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DEPARTMENT OF ENVIRONMENTAL QUALITY
AIR QUALITY DIVISION
ACTIVITY REPORT: On-site Inspection

N751967554

FACILITY: ASH STEVENS LLC		SRN / ID: N7519
LOCATION: 18655 KRAUSE, RIVERVIEW		DISTRICT: Detroit
CITY: RIVERVIEW		COUNTY: WAYNE
CONTACT: Jay Brown , Associate Director of EHS		ACTIVITY DATE: 05/31/2023
STAFF: Jonathan Lamb	COMPLIANCE STATUS: Compliance	SOURCE CLASS: SM OPT OUT
SUBJECT: Scheduled inspection, FY '23		
RESOLVED COMPLAINTS:		

INSPECTED BY: Jonathan Lamb, EGLE-AQD

PERSONNEL PRESENT: Jay Browns, Assistant Director EHS; Joe Monkiewicz, North American Regional Headquarters EHS; Jafar Momani, Senior EHS Specialist

FACILITY PHONE NUMBER: 313-282-3370

FACILITY WEBSITE: www.piramalpharmasolutions.com/our-facilities/riverview

SAFETY EQUIPMENT REQUIRED: safety glasses; lab coat provided during inspection.

FACILITY BACKGROUND:

Ash Stevens, LLC is a pharmaceutical manufacturer specializing in the manufacturing of active pharmaceutical ingredients; the facility also performs some drug 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. The facility operates 7 days per week, 24 hours per day. There are currently 194 employees at this facility.

COMPLAINT/COMPLIANCE HISTORY:

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

INSPECTION NOTES:

Since the last inspection in 2020, the facility expanded operations into an adjacent building to include an additional reactor bay (Bay 1400). Permit to Install (PTI) No. 31-12B was issued in October 2021 to permit the additional equipment and the equipment was put into operation in February 2023. The facility has plans to install additional equipment in this building in the near future, which will likely require another permit modification.

Additional note: Ash Stevens operated a lab facility located at 5861 John C. Lodge Freeway in Detroit. This site has been closed and the company is currently in the process of putting the building up for sale. Mr. Brown does not believe there are any air permits associated with this address, as the operations at this site would have been exempt from permitting requirements. There does not appear to be a file for this facility nor was it found in AQD databases.

PROCESS DESCRIPTION AND EQUIPMENT:

Ash Stevens produces the active pharmaceutical ingredients (APIs) used in the full-scale production of pharmaceuticals. These APIs may be used to create a final pharmaceutical product at this facility or are produced for other pharmaceutical companies on a contract basis.

APIs are produced based on contract specifications using batch processes 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. 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 are permitted under Permit to Install No. 31-12B:

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).</p> <p>Reactors and nominal capacities are: R-4, 100 gallons R-10, 500 gallons R-12, 300 gallons R-15, 100 gallons</p> <p>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</p> <p>This equipment may be subject to 40 CFR Part 63 Subpart VVVVVV when processing HAPs listed in Table 1 of Subpart VVVVVV.</p>	FG-MfgAPIs
EU-Bay1100	<p>Bay 1100 process area. Includes one glass-lined reactor, one Hastelloy reactor, and three process condensers.</p> <p>Reactors and nominal capacities are: RX-1101, 100 liters RX-1102, 100 liters</p> <p>Process condensers and lowest coolant operating temperatures are: HX-1101, -15 degrees C HX-1102, -15 degrees C HX-1103, -15 degrees C</p> <p>This equipment exhausts to an emission control condenser with the stated exit temperature: HX-2014, 6 degrees F</p> <p>This equipment may be subject to 40 CFR Part 63 Subpart VVVVVV when processing HAPs listed in Table 1 of Subpart VVVVVV.</p>	FG-MfgAPIs

