

**DEPARTMENT OF ENVIRONMENTAL QUALITY  
AIR QUALITY DIVISION  
ACTIVITY REPORT: Scheduled Inspection**

P102749555	
<b>FACILITY:</b> DDP Specialty Electronic Materials US, Inc.	<b>SRN / ID:</b> P1027
<b>LOCATION:</b> 633 Washington Street, MIDLAND	<b>DISTRICT:</b> Saginaw Bay
<b>CITY:</b> MIDLAND	<b>COUNTY:</b> MIDLAND
<b>CONTACT:</b> Sara Bennett , Environmental, Health, & Safety Specialist	<b>ACTIVITY DATE:</b> 07/09/2019
<b>STAFF:</b> Kathy Brewer	<b>COMPLIANCE STATUS:</b> Non Compliance
	<b>SOURCE CLASS:</b> MEGASITE
<b>SUBJECT:</b> EU88 associated Rule 290 Fine mesh anion and cation. Facility will submit a PTI application after sampling of two atmospheric tank vents (SV88002 and SV88014) found previously unidentified SO2 emissions that exceeded the significant impact levels at fence line distance and also MeCl2 emissions greater than levels provided in the PTI application (PTI No. 374-08) but below the screening level.	
<b>RESOLVED COMPLAINTS:</b>	

**July 9, 2019 EU88 and associated Rule 290 Fine Mesh Anion and Cation**

The ownership of EU88 assets were recently transferred from Dow Chemical to DDP.

DDP contact Sara Bennett

The Cation process in the ion exchange resins manufacturing complex with reactors, separators, storage tanks/silos and related equipment. Pollution control equipment includes a water scrubber (Cation scrubber T-301) , methylene chloride tank vapor balance system, and afterburner (963THROX – thermal heat recovery oxidation unit followed by a quench and scrubber). Most process vents are sent to the 963 THROX.

This emission unit is subject to the requirements of 40 CFR Part 63, Subpart FFFF (MON) and 40 CFR Part 63, Subpart EEEE (OLD). EU88 is also subject to the equipment leak provisions of the HON (i.e., 40 CFR Part 63, Subpart H).

This emission unit was permitted in PTI 374-08. The Special Conditions included in the PTI do not contain requirements different from the current ROP.

Inspection included a review of the process flow diagram, including control devices and vents, on site records, control room process screen, Cation scrubber T-301, tanks and truck loading area for MeCl2 and Oleum, and, ROP required emission control and metering devices. AQD records were also reviewed.

No direct releases found post 2016 for EU88 in AQD release report database

On Aug 3, 2017 the AQD sent the facility a Violation Notice for 963 Throx chlorine emission exceedance to 963THROX due to failure to control pH of T-301 scrubber (scrubber 6) on May 22 and May 25, 2017. The 963 THROX digital input sensor for the pump was replaced. The pump and pH controller programming and supporting documentation has been upgraded.

Emissions reported to MAERS for 2018 were

Pollutant	Reported emissions	Basis
VOC	15 LBS	Other
PM10, Primary	120 LBS	Other
SO2	0 LBS	Other

The documentation for the emission calculation basis submitted with the 2018 emissions reported to MAERS is attached.

Based on the site visit and records review the facility appears to be in compliance with the ROP Special conditions for EU88 except for the following:

December 2018 emissions of SO2 from tank VE-101 not previously reported and emission levels of MeCl2 from tank VE-304 present at levels great than included it the PTI application for EU88. The emissions were included in the March 2019 ROP Annual Deviation Report.

On Site Records Review

- MeCl2 Tank truck unloading procedure
- MeCl2 storage tank loading operations data
- EU88 Cation shutdown procedure for planned and abrupt 963THROX shutdown
- EU88 Block valve to 963THROX curing 963THROX shutdown periods
- EU88 Cation T-301 scrubber liquid flow meter calibration April 2018 and May 2019
- Process diagram with control devices and vents
- Batch emissions from vents for IsoOctane and MeCl2 used in Feb 2019 emission calculations
- Rule 290 emissions from Fine mesh anion and cation

AQD File Review

- MAERS 2018 emissions
- CAM reports March 2018, September 2018, March 2019

MACES- Activity Report

ROP Annual and semi annual Deviation reports March 2018, September 2018, March 2019  
 MACT Reports Subpart FFFF reports March 2018, September 2018, March 2019  
 No Permit EVAL form was posted on the AQD common drive for PT1 374-08.

EU88 operates as a batch process which runs 3-5 batches /day for 24 hours 7 days a week most of the year. The process involves mixing and reaction vessels, washing, filter/dewatering and packaging. Recycle and recovery of raw materials including MeCl2 (using condensers) and a variety of acid concentrations.

Raw materials include Oleum, sulfuric acid, copolymer, MeCl, NaOH, CaCl, KOH, NaHCO3 (sodium bicarbonate), and lime. Raw materials are received from tank trucks, site pipeline and common header. Typically receive or send material by truck 5 days/week

Oleum tank vents to Cation scrubber. Process scrubber is for oleum tank and as back up for some vents (T-301 cation scrubber /458 scrubber).

**Emission limits**

EU88 is subject to the following emission limits:

Pollutant	Limit	Time Period /Operating Scenario	Monitoring/ Testing Method
1. Chlorine	3.1 pph	Test method	SC VI.2 liquid flow rate records for the water scrubber
2. VOC	9 tpy	12-month rolling time period as determined at the end of each calendar month	SC VI.3 calculate and record 12-month rolling emissions

Records reviewed indicate the facility is in compliance with the emission limits.

Parameter	Limit (12-month rolling )	date	Value (12-month rolling )
SC I.2 VOC	9 tpy	Sept 2017	5.4
		April 2018	4.8
		Feb 2019	5.1

On site records of chlorine emissions (lbs/hr) from Vent No. 5 (Cation combined vent) were the following:

Mar 2018	Apr 2018	May 2018	Jun 2018	Jul 2018	Aug 2018	Sep 2018	Oct 2018	Nov 2018	Dec 2018	Jan 2018	Feb 2018
0.02	0.04	0.05	0.10	0.07	0.08	0.07	0.06	0.07	0.07	0.07	0.07

**Material limits**

The ROP does not contain any specified material limits

**Process/Operational Restrictions**

SC III.1 prohibits venting to the 963THROX unless the 963THROX is operating as required. If 963THROX malfunctions portions of the system ducted to 963THROX shall be vented to the water scrubber until the process can be safely shutdown. Most vents are Group 1 under MON so must go to 963THROX. During shutdown of 963THROX the EU88 process shutdowns.

Review of the operating information shows Cation combined vent to 963THROX closed during periods when 963THROX shutdown. Production activity venting to the 963THROX can be indicated by the status of the hydrator weight. Hydrator weight goes to zero or steady state (if contents held) during a 963THROX shutdown. Two valves with the option to bypass to Cation scrubber T-301 are normally closed and remain closed during 963THROX shutdown periods reviewed.

Attached operating records were reviewed for 963THROX operating periods when temperatures were below 701 C.

I also reviewed valve status and control procedure during a 963THROX shutdown for process vents exhausting to 963THROX. Shutdown action for numerous process steps are described, including record keeping and notification requirements where appropriate. Records during recent shutdown period for 963THROX temperatures, Cation scrubber status, hydrator tank status, and the two valves that could be vented to scrubber or 963THROX were reviewed to show the process was not operating and did not exhaust to the vents going to the 963THROX or Cation scrubber.

SC III. 2. Limits the transfer of oleum into the storage tank to only when the water scrubber is installed and operating properly.

SC III.4 requires the liquid flow rate of the water scrubber not be less than 5 gallons per minute (gpm).

The records reviewed indicate the facility is in compliance with these requirement.

Operation	Requirement	Sept 20	Aug 22	Feb 20	July 9,
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		2017 Cation T-301 flow	2018	2019	2019 (instantaneous @1:30 PM)
Oleum transfer	Only if Cation T-301 scrubber operating > 5 gpm	>9 gpm	>5 gpm	>5 gpm	24.9 gpm

Operating records with the oleum tank loading status and the T-301 scrubber flow are attached.

SC III.3 requires proper operation of the vapor balance system for transfer of MeCl<sub>2</sub> into the MeCl<sub>2</sub> storage tank (MeCl<sub>2</sub> Tank Cation (VE-106)). The procedure for unloading MeCl<sub>2</sub> into the VE-106 storage was reviewed. The procedure includes a requirement to manage vents so VE-106 displaced vapors are returned back to the truck. Frequency of Me Cl<sub>2</sub> loading events between July 2018 and July 2019 is shown in an attached operating graph.

To relieve pressure, the MeCl<sub>2</sub> Tank Cation VE-106 vents to 963THROX. The only time the MeCl<sub>2</sub> tank vents to the atmosphere is during planned maintenance. The MON MACT allows venting to the atmosphere for up to 240 hours per year for planned maintenance. To be conservative, the site tracks and reports entire time MeCl<sub>2</sub> Tank Cation VE-106 is not venting to 963THROX, as time the tank vent exhausted to atmosphere. Actual venting through pressure relief valve only occurs during warmest part of a day and intermittent. The Annual and Semi Annual MON MACT reports contain the total number of hours the MeCl<sub>2</sub> Tank Cation VE-106 are venting to the atmosphere during periods of planned maintenance. The reported hours of venting to the were below 240 hours.

**Design/Equipment Parameters**

SC IV.1 requires that water scrubber (T-301) to be equipped with a maintained liquid flow indicator. At the time of the inspection the water scrubber had an operating liquid flow indicator operating. The calibration records for the water scrubber for 2018 and 2019 are attached.

**Testing/Sampling**

The ROP for EU89 contain no specified Testing/sampling special conditions

**Monitoring and Recordkeeping**

SC VI 1. requires the permittee to monitor and record, on a continuous basis the liquid flow rate of the scrubber. SC VI.2 requires the permittee to keep the continuous liquid flow rate records. Operating records of the T-301 scrubber flow are were reviewed and are attached for September 20, 2017, August 22, 2018, and February 20, 2019.

SC VI.3 requires the permittee to calculate and record the 12 month rolling averages for VOCs. The emission calculations and records were reviewed for September 2017, August 2018, and February 2019. Detailed information on the emissions and calculations for several vents were reviewed for the February 2019 emissions. Initially the records presented for Vent No. 5 were incorrect, all showing zero emissions for IsoOctane. The site relayed that that value shown was incorrect due to a spreadsheet cell malfunction and they would determine the cause of the erroneous emission value and provide the correct values. I received the correct values on July 16. The change in value of the Vent 5 IsoOctane emissions changed the 12 Month Rolling average by the last three significant digits (e.g. September 2017 12 month rolling average changed from 5.3770 TPY to 5.3958 TPY), which resulted in no change to the numbers in the emission values rounded up to the nearest tenth of a ton.

Parameter	Limit (12-month rolling )	date	Value (12-month rolling )
SC I.2 VOC	9 tpy	Sept 2017	5.4
		April 2018	4.8
		Feb 2019	5.1

**Reporting**

The March and September ROP Deviation reports did include the May 22 and May 25, 2017 chlorine emission exceedance for 963THROX due to failure to control pH of T-301 scrubber (scrubber 6). The AQD sent the facility a Violation Notice On Aug 3, 2017 for the 963THROX scrubber chlorine exceedances.

The March 2019 ROP Deviation report and a March 14, 2019 Audit Disclosure Report included a deviation for EU88 due findings from sampling of two atmospheric tank vents (SV88002 and SV88014). The sampling results found previously unidentified SO<sub>2</sub> emissions that exceeded the significant impact levels at fence line distance and also MeCl<sub>2</sub> emissions greater than levels provided in the PTI application (PTI No. 374-08) but below the screening level. In response to the deviation I requested and received the 12 month rolling VOC emissions and the methylene chloride emissions for October, November and December 2018.

Pollutant	ROP Limit TPY (12 month rolling average)	October 2018 TPY emissions (12 month rolling average)	November 2018 TPY emissions (12 month rolling average)	December 2018 TPY emissions (12 month rolling average)
VOC	9	4.84	4.88	4.98
Methylene chloride	NA	0.58	0.42	0.38

The monthly VOC emissions above and the 2018 emissions that were reported do not include the increase in methylene chloride emissions reported in the March 2019 Title V Deviation Report.

In the March 2019 Deviation report the company stated they would be submitting a PTI application. The company has not yet submitted a PTI application. They relayed that while initial testing data did show that the plant could have exceeded the emission levels included in the permit application for PTI 374-08 under certain process conditions, the results were limited in scope and more variable than anticipated making it difficult to accurately quantify the increase in emissions. Additional sampling was conducted in April 2018 to collect emissions data across a broad range of process conditions. This data will be used by the design team to develop a PTI application (not yet submitted) for EU88 and will also be incorporated into 2019 emissions. They continue to have weekly update meetings for completing information required for the PTI application submittal.

A VN will be written for the deviation if an administratively complete PTI application has not been received by September 30, 2019. The time allocated for submittal of the PTI is based on the ongoing effort and technical difficulty in directing the exhaust from VE-101 (SV88002) and VE-304 (SV88014) to 963THROX in a functional and safe manner.

Per the MON MACT, for planned periods of routine maintenance, Me Cl<sub>2</sub> Tank Cation VE-106 per MON allowed venting to atmosphere to relieve pressure for no more than 240 hours when Cation VE-106 not venting to 963THROX. The site tracks and reports entire time not venting to 963THROX as time vent exhausted to atmosphere to be conservative. Actual venting through a pressure relief valve only during warmest part of a day and is intermittent.

No other Deviations or CAM excursion or exceedances were reported for EU88 for the time period reviewed.

#### Stack/Vent Restrictions

The following descriptions were provided during the inspection:

Stack & Vent ID	Maximum Exhaust Dimensions (inches)	Minimum Height Above Ground (feet)	Description
1. SV88002	8	40	(99% acid) Feed acid tank vent to atmosphere
2. SV88004	3	70	Cation scrubber T-301
3. SV963THROX	24	80	
4. SV88010	6	50	Neutralization tank after some Neutralization steps and no longer venting to 963THROX
5. SV88011	6	60	Neutralization tank after some Neutralization steps and no longer venting to 963THROX
6. SV88012	8	40	Spent acid tank to atmosphere
7. SV88014	8	60	Sump to WWTP

#### Other Requirements

The MeCl<sub>2</sub> raw storage tank VE-106 is subject to 40 CFR Part 60, Subpart Kb. The permittee is utilizing the Group 1 storage vessel provisions under 40 CFR Part 63, Subpart FFFF to supersede the NSPS Kb requirements. The on site records and MON MACT reports indicate the facility is in compliance with requirements applicable to tank VE106.

#### RULE 290 Fine Mesh Cation and Anion

September 2017, August 2018, and February 2019 monthly emissions and calculations were reviewed for the Rule 290 Fine Mesh Anion and Fine Mesh Cation. The emissions are based on the pounds of pollutants released per each batch. For the MeCl<sub>2</sub> emissions sent to the 963THROX from the Fine Mesh Cation and from the Fine Mesh Anion a DRE of >99.9% is applied. A DRE of >99.9% is also applied to the HCL emissions from the Fine Mesh Anion sent to the 963THROX.

The records reviewed indicate the facility is in compliance with Rule 290 record keeping requirements and emission limits.

Date	Fine Mesh Anion HCL (limit of 500 lbs/month)	Fine Mesh Anion MeCl <sub>2</sub> (limit of 10 lbs/month)	Fine Mesh Cation MeCl <sub>2</sub> (limit of 10 lbs/month)
Sept 2017	0 lbs/month	0 lbs/month	0.002 lbs/month
Aug 2018	0.09 lbs/month	0.03 lbs/month	0 lbs/month
Feb 2019	0.06 lbs/month	0.02 lbs/month	0 lbs/month

NAME KEB

DATE 8/9/2019

SUPERVISOR C. Kane