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

N751952491

FACILITY: ASH STEVENS LLC		SRN / ID: N7519
LOCATION: 18655 KRAUSE, RIVERVIEW		DISTRICT: Detroit
CITY: RIVERVIEW		COUNTY: WAYNE
CONTACT: Sheryl Goddard , EHS Manager		ACTIVITY DATE: 02/21/2020
STAFF: Jonathan Lamb	COMPLIANCE STATUS: Compliance	SOURCE CLASS: SM OPT OUT
SUBJECT: Scheduled inspection, FY 2020		
RESOLVED COMPLAINTS:		

REASON FOR INSPECTION: Scheduled Inspection  
INSPECTED BY: Jonathan Lamb, AQD-Detroit Office  
PERSONNEL PRESENT: Sheryl Goddard - Manager, Environmental Health and Safety; John Iverson - Operations & Engineering Manager; Manoj Zalpuri - Director of Operation & Operation Excellence Lead; Vince Ammoscato - Vice President, Site Head; Paola Prieto - Associate, Environmental Health and Safety  
FACILITY PHONE NUMBER: 313-282-3370  
FACILITY WEBSITE: www.ashstevens.com  
SAFETY EQUIPMENT REQUIRED: safety glasses; lab coat provided during inspection.

**FACILITY BACKGROUND:**

Ash Stevens, LLC is a pharmaceutical contract manufacturer specializing in the manufacturing of active pharmaceutical ingredients; the facility also performs some research and development. The company was founded in 1962 and the facility has operated at this location since 1988. Ash Stevens is a subsidiary of India-based Piramal Pharma Solutions, which acquired the facility in September 2016. As of December 2019, the facility started operating 7 days per week, 24 hours per day (two 12-hour shifts). There are currently around 140 employees at the facility.

**COMPLAINT/COMPLIANCE HISTORY:**

There have been no complaint or compliance issues at this facility.

**INSPECTION NOTES:**

Since the last inspection in 2016, the facility has doubled the staff on site and increased hours of operation. Throughput has increased, but the products have remained consistent. The facility has increased lab space and R&D equipment (hoods and glassware). The facility currently has plans to expand the building footprint and add equipment within the next two years; the additional equipment is expected to require a modification to the current permit.

**PROCESS DESCRIPTION AND EQUIPMENT:**

Ash Stevens produces the active pharmaceutical ingredients (APIs) used in the full-scale production of pharmaceuticals by other drug manufacturers; there is no final drug or pill produced on site, just the intermediary product.

APIs are produced based on contract specifications using a batch process performed in "bays", which are small rooms containing reactors and other process equipment. The process begins by "charging" a reactor: combining a solid starting material (generally in powder form) with solvents and reactive ingredients in the reactor, forming a slurry. All raw materials are produced off site. The solid material is categorized as FDA-approved complex organic molecules. Solvents include methylene chloride, methanol, acetone, heptane, and ethyl acetate, while the most common reactive agent used is hydrochloric acid.

During processing, condensers may be used to either distill off solvents or to condense vapors, if the

process involves boiling; process condensers are located in the bays while vent condensers are located by the scrubbers. The condensers operate in a closed-loop system, with collected condensate introduced back into the process or sent to another reactor. Once the reactions are complete and the material is tested to make sure it meets contract specifications, the material is extracted from the reactor by either centrifuge or vacuum filtration. The extracted material is allowed to cool and crystallize before being dried in ovens. The synthesis of raw material to final product can involve one to five "activities" (steps of the process), with each activity taking up to a few days. There can be wide variation in the raw materials used for each batch. The quantity of APIs produced per batch can range from less than a kilogram to 50 kilograms. Any solvents distilled off the process are considered a waste product and are collected and shipped off for disposal; currently, the facility sends distilled waste solvents to either Stericycle or US Ecology – Detroit South, both located in Detroit.

There are six bays, all of which contain reactors and condensers. There are five portable filter dyers, which can be moved between bays when needed. The following table lists the emission units at the facility which is permitted under Permit to Install No. 31-12A:

Emission Unit ID	Emission Unit Description (Process Equipment & Control Devices)	Flexible Group ID
EU-MainBay	<p>Main bay process area. Includes four glass-lined reactors, four process condensers, a 40-inch vertical basket centrifuge (ID C5), and a wet scrubber (ID CS1).                      Reactors and nominal capacities are:                          R-4, 100 gallons                      R-10, 500 gallons                          R-12, 300 gallons                    R-15, 100 gallons                      Process condensers and operating temperatures are:                          HX-4, 5 degrees C                    HX-10, 5 degrees C                          HX-12, 5 degrees C                   HX-15, 5 degrees C                      This equipment may be subject to 40 CFR Part 63 Subpart VVVVV when processing HAPs listed in Table 1 of Subpart VVVVV.</p>	FG-MfgAPIs
EU-Bay1100	<p>Bay 1100 process area. Includes one glass-lined reactor, one Hastelloy reactor, and three process condensers.                      Reactors and nominal capacities are:                          RX-1101, 100 liters                    RX-1102, 100 liters                      Process condensers and lowest coolant operating temperatures are:                          HX-1101, -15 degrees C            HX-1102, -15 degrees C                          HX-1103, -15 degrees C                      This equipment exhausts to an emission control condenser with the stated exit temperature:                          HX-2014, 6 degrees F                      This equipment may be subject to 40 CFR Part 63 Subpart VVVVV when processing HAPs listed in Table 1 of Subpart VVVVV.</p>	FG-MfgAPIs
EU-Bay300	<p>Bay 300 process area. Includes three glass-lined reactors and three process condensers.                      Reactors and nominal capacities are:                          RX-0301, 100 gallons                RX-0302, 100 gallons                          RX-0303, 100 gallons                      Process condensers and lowest coolant operating temperatures are:                          HX-0301, -15 degrees C            HX-0302, -15 degrees C                          HX-0303, -15 degrees C                      This equipment exhausts to an emission control condenser with the stated exit temperature:                          HX-2014, 6 degrees F                      This equipment may be subject to 40 CFR Part 63 Subpart</p>	FG-MfgAPIs

	<p>VVVVV when processing HAPs listed in Table 1 of Subpart VVVVV.</p>	
EU-Bay400	<p>Bay 400 process area. Contains a 40-inch vertical basket centrifuge (ID CTFG-0401). This equipment exhausts to an emission control condenser with the stated exit temperature: HX-2014, 6 degrees F This equipment may be subject to 40 CFR Part 63 Subpart VVVVV when processing HAPs listed in Table 1 of Subpart VVVVV.</p>	FG-MfgAPIs
EU-Bay500	<p>Bay 500 process area. Includes three glass-lined reactors and two process condensers. Reactors and nominal capacities are: RX-0501, 100 gallons      RX-0502, 50 gallons RX-0503, 100 gallons Process condensers and lowest coolant operating temperatures are: HX-0501, -15 degrees C      HX-0502, -15 degrees C This equipment exhausts to an emission control condenser with the stated exit temperature: HX-2014, 6 degrees F This equipment may be subject to 40 CFR Part 63 Subpart VVVVV when processing HAPs listed in Table 1 of Subpart VVVVV.</p>	FG-MfgAPIs
EU-Bay600	<p>Bay 600 process area. Includes three glass-lined reactors, four process condensers, a filter dryer with 1.5 square meter filter area (ID FD-0701), and two wet scrubbers (IDs CS2 and SC-2010). Reactors and nominal capacities are: RX-0601, 1000 gallons      RX-0602, 750 gallons RX-0603, 500 gallons Process condensers and lowest coolant operating temperatures are: HX-0601, -15 degrees C      HX-0602, -15 degrees C HX-0603, -15 degrees C      HX-0703, -15 degrees C This equipment exhausts to an emission control condenser with the stated exit temperature: HX-2017, -10 degrees C This equipment may be subject to 40 CFR Part 63 Subpart VVVVV when processing HAPs listed in Table 1 of Subpart VVVVV.</p>	FG-MfgAPIs
EU-VacOven	<p>Vacuum tray dryer with 7 shelves. This equipment exhausts to an emission control condenser with the stated exit temperature: HX-2014, 6 degrees F This equipment may be subject to 40 CFR Part 63 Subpart VVVVV when processing HAPs listed in Table 1 of Subpart VVVVV.</p>	FG-MfgAPIs
EU-FilterDryer1	<p>Portable filter dryer FD-01, 0.3 square meter filter area. This equipment may exhaust without emission control or to an emission control condenser with the stated exit temperature: HX-2014, 6 degrees F This equipment may be subject to 40 CFR Part 63 Subpart VVVVV when processing HAPs listed in Table 1 of Subpart VVVVV.</p>	FG-MfgAPIs
	<p>Portable filter dryer FD-02, 0.03 square meter filter area. This equipment may exhaust without emission control or to an</p>	

EU-FilterDryer2	emission control condenser with the stated exit temperature: HX-2014, 6 degrees F This equipment may be subject to 40 CFR Part 63 Subpart VVVVV when processing HAPs listed in Table 1 of Subpart VVVVV.	FG-MfgAPIs
EU-FilterDryer3	Portable filter dryer FD-03, 0.3 square meter filter area. This equipment may exhaust without emission control or to an emission control condenser with the stated exit temperature: HX-2014, 6 degrees F This equipment may be subject to 40 CFR Part 63 Subpart VVVVV when processing HAPs listed in Table 1 of Subpart VVVVV.	FG-MfgAPIs
EU-FilterDryer4	Portable Rosenmund filter dryer FD-04, 0.1 square meter filter area. This equipment may exhaust without emission control or to an emission control condenser with the stated exit temperature: HX-2014, 6 degrees F This equipment may be subject to 40 CFR Part 63 Subpart VVVVV when processing HAPs listed in Table 1 of Subpart VVVVV.	FG-MfgAPIs
EU-FilterHousng3	Portable Nutsche filter housing FH-03, 0.2 square meter filter area. This equipment exhausts without emission control. This equipment may be subject to 40 CFR Part 63 Subpart VVVVV when processing HAPs listed in Table 1 of Subpart VVVVV.	FG-MfgAPIs
EU-ConvOven	Gruenberg convection oven O-20, with 20 trays. This equipment does not exhaust to an emission control device. This equipment may be subject to 40 CFR Part 63 Subpart VVVVV when processing HAPs listed in Table 1 of Subpart VVVVV.	FG-MfgAPIs

The following additional equipment is exempt from permitting requirements:

- There are three natural gas fired boilers, with heat input capacities of 6 MMBtu, 4.5 MMBtu, and 3.5 MMBtu, respectively, which are exempt from permitting per R.282(2)(b)(i);
- One 6,000-gallon nitrogen tank, which is exempt per R.284(2)(j);
- One 2,500-gallon closed-loop propylene glycol tank used for cooling the processes, which is exempt per R.284(2)(i). The tank does have a conservation vent to relieve pressure when the volume of the tank increases, and the tank has a nitrogen blanket.
- One 387 hp diesel-fired emergency generator, installed in 2003, which is exempt per R.285(2)(g). Facility maintains records of maintenance and hours of operation; based on a review of the operational records, the generator is operated at least one hour per month to test the unit but is otherwise only used in case of power outage. Based on the information provided, this generator appears to meet the definition of an emergency stationary RICE, as defined in 40 CFR 63.6675, including operating according to the provisions specified in 40 CFR 63.6640(f), and is therefore not subject to 40 CFR Part 63, Subpart ZZZZ.
- National Emission Standards for Hazardous Air Pollutants for Stationary Reciprocating Internal Combustion Engines.
- One 1,000-gallon diesel tank for the storage of fuel for the emergency generator, which is exempt per R.284(2)(d).

**PROCESS CONTROLS:**

Depending on the API being produced, emissions may be controlled or uncontrolled. When control is necessary, emissions from processing in the bays are controlled by the condensers, which are operated in a closed-loop system, or by scrubbers. All bays are equipped with condensers; Bay 1100

and Bay 400 are controlled by the condensers only.

The scrubbers are only used when potential emissions include hydrogen chloride, hydrogen sulfide, or ammonia; as such, the scrubbers are not used during the production of most APIs. There are three scrubbers installed: CS-2010, CS-1, and CS-2. CS-2010 controls emissions from Bay 300, Bay 500, and Bay 600. CS-1 controls emissions from the Main Bay. CS-2 has not been used in several years but is installed and capable of being operated, if necessary.

The portable filter dryers can exhaust uncontrolled or through the condensers. Emissions from the vacuum oven are controlled by a condenser.

HEPA filters are used at the end of the process during drying to control particulate emissions.

**APPLICABLE RULES/ PERMIT CONDITIONS:**

Ash Stevens is a synthetic minor source operating under PTI No. 31-12A, issued on May 10, 2016. This permit modification added conditions to limit HCl emissions before control and the amount of reactive chloride atoms processed in the reactors per 12-month rolling time period to allow the facility to opt out of the Title V permitting requirements of 40 CFR Part 63, Subpart VVVVVV – National Emission Standards for Hazardous Air Pollutants for Chemical Manufacturing Area Sources.

For this inspection, production and emission records from January 2018 through January 2020 were reviewed in determining compliance with the conditions of PTI No. 31-12A. These records can be found in the orange facility file.

PTI No. 31-12A, Special Conditions:

FG-MfgAPIs: Equipment used to manufacture active pharmaceutical ingredients. Associated Emission Unit IDs include EU-MainBay, EU-Bay1100, EU-Bay300, EU-Bay400, EU-Bay500, EU-Bay600, EU-VacOven, EU-FilterDryer1, EU-FilterDryer2, EU-FilterDryer3, EU-FilterDryer4, EU-FilterHousng3, and EU-ConvOven.

I. Emissions

Pollutant	Emission Limit	Highest Reported Emissions	Compliance Status
1. VOC	6 tons per 12-month rolling time period	0.34 tons (678 pounds) in 12-month rolling time period ending December 2019.	IN COMPLIANCE
2. Organic compounds that are not VOCs	6 tons per 12-month rolling time period	0.15 tons (292 pounds) in 12-month rolling time period ending Sept. 2019.	IN COMPLIANCE
3. Inorganic Acids	3 tons per 12-month rolling time period	0.01 tons (25 pounds) in 12-month rolling time period ending May 2019.	IN COMPLIANCE
4. Inorganic Bases	3 tons per 12-month rolling time period	0.01 tons (17 pounds) in 12-month rolling time period ending January 2020.	IN COMPLIANCE
5. PM	Less than 0.14 pph	PM testing has not been performed; however, since the maximum monthly total PM reported was 0.002 pounds (April 2019), this condition is assumed to be in compliance.	IN COMPLIANCE
6. PM	Less than 10 lbs/month	0.002 pounds reported in April 2019.	IN COMPLIANCE

7. IN COMPLIANCE. Facility calculates the emission rate of each individual TAC emitted for every batch produced to assure that no TAC exceeds its maximum emission rate (MER). These TACs are tracked on a per-batch basis and recorded in monthly reports, which were reviewed to determine compliance for this inspection. Because the TAC emissions vary for each batch, the individual TACs are not listed in this report, but copies of the monthly reports can be found in the orange facility file.

### III. Process/Operational Restrictions

1. IN COMPLIANCE. A Malfunction Abatement Plan (MAP) for FG-MfgAPIs has been approved by AQD and is implemented and maintained by the facility during operation.
2. IN COMPLIANCE. A scrubber operating plan has been approved by AQD and is implemented and maintained by the facility during operations which require the use of a scrubber.

### IV. Design/Equipment Parameters

1. IN COMPLIANCE. Scrubbers CS1, CS2, and CS-2010 are equipped with liquid flow meters.
2. IN COMPLIANCE. Condensers HX-2014 and HX-2017 are equipped with temperature indicators for exhaust vapors.

### VI. Monitoring/Recordkeeping

1. IN COMPLIANCE. All required calculations are maintained on a monthly basis.
2. IN COMPLIANCE. VOC emission rate from FG-MfgAPIs is calculated on a monthly and 12-month rolling time period basis. Records of emission calculations were provided to AQD.
3. IN COMPLIANCE. Emission rates for organic compounds that are not VOCs, inorganic acids, and inorganic bases are calculated on a monthly and 12-month rolling time period basis. Records of emission calculations were provided to AQD.
4. IN COMPLIANCE. PM emission rates from FG-MfgAPIs are calculated on a monthly basis. Records of PM emission calculations were provided to AQD.
5. IN COMPLIANCE. Facility maintains a description of all processes carried out in FG-MfgAPIs. This information includes the following for each process:
  - a. Raw materials used;
  - b. Products, byproducts, and wastes generated;
  - c. Process step descriptions;
  - d. Process operating variable set points;
  - e. TACs emitted;
  - f. Emission calculations;
  - g. The screening levels and associated averaging times that apply to each TAC.
6. IN COMPLIANCE. Facility maintains monthly records of all processes carried out in FG-MfgAPIs, including dates and times for each process batch and dates and times when pollutants were emitted. A cursory review of these records was performed during the on-site inspection.
7. IN COMPLIANCE. Facility maintains a list of materials used in FG-MfgAPIs that are determined to be exempt from the health-based screening level requirement of Rule 225.
8. IN COMPLIANCE. Facility manually records the process and scrubber parameters whenever a scrubber is used during processing. Scrubber logs for CS-2010 were reviewed on site during the inspection; however, the facility could not locate the written logs for CS-1 for 2018 or 2019 (but did provide the logs for 2016 and 2017). Following the inspection, the facility implemented new tracking procedures to assure that all scrubber records are electronically recorded and maintained as required. Based on the corrective actions taken and the infrequent use of Scrubber CS-1, this condition is determined to be in substantial compliance at this time.
9. IN COMPLIANCE. Facility monitors and records the condenser temperature at least once per shift whenever the process exhausts to either condenser HX-2014 or HX-2017. Condenser temperature logs were reviewed on site during the inspection.

## FG-FACILITY

### 1. Emission Limits

Pollutant	Emission Limit	Emissions	Compliance Status
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1. Individual HAP	Less than 9 tons per 12-month rolling time period	0.09 tons (178 pounds) of Methylene Chloride in the 12-month rolling time period ending June 2019.	IN COMPLIANCE
2. Aggregate HAPs	Less than 22.5 tons per 12-month rolling time period	0.2 tons (409 pounds) in the 12-month rolling time period ending June 2019.	IN COMPLIANCE
3. HCl (before emission control)	Less than 9 tons per 12-month rolling time period	0.12 tons (235 pounds) in the 12-month rolling time period ending January 2020.	IN COMPLIANCE

II. Material Limits

1. IN COMPLIANCE. Mass of reactive chlorine atoms fed to the reactors was below the permit limit of 17,000 pounds per 12-month rolling time period. The highest 12-month rolling total since issuance of PTI No. 31-12A was 2,958 pounds in the 12-month rolling time period ending January 2020.

VI. Monitoring/Recordkeeping

1. IN COMPLIANCE. The facility maintains monthly and 12-month rolling time period records of the material feeds listed below:

- a. The identity and quantity of each reagent containing reactive chlorine fed to reactors in FG-FACILITY during each month and 12-month rolling time period.
- b. The mass of reactive chlorine atoms fed to reactors in FG-FACILITY during each month and 12-month rolling time period.

2. IN COMPLIANCE. The facility calculates the following emission rates on a 12-month rolling time period basis. Records were provided to AQD during the inspection:

- a. Each individual HAP from FG-FACILITY
- b. Aggregate HAPs from FG-FACILITY
- c. HCl before emission control from FG-FACILITY, based on mass balance of reactive chlorine atoms fed to reactors in FG-FACILITY.

VIII. Stack/Vent Restrictions

1 through 7. IN COMPLIANCE. According to facility documentation, stacks SV\_EF-4, SV\_EF-5, SV\_V-2014, SV\_V-2017, SV\_V201020, SV\_V201025, and SV\_OvenO-20 meet permit specifications.

IX. Other Requirements

1. IN COMPLIANCE. Facility complies with the provisions of 40 CFR Part 63, Subpart VVVVVV, as they apply to the emission units in FG-FACILITY.

**FINAL COMPLIANCE DETERMINATION:**

At the time of inspection, Ash Stevens Inc. was determined to be in substantial compliance with the conditions of PTI No. 31-12A and other applicable state and federal air regulations at the time of inspection.

NAME 

DATE 8-24-2020

SUPERVISOR 