Emission Unit ID	Emission Unit Description (Process Equipment & Control Devices)	Flexible Group ID
EU-Bay300	<p>Bay 300 process area. Includes three glass-lined reactors and three process condensers.</p> <p>Reactors and nominal capacities are: RX-0301, 100 gallons RX-0302, 100 gallons RX-0303, 100 gallons</p> <p>Process condensers and lowest coolant operating temperatures are: HX-0301, -15 degrees C HX-0302, -15 degrees C HX-0303, -15 degrees C</p> <p>This equipment exhausts to an emission control condenser with the stated exit temperature: HX-2014, 6 degrees F</p> <p>This equipment may be subject to 40 CFR Part 63 Subpart VVVVVV when processing HAPs listed in Table 1 of Subpart VVVVVV.</p>	FG-MfgAPIs
EU-Bay400	<p>Bay 400 process area. Contains a 40-inch vertical basket centrifuge (ID CTFG-0401).</p> <p>This equipment exhausts to an emission control condenser with the stated exit temperature: HX-2014, 6 degrees F</p> <p>This equipment may be subject to 40 CFR Part 63 Subpart VVVVVV when processing HAPs listed in Table 1 of Subpart VVVVVV.</p>	FG-MfgAPIs
EU-Bay500	<p>Bay 500 process area. Includes three glass-lined reactors and two process condensers.</p> <p>Reactors and nominal capacities are: RX-0501, 100 gallons RX-0502, 50 gallons RX-0503, 100 gallons</p> <p>Process condensers and lowest coolant operating temperatures are: HX-0501, -15 degrees C HX-0502, -15 degrees C</p> <p>This equipment exhausts to an emission control condenser with the stated exit temperature: HX-2014, 6 degrees F</p> <p>This equipment may be subject to 40 CFR Part 63 Subpart VVVVVV when processing HAPs listed in Table 1 of Subpart VVVVVV.</p>	FG-MfgAPIs

Emission Unit ID	Emission Unit Description (Process Equipment & Control Devices)	Flexible Group ID
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).</p> <p>Reactors and nominal capacities are: RX-0601, 1000 gallons RX-0602, 750 gallons RX-0603, 500 gallons</p> <p>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</p> <p>This equipment exhausts to an emission control condenser with the stated exit temperature: HX-2017, -10 degrees C</p> <p>This equipment may be subject to 40 CFR Part 63 Subpart VVVVVV when processing HAPs listed in Table 1 of Subpart VVVVVV.</p>	FG-MfgAPIs
EU-Bay1400	<p>Bay 1400 process area. Includes two glass-lined reactors, one Hastelloy reactor, a filter dryer with a 1.0 square meter filter area (ID FD01501), and four process condensers.</p> <p>Reactors and nominal capacities are: RX-1401, 1,000 gallons; RX-1402, 750 gallons; RX-1403, 750 gallons</p> <p>Process condensers and lowest coolant operating temperatures are: HX-1401, -15 degrees C HX-1402, -15 degrees C HX-1403, -15 degrees C HX-1501, -15 degrees C</p> <p>This equipment exhausts to an emission control condenser with the stated exit temperature: HX-3014, -14 degrees C</p> <p>This equipment may be subject to 40 CFR Part 63 Subpart VVVVVV when processing HAPs listed in Table 1 of Subpart VVVVVV.</p>	FG-MfgAPIs
EU-VacOven	<p>Vacuum tray dryer with 7 shelves.</p> <p>This equipment exhausts to an emission control condenser with the stated exit temperature: HX-2014, 6 degrees F</p> <p>This equipment may be subject to 40 CFR Part 63 Subpart VVVVVV when processing HAPs listed in Table 1 of Subpart VVVVVV.</p>	FG-MfgAPIs

Emission Unit ID	Emission Unit Description (Process Equipment & Control Devices)	Flexible Group ID
EU-FilterDryer1	<p>Portable filter dryer FD-01, 0.3 square meter filter area.</p> <p>This equipment may exhaust without emission control or to an emission control condenser with the stated exit temperature: HX-2014, 6 degrees F</p> <p>This equipment may be subject to 40 CFR Part 63 Subpart VVVVVV when processing HAPs listed in Table 1 of Subpart VVVVVV.</p>	FG-MfgAPIs
EU-FilterDryer2	<p>Portable filter dryer FD-02, 0.03 square meter filter area.</p> <p>This equipment may exhaust without emission control or to an emission control condenser with the stated exit temperature: HX-2014, 6 degrees F</p> <p>This equipment may be subject to 40 CFR Part 63 Subpart VVVVVV when processing HAPs listed in Table 1 of Subpart VVVVVV.</p>	FG-MfgAPIs
EU-FilterDryer3	<p>Portable filter dryer FD-03, 0.3 square meter filter area.</p> <p>This equipment may exhaust without emission control or to an emission control condenser with the stated exit temperature: HX-2014, 6 degrees F</p> <p>This equipment may be subject to 40 CFR Part 63 Subpart VVVVVV when processing HAPs listed in Table 1 of Subpart VVVVVV.</p>	FG-MfgAPIs
EU-FilterDryer4	<p>Portable Rosenmund filter dryer FD-04, 0.1 square meter filter area.</p> <p>This equipment may exhaust without emission control or to an emission control condenser with the stated exit temperature: HX-2014, 6 degrees F</p> <p>This equipment may be subject to 40 CFR Part 63 Subpart VVVVVV when processing HAPs listed in Table 1 of Subpart VVVVVV.</p>	FG-MfgAPIs
EU-FilterDryer5	<p>Portable Rosenmund filter dryer FD-05, 0.3 square meter filter area.</p> <p>This equipment may exhaust without emission control or to an emission control condenser with the stated exit temperature: HX-2014, 6 degrees F; HX-2017, 6 degrees F</p> <p>This equipment may be subject to 40 CFR Part 63 Subpart VVVVVV when processing HAPs listed in Table 1 of Subpart VVVVVV.</p>	FG-MfgAPIs
EU-FilterHousng3	<p>Portable Nutsche filter housing FH-03, 0.2 square meter filter area.</p> <p>This equipment exhausts without emission control.</p> <p>This equipment may be subject to 40 CFR Part 63 Subpart VVVVVV when processing HAPs listed in Table 1 of Subpart VVVVVV.</p>	

Emission Unit ID	Emission Unit Description (Process Equipment & Control Devices)	Flexible Group ID
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 VVVVVV when processing HAPs listed in Table 1 of Subpart VVVVVV.	FG-MfgAPIs
EU-MixTank	Portable mixing tank, 100 gallon capacity. This equipment may be subject to 40 CFR Part 63 Subpart VVVVVV when processing HAPs listed in Table 1 of Subpart VVVVVV.	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.
- Two 387 hp diesel-fired emergency generators, installed in 2003 and 2023, respectively, which are 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 for readiness testing but is otherwise only used in case of power outage. Based on the information provided, these generators appear 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 controls are used, 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 four scrubbers installed: CS-1, CS-2, SC-2010, and SC-3010. SC-2010 controls emissions from Bay 300, Bay 500, and Bay 600. SC-3010 controls emissions from Bay 1400. 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. 31-12B, issued on October 14, 2021. This modification permitted equipment associated with Bay 1400. This permit limits HAP emissions below major source thresholds, plus has conditions to limit the emissions of HCl (before control) and the amount of reactive chloride atoms processed in the reactors per 12-month rolling time period, allowing 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 2020 through April 2023 were reviewed in determining compliance with the conditions of PTI No. 31-12B. These records can be found in the orange facility file.

PTI No. 31-12B, 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-Bay1400; EU-VacOven, EU-FilterDryer1, EU-FilterDryer2, EU-FilterDryer3, EU-FilterDryer4, EU-FilterDryer5, EU-FilterHousng3, EU-ConvOven, and EU-MixTank.

I. Emissions

Pollutant	Emission Limit	Highest Reported Emissions	Compliance Status
1. VOC	6 tons per 12-month rolling time period	0.33 tons (668 pounds) in 12-month rolling time period ending January 2020; 0.26 tons (515 pounds) in the 12-month rolling time period ending April 2023.	IN COMPLIANCE
2. Organic compounds that are not VOCs	6 tons per 12-month rolling time period	0.15 tons (304 pounds) in 12-month rolling time period ending May 2020; 0.09 tons (178 pounds) in the 12-month rolling time period ending April 2023.	IN COMPLIANCE
3. Inorganic Acids	3 tons per 12-month rolling time period	0.04 tons (81 pounds) in the 12-month rolling time period ending April 2023.	IN COMPLIANCE
4. Inorganic Bases	3 tons per 12-month rolling time period	0.01 tons (20 pounds) in 12-month rolling time period ending April 2021; 0.003 tons (6 pounds) in the 12-month rolling time period ending April 2023.	IN COMPLIANCE
5. PM	Less than 0.14 pph	PM testing has not been performed; however, since the maximum monthly total PM emitted was 0.004 pounds (April 2023), this condition is assumed to be in compliance.	IN COMPLIANCE
6. PM			IN COMPLIANCE

	Less than 10 pounds per calendar month	0.004 pounds emitted during the month of April 2023.	
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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, SC-2010, and SC-3010 are equipped with liquid flow meters.
2. IN COMPLIANCE. Condensers HX-2014, HX-2017, and HX-3014 are equipped with temperature indicators for exhaust vapors.

VI. Monitoring/Recordkeeping

1. NOT IN COMPLIANCE. Facility failed to provide monthly emission calculations for the time period January 2023 through April 2023. Records from January 2020 through December 2022 were provided.
2. NOT IN COMPLIANCE. Facility failed to provide calculations of the VOC emission rate from FG-MfgAPIs on a monthly and 12-month rolling time period basis for the time period January 2023 through April 2023. Records from January 2020 through December 2022 were provided.
3. NOT IN COMPLIANCE. Facility failed to provide calculations of the emission rates for organic compounds that are not VOCs, inorganic acids, and inorganic bases on a monthly and 12-month rolling time period basis for the time period January 2023 through April 2023. Records from January 2020 through December 2022 were provided.
4. NOT IN COMPLIANCE. Facility failed to maintain a record of the data and calculations used to demonstrate compliance with S.C. 1.5 for each activity generating PM emissions for the time period January 2023 through April 2023. Records from January 2020 through December 2022 were maintained.
5. NOT IN COMPLIANCE. Facility failed to provide PM emission rates from FG-MfgAPIs on a monthly basis for the time period January 2023 through April 2023. Records from January 2020 through December 2022 were provided.
6. NOT IN COMPLIANCE. Facility failed to maintain a description of all processes carried out in FG-MfgAPIs for the time period January 2023 through April 2023. Descriptions of all processes carried out in FG-MfgAPIs for the time period January 2020 through December 2022 were provided. 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.
7. 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.
8. 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.

9. IN COMPLIANCE. Facility manually records the process and scrubber parameters whenever a scrubber is used during processing. Scrubber logs for were reviewed on site during the inspection.

10. IN COMPLIANCE. Facility monitors and records the condenser temperature at least once per shift whenever the process exhausts to either condenser HX-2014, HX-2017, or HX-3014. Condenser temperature logs were reviewed on site during the inspection.

FG-FACILITY

1. Emission Limits

Pollutant	Emission Limit	Emissions	Compliance Status
1. Individual HAP	Less than 9 tons per 12-month rolling time period	Methylene Chloride is the individual HAP with the highest emission rate on an annual basis. 0.13 tons (259 pounds) of Methylene Chloride in the 12-month rolling time period ending December 2020; 0.07 tons (143 pounds) in the 12-month rolling time period ending April 2023.	IN COMPLIANCE
2. Aggregate HAPs	Less than 22.5 tons per 12-month rolling time period	0.24 tons (472 pounds) in the 12-month rolling time period ending December 2020; 0.22 tons (431 pounds) in the 12-month rolling time period ending April 2023.	IN COMPLIANCE
3. HCl (before emission control)	Less than 9 tons per 12-month rolling time period	0.24 tons (490 pounds) in the 12-month rolling time period ending April 2023; 0.24 tons (490 pounds) in the 12-month rolling time period ending April 2023.	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 was 2,738 pounds in the 12-month rolling time period ending December 2022. A total of 2,271 pounds were fed to the reactors for the 12-month rolling time period ending April 2023.

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.

2a. and b. NOT IN COMPLIANCE. The facility failed to provide emission calculations for individual and aggregate HAPs for the 12-month rolling time periods ending January 2023 through April 2023. Emission calculations for the time period January 2020 through December 2022 were provided.

2c. IN COMPLIANCE. Facility calculates the emission rate of HCl (before emission control) from FG-FACILITY on a monthly and 12-month rolling time period basis, based on mass balance of reactive chlorine atoms fed to reactors in FG-FACILITY.

VIII. Stack/Vent Restrictions

1 through 9. IN COMPLIANCE. According to facility documentation, stacks SV_EF-4, SV_EF-5, SV_V-2014, SV_V-2017, SV_V201020, SV_V201025, SV_OvenO-20, SV_3014, and SV_3010 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 was determined to be in noncompliance with the conditions of PTI No. 31-12B. Specifically, the facility was in noncompliance with FG-MfgAPIs, Special Conditions VI.1 through 6 and FGFACILITY, VI.2a. and b., for failure to calculate various emission rates for the time period January 2023 through April 2023. As a result, a Violation Notice was issued to Ash Stevens on October 26, 2023.

The facility provided the emission records to AQD on November 29, 2023, resolving the violations. AQD has amended the inspection report to reflect the updated emission data. There were no emission exceedances during the compliance period and the violations cited in the October 26, 2023 Violation Notice are now considered to be resolved.

NAME 

DATE 5-29-24

SUPERVISOR JK