United States Environmental Protection Agency
Office of Enforcement and Compliance Assurance
Office of Criminal Enforcement, Forensics and Training

National Enforcement Investigations Center

NEICVP1508E01

NEIC CIVIL INVESTIGATION REPORT
Calgon Carbon – Big Sandy Plant
Catlettsburg, Kentucky 41129

Investigation Dates:
March 21-29, 2023

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APPENDICES (*NEIC-created documents)

A Calgon Carbon Big Sandy Plant Final Permit KY0022276 (27 pages)
B Wastewater Flow Diagram (2 pages)
C Calgon Carbon RCRA Permit (126 pages)
D Prime Lines Process Diagram (3 pages)
E Acid Wash Process Diagram (1 page)
F FluePAC Process Diagram (1 page)
G Reactivation Process Diagram (1 page)
H VP1508 Calgon Carbon- Big Sandy Plant – Photographs (91 pages)*
I Calgon Carbon ERG Sampling Report (28 pages)
J Sample Chain of Custody Record (2 pages)*
K Calgon Carbon - Big Sandy Plant - Laboratory Photos (6 pages)*
L Calgon Carbon-SWPPP-BMP (259 pages)
M REACT Trench Photographs (1 page)
N 3-22-23 Spent Carbon Spill (1 page)
O Calgon Carbon RCRA Permit Application (1527 pages)

P REACT Dike Cleanup Photograph (1 page)

These Contents pages shows all the sections contained in this report and provides a clear indication of the end of this report.

INVESTIGATION OVERVIEW

PROJECT OBJECTIVE

U.S. Environmental Protection Agency (EPA) Office of Civil Enforcement (OCE) Water Enforcement Division (WED) and Waste and Chemical Enforcement Division (WCED), and Region 4 (Region), requested EPA's National Enforcement Investigations Center (NEIC) to conduct a multimedia compliance investigation of the Calgon Carbon – Big Sandy Plant (Calgon) facility located at 15024 Old United States Route 23 South, in Catlettsburg, Kentucky, 41129.

The primary purpose of the inspection was to conduct a comprehensive compliance evaluation inspection of the facility under the Clean Water Act (CWA) and the Resource Conservation and Recovery Act (RCRA). NEIC inspectors also conducted an evaluation of per and polyfluoroalkyl substances (PFAS) wastewater generation, management, treatment, and effluent discharge. Appropriate samples of effluent wastewater were collected for analysis by the NEIC laboratory for PFAS and submission to EPA Office of Research and Development (ORD) for non-targeted analysis of PFAS compounds. Soil and sediment samples were collected by CWA team members for analysis at the ORD laboratory in Narragansett, Rhode Island. NEIC inspectors also evaluated the facility's RCRA compliance including compliance with the requirement to make hazardous waste determinations, evaluation of waste management practices including inspections of process areas, satellite hazardous waste accumulation areas, and less-than-90-day hazardous waste accumulation areas and tanks. RCRA team members also conducted an evaluation of waste management practices of solid and hazardous wastes containing PFAS.

Table 1 lists the project team members.

Table 1. PROJECT TEAM MEMBERS		
Team Member	**Organization**	**Project Role**
Mike Solter	NEIC	Project manager (PM)
Alex Flevarakis	NEIC	Field team – CWA inspector
Craig Lutz	NEIC	Field team – RCRA team lead and inspector
Trent Rainey	NEIC	Field team – CWA inspector
Zach Schlachter	NEIC	Field team – RCRA inspector
Daniel Hurlbut	NEIC	Analytical project manager (APM)
Jessica Krank	NEIC	Laboratory analyst
Daya Mitchell	NEIC	Laboratory analyst
REGIONAL AND OTHER CONTACTS		
Scott Gerstner	Kentucky Department for Environmental Protection (KY-DEP)	RCRA team member
Laurence Libelo	EPA-OCE-WED	CWA team member
Robert Nakamoto	EPA Region 4	RCRA team member
Nikki Radford	EPA Region 4	CWA team member
Ben Walter	KY-DEP	RCRA team member
Justin Young	EPA-OCE-WCED	RCRA team member

FACILITY CONTACT INFORMATION

Table 2 lists the primary facility contacts.

Table 2. FACILITY CONTACT INFORMATION		
Name, Title	**Phone No.**	**Email Address**
Eric Nash, Regional EH&S manager	606-471-4233	eric.nash@kuraray.com
Marvin Church, Director, EH&S	412-787-6724	mchurch@calgoncarbon.com
Brian Lauerman, Plant manager	606-739-2326	blauerman@calgoncarbon.com
Charles Brown, Assistant plant manager	606-739-2308	charles.brown@kuraray.com

FACILITY OVERVIEW

Calgon is an activated carbon manufacturing and regeneration facility located on the west bank of the Big Sandy River, on the border between Kentucky and West Virginia. The total site is approximately 157 acres in size, and the active operation occupies approximately 40 acres. There is an additional 7-acre landfill located on the western side of U.S. Highway 23. The landfill is active but has not received waste since approximately 2002. The facility has been operating at its current location since the 1960s and has expanded several times over its operational life. Calgon has a staff of approximately 210 personnel and operates in three shifts, 24 hours per day, 7 days per week. The facility produces roughly 100 million pounds per year of virgin activated carbon products and an additional 35 million pounds per year of reactivated carbon products.

CWA Applicability

Calgon is authorized to discharge process wastewater, stormwater, and non-contact cooling water to the Big Sandy River and White's Creek under National Pollutant Discharge Elimination System (NPDES) issued by the State of Kentucky as the Kentucky Pollutant Discharge Elimination System (KYPDES) permit No. KY0022276 (**Appendix A**). The permit does not contain any requirements to monitor for PFAS compounds in the discharges. According to EPA's Enforcement and Compliance History Online (ECHO) website, Calgon was last inspected for CWA requirements on September 14, 2021, as a state-conducted base program evaluation. The facility has received seven CWA notices of violation from the state of Kentucky since October 2019.

Table 3 summarizes the outfalls authorized by the permit.

Table 3. KYPDES Permit No. KY0022276 Outfalls

Outfall	Direct/Internal	Receiving Water	PFAS Compound Monitoring	Description of Discharge
001	Direct	Big Sandy River	No	Boiler blowdown and backwash Prime line bakers wet scrubbers Acid wash / neutralization wastewater Wash down Non-contact Cooling Stormwater Reactivation (REACT) wastewater (Outfall 004)
002	Direct	Big Sandy River	No	Domestic sanitary wastewater
003	Direct	White's Creek	No	Stormwater runoff from closed and capped landfill
004	Internal	Outfall 001	No	Motive water from REACT Stormwater from REACT area

The outfalls listed in Table 3 are depicted in the site wastewater flow diagram attached as **Appendix B.** The KYPDES permit expands on the monitoring requirements to specify that the monthly average discharge concentration for total recoverable selenium of 0.179 milligrams per liter (mg/L) is a trigger. If exceeded, the permit requires the permittee to collect and analyze fish tissue for selenium residue., If the fish tissue analysis results in a selenium concentration less than or equal to 8.6mg/Kg dry weight, then there is no permit violation.

RCRA Applicability

Calgon (EPA RCRA ID No. KYD005009923) is a hazardous waste treatment and storage facility and a large quantity generator of hazardous waste. Calgon has been permitted by the Kentucky Department of Environmental Protection (KY-DEP) to receive, store, consolidate, and treat a variety of hazardous and non-hazardous waste streams associated with receiving and reactivating spent carbon. A RCRA permit renewal was issued on December 19, 2019 (**Appendix C**). Typical waste streams generated on-site include wastewater fines, wastewater sludge, spent soda, furnace slag, paint residues, solvents, and lab packs.

FACILITY OPERATIONS SUMMARY

Calgon operations consist of three prime line (virgin) activated carbon manufacturing lines, one flue gas powdered activated carbon or "FluePAC®" line, one fine mesh line, one acid wash line, and the reactivation (REACT) line. The facility also operates an on-site wastewater treatment plant and a domestic wastewater treatment package plant.

Prime Lines

The prime lines, labeled as B, D, and E, are the primary production lines for newly manufactured activated carbon products. With slight variations noted in the production flow diagrams, all three of the prime lines consist of similar processes. Raw coal or other carbon is added to bakers and heated before shifting into afterburners for additional heating in a spiral furnace. Once the carbon is fully activated, it is cooled in a cooler drum before being routed to other lines or for sale to customers. Non-contact cooling water is used in the prime lines to cool the carbon products after activation. A process flow diagram of the prime lines is included in **Appendix D**.

Fine Mesh Line

The fine mesh manufacturing process consists of a series of screens intended to screen the activated carbon product into various sizes based on customer specifications for the finished product. The fine mesh process does not use any process water.

Acid Wash Line

The acid wash product line takes finished activated carbon from the D or E prime lines and turns it into an acid washed product. The primary purpose of acid washing carbon is to remove water soluble ash and other materials so the product may be used in drinking water and other food-grade applications.

The carbon is first introduced into charge bins before being added to reactor vessels where it is exposed to a hydrochloric acid solution. Once the reaction is complete, the product is moved to dewatering bins before being dried and prepared for packaging. Water from the process is discharged to the on-site wastewater treatment system through the on-site sewers. A process flow diagram of the acid wash line is included in **Appendix E**.

FluePAC® Line

The FluePAC® process consists of proprietary additions of material to the activated carbon from the D and E lines to make specialty products for industrial applications. No water is used in the FluePAC® production process, and dehumidifiers are used to minimize the moisture in the processing areas. Water from the dehumidifiers is routed to the on-site sewers. A process flow diagram of the FluePAC® line is included in **Appendix F.**

Reactivation Line

The reactivation area consists of hazardous waste storage tanks, associated lines, and process units to treat spent activated carbon for reuse. The main treatment unit is a regeneration furnace that removes the organics from the spent carbon. The furnace and associated pollution control equipment are regulated under a Clean Air Act permit (not reviewed as part of this inspection). The hazardous waste storage tanks and associated piping are regulated under the RCRA permit. A process flow diagram of the reactivation line is included in **Appendix G**.

Water Treatment

The facility obtains its process water from an intake structure located on the Big Sandy River (**Appendix H**, photo 34). The approximate daily intake is 3.2 million gallons, to which chemical flocculants are first added to help precipitate solids out of the water in the clarifiers (**Appendix H**, photos 35 and 36). Water is then sent to the process areas for use. Additional sand filters, ultra filtration, reverse osmosis, and water softening may occur depending on the process for which the water is intended. Water for domestic purposes is provided by the Big Sandy Water District.

Wastewater Treatment Systems

Calgon has two primary wastewater treatment systems including a main system for process wastewater and a package wastewater treatment plant for domestic wastewater.

Process Wastewater Treatment System

The process wastewater treatment system consists of a fines collection system using a fines pit and fines box (**Appendix H**, photos 30 and 31), a clarifier tank, mix tank, and filter press (**Appendix H**, photo 32). After the treatment works, wastewater is directed through a series of three settling ponds (lagoons) in sequence. At the time of the inspection, only two of the settling ponds were in use as the first pond in the sequence was being dredged and rehabilitated. Overflows from the fines pit and clarifier tanks are directed into lagoon 2 (**Appendix H**, photo 27). Final effluent is discharged through an outlet structure in lagoon 3 (**Appendix H**, photo 24). There is also a spillway overflow on lagoon 3 (**Appendix H**, photo 25) and Calgon representatives stated the sandbags that were observed by NEIC inspectors are to prevent inflow from the Big Sandy River in the event of flooding.

Settled material removed from the settling ponds are sent to either the Boyd County sanitary landfill or the Big Run landfill and may be used as coal industry bed material. Eastern Research Group (ERG) was contracted by EPA OCE-WCED to collect waste stream samples for hazardous waste and PFAS analysis and this material. ERG collected samples of the settled material. The sample are SP-4 and SP-5 and the analysis results are in **Appendix I**.

A process flow diagram of the wastewater treatment system, with estimated water usage, is included in **Appendix B**.

Domestic Wastewater Treatment Package Plant

Domestic wastewater for the entire facility is treated by a secondary treatment plant (**Appendix H**, photo 33). Calgon uses a steel package plant rated at 20,000 gallons per day. Treatment process units include a lift station equipped with two grinder pumps, an equalization basin, an aeration tank with solids recirculation, a rectangular clarifier, and ultraviolet disinfection. Diffused air is provided for aeration and mixing. The aeration system operates intermittently. During the summer months, it is on for 45 minutes out of every hour and off for 15 minutes. During the winter, the operation cycle is 30 minutes on and 30 minutes off each hour. Solids in the clarifier are not continuously wasted. When the blanket depth reaches 3 feet above the bottom of the clarifier, the solids are pumped down into a holding tank until the blanket depth is within 1 foot of the clarifier bottom. Effluent from the clarifier is disinfected and then discharged through Outfall 002. AZ Septic is contracted by Calgon to remove the sanitary waste sludge.

FIELD ACTIVITIES SUMMARY

NEIC conducted the inspection from March 21-29, 2023. NEIC, OCE-WED, OCE-WCED, EPA Region 4, and State of Kentucky inspectors, arrived at the facility at 9:00 a.m. on March 21, 2023, at which time credentials were presented to the facility staff listed in Table 2 of this report. NEIC conducted an opening conference that explained the purpose and scope of the inspection.

During the inspection of Calgon, NEIC inspectors conducted the following activities:

- Evaluated on-site processes with an emphasis on waste management and PFAS wastewater generation, management, treatment, and effluent discharge, including meeting with facility personnel to discuss facility operations;

- Inspected and observed facility process operations, wastewater generation sources, treatment facilities, outfall locations, and sampling and monitoring locations;

- Photographed and documented site conditions;

- Reviewed facility documents, including operating plans, records, monitoring reports, and analytical data;

- Collected samples of stormwater and wastewater effluent discharges for PFAS compound analysis, including storm water runoff samples;

- Collected duplicate samples of wastewater for analysis by EPA ORD;

- Collected samples of soil/sediments for analysis by EPA ORD;

- Reviewed hazardous waste determinations;
- Inspected hazardous and solid waste management and storage practices;
- Performed leak detection and repair (LDAR) monitoring using a flame ionization detector of tank openings and closed vent systems;
- Surveyed for volatile organic compound emissions with an infrared camera;
- Performed analysis of a portion of collected samples at the NEIC laboratory in Denver, Colorado.

At the request of EPA OCE-WED, duplicate samples were not collected from locations that were determined to be hazardous because EPA ORD is unable to process samples that meet the definition of RCRA hazardous waste. NEIC's investigation did not include sampling of groundwater wells or sampling in surface waters.

ERG was contracted by EPA OCE-WCED to collect waste stream samples for hazardous waste and PFAS analysis. ERG's sampling report and analytical results can be found in **Appendix I**. ERG collected samples from four locations. Samples SP-1 and SP-2 were from drums with the waste profiles of 3551N and 4012N respectively. Sample SP-3 was collected from a filter press in the reactivation area. Sample SP-4was collected from a pile of material dredged from the wastewater lagoons. As described above, this is the material removed from the settling ponds and sent to either the Boyd County sanitary landfill or the Big Run landfill where it may be used as coal industry bed material.

Measurement and Sampling Activities

The NEIC field team provided support to this investigation by collecting samples of process wastewater and stormwater runoff. Samples were provided to the NEIC laboratory for PFAS analysis and the ORD laboratory for non-targeted PFAS analysis. All samples were screened in the field for radionuclide levels to ensure no radioactive materials were sent to the NEIC laboratory. The NEIC field team also provided support to this investigation by conducting LDAR monitoring of regulated process areas and equipment. **Table 4** summarizes field measurement and field sampling activities. A copy of the chain of custody record is provided in **Appendix J**.

All environmental measurement activities were performed in accordance with the NEIC quality system. All field sampling, field measurements/monitoring, and laboratory measurements described in this report are within the scope of NEIC's ISO/IEC 17025 accreditation issued by the ANSI National Accreditation Board (certificate No. FT-0303). Sampling performed by ERG and analysis performed by Eurofins Lancaster Laboratories are not within the scope of NEIC's ISO/IEC 17025 accreditation.

On March 24, 2023, conditions were rainy and overcast. This allowed for the collection of stormwater runoff samples.

<table>
<tr><th colspan="6">Table 4. FIELD MEASUREMENT AND FIELD SAMPLING ACTIVITIES</th></tr>
<tr><th colspan="2">Location Identifier</th><th colspan="2">Dates</th><th>Method, and/or Procedure[1], and Equipment</th><th>Measurer Name</th></tr>
<tr><th colspan="6">MEASUREMENTS</th></tr>
<tr><td colspan="2">MSA Altair used to screen location for safety</td><td colspan="2">3-24-23
and
3-27-23</td><td>NEIC procedure: Safety and Sample Screening Instruments, NEICPROC/17-002
Instrument guide:
MSA Altair 5X Multi-Gas Monitoring Equipment

Equipment: MSA Altair 5X (SN2420)</td><td>Mike Solter
Craig Lutz</td></tr>
<tr><td colspan="2">Radeye used to screen samples for radiation</td><td colspan="2">3-24-23
and
3-27-23</td><td>NEIC procedure: Safety and Sample Screening Instruments, NEICPROC/17-002
Instrument guide:
RadEye B20-ER – Radiation Detection Equipment

Equipment: Radeye B20-ER (SN84634)</td><td>Trent Rainey</td></tr>
<tr><td colspan="2">Tank farm B

Unloading sump

Close vent system</td><td colspan="2">3-27-23</td><td>Method: EPA Method 21: Determination of Volatile Organic Compound Leaks
NEIC procedure: Toxic Vapor Analyzer (TVA), NEICPROC/00-016

Equipment: Thermo Scientific TVA 2020 (B24256)</td><td>Zach Schlachter</td></tr>
<tr><th colspan="6">SAMPLING</th></tr>
<tr><th>Station No. and Sample Tag No.</th><th>Appendix H Photo Nos.</th><th>Date and Time</th><th>Sampling Technique</th><th>Method, and/or Procedure, and Equipment</th><th>Sampler Name</th></tr>
<tr><td>S01 – Trip Blank
VP1508-01</td><td rowspan="6">N/A</td><td rowspan="6">Received on 3-15-2023 at 0931</td><td rowspan="6">Prepared by NEIC laboratory</td><td rowspan="6">Trip blanks and field spikes
Sample containers: 1-liter high density polyethylene (HDPE) plastic container (without Teflon™ -lined lids); nitric acid preservative</td><td rowspan="6">Prepared by Dan Hurlbut, NEIC Chemist in advance of the inspection and provided to Mike Solter, NEIC Project manager to bring to the field</td></tr>
<tr><td>S01 – Trip Blank
VP1508-02</td></tr>
<tr><td>S01 – Trip Blank
VP1508-03</td></tr>
<tr><td>S01 – Low Spike
VP1508-04</td></tr>
<tr><td>S01 – Mid Spike
VP1508-05</td></tr>
<tr><td>S01 – High Spike
VP1508-06</td></tr>
</table>

Table 4. FIELD MEASUREMENT AND FIELD SAMPLING ACTIVITIES

S02 – Outfall 003 VP1508-07	44 and 45	03-24-2023 at 0855	Grab sample using direct fill into sample container	**NEIC procedure**: *Water and Wastewater Sampling*, NEICPROC/00-051 **Equipment**: None **Sample container**: 1-liter HDPE plastic container (without Teflon™ -lined lids); nitric acid preservative	Mike Solter Trent Rainey
S03 – Field Blank VP1508-08	46	03-24-2023 at 0905	Grab sample using direct fill into sample container	**NEIC procedure**: *Water and Wastewater Sampling*, NEICPROC/00-051 **Equipment**: None **Sample container**: 1-liter HDPE plastic container (without Teflon™ -lined lids); nitric acid preservative	Mike Solter Trent Rainey
S04 – Staging area north of thaw building runoff VP1508-09	47 and 48	03-24-2023 at 0953	Grab sample using direct fill into sample container	**NEIC procedure**: *Water and Wastewater Sampling*, NEICPROC/00-051 **Equipment**: None **Sample container**: 1-liter HDPE plastic container (without Teflon™ -lined lids); nitric acid preservative	Mike Solter Trent Rainey
S05 – Retention basin north and east of REACT VP1508-10	49 and 50	03-24-2023 at 1004	Grab sample using direct fill into sample container	**NEIC procedure**: *Water and Wastewater Sampling*, NEICPROC/00-051 **Equipment**: None **Sample container**: 1-liter HDPE plastic container (without Teflon™ -lined lids); nitric acid preservative	Mike Solter Trent Rainey

Table 4. FIELD MEASUREMENT AND FIELD SAMPLING ACTIVITIES

S06 – Equipment rinse blank outside of 001 VP1508-11	51	03-24-2023 at 1039	Grab sample using direct fill into sample container	**NEIC procedure**: *Water and Wastewater Sampling*, NEICPROC/00-051 **Equipment**: Telescoping rod **Sample container**: 1-liter HDPE plastic container (without Teflon™ -lined lids); nitric acid preservative	Mike Solter Trent Rainey
S07 – Outfall 001 VP1508-12	52 and 53	03-24-2023 at 1044	Grab sample using direct fill into sample container	**NEIC procedure**: *Water and Wastewater Sampling*, NEICPROC/00-051 **Equipment**: Sample container affixed to telescoping rod **Sample container**: 1-liter HDPE plastic container (without Teflon™ -lined lids); nitric acid preservative	Mike Solter Trent Rainey
S08 – Outfall 002 VP1508-13	55 and 56	03-24-2023 at 1120	Grab sample using direct fill into sample container	**NEIC procedure**: *Water and Wastewater Sampling*, NEICPROC/00-051 **Equipment**: None **Sample container**: 1-liter HDPE plastic container (without Teflon™ -lined lids); nitric acid preservative	Mike Solter Trent Rainey
S09 – Raw water intake VP1508-14	57 and 58	03-24-2023 at 1402	Grab sample using direct fill into sample container	**NEIC procedure**: *Water and Wastewater Sampling*, NEICPROC/00-051 **Equipment**: None **Sample containers**: 1-liter HDPE plastic container (without Teflon™ -lined lids); nitric acid preservative	Mike Solter Trent Rainey
S09 – Raw water intake VP1508-15		03-24-2023 at 1403			
S09 – Raw water intake VP1508-16		03-24-2023 at 1404			

Table 4. FIELD MEASUREMENT AND FIELD SAMPLING ACTIVITIES

S10 – City water inlet VP1508-17	59 and 60	03-24-2023 at 14354	Grab sample using direct fill into sample container	**NEIC procedure**: *Water and Wastewater Sampling*, NEICPROC/00-051 **Equipment**: None **Sample container**: 1-liter HDPE plastic container (without Teflon™ -lined lids); nitric acid preservative	Mike Solter Trent Rainey
S11 – Tank 707 VP1508-18	61 and 62	3-27-2023 at 1108	Grab sample using direct fill into sample container	**NEIC procedure**: *Water and Wastewater Sampling*, NEICPROC/00-051 **Equipment**: None **Sample container**: 1-liter HDPE plastic container (without Teflon™ -lined lids); nitric acid preservative	Mike Solter Trent Rainey
S12 – Tank 361 VP1508-19	63 and 64	3-27-2023 at 1121	Grab sample using direct fill into sample container	**NEIC procedure**: *Water and Wastewater Sampling*, NEICPROC/00-051 **Equipment**: None **Sample container**: 1-liter HDPE plastic container (without Teflon™ -lined lids); nitric acid preservative	Mike Solter Trent Rainey
S13 – Frac box VP1508-20	65 and 66	3-27-2023 at 1127	Grab sample using direct fill into sample container	**NEIC procedure**: *Water and Wastewater Sampling*, NEICPROC/00-051 **Equipment**: None **Sample container**: 1-liter HDPE plastic container (without Teflon™ -lined lids); nitric acid preservative	Mike Solter Trent Rainey
S14 – Equivalent to 004 after the 7 ½ tanks VP1508-21	67 and 68	3-27-2023 at 1135	Grab sample using direct fill into sample container	**NEIC procedure**: *Water and Wastewater Sampling*, NEICPROC/00-051	Mike Solter Trent Rainey

Table 4. FIELD MEASUREMENT AND FIELD SAMPLING ACTIVITIES					
S14 – Equivalent to 004 after the 7 ½ tanks VP1508-22,		3-27-2023 at 1136		**Equipment**: None **Sample containers**: 1-liter HDPE plastic container (without Teflon™ -lined lids); nitric acid preservative	
S14 – Equivalent to 004 after the 7 ½ tanks VP1508-23		3-27-2023 at 1138			
S15 – Field blank VP1508-24	69	3-27-2023 at 1146	Grab sample using direct fill into sample container	**NEIC procedure**: *Water and Wastewater Sampling*, NEICPROC/00-051 **Equipment**: None **Sample container**: 1-liter HDPE plastic container (without Teflon™ -lined lids); nitric acid preservative	Mike Solter Trent Rainey
VP1508-01-Soils (Retention Basin near REACT area)	N/A	3-27-2023 at 1326	Grab sample using direct fill into sample container	**Method:** ASTM D5633-21: Standard Practice for Sampling with a Scoop **Equipment**: Grab samples using scoops/containers provided by the analyzing laboratory.	Mike Solter Trent Rainey
VP1508-02-Soils (Field Blank)	N/A	3-27-2023 at 1346	Grab sample using direct fill into sample container	**Method:** ASTM D5633-21: Standard Practice for Sampling with a Scoop **Equipment**: Grab samples using scoops/containers provided by the analyzing laboratory.	Mike Solter Trent Rainey

Table 4. FIELD MEASUREMENT AND FIELD SAMPLING ACTIVITIES					
VP1508-03-Soils (Spillway near lagoon 3)	N/A	3-27-2023 at 1347	Grab sample using direct fill into sample container	**Method:** ASTM D5633-21: Standard Practice for Sampling with a Scoop **Equipment**: Grab samples using scoops/containers provided by the analyzing laboratory.	Mike Solter Trent Rainey
VP1508-04-Soils (Mike Taylor Lute Memorial Park)	N/A	3-27-2023 at 1413	Off-site background soil Grab sample using direct fill into sample container	**Method:** ASTM D5633-21: Standard Practice for Sampling with a Scoop **Equipment**: Grab samples using scoops/containers provided by the analyzing laboratory.	Mike Solter Trent Rainey
[1] The current version of each procedure, at the time of the investigation, was followed.					

LDAR monitoring was performed on tank openings in Tank Farm B and the associated closed vent system along with the unloading sumps. No emissions above 500 ppm were detected. Valves were not monitored because the valves are in heavy liquid service and not subject to Method 21 monitoring.

Site conditions and activities were documented in field records, and field photographs are attached as **Appendix H**. Samples collected during the field activities for analysis by NEIC were driven by Trent Rainey to the NEIC laboratory in Denver, Colorado. Water and soil/sediment samples collected for analysis by ORD were shipped to the ORD laboratories via FedEx. The ORD analytical results are not included in this report.

LABORATORY ACTIVITIES SUMMARY

The field samples were delivered to the NEIC laboratory by Trent Rainey on March 31, 2023, and were placed in a secured walk-in cooler. Sample custody was relinquished by Trent Rainey to NEIC analyst Daniel Hurlbut on April 3, 2023. Tag information for each sample was compared to the accompanying chain of custody records in **Appendix J**. The NEIC laboratory team analyzed the samples for the presence of selected PFAS compounds.

Table 5 summarizes the analytical objective, technique, and methods followed, as well as the analysts and dates of the analyses. Data quality summaries, including uncertainty statements for all laboratory measurements, are maintained in the project file.

Table 5. ANALYTICAL OBJECTIVE, TECHNIQUE, AND METHOD; ANALYST; AND DATE PERFORMED

Analytical Objective, Technique, and Method	NEIC Analyst	Samples Analyzed by Method (Station Nos.)	Date(s) Performed
<u>Short Chain PFAS Analysis:</u> Restek application note, "A Novel Approach for Ultrashort-Chain PFAS Analysis in Water Samples," by Dr. Shun-Hsin Liang (<u>www.restek.com</u>). • Methanol diluted samples analyzed by liquid chromatography/mass spectrometry (LC/MS) using a triple-quadrupole (QQQ) mass spectrometer for identification of targeted, short chain PFAS.	Daya Mitchell	S01-S18. S20-S24	August 9 to September 18, 2023
<u>Long Chain PFAS Analysis:</u> • Improved Method for Extraction and Analysis of Perfluorinated Compounds (PFCs) from Surface Waters and Well Water by Ultra-High Performance Liquid Chromatography (UPLC)-Tandem Mass Spectrometry (MS/MS). SOP ID: D-EMMDPHCB043-SOP-03, August 2016. • Method for Extraction and Analysis of Perfluoroethercarboxylic acids (PFECAs) from Surface Water, Well Water and Waste Water by Ultra-High Performance Liquid Chromatography (UPLC)-Tandem Mass Spectrometry (MS/MS). SOP ID:D-EMMD-PHCB-062-SOP-01, July 2017. • Both of these methodologies require filtration followed by loading the sample on a cartridge. At a later date, the samples were eluted off the cartridge and concentrated by evaporation for sample extracts.	Filtration and sample loading on cartridge: Daya Mitchell Jessica Krank Sample elution and concentration: Daya Mitchell Jessica Krank	S01-S18, S20-S24	May 3 to 9, 2023 July 19, 2023
• Long chain PFAS sample extracts analyzed by liquid chromatography/mass spectrometry (LC/MS) using a triple-quadrupole (QQQ) mass spectrometer for identification and quantitation of targeted, long chain PFAS.	Daya Mitchell	S01-S18, S20-S24	August 9 to September 18, 2023
• Long chain PFAS sample extracts analyzed by LC/MS using a quadrupole time of flight (QTOF) high resolution mass spectrometer for identification of semi-target PFAS.	Jessica Krank	S01-S18, S20-S24	July 26 to August 21, 2023

Table 5. ANALYTICAL OBJECTIVE, TECHNIQUE, AND METHOD; ANALYST; AND DATE PERFORMED			
Analytical Objective, Technique, and Method	**NEIC Analyst**	**Samples Analyzed by Method (Station Nos.)**	**Date(s) Performed**
[1] Data quality summaries, including uncertainty measurements, for all laboratory measurements are maintained in the project file.			

The mass spectrometers listed in Table 6 use electrospray ionization to detect negatively charged mass ions. The triple quadrupole (QQQ) mass spectrometer also incorporates analyte specific parameters (retention time, precursor and fragment mass ions, etc.), along with certified reference materials in order to identify and quantify targeted PFAS. The quadrupole time of flight (QTOF) high resolution mass spectrometer collects accurate mass data for all negatively charged mass ions detected (PFAS and non-PFAS) for a specified mass range during the analysis run, and analyte identifications were based on comparing observed accurate mass values of precursor mass ions to the exact mass values of the semi-targeted PFAS, along with other mass ions (fragments, adducts, etc.), and if available, retention time and accurate mass value comparisons to reference materials. Therefore, reported results will differ based on the functionality of the mass spectrometer being used.

In addition, some PFAS are available in various chemical forms, and each form has its own associated Chemical Abstract Services (CAS) number. For example, perfluorobutanesulfonic acid (CAS No. 375-73-5) and perfluorobutanesulfonate potassium salt (CAS No. 29420-49-3) have unique CAS numbers, and both chemicals produce the same negative mass ion. The mass spectrometer cannot determine which chemical form (acid, potassium salt, etc.) produced the observed PFBS response. Therefore, if applicable, results from the short chain and long chain analyses were based on the acid form of the reported PFAS.

ANALYTICAL RESULTS

Table 6 contains the station number, NEIC sample tag, station location, photograph file name and physical description of the samples collected by the NEIC field team. Photographs of the samples as observed in the NEIC laboratory are in **Appendix K**. Three trip blanks (NEIC sample tag Nos. VP1508-01 to VP1508-03) and three trip spike samples (NEIC sample tag Nos. VP1508-04 to VP1508-06) were prepared by the NEIC laboratory and given to the NEIC field team who transported them to the facility and back to NEIC. The extraction and analysis methods described in Table 5 were developed and validated by EPA analysts at the ORD National Exposure Research Laboratory for PFAS determinations in water matrices similar to the samples described in Table 7. The project file contains additional laboratory activities information, including sample description, preparation, filtration, extraction spike recoveries, and reporting limits, along with notable observations, method modifications, and comments. The analytical results presented apply to the samples as received.

Table 6. SUMMARY OF SAMPLE IDENTIFICATION AND DESCRIPTION				
Station No.	**NEIC Tag No.**	**Station Location/ Description of Sample Location**	**Appendix K File Name**	**Physical Description**
S01	VP1508-01	NEIC laboratory/Trip Blanks	DSC_0247.JPG	Colorless, non-viscous, liquid
	VP1508-02			
	VP1508-03			
	VP1508-04	NEIC laboratory/ Low Trip Spike	DSC_0248.JPG	Colorless, non-viscous, liquid
	VP1508-05	NEIC laboratory/ Mid Trip Spike		
	VP1508-06	NEIC laboratory/ High Trip Spike		
S02	VP1508-07	Outfall 003	DSC_0249.JPG	Non-viscous liquid, light brown in color
S03	VP1508-08	Field blank	DSC_0250.JPG	Colorless, non-viscous, liquid
S04	VP1508-09	Staging area north of thaw building-lot runoff	DSC_0251.JPG	Colorless, non-viscous, liquid with black sediment present
S05	VP1508-10	Retention basin north and east of REACT.	DSC_0252.JPG	Non-viscous liquid, light brown in color, with black sediment present
S06	VP1508-11	Equipment rinse blank	DSC_0253.JPG	Colorless, non-viscous, liquid
S07	VP1508-12	Outfall 001	DSC_0254.JPG	Colorless, non-viscous, liquid with black sediment present
S08	VP1508-13	Outfall 002	DSC_0255.JPG	Non-viscous liquid, light brown color with sediment present
S09	VP1508-14	Raw water intake	DSC_0256.JPG	Colorless, non-viscous, liquid
	VP1508-15			
	VP1508-16			
S10	VP1508-17	City water inlet	DSC_0257.JPG	Colorless, non-viscous, liquid
S11	VP1508-18	Tank 707	DSC_0258.JPG	Colorless, non-viscous liquid with a layer of black sediment on container bottom and adhering to inner container walls and cap.
S12	VP1508-19	Tank 361	DSC_0259.JPG	Black, semi-viscous liquid containing a substantial amount of black material , opaque liquid and solid layers.
S13	VP1508-20	Frac box	DSC_0260.JPG	Colorless, non-viscous, liquid containing a substantial amount of black sediment.
S14	VP1508-21	Equivalent to 004 after 7 ½ tanks	DSC_0261.JPG	Colorless, non-viscous, liquid
	VP1508-22			
	VP1508-23			
S15	VP1508-24	Field blank	DSC_0262.JPG	Colorless, non-viscous, liquid

Prior to analysis, a 750 milliliter (mL) volume from each sample was passed through a glass fiber filter to remove any particulates present, except for sample VP1508-19 which was mainly comprised of a black, opaque solid material, and could not be analyzed using the methods shown in Table 6. Samples VP1508-09, VP1508-18 and VP1508-20 were passed through multiple filters because of the particulate amount present in these samples. A diluted aliquot from each filtrate was analyzed for the targeted short chain PFAS using the LC/MS QQQ methodology shown in Table 6. A 500-mL volume from each filtrate was passed through a solid phase extraction (SPE) cartridge, and the resulting extract was analyzed for the long chain PFAS using the LC/MS QQQ and LC/MS QTOF methods shown in Table 6.

Targeted PFAS Analysis

Table 7 contains the targeted PFAS analytes along with their NEIC acronym, CAS number, and molecular formula. The SPE extracts were analyzed for the long chain targeted PFAS while the diluted aliquots were analyzed for the short chain target PFAS, using the LC/MS-QQQ methods shown in Table 5.

Table 7. TARGETED PFAS

Analyte	NEIC Acronym	CAS No.	Formula
Long Chain			
Perfluorobutyric acid	PFBA	375-22-4	$C_4HF_7O_2$
Perfluorodecanoic acid	PFDA	335-76-2	$C_{10}HF_{19}O_2$
Perfluorododecanoic acid	PFDoA	307-55-1	$C_{12}HF_{23}O_2$
Perfluoroheptanoic acid	PFHpA	375-85-9	$C_7HF_{13}O_2$
Perfluorohexanoic acid	PFHxA	307-24-4	$C_6HF_{11}O_2$
Perfluorononanoic acid	PFNA	375-95-1	$C_9HF_{17}O_2$
Perfluorooctanoic acid	PFOA	335-67-1	$C_8HF_{15}O_2$
Perfluoropentanoic acid	PFPeA	2706-90-3	$C_5HF_9O_2$
Perfluorotridecanoic acid	PFTrA	72629-94-8	$C_{13}HF_{25}O_2$
Perfluoroundecanoic acid	PFUnA	2058-94-8	$C_{11}HF_{21}O_2$
Hexafluoropropylene oxide dimer acid (a.k.a. *Undecafluoro-2-methyl-3-oxahexanoic acid*)	HFPO-DA (a.k.a. GenX)	13252-13-6	$C_6HF_{11}O_3$
Butanoic acid, 2,2,3,3,4,4-hexafluoro-4-(trifluoromethoxy)-	PFECA A (a.k.a. PFMBA)	863090-89-5	$C_5HF_9O_3$
Acetic acid, 2,2-difluoro-2-[1,1,2,2-tetrafluoro-2-(trifluoromethoxy)ethoxy]-	PFECA B (a.k.a. NFDHA)	151772-58-6	$C_5HF_9O_4$
Butanoic acid, 2,2,3,3,4,4-hexafluoro-4-[1,2,2,2-tetrafluoro-1-(trifluoromethyl)ethoxy]-	PFECA G	801212-59-9	$C_7HF_{13}O_3$
N-ethylperfluoro-1-octanesulfonamide	EtFOSA	4151-50-2	$C_{10}H_6F_{17}NO_2S$
N-methylperfluoro-1-octanesulfonamide	MeFOSA	31506-32-8	$C_9H_4F_{17}NO_2S$
Perfluorooctanesulfonamide	PFOSA	754-91-6	$C_8H_2F_{17}NO_2S$
N-ethyl perfluorooctanesulfonamidoacetic acid (*a.k.a. N-ethyl-N-((heptadecafluorooctyl)sulfonyl)glycine*)	NEtFOSAA	2991-50-6	$C_{12}H_8F_{17}NO_4S$
N-methyl perfluorooctanesulfonamidoacetic acid (*a.k.a. N-(Heptadecafluorooctylsulfonyl)-N-methylglycine*)	NMeFOSAA	2355-31-9	$C_{11}H_6F_{17}NO_4S$
Perfluorobutanesulfonic acid	PFBS	375-73-5	$C_4F_9O_3SH$
Perfluorodecanesulfonic acid	PFDS	335-77-3	$C_{10}F_{21}O_3SH$
Perfluorododecanesulfonic acid	PFDoS	79780-39-5	$C_{12}F_{25}O_3SH$
Perfluoroheptanesulfonic acid	PFHpS	375-92-8	$C_7F_{15}O_3SH$
Perfluorohexanesulfonate	$PFHxS_{Total}$[3]	355-46-4	$C_6F_{13}O_3SH$
Perfluorononanesulfonic acid	PFNS	68259-12-1	$C_9F_{19}O_3SH$

Table 7. TARGETED PFAS			
Analyte	**NEIC Acronym**	**CAS No.**	**Formula**
Perfluorooctanesulfonic acid	$PFOS_{Total}$[3]	1763-23-1	$C_8F_{17}O_3SH$
Perfluoropentanesulfonic acid	PFPeS	2706-91-4	$C_5F_{11}O_3SH$
4:2 Fluorotelomer sulfonic acid	4:2 FTS	757124-72-4	$C_6H_5F_9O_3S$
6:2 Fluorotelomer sulfonic acid	6:2 FTS	27619-97-2	$C_8H_5F_{13}O_3S$
8:2 Fluorotelomer sulfonic acid	8:2 FTS	39108-34-4	$C_{10}H_5F_{17}O_3S$
10:2 Fluorotelomer sulfonic acid	10:2 FTS	120226-60-0	$C_{12}H_5F_{21}O_3S$
Short Chain			
2,3,3,3-Tetrafluoropropionic acid	2,3,3,3-TPrA	359-49-9	$C_3H_2O_2F4$
3H-Tetrafluoropropionic acid	2,2,3,3-TPrA	756-09-2	$C_3H_2O_2F_4$
Lithium bis(trifluoromethanesulfonyl)amide[1]	HQ-115	90076-65-6	$C_2F_6S_2O_4NLi$
Pentafluoropropionic acid	PFPA	422-64-0	$C_3HO_2F_5$
Perfluoro-4-ethylcyclohexanesulfonic acid	PFECHS	646-83-3	$C_8F_{15}SO_3H$
Perfluoroethanesulfonic Acid	PFES	354-88-1	$C_2F_5SO_3H$
Potassium Bis(perfluorobutanesulfonyl)imide[2]	DBI	129135-87-1	$C_8F_{18}S_2O_4NK$
Trifluoroacetic acid	TFAA (or TFA)	76-05-1	$C_2HO_2F_3$
Trifluoromethanesulfonic Acid	TFMS	1493-13-6	CF_3SO_3H

1- Reported results are based on *bis(trifluoromethanesulfonyl)amide* form.
2- Reported results are based on *bis(perflurobutanesulfonyl)imide* form.
3- Analyte contains linear (L) and branched (B) isomers. Therefore, reported "Total" results = (L+B) isomer results.

As shown in Table 8, perfluoroethanesulfonic acid (PFES) was included in the short chain targeted PFAS list, however, reference materials for this analyte were not readily available, therefore, the diluted filtrates were not analyzed for PFES. The SPE extracts were analyzed for perfluorododecanesulfonic acid (PFDoS) but these results were not reported since PFDoS was not observed in the quality control samples that were prepared and analyzed.

Tables 8 through **11** contain the observed targeted PFAS concentrations in nanograms per liter (ng/L), and the corresponding reporting limits (RL), for samples VP1508-07 through VP1508-24, excluding VP1508-19. Four additional targeted PFAS (PFDS, PFECA-B, PFECA-G and PFNS) are not reported in Tables 9 through 12, as these analytes were not observed in any site samples analyzed. . The RL value for each analyte was based on the lowest standard solution in its calibration curve, along with the sample preparation technique used (i.e., methanol diluted aliquot or SPE). If the analyte was identified in any of the blank samples, the analyte response of the lowest standard solution was greater than the observed responses from the blank samples. These RL criteria were applied to all but seven of the reported PFAS. Low analyte recoveries from spike samples resulted in the significantly higher RL values for 10:2 FTS, PFDoA, TFAA, PFPA, 2,2,3,3-TFPA and 2,3,3,3-TPrA. An RL value could not be determined for PFTrA since this analyte was not recovered from any of the spike samples, although it was identified in sample VP1508-18. As mentioned previously, samples VP1508-01, VP1508-02 and VP1508-03 (trip blanks) and samples VP1508-04, VP1508-05, VP1508-06 (trip spikes) were prepared at the NEIC laboratory. The PFAS results for the trip blanks were less than the RL value for each target PFAS. The trip spike results were included in determining the expanded measurement uncertainty (EMU) ranges that are shown as a footnote in Tables 9 through12.

Table 8. RESULTS FOR VP1508-07 THROUGH VP1508-11, AND REPORTING LIMITS

Analyte	Analyte Concentration (ng/L)					
	Outfall 003 (VP1508-07)	Field blank (VP1508-08)	Staging area (VP1508-09)	Ret. basin (VP1508-10)	Equip. rinse (VP1508-11)	Reporting Limit (RL)
Long Chain						
10:2 FTS[1]	< RL	< RL	< RL	< RL	< RL	50.0
4:2 FTS	< RL	< RL	< RL	< RL	< RL	2.07
6:2 FTS	< RL	< RL	34.0	< RL	< RL	2.10
8:2 FTS	< RL	< RL	< RL	< RL	< RL	2.12
HFPO-DA	< RL	< RL	4750	2990	< RL	5.53
N-EtFOSA	< RL	< RL	< RL	< RL	< RL	2.21
N-EtFOSAA	< RL	< RL	< RL	< RL	< RL	2.21
N-MeFOSA	< RL	< RL	< RL	< RL	< RL	2.21
N-MeFOSAA	< RL	< RL	< RL	< RL	< RL	2.21
PFBA	6.22	< RL	110	61.3	< RL	2.21
PFBS	< RL	< RL	86.9	79.7	< RL	1.96
PFDA	< RL	< RL	< RL	< RL	< RL	2.21
PFDoA	< RL	< RL	< RL	< RL	< RL	50.0
PFECA-A	< RL	< RL	< RL	< RL	< RL	2.21
PFHpA	< RL	< RL	9.74	24.7	< RL	2.21
PFHpS	< RL	< RL	< RL	< RL	< RL	2.10
PFHxA	< RL	< RL	36.1	57.4	< RL	2.21
$PFHxS_{Total}$	< RL	< RL	< RL	6.61	< RL	2.02
PFNA	< RL	< RL	< RL	6.24	< RL	2.21
PFOA	< RL	< RL	74.4	871	< RL	2.21
$PFOS_{Total}$	< RL	< RL	14.3	19.2	< RL	2.05
PFOSA	< RL	< RL	< RL	< RL	< RL	2.21
PFPeA	< RL	< RL	78.0	71.0	< RL	22.1
PFPeS	< RL	< RL	< RL	< RL	< RL	2.08
PFTrA[2]	NA	NA	NA	NA	NA	NA
PFUnA	< RL	< RL	< RL	< RL	< RL	2.21
Short Chain						
2,2,3,3-TPrA	< RL	< RL	< RL	< RL	< RL	1000
2,3,3,3-TPrA	< RL	< RL	< RL	DET	< RL	200
DBI	< RL	< RL	< RL	< RL	< RL	10.0
HQ-115	< RL	< RL	11.8	< RL	< RL	10.0
PFECHS	< RL	< RL	< RL	< RL	< RL	10.0
PFPA	< RL	< RL	DET	< RL	< RL	800
TFAA	< RL	< RL	DET	DET	< RL	600
TFMS	< RL	< RL	12.0	9.94[3]	< RL	10.0

Table 8. RESULTS FOR VP1508-07 THROUGH VP1508-11, AND REPORTING LIMITS						
Analyte	Analyte Concentration (ng/L)					Reporting Limit (RL)
	Outfall 003 (VP1508-07)	Field blank (VP1508-08)	Staging area (VP1508-09)	Ret. basin (VP1508-10)	Equip. rinse (VP1508-11)	

[1] Results are estimated due to low spike recovery.
[2] PFTrA was not detected in the spike samples, thus is reported as NA (Not Applicable).
[3] Response within calibration curve response range, but concentration calculates as less than lowest calibrator.
DET – Analytes are detected above the stated RL but could not be quantified due to poor calibration and/or QC results or significant ion suppression. interfering with the MassHunter software's ability to quantify the result.
EMU- Expanded measurement uncertainty range for reported PFAS concentration values: 64% to 170%, except for:

- N-EtFOSAA, N-MeFOSAA, EtFOSA, PFHxA: EMU range: 36% to 230%
- 10:2 FTS, DBI: EMU range: 51% to 1100%
- 2,2,3,3-TPrA, 2,3,3,3-TPrA, PFPA, TFAA, PFTrA: No EMU range calculated due to insufficient quality control data.

(Using a coverage probability of 95% and coverage factor of 2.09 for short chain PFAS, or 2.57 for long chain PFAS.)

Table 9. RESULTS FOR VP1508-12 THROUGH VP1508-16, AND REPORTING LIMITS						
Analyte	Analyte Concentration (ng/L)					Reporting Limit (RL)
	Outfall 001	Outfall 002	Raw water intake			
	(VP1508-12)	(VP1508-13)	(VP1508-14)	(VP1508-15)	(VP1508-16)	
Long Chain						
10:2 FTS[1]	< RL	< RL	< RL	< RL	< RL	50.0
4:2 FTS	< RL	< RL	< RL	< RL	< RL	2.07
6:2 FTS	< RL	< RL	< RL	< RL	< RL	2.10
8:2 FTS	< RL	< RL	< RL	< RL	< RL	2.12
HFPO-DA	< RL	< RL	< RL	< RL	< RL	5.53
N-EtFOSA	< RL	< RL	< RL	< RL	< RL	2.21
N-EtFOSAA	< RL	< RL	< RL	< RL	< RL	2.21
N-MeFOSA	< RL	< RL	< RL	< RL	< RL	2.21
N-MeFOSAA	< RL	< RL	< RL	< RL	< RL	2.21
PFBA	169	< RL	< RL	< RL	< RL	2.21
PFBS	< RL	< RL	< RL	< RL	< RL	1.96
PFDA	< RL	< RL	< RL	< RL	< RL	2.21
PFDoA	< RL	< RL	< RL	< RL	< RL	50.0
PFECA-A	< RL	< RL	< RL	< RL	< RL	2.21
PFHpA	< RL	< RL	< RL	< RL	< RL	2.21
PFHpS	< RL	< RL	< RL	< RL	< RL	2.10
PFHxA	< RL	6.89	< RL	< RL	< RL	2.21
PFHxS$_{Total}$	< RL	< RL	< RL	< RL	< RL	2.02
PFNA	< RL	< RL	< RL	< RL	< RL	2.21
PFOA	< RL	< RL	2.99	< RL	< RL	2.21
PFOS$_{Total}$	< RL	< RL	< RL	< RL	< RL	2.05
PFOSA	< RL	< RL	< RL	< RL	< RL	2.21
PFPeA	< RL	< RL	< RL	< RL	< RL	22.1
PFPeS	< RL	< RL	< RL	< RL	< RL	2.08
PFTrA[2]	NA	NA	NA	NA	NA	NA
PFUnA	< RL	< RL	< RL	< RL	< RL	2.21
Short Chain						
2,2,3,3-TPrA	< RL	< RL	< RL	DET	< RL	1000
2,3,3,3-TPrA	DET	< RL	< RL	< RL	< RL	200
DBI	< RL	< RL	< RL	< RL	< RL	10.0

Table 9. RESULTS FOR VP1508-12 THROUGH VP1508-16, AND REPORTING LIMITS						
Analyte	Analyte Concentration (ng/L)					
	Outfall 001	Outfall 002	Raw water intake			Reporting Limit (RL)
	(VP1508-12)	(VP1508-13)	(VP1508-14)	(VP1508-15)	(VP1508-16)	
HQ-115	< RL	< RL	< RL	< RL	< RL	10.0
PFECHS	< RL	< RL	< RL	< RL	< RL	10.0
PFPA	DET	< RL	< RL	DET	< RL	800
TFAA	DET	< RL	< RL	DET	< RL	600
TFMS	21.0	< RL	< RL	< RL	< RL	10.0

[1] Results are estimated due to low spike recovery.
[2] PFTrA was not detected in the spike samples, thus is reported as NA (Not Applicable).
DET – Analytes are detected above the stated RL but could not be quantified due to poor calibration and/or QC results or significant ion suppression interfering with the MassHunter software's ability to quantify the result.
EMU- Expanded measurement uncertainty range for reported PFAS concentration values: 64% to 170%, except for:

- N-EtFOSAA, N-MeFOSAA, EtFOSA, PFHxA: EMU range: 36% to 230%
- 10:2 FTS, DBI: EMU range: 51% to 1100%
- 2,2,3,3-TPrA, 2,3,3,3-TPrA, PFPA, TFAA, PFTrA: No EMU range calculated due to insufficient quality control data.

(Using a coverage probability of 95% and coverage factor of 2.09 for short chain PFAS, or 2.57 for long chain PFAS.)

Table 10. RESULTS FOR VP1508-17, VP1508-18, VP1508-20, AND REPORTING LIMITS				
Analyte	Analyte Concentration (ng/L)			
	City water inlet	Tank 707	Frac Box	Reporting Limit (RL)
	(VP1508-17)	(VP1508-18)[2]	(VP1508-20)	
Long Chain				
10:2 FTS[1]	< RL	62.8	< RL	50.0
4:2 FTS	< RL	19.3	< RL	2.07
6:2 FTS	< RL	DET	275	2.10
8:2 FTS	< RL	810	< RL	2.12
HFPO-DA	< RL	> 5500[3]	> 5500[3]	5.53
N-EtFOSA	< RL	647	< RL	2.21
N-EtFOSAA	< RL	2780	< RL	2.21
N-MeFOSA	< RL	840	< RL	2.21
N-MeFOSAA	< RL	2130	< RL	2.21
PFBA	< RL	30,400	1220	2.21
PFBS	< RL	7020	597	1.96
PFDA	< RL	2110	15.4	2.21
PFDoA	< RL	745	< RL	50.0
PFECA-A	< RL	313	< RL	2.21
PFHpA	< RL	3590	37.2	2.21
PFHpS	< RL	8290	< RL	2.10
PFHxA	< RL	4720	59.5	2.21
$PFHxS_{Total}$	< RL	29,000	40.7	2.02
PFNA	< RL	10,400	62.9	2.21
PFOA	< RL	DET	1350	2.21
$PFOS_{Total}$	< RL	> 5500[3]	157	2.05
PFOSA	< RL	6950	< RL	2.21
PFPeA	< RL	3270	186	22.1
PFPeS	< RL	189	3.85	2.08
PFTrA[4]	NA	DET	NA	NA

Table 10. RESULTS FOR VP1508-17, VP1508-18, VP1508-20, AND REPORTING LIMITS

Analyte	Analyte Concentration (ng/L)			Reporting Limit (RL)
	City water inlet (VP1508-17)	Tank 707 (VP1508-18)[2]	Frac Box (VP1508-20)	
PFUnA	< RL	24,500	48.5	2.21
Short Chain				
2,2,3,3-TPrA	< RL	< RL	< RL	1000
2,3,3,3-TPrA	< RL	DET	DET	200
DBI	< RL	50.3	< RL	10.0
HQ-115	< RL	5280	27.8	10.0
PFECHS	< RL	288	< RL	10.0
PFPA	< RL	DET	DET	800
TFAA	< RL	DET	DET	600
TFMS	< RL	534	3480	10.0

[1] Results are estimated due to low spike recovery.
[2] Significant ion suppression of internal standards was seen in multiple compounds in VP1508-18. Due to low internal standard recovery of their corresponding internal standards, some PFAS compounds did not quantify via the Masshunter software and are thus reported as detected (DET).
[3] Reported as a "greater than" value because analyte response was above the response of the highest calibrator.
[4] PFTrA was not detected in the spike samples but was detected in VP1508-18 at above the 250 ng/L equivalent calibrator, thus is reported as DET in VP1508-18 and NA (Not Applicable) in all other samples.
DET – Analytes are detected above the stated RL but could not be quantified due to poor calibration and/or QC results. or significant ion suppression interfering with the MassHunter software's ability to quantify the result.
EMU- Expanded measurement uncertainty range for reported PFAS concentration values: 64% to 170%, except for:

- N-EtFOSAA, N-MeFOSAA, EtFOSA, PFHxA: EMU range: 36% to 230%
- 10:2 FTS, DBI: EMU range: 51% to 1100%
- 2,2,3,3-TPrA, 2,3,3,3-TPrA, PFPA, TFAA, PFTrA: No EMU range calculated due to insufficient quality control data.

(Using a coverage probability of 95% and coverage factor of 2.09 for short chain PFAS, or 2.57 for long chain

Table 11. RESULTS FOR VP1508-21 THROUGH VP1508-24, AND REPORTING LIMITS

Analyte	Analyte Concentration (ng/L)				Reporting Limit (RL)
	Equivalent to 004 after 7 ½ tanks			Field blank	
	(VP1508-21)	(VP1508-22)	(VP1508-23)	(VP1508-24)	
Long Chain					
10:2 FTS[1]	< RL	< RL	< RL	< RL	50.0
4:2 FTS	< RL	< RL	< RL	< RL	2.07
6:2 FTS	< RL	< RL	< RL	< RL	2.10
8:2 FTS	< RL	< RL	< RL	< RL	2.12
HFPO-DA	< RL	< RL	41.9	< RL	5.53
N-EtFOSA	< RL	< RL	< RL	< RL	2.21
N-EtFOSAA	< RL	< RL	< RL	< RL	2.21
N-MeFOSA	< RL	< RL	< RL	< RL	2.21
N-MeFOSAA	< RL	< RL	< RL	< RL	2.21
PFBA	4450	4410	4390	< RL	2.21
PFBS	< RL	< RL	< RL	< RL	1.96
PFDA	< RL	< RL	< RL	< RL	2.21
PFDoA	< RL	< RL	< RL	< RL	50.0
PFECA-A	< RL	< RL	< RL	< RL	2.21
PFHpA	< RL	2.66	< RL	< RL	2.21
PFHpS	< RL	< RL	< RL	< RL	2.10

Table 11. RESULTS FOR VP1508-21 THROUGH VP1508-24, AND REPORTING LIMITS

Analyte	Analyte Concentration (ng/L)				
	Equivalent to 004 after 7 ½ tanks			Field blank	Reporting Limit (RL)
	(VP1508-21)	(VP1508-22)	(VP1508-23)	(VP1508-24)	
PFHxA	< RL	3.77	< RL	< RL	2.21
$PFHxS_{Total}$	< RL	< RL	< RL	< RL	2.02
PFNA	< RL	< RL	< RL	< RL	2.21
PFOA	< RL	12.6	14.6	< RL	2.21
$PFOS_{Total}$	< RL	< RL	< RL	< RL	2.05
PFOSA	< RL	< RL	< RL	< RL	2.21
PFPeA	< RL	< RL	< RL	< RL	22.1
PFPeS	< RL	< RL	< RL	< RL	2.08
PFTrA[2]	NA	NA	NA	NA	NA
PFUnA	< RL	< RL	< RL	< RL	2.21
Short Chain					
2,2,3,3-TPrA	< RL	< RL	< RL	DET	1000
2,3,3,3-TPrA	DET	DET	DET	< RL	200
DBI	< RL	< RL	< RL	< RL	10.0
HQ-115	< RL	< RL	< RL	< RL	10.0
PFECHS	< RL	< RL	< RL	< RL	10.0
PFPA	DET	DET	DET	< RL	800
TFAA	DET	DET	DET	< RL	600
TFMS	3140	2990	3000	< RL	10.0

[1] Results are estimated due to low spike recovery.
[2] PFTrA was not detected in the spike samples, thus is reported as NA (Not Applicable).
DET – Analytes are detected above the stated RL but could not be quantified due to poor calibration and/or QC results or significant ion suppression interfering with the MassHunter software's ability to quantify the result.
EMU- Expanded measurement uncertainty range for reported PFAS concentration values was 64% to 170%, except for:

- N-EtFOSAA, N-MeFOSAA, EtFOSA, PFHxA: EMU range: 36% to 230%
- 10:2 FTS, DBI: EMU range: 51% to 1100%
- 2,2,3,3-TPrA, 2,3,3,3-TPrA, PFPA, TFAA, PFTrA: No EMU range calculated due to insufficient quality control data.

(Using a coverage probability of 95% and coverage factor of 2.09 for short chain PFAS, or 2.57 for long chain PFAS.)

EMU ranges were calculated for each PFAS shown in Tables 9 through 12 and were based on their quality control (QC) data, including analyte recoveries from the trip and laboratory spike samples. For 2,2,3,3-TPrA, 2,3,3,3-TPrA, PFPA, TFAA, and PFTrA, EMU ranges could not be determined due to insufficient QC data. EMU ranges were calculated for the remaining PFAS and each were placed into one of the range groupings shown in Tables 9 through 12, depending on the accuracy and precision of their QC data. For example, the accuracy of the QC data for 10:2 FTS, and DBI were either much lower than expected, or the precision was highly variable which placed them in the 51% to 1100% EMU range grouping.

Semi-Targeted PFAS Analysis

The SPE extracts were also analyzed using the LC/MS QTOF method from Table 6, for the semi-targeted PFAS shown in **Table 12**, which includes the NEIC acronyms, molecular formula, and exact mass values.

Table 12. SEMI-TARGETED PFAS, NEIC ACRONYM, FORMULAS and EXACT MASS VALUES

Analyte	NEIC Acronym	Formula	Exact Mass Value (Dalton)[1]
4,8-Dioxa-3H-perfluorononanoic acid	ADONA	$C_7H_2F_{12}O_4$	377.9762
4,8-Dioxa-3H-perfluorononanoate sodium salt	NaDONA	$C_7HF_{12}O_4Na$	399.9581
Perfluorobutane sulfonamide (C4 amide) 1,1,2,2,3,3,4,4,4-Nonafluorobutane-1-sulfonamide	FBSA	$C_4H_2F_9NO_2S$	298.9663
Perfluorobutyl sulfonamido acetic acid	FBSAA (1)	$C_6H_4F_9NO_4S$	356.9717
Nonafluoro-N-(2-hydroxyethyl)butane-1-sulfonamide	FBSE = HFBSE Alcohol	$C_6H_6F_9NO_3S$	342.9925
[(Nonafluorobutane-1-sulfonyl)-carboxymethylamino] acetic acid	FBSEE diacid	$C_8H_6F_9NO_6S$	414.9772
Nonafluoro-N,N-bis(2-hydroxyethyl)butane-1- sulfonamide	FBSEE diol	$C_8H_{10}F_9NO_4S$	387.0187
N-methyl-perfluorobutane sulfonamide or 1,1,2,2,3,3,4,4,4-nonafluoro-N-methyl-1-Butanesulfonamide	MeFBSA	$C_5H_4F_9NO_2S$	312.9819
Perfluorobutyl-methyl sulfonamido acetic acid	MeFBSAA	$C_7H_6F_9NO_4S$	370.9874
Nonafluoro-N-(2-Hydroxyethyl)-N-methyl-1-butanesulfonamide	MeFBSE	$C_7H_8F_9NO_3S$	357.0081
2-(N-ethylperfluoro-1-octanesulfonamido)-ethanol	N-EtFOSE	$C_{12}H_{10}F_{17}NO_3S$	571.0110
2-(N-methylperfluoro-1-octanesulfonamido)-ethanol	N-MeFOSE	$C_{11}H_8F_{17}NO_3S$	556.9953
Perfluorobutanesulfinic Acid	PFBSi	$C_4HF_9O_2S$	283.9554
2,2,3,3-tetrafluoro-3-(trifluoromethoxy)-propanoic acid	PFECA F or PMPA	$C_4HF_7O_3$	229.9814
Perfluoro(2-ethoxyethane) sulfonic acid	PFEESA	$C_4HF_9O_4S$	315.9452
2-[1-[difluoro(1,2,2,2-tetrafluoroethoxy)methyl]-1,2,2,2-tetrafluoroexthoxy]-1,1,2,2-tetrafluoro-ethanesulfonic acid	PFESA BP2 (Nafion)	$C_7H_2F_{14}O_5S$	463.9399
Perfluoro-3,6-dioxa-4-methyl-7-octene-1-sulfonic acid	PFESA BP1	$C_7HF_{13}O_5S$	443.9337
2,2-difluoro-2-(trifluoromethoxy) acetic acid	PFMOAA	$C_3HF_5O_3$	179.9846
Perfluoro-3,5-dioxahexanoic acid	PFO2HxA	$C_4HF_7O_4$	245.9763
Perfluoro-3,5,7-trioxaoctanoic acid	PFO3OA	$C_5HF_9O_5$	311.9680
Perfluoro-3,5,7,9-tetraoxadecanoic acid	PFO4DA	$C_6HF_{11}O_6$	377.9597
Perfluoro-3,5,7,9,11-pentaoxadodecanoic acid	PFO5OA or TAFN4	$C_7HF_{13}O_7$	443.9515
Overlap targeted analytes for confirmation of spikes and samples.			

Table 12. SEMI-TARGETED PFAS, NEIC ACRONYM, FORMULAS and EXACT MASS VALUES			
Analyte	**NEIC Acronym**	**Formula**	**Exact Mass Value (Dalton)[1]**
Perfluorooctanesulfonic Acid[2]	PFOS	$C_8HF_{17}O_3S$	499.9375
Perfluorooctanoic Acid[2]	PFOA	$C_8HF_{15}O_2$	413.9737
[1] – Exact mass value is the summation of the principal isotope for each element that make up the compound. [2] – PFOA and PFOS were also analyzed and quantified using the LC/MS QQQ analysis.			

Reference materials for 13 of the semi-targeted PFAS were used to prepare calibration standards that were analyzed with the SPE extracts. The identifications of these analytes were based on comparing responses (retention time, accurate mass value, etc.) from the extracts and calibration standards. Once identified, calibration data was used to approximate these analyte concentrations in the samples. Tentative identifications of the remaining semi-targeted PFAS in the extracts were based solely on observed accurate mass value from precursor, fragment and adducts ions, and comparing these to their exact mass values, as no reference material was readily available for these analytes.

Table 13 contains the semi-targeted PFAS results. Samples not shown in this table indicate that none of the semi-targeted PFAS were identified.

Table 13. SEMI-TARGETED PFAS RESULTS																	
Compound Acronym	NEIC Tag Number																
	VP1508-04	VP1508-05	VP1508-06	VP1508-07	VP1508-09	VP1508-10	VP1508-14	VP1508-15	VP1508-17	VP1508-18	VP1508-20	VP1508-21	VP1508-22	VP1508-23	VP1508-28	VP1508-29	VP1508-30
Semi-Targeted Identifications (ng/L)[1]																	
PFECA F	---	---	---	---	---	---	---	---	---	*>240*	---	*>65*	---	---	---	---	---
PFBSi	---	---	---	---	---	---	---	---	---	*>800*	*X*	---	---	---	---	---	---
FBSA	---	---	---	---	---	---	---	---	---	*X*	*X*	---	---	---	---	---	---
PFESA BP2	---	---	---	---	*X*	---	---	*X*	*X*	*X*	*X*	---	---	---	---	---	---
MeFBSA	---	---	---	---	---	---	---	---	---	*>680*	*X*	---	---	---	---	---	---
Tentative Identifications																	
PFO5OA	---	---	---	---	---	---	---	---	---	O	---	---	---	---	---	---	---
PFO2HxA	---	---	---	---	---	---	---	---	---	O	O	---	---	---	---	---	---
PFO3OA	---	---	---	---	---	---	---	---	---	O	O	---	---	---	---	---	---
PFMOAA	---	---	---	---	---	---	---	---	---	O	O	---	---	---	---	---	---
PFOA	√	√	√	√	√	√	√	---	---	√	√	√	√	√	√	√	√
PFOS	√	√	√	---	√	√	---	---	---	√	√	√	---	---	√	√	√
6H-Perfluorohex-1-ene	---	---	---	---	O	O	---	---	---	O	O	---	---	---	---	---	---
Perfluoro-3-(1H-perfluoroethoxy)	---	---	---	---	---	O	---	---	---	---	---	---	---	---	---	---	---

Table 13. SEMI-TARGETED PFAS RESULTS

Compound Acronym	NEIC Tag Number																
	VP1508-04	VP1508-05	VP1508-06	VP1508-07	VP1508-09	VP1508-10	VP1508-14	VP1508-15	VP1508-17	VP1508-18	VP1508-20	VP1508-21	VP1508-22	VP1508-23	VP1508-28	VP1508-29	VP1508-30
propane																	
2-Vinylperfluorobutane	---	---	---	---	---	---	---	---	---	O	O	---	---	---	---	---	---
3-(Perfluoro-2-butyl)propane-1,2-diol	---	---	---	---	---	---	---	---	---	O	---	---	---	---	---	---	---
3-(Perfluoropropyl) propanol	---	---	---	---	---	---	---	---	---	O	---	---	---	---	---	---	---
Allyl perfluoroisopropyl ether	---	---	---	---	---	---	---	---	---	O	---	---	---	---	---	---	---
FDEA	---	---	---	---	---	---	---	---	---	O	---	---	---	---	---	---	---
Methyl perfluorohexanoate	---	---	---	---	---	---	---	---	---	O	---	---	---	---	---	---	---
PFecHS	O	O	O	---	---	---	---	---	---	O	---	---	---	---	O	O	O
Perfluorohexane sulfonamide	---	---	---	---	---	---	---	---	---	O	---	---	---	---	---	---	---

[1] - Identification based on reference material comparison.

- "*X*" - observed response was less than the lowest calibration standard response.
- ">" - observed response was within calibration standard response range and the reported concentration value in nanograms per liter (ng/L) is a minimal value, as the extraction efficiency for these analytes were not evaluated during the semi-targeted analysis.

O – Tentative identification based on the accurate mass value of observed precursor, fragment and adduct ions. No calibration standard was analyzed as reference material was not readily available.

√ - Calibration standards for PFOA and PFOS were not analyzed with these samples, but these analytes were identified and quantified in the SPE extracts using the LC/MS QQQ method shown in Table 6.

--- Indicates analyte was not observed.

INVESTIGATION OBSERVATIONS

NEIC made the following observations during the multimedia compliance inspection. NEIC field team members discussed all observations with facility representatives during the closeout meeting unless otherwise noted in the observation description below. Additional observations were made based on the analytical results of the samples collected during the inspection that were not discussed with the facility representatives.

These observations are not final compliance determinations. OCE-WED, OCE-WCED, and Region 4 will make the final compliance determinations based on their review of this report and other technical, regulatory, and facility information.

Observation: 1 CWA
Observation Summary: Analytical results provided by NEIC laboratory for the wastewater samples collected by NEIC during the inspection at outfall 001, 002, 003, and internal outfall 004 show the discharge of several PFAS that are not listed in the KYPDES permit and are not monitored in the discharge.
Citation: **40 Code of Federal Regulations (CFR) § 122.1(b)** – *Scope of the NPDES permit requirement. (1) The NPDES program requires permits for the discharge of "pollutants" from any "point source" into "waters of the United States." The terms "pollutant", "point source" and "waters of the United States" are defined at §122.2.* **Kentucky Revised Statutes (KRS) 224.70-110** – *No person shall, directly or indirectly, throw, drain, run or otherwise discharge into any of the waters of the Commonwealth, or cause, permit or suffer to be thrown, drained, run or otherwise discharged into such waters any pollutant, or any substance that shall cause or contribute to the pollution of the waters of the Commonwealth in contravention of the standards adopted by the cabinet or in contravention of any of the rules, regulations, permits, or orders of the cabinet or in contravention of any of the provisions of this chapter.*
Evidence: **Appendix A –** Calgon Carbon Big Sandy Plant Final Permit KY0022276 **Appendix H –** VP1508 Calgon Carbon- Big Sandy Plant – Photographs
Description of Observation: Calgon is authorized to discharge boiler blowdown and backwash water, prime line bakers wet scrubbers, acid wash/neutralization wastewater, wash down, non-contact cooling, stormwater, REACT wastewater (including motive water from REACT and stormwater from the REACT area (Outfall 004)) domestic sanitary wastewater, and stormwater runoff from closed and capped landfill through outfalls 001, 002, and 003 per its KYPDES permit (**Appendix A)** NEIC collected wastewater samples from outfalls 001, 002, 003, and equivalent to outfall 004 identified as sample tag numbers VP1508-07, VP1508-12, VP1508-13, VP1508-21, VP1508-22, and VP1508-23 (**Appendix H**, photos 45, 53, 56, and 68).

Observation: 1 CWA

The table below displays the quantitative analytical results for samples VP1508-07, VP1508-12, VP1508-21, VP1508-22, and VP1508-23 reported in ng/L (equivalent to parts per trillion [ppt]).

Analyte	Analyte Concentration (ng/L)						
	Outfall 001	Outfall 002	Outfall 003	Equivalent to 004 after 7 ½ tanks			Reporting Limit (RL)
	(VP1508-12)	(VP1508-13)	(VP1508-07)	(VP1508-21)	(VP1508-22)	(VP1508-23)	
	Long Chain						
10:2 FTS[1]	< RL	< RL	< RL	< RL	< RL	< RL	50.0
4:2 FTS	< RL	< RL	< RL	< RL	< RL	< RL	2.07
6:2 FTS	< RL	< RL	< RL	< RL	< RL	< RL	2.10
8:2 FTS	< RL	< RL	< RL	< RL	< RL	< RL	2.12
HFPO-DA	< RL	< RL	< RL	< RL	< RL	41.9	5.53
N-EtFOSA	< RL	< RL	< RL	< RL	< RL	< RL	2.21
N-EtFOSAA	< RL	< RL	< RL	< RL	< RL	< RL	2.21
N-MeFOSA	< RL	< RL	< RL	< RL	< RL	< RL	2.21
N-MeFOSAA	< RL	< RL	< RL	< RL	< RL	< RL	2.21
PFBA	169	< RL	6.22	4450	4410	4390	2.21
PFBS	< RL	< RL	< RL	< RL	< RL	< RL	1.96
PFDA	< RL	< RL	< RL	< RL	< RL	< RL	2.21
PFDoA	< RL	< RL	< RL	< RL	< RL	< RL	50.0
PFECA-A	< RL	< RL	< RL	< RL	< RL	< RL	2.21
PFHpA	< RL	< RL	< RL	< RL	2.66	< RL	2.21
PFHpS	< RL	< RL	< RL	< RL	< RL	< RL	2.10
PFHxA	< RL	6.89	< RL	< RL	3.77	< RL	2.21
$PFHxS_{Total}$	< RL	< RL	< RL	< RL	< RL	< RL	2.02
PFNA	< RL	< RL	< RL	< RL	< RL	< RL	2.21
PFOA	< RL	< RL	< RL	< RL	12.6	14.6	2.21
$PFOS_{Total}$	< RL	< RL	< RL	< RL	< RL	< RL	2.05
PFOSA	< RL	< RL	< RL	< RL	< RL	< RL	2.21
PFPeA	< RL	< RL	< RL	< RL	< RL	< RL	22.1
PFPeS	< RL	< RL	< RL	< RL	< RL	< RL	2.08
PFTrA[2]	NA	NA	NA	NA	NA	NA	NA
PFUnA	< RL	< RL	< RL	< RL	< RL	< RL	2.21
	Short Chain						
2,2,3,3-TPrA	< RL	< RL	< RL	< RL	< RL	< RL	1000
2,3,3,3-TPrA	DET	< RL	< RL	DET	DET	DET	200
DBI	< RL	< RL	< RL	< RL	< RL	< RL	10.0
HQ-115	< RL	< RL	< RL	< RL	< RL	< RL	10.0
PFECHS	< RL	< RL	< RL	< RL	< RL	< RL	10.0
PFPA	DET	< RL	< RL	DET	DET	DET	800
TFAA	DET	< RL	< RL	DET	DET	DET	600
TFMS	21.0	< RL	< RL	3140	2990	3000	10.0

Observation: 1 CWA

[1] Results are estimated due to low spike recovery.
[2] PFTrA was not detected in the spike samples, thus is reported as NA (Not Applicable).
DET – Analytes are detected above the stated RL but could not be quantified due to poor calibration and/or QC results or significant ion suppression interfering with the MassHunter software's ability to quantify the result.
EMU- Expanded measurement uncertainty range for reported PFAS concentration values was 64% to 170%, except for:

- N-EtFOSAA, N-MeFOSAA, EtFOSA, PFHxA: EMU range: 36% to 230%
- 10:2 FTS, DBI: EMU range: 51% to 1100%
- 2,2,3,3-TPrA, 2,3,3,3-TPrA, PFPA, TFAA, PFTrA: No EMU range calculated due to insufficient quality control data.

(Using a coverage probability of 95% and coverage factor of 2.09 for short chain PFAS, or 2.57 for long chain PFAS.)

None of the above analytes are subject to monitoring requirements in the permit.

In addition to the quantitative analysis, the semi- targeted and tentative identifications show the presence of PFECA F in the outfall 004 equivalent samples.

Observation: 2 CWA

Observation Summary: NEIC inspectors observed evidence that unmonitored stormwater discharges were occurring at multiple locations in the southeast portion of the facility during rain events. These locations are not included in the KPDES permit as locations for allowable stormwater discharges.

Citation:

KPDES Permit – KY0022276

Pursuant to Authority in KRS 224,…

Calgon Carbon Corporation…is authorized to discharge from a facility located at Calgon Carbon Corporation – Big Sandy Plant, 15025 US Route 23 S, Catlettsburg, Boyd County, Kentucky to receiving waters named: Big Sandy River and White's Creek in accordance with effluent limitations, monitoring requirements and other conditions set forth in this permit.

Section 1.1 – 1.1. Compliance Monitoring Locations (Outfalls)

The following table lists the outfalls authorized by this permit, the location and description of each, and the DOW assigned KPDES outfall number:

Outfall No.	*Outfall Type*	*Latitude (N)*	*Longitude (W)*	*Receiving Water*	*Description*
001	*External*	*38.337500°*	*82.587500°*	*Big Sandy River*	*Boilers blowdown and backwash* *Prime Line Bakers Wet Scrubbers* *Acid Wash / Neutralization wastewater* *Wash Down* *Non-Contact Cooling* *Stormwater*

Observation: 2 CWA

					REACT Wastewater (Outfall 004)
002	*External*	*38.337500°*	*82.587500°*	*Big Sandy River*	*Domestic Sanitary Wastewater*
003	*External*	*38.336138°*	*82.598799°*	*White's Creek*	*Stormwater runoff from closed and capped landfill*
004	*Internal*	*38.337682°*	*82.588372°*	*Outfall 001*	*Motive water from REACT* *Stormwater from REACT area*

CWA § 301(a): *except as in compliance with this section and sections 302, 306, 307, 318, 402 and 404 of this Act, the discharge of any pollutant by any person shall be unlawful.*

40 CFR § 122.1(b)(1): *The NPDES program requires permits for the discharge of "pollutants" from any "point source" into "waters of the United States."*

40 CFR § 122.2*: Pollutant: means dredged spoil, solid waste, incinerator residue, filter backwash, sewage, garbage, sewage sludge, munitions, chemical wastes, biological materials, radioactive materials (except those regulated under the Atomic Energy Act of 1954, as amended (42 U.S.C. 2011 et seq.)), heat, wrecked or discarded equipment, rock, sand, cellar dirt and industrial, municipal, and agricultural waste discharged into water. It does not mean: (a) Sewage from vessels; or (b) Water, gas, or other material which is injected into a well to facilitate production of oil or gas, or water derived in association with oil and gas production and disposed of in a well, if the well used either to facilitate production or for disposal purposes is approved by authority of the State in which the well is located, and if the State determines that the injection or disposal will not result in the degradation of ground or surface water resources.*

40 CFR § 122.26(b)(14*): Storm water discharge associated with industrial activity means the discharge from any conveyance that is used for collecting and conveying storm water and that is directly related to manufacturing, processing or raw materials storage areas at an industrial plant. The term does not include discharges from facilities or activities excluded from the NPDES program under this part 122. For the categories of industries identified in this section, the term includes, but is not limited to, storm water discharges from industrial plant yards; immediate access roads and rail lines used or traveled by carriers of raw materials, manufactured products, waste material, or by-products used or created by the facility; material handling sites; refuse sites; sites used for the application or disposal of process waste waters (as defined at part 401 of this chapter); sites used for the storage and maintenance of material handling equipment; sites used for residual treatment, storage, or disposal; shipping and receiving areas; manufacturing buildings; storage areas (including tank farms) for raw materials, and intermediate and final products; and areas where industrial activity has taken place in the past and significant materials remain and are exposed to storm water. For the purposes of this paragraph, material handling activities include storage, loading and unloading, transportation, or conveyance of any raw material, intermediate product, final product, by-product or waste product. The term excludes areas located on plant lands separate from the plant's industrial activities, such as office buildings and accompanying parking lots as long as the drainage from the excluded areas is not mixed with storm water*

Observation: 2 CWA
drained from the above described areas. Industrial facilities (including industrial facilities that are federally, State, or municipally owned or operated that meet the description of the facilities listed in paragraphs (b)(14)(i) through (xi) of this section) include those facilities designated under the provisions of paragraph (a)(1)(v) of this section.
Evidence: **Appendix H –** VP1508 Calgon Carbon- Big Sandy Plant – Photographs
Description of Observation: During the walkthrough of the site, NEIC inspectors observed evidence of stormwater discharges along the southeast side of the property. Photos 38, 39, 40, 42, and 43 (**Appendix H**) display evidence of stormwater flow leaving the industrial containment areas and running down the banks of the Big Sandy River. The apparent flow paths of the storm water indicate that coal may have been present in the discharges. Calgon is only authorized to discharge industrial stormwater through outfalls 001, 003, and 004. The facility started remediation efforts during NEIC's inspection to stop future discharges (**Appendix H,** photos 70, 71, 72, and 73) and was developing a plan to fully resolve the issue.

Observation: 3 CWA
Observation Summary: Best Management Practices (BMP) inspection reports did not include stormwater assessments on the southeast portion of the facility where NEIC inspectors found evidence of unmonitored stormwater discharges.
Citation: ***KYPDES Permit KY0022276 - 3.5 Specific Requirements*** *– The plan shall be consistent with the general guidance contained in the publication entitled "NPDES Best Management Practices Guidance Document" and shall include the following baseline BMPs as a minimum:… (7) Good Housekeeping.* ***BMP Plan Section 4.1.3*** *– …Any material with the potential to contaminate storm water is stored on an impervious surface with drainage to the treatment facility or secondary containment will be utilized.* ***BMP Plan Section 4.1.6*** *– Monthly and quarterly visual inspections are conducted to audit that all elements of the SWP3/BMP Plan are in place and working properly. The Big Sandy Plant Pollution Prevention Team is responsible for conducting and recording the results of all visual inspections. Appendix D, or Section J of the Comprehensive Environmental BMP include the Inspection Report Forms that are used to record inspection findings. Periodic housekeeping audits are also performed to assist with each area's compliance.*
Evidence: **Appendix L –** Calgon Carbon-BMP-SWPPP

Observation: 3 CWA
Description of Observation: In the Calgon Carbon BMP/Storm Water Pollution Prevention Plan (SWPPP), there are inspection forms available for facility representatives to conduct monthly and quarterly monitoring of the site (**Appendix L,** pages 35 – 41). The inspection forms do not include monitoring for stormwater discharges on the southeast portion of the property and the monthly form appears focused on the "oil storage and loading/unloading areas," "oil-filled electrical equipment areas," and "chemical storage tanks." The only language on the quarterly form addressing stormwater states, "Is storm water in areas around mobile fueling vehicle, diesel fuel pumps, and above ground storage tanks adequately addressed?" During the inspection, NEIC inspectors observed evidence of unmonitored stormwater discharges occurring along the bank of the Big Sandy River on the southeastern portion of the site. The KYPDES permit only allows for the discharge of stormwater through the permitted outfalls.

Observation: 4 CWA
Observation Summary: The BMP/SWPPP plan refers to sheet flows leaving the industrial material storage areas. NEIC inspectors observed evidence of industrial stormwater discharges occurring in the material storage areas.
Citation: ***40 CFR § 122.26(b)(14)****: Storm water discharge associated with industrial activity means the discharge from any conveyance that is used for collecting and conveying storm water and that is directly related to manufacturing, processing or raw materials storage areas at an industrial plant…*
Evidence: **Appendix L –** Calgon Carbon-BMP-SWPPP **Appendix H –** VP1508 Calgon Carbon- Big Sandy Plant - Photographs
Description of Observation: Page 6 of Calgon's BMP/SWPPP plan (**Appendix L**) states: *Small portions of the extreme southern and eastern areas of the property which drain by sheet flow to the Big Sandy River. These areas total less than 1.5 acres and are made up primarily of the raw material storage area outside slope and the southern edge of the property, which naturally slopes in that direction. No oils are stored or utilized in this area.* Discharges from the industrial processing areas that are exposed to stormwater are to be prevented unless the water is discharged through the approved existing outfalls. The specific reference to sheet flows occurring and discharging from the industrial areas indicates that the facility must control the stormwater flows and eliminate discharges. The facility had conducted some remedial actions by the close of the NEIC inspection to prevent additional run off from the storage areas to the Big Sandy River (**Appendix H,** photos 70, 71, 72, and 73).

Observation: 5 CWA
Observation Summary: The collection drain between the REACT area and the north staging area was clogged.
Citation: *None*
Evidence: **Appendix H –** VP1508 Calgon Carbon- Big Sandy Plant – Photographs **Appendix M –** REACT Trench Photographs
Description of Observation: NEIC inspectors observed that the collection drain on the border of the REACT area adjacent to the north staging area was clogged with, what appeared to be, spent carbon (**Appendix H**, photo 17). The clogged drain would prevent the collection of water and might allow the discharge of materials outside of the REACT containment area. This issue was corrected before the end of the inspection (**Appendix M**).

Observation: 6 CWA
Observation Summary: A small spill of carbon was observed by NEIC inspectors outside of the REACT containment area.
Citation: *None*
Evidence: **Appendix H –** VP1508 Calgon Carbon- Big Sandy Plant – Photographs **Appendix N –** 3-22-23 Spent Carbon Spill
Description of Observation: NEIC inspectors observed evidence of a small spill of untreated, used, activated carbon extending from the north side of the REACT area into the north staging area (**Appendix H,** photo 15 and 16). This spill was cleaned up before the end of the inspection (**Appendix N**).

Observation: 1 RCRA
Observation Summary: Calgon stored hazardous waste in four frac tanks in the REACT area. Calgon did not have a written assessment, reviewed, and certified by a qualified Professional Engineer, attesting that the four tank systems have sufficient structural integrity and are acceptable for the storing and treating of hazardous waste. The assessment is required before placing the tanks into service.
Citation*:* ***401 Kentucky Administrative Regulations (KAR) 39:090. Hazardous waste permit program.*** *Section 1. Owners and Operators of Hazardous Waste Treatment, Storage, and Disposal Facilities. Except as established in subsections (1) through (7) of this section and Sections 5 through 9 of this administrative regulation, standards for owners and operators of hazardous waste treatment, storage, and disposal facilities shall be as established in 40 C.F.R. Part 264, except 40 C.F.R. 264.1(f), 40 C.F.R. 264.1(g)(12), 40 C.F.R. 264.15(b)(5), 40 C.F.R. 264.149, 40 C.F.R. 264.150, 40 C.F.R. 264.301(l), 40 C.F.R. 264.1030(d), 40 C.F.R. 264.1050(g), and 40 C.F.R. 264.1080(e) through (g).*

Observation: 1 RCRA

401 KAR 39:060, Section 3(1) [40 CFR § 261.3] Definition of hazardous waste.

(a) A solid waste, as defined in §261.2, is a hazardous waste if:

(2) It meets any of the following criteria:

(iv) It is a mixture of solid waste and one or more hazardous wastes listed in subpart D of this part and has not been excluded from paragraph (a)(2) of this section under §§260.20 and 260.22,

401 KAR 39:005, Section 1 [40 CFR § 260.10] Definitions

Tank means a stationary device, designed to contain an accumulation of hazardous waste which is constructed primarily of non-earthen materials (e.g., wood, concrete, steel, plastic) which provide structural support.

401 KAR 39:090, Section 1 [40 CFR § 264.192] Design and installation of new tank systems or components.

(a) Owners or operators of new tank systems or components must obtain and submit to the Regional Administrator, at time of submittal of part B information, a written assessment, reviewed and certified by a qualified Professional Engineer, in accordance with §270.11(d) of this chapter, attesting that the tank system has sufficient structural integrity and is acceptable for the storing and treating of hazardous waste. The assessment must show that the foundation, structural support, seams, connections, and pressure controls (if applicable) are adequately designed and that the tank system has sufficient structural strength, compatibility with the waste(s) to be stored or treated, and corrosion protection to ensure that it will not collapse, rupture, or fail.

Evidence:
Appendix H – VP1508 Calgon Carbon- Big Sandy Plant - Photographs
Appendix O – Calgon Carbon RCRA Permit Application
Interviews

Description of Observation: At the time of the inspection, Calgon was storing hazardous waste in four frac tanks (**Appendix H**, photo 5). The frac tanks were labeled as hazardous waste and contained water from the sumps and secondary containment area that was in contact with motive water generated during storm events. The rainwater in the secondary containment is channeled by trenches into the main sump mixing with motive water. The excess water is stored in storage tanks because the volume is too much for the wastewater treatment system to process at one time.

Motive water is used to move spent carbon from the loading sumps into the hazardous waste tanks and, ultimately, to the regeneration furnace. Calgon mixes non-hazardous and hazardous spent carbons (including listed hazardous waste carbon) when received, which, per the mixture rule, would make all spent carbon handled by Calgon a listed hazardous waste. A full list of the hazardous waste codes received by Calgon is in the RCRA permit application (**Appendix O,** pages 153-185). The list includes F, K, P and U-listed hazardous wastes. Motive water that is mixed with the listed spent carbon is also a listed hazardous waste. As more water is needed in the motive water system to make up for lost water, the hazardous waste stored in the frac tanks is returned to main sump and transferred to permitted hazardous waste tank D-707, the carbon motive water tank.

Observation: 1 RCRA
Frac tanks are designed for use as stationary tanks. Frac tanks are not designed to transport material when material is stored in the tanks. Because the tanks are designed and intended for use when stationary, frac tanks meet the definition of a tank in 40 CFR §260.10. According to Calgon representatives, the frac tanks were placed into service two or three years before the NEIC inspection. At the time of the inspection, Calgon could not provide a written assessment, reviewed, and certified by a qualified Professional Engineer, in accordance with 40 CFR § 270.11(d), attesting that the four tank systems have sufficient structural integrity and is acceptable for the storing and treating of hazardous waste. Calgon representatives stated that such an assessment has not been performed.

Observation: **2 RCRA**

Observation Summary: Calgon stored listed hazardous waste sludge from the motive water system in a secondary containment system. The containment system meets the definition of a tank. This unit is not included as a hazardous waste management unit in Calgon's hazardous waste permit and did not have an engineering assessment performed before the unit was placed into service.

Citation*:*

401 KAR 39:090. Hazardous waste permit program.

Section 1. Owners and Operators of Hazardous Waste Treatment, Storage, and Disposal Facilities. Except as established in subsections (1) through (7) of this section and Sections 5 through 9 of this administrative regulation, standards for owners and operators of hazardous waste treatment, storage, and disposal facilities shall be as established in 40 C.F.R. Part 264, except 40 C.F.R. 264.1(f), 40 C.F.R. 264.1(g)(12), 40 C.F.R. 264.15(b)(5), 40 C.F.R. 264.149, 40 C.F.R. 264.150, 40 C.F.R. 264.301(l), 40 C.F.R. 264.1030(d), 40 C.F.R. 264.1050(g), and 40 C.F.R. 264.1080(e) through (g).

401 KAR 39:060, Section 3(1) [40 CFR § 261.3] Definition of hazardous waste.

(a) A solid waste, as defined in §261.2, is a hazardous waste if:

(2) It meets any of the following criteria:

(iv) It is a mixture of solid waste and one or more hazardous wastes listed in subpart D of this part and has not been excluded from paragraph (a)(2) of this section under §§260.20 and 260.22,

401 KAR 39:005, Section 1 [40 CFR § 260.10] Definitions

Tank means a stationary device, designed to contain an accumulation of hazardous waste which is constructed primarily of non-earthen materials (e.g., wood, concrete, steel, plastic) which provide structural support.

401 KAR 39:090, Section 1 [40 CFR § 264.192] Design and installation of new tank systems or components.

(a) Owners or operators of new tank systems or components must obtain and submit to the Regional Administrator, at time of submittal of part B information, a written assessment, reviewed and certified by a qualified Professional Engineer, in accordance with §270.11(d) of this chapter, attesting that the tank system has sufficient structural

Observation: **2 RCRA**
integrity and is acceptable for the storing and treating of hazardous waste. The assessment must show that the foundation, structural support, seams, connections, and pressure controls (if applicable) are adequately designed and that the tank system has sufficient structural strength, compatibility with the waste(s) to be stored or treated, and corrosion protection to ensure that it will not collapse, rupture, or fail.
Evidence: **Appendix H –** VP1508 Calgon Carbon- Big Sandy Plant – Photographs **Appendix O –** Calgon Carbon RCRA Permit Application **Appendix P –** REACT Dike Interviews
Description of Observation: Calgon was storing sludge from storm water that fell within the carbon activation process area containment in the secondary containment of a tank that was removed (**Appendix H,** photo 74). Calgon refers to the area as the dike north of the old hose-in sump (**Appendix P**), and the area has a secondary containment system that was constructed for a tank that has since been removed. The sludge-containing secondary containment system is located next to a small container unloading sump. The sludge in the containment system observed by NEIC inspectors consisted of spent carbon particles from inside the reactivation process area. Calgon mixes non-hazardous and hazardous spent carbons (including listed hazardous waste carbon) when received, which, per the mixture rule, would make all spent carbon handled by Calgon a listed hazardous waste. A full list of the hazardous waste codes received by Calgon is in the RCRA permit application (**Appendix O,** pages 153-185). The list includes F, K, P and U-listed hazardous wastes. The sludge managed in the secondary containment system described above is also a listed hazardous waste because it consists of particles of spent activated carbon. Calgon representatives stated that the sludge was the residual solids from storing storm water from process drains and the process area. This is the same material that was stored in the frac tanks in RCRA observation 1 that is managed as hazardous waste. The containment system is constructed with concrete sides and floor that meets the definition of a tank. At the time of the inspection, a written assessment, reviewed and certified by a qualified Professional Engineer, in accordance with §270.11(d) of this chapter, attesting that the tank system has sufficient structural integrity and is acceptable for the storing and treating of hazardous waste was not available. Calgon representatives stated that such an assessment has not been performed. The facility provided a picture showing the sludge was removed from the dike area before the end of NEIC's inspection (**Appendix P**).

Observation: **3 RCRA**
Observation Summary: The closed vent system for Calgon's hazardous waste tanks contained one open-ended line that operates as a bypass of the activated carbon control device. A closed vent system bypass must be equipped with a flow indicator or a locking device to ