one-hour runs. The unit was previously tested on October 21, 2013 and this testing is required to be completed every 5 years.

The following table summarizes the pertinent data for this compliance test:

Responsible Groups	• Dow Silicones Corporation • Michigan Department of Environmental Quality (MDEQ) • Environmental Protection Agency (EPA)
Applicable Regulations	• MI-ROP-A4043-2008 • 40 CFR 63 NSPS Subpart NNNNN
Industry / Plant	• 356 Building
Plant Location	• Dow Silicones Corporation Midland, Michigan 48667
Date of Last Test	• October 21, 2013
Air Pollution Control Equipment	• Scrubber 24388
Emission Points	• Scrubber 24388 (EU 356-01)
Pollutants/Diluent Measured	HCl process Vents: • Hydrogen Chloride (HCl) 6 ppmv • Chlorine (Cl_2) < 1 ppmv HCl storage tanks: • Hydrogen Chloride (HCl) 6 ppmv
Test Dates	• September 18, 2018 –CMS Performance Evaluation (e.g. Instrument calibration) • October 30th, 2018 – Performance Test

1.2 Key Personnel

The key personnel who coordinated the test program are:

- Chris Dinh is the Process Focal Point. The Process Focal Point is responsible for coordinating the plant operation during the test and ensuring the unit was operating at the agreed upon conditions in the test plan. They also serve as the key contact for collecting any process data required and providing all technical support related to process operation. Contact information is 989-496-5857

- Michael Gruber is the Environmental Focal Point for this unit. The Environmental Focal Point is responsible for ensuring that all regulatory requirements and citations are reviewed and considered for the testing. Contact information is 989-496-5539.

- Chuck Glenn is the Test Plan Coordinator. The Test Plan Coordinator is responsible for the overall leadership of the sampling program. They also develop the overall testing plan and determine the correct sample methods.

- Spencer Hurley is the back-up for the Test Plan Coordinator. He also served as the technical review role of the test data.

- Michael Abel is the Technical Reviewer of the test information.

- Daniel Nunez is the Project Manager. The Project Manager is responsible for ensuring the data generated meets the quality assurance objectives of the plan.

2.0 PLANT AND SAMPLING LOCATION DESCRIPTION

2.1 Facility Description

The main purpose of the Dual Pressure Distillation Process (DPD) process at the 356 building is to produce anhydrous HCl. Part of the DPD process is considered a HCl production facility subject to HCl MACT, because it produces a liquid HCl product at a concentration of 30 weight percent or greater during its normal operations.

The primary equipment for the DPD process at the 356 Building consists of distillation columns, reboilers, plate and frame interchangers, condensers, sample coolers, demisters, an absorber, and two different types of scrubbers, and storage tanks to allow storage of feed materials and intermediates.

The aqueous manufacturing process (e.g., the HCl production facility) begins at the outlet of the 24383 demister, which feeds anhydrous HCl to the 24387 absorber. The HCl production facility also begins at the hand valve where the highline that supplies anhydrous HCl to other processes in the plant splits off to supply the aqueous HCl production process.

The 30-36% aqueous HCl generated in the HCl Vent Absorber (24387) is sent to the two aqueous HCl storage tanks (T-24345 / T-24346). The aqueous HCl storage tanks (T-24345/T-24346) and other equipment not applicable to the HCl MACT (e.g. bottoms receivers for distillation columns, and the vent from a steam jet ejector system) vent to the Venturi (24386).

The water (containing ~0.1 wt% HCl) flowing through the Venturi is recycled to the HCl Vent Absorber (24387). The 0.1 wt% HCl in the liquid stream from the Venturi combines with a gaseous anhydrous HCl stream (100 wt% HCl) in the HCl Vent Absorber (24387), which is the HCl production unit that produces the liquid aqueous HCl at a concentration of 30 weight percent or greater.

The process vent from the HCl Vent Absorber (24387) and the gaseous stream from the Venturi (24386) are routed to the final water scrubber control device (24388) and then the atmosphere.

2.2 Control Equipment Descriptions

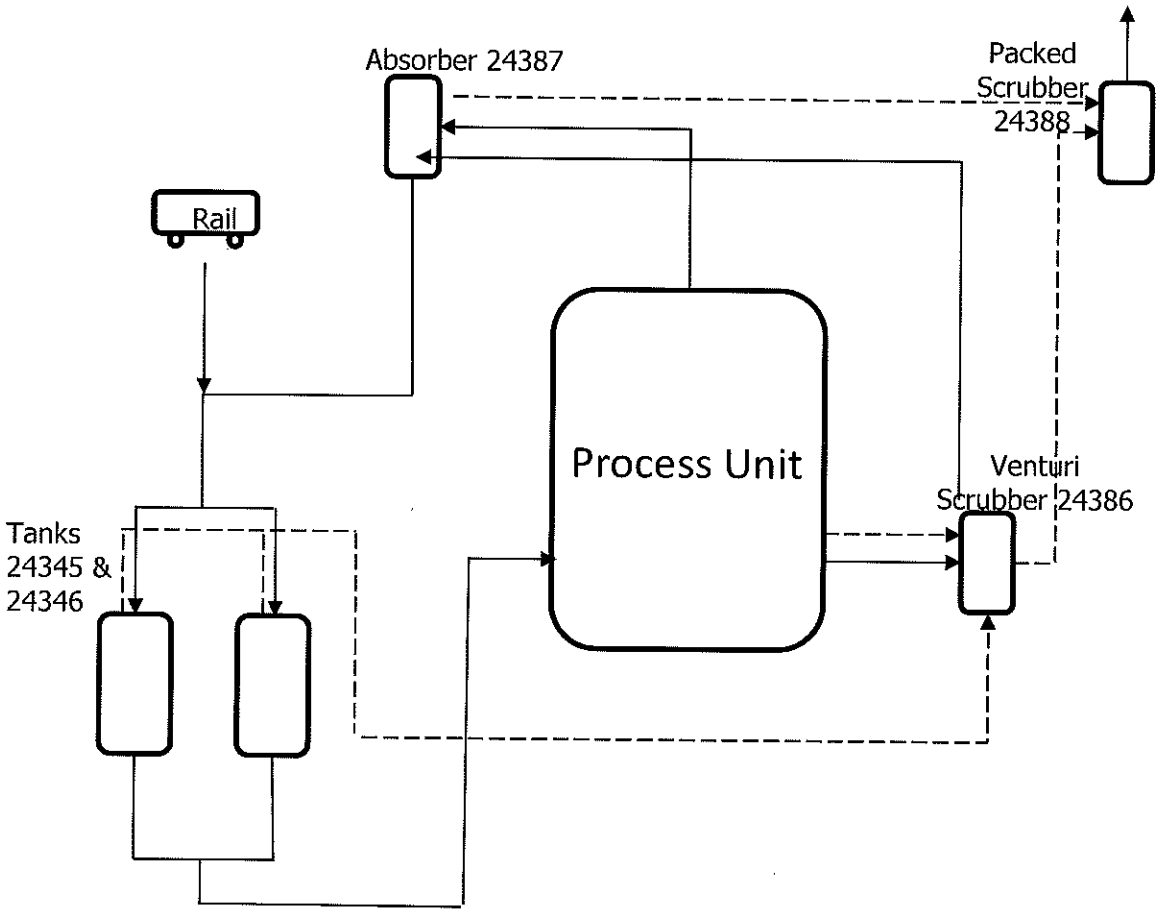
The DPD process tail gas scrubber, 24388, is a packed section scrubber tower that removes HCl and Chlorine from the exhaust gas.

The DPD process also includes a Venturi Scrubber (24386), which is a two stage venturi scrubber that currently adsorbs some HCl. However, the Venturi is considered to be part of the HCl production unit as defined by §63.9075 and is not a control device for HCl MACT. In a letter dated September 4, 2018, the Michigan Department of Environmental Quality (MDEQ), Air Quality Division (AQD), concurs with the DSC interpretation of the HCl MACT that the Venturi (24386) is not a control device used to reduce HCl at an HCl production facility.

2.3 Flue Gas Sampling Locations

Outlet of Scrubber 24388
Emission sampling was conducted from the air emissions Scrubber 24388 outlet stack. There was a sample port installed on the 3" duct.

FIGURE 2.1: FACILITY DIAGRAM



3.0 SUMMARY AND DISCUSSION OF TEST RESULTS

3.1 Objectives and Test Matrix

The purpose of this test was to demonstrate compliance with the following regulations and requirements of HCl MACT (40 CFR 63 NNNNN).

The specific objectives of the tests were:

- Measure HCl/Cl2 outlet emissions from Scrubber 24388
  - Establish the minimum operating limit for the scrubber fresh water inlet flow rate based on values measured during the performance test

Notes:

- Ongoing compliance is based on a daily average compared to these established operating limits.
- On March 20, 2014, EPA Region 5 approved a request from DSC dated February 6, 2014 to waive monitoring effluent pH on Scrubber 24388 under the condition that DSC used "once through" water in the scrubber.

3.2 Results

Scrubber 24388 yielded results that meet regulatory requirements during the conditions of operation.

3.3 Facility Operations

During the test, Scrubber 24388 was operated at the current maximum vent flow rate. In addition, the scrubber inlet fresh water flow rate was reduced thus establishing a minimum scrubber inlet liquid flow rate. This resulted in the scrubber operating at the worst case HCl/Cl_2 emission rate possible during normal maximum representative operating conditions.

During the test, the 24386 Venturi was operated at the current maximum vent flow rate.

TABLE 3.1: Test Results

Emission Results				
SAMPLE TYPE	**TEST METHOD**	**SAMPLING TIME (MINUTES)**	**ACTUAL EMISSION CONC. (AVG PPM)**	**ALLOWABLE EMISSION CONC.**
HCl (ppmv)	EPA Method 26A	64	6 ppmv	12 ppmv
Cl2 (ppmv)	EPA Method 26A	64	< 1 ppmv	20 ppmv

Field Sample Data				
PARAMETER	**RUN 1**	**RUN 2**	**RUN 3**	**AVERAGE**
Run Date	10/30/18	10/30/18	10/30/18	n/a
Run Times	1049/1153	1212/1316	1337/1441	n/a
Average Stack Temperature (deg F)	81	90	82	84
Moisture Fraction (%)	3.1%	3.3%	3.8%	3.4%
Outlet Stack Gas Dry Flow (dscfh)	1652	1602	1639	1631
Sample Volume in Liters	883.9	844.8	877.0	868.6
Catch Wt. HCl in Outlet (ug)	5976	8543	9413	7977
Emissions HCl (ppmv)	**4**	**7**	**7**	**6**
Catch Wt. Cl2 in Outlet (ug)	522	484	283	430
Emissions Cl2 (ppmv)	**< 1**	**< 1**	**< 1**	**< 1**

Process Data				
Water Flow Rate (lb/hr) (FT28651B)	1010	1011	1015	1012

Operational Parameter Data			
Scrubber	**Design Minimum Operating Rate**	**Normal Operating Rate**	**Actual Operating Rate**
AHCl Feed (Lb/Hr)	1500 lb/hr	~ 1300 lb/hr	~ 1554 lb/hr

Please note the following:

During the compliance test, DSC operated the HCl production unit at ~1,500 lbs/per hour as listed above. However, DSC did not simultaneously unload aqueous HCl from railcars into the aqueous HCl storage tanks (T-24345 and T-24346) during the vent testing. The reason DSC did not unload aqueous HCl from railcars during the test is because results of ASPEN modeling demonstrate that worst-case operating conditions for Scrubber 24388 occur when the head space vapors in the aqueous HCl storage tanks (T-24345 / T-24346) are removed by the Venturi (24386). Conditions are not worst-case for Scrubber 24388 when the aqueous HCl storage tanks (T-24345 and T-24346) are being filled from railcars while simultaneously removing the tanks' vapors by the Venturi (24386).

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4.0 SAMPLING AND ANALYTICAL PROCEDURES

4.1 Test Methods

The following EPA methods were used during the sampling and analysis:

4.1.1 Flue Gas Velocity and Volumetric Flow Rate - EPA Method 2

Determine flue gas velocity and volumetric flow rate using the procedure outline in 40 CFR 60, Appendix A, EPA Method 2. Measure and record both velocity and temperature using S-type pitot tubes conforming to the geometric specifications outlined in EPA Method 2, a manometer and a calibrated thermocouple.

4.1.2 Flue Gas Composition and Molecular Weight - EPA Method 3

Flue gas composition and molecular weight was assumed to be that of ambient air (MW-29) as allowed by 40 CFR Part 63 Subpart NNNNN.

4.1.3 Flue Gas Moisture Content - EPA Method 4

Determine the flue gas moisture content was determined in conjunction with the Method 26A sampling train according to the procedures outlined in 40 CFR 60, Appendix A, EPA Method 4. Method 4 requires all impingers connected in series and contained reagents as described in. Method 4 also requires impingers be contained in an ice bath in order to assure condensation of the moisture in the flue gas stream. Any moisture not condensed in the impingers is captured in the silica gel; therefore, all moisture is weighed and entered into moisture content calculations.

4.1.4 HCl/Cl2 Sampling - EPA Method 26A

The EPA Method 26A sampling train was used to determine HCl/Cl2 emissions. The average sampling rate for each run was within 10% of 100% isokinetic conditions. Each test run was at least one-hour in duration. The sampling train is described: the first and second impinger contained 0.1N H_2SO_4, the third impinger and fourth impinger contained 0.1N NaOH. The fifth impinger was filled with silica gel to prevent water from getting to the dry gas meter. All impingers were analyzed for chloride by Ion Chromatography (EPA Method 26A). As required by 40 CFR Part 63 Subpart NNNNN, an audit sample was requested by The Dow Chemical Company and submitted by Aecom Corporation sample team with the scrubber samples to the laboratory.

FIGURE 4.1: SAMPLING TRAIN FOR HCl/Cl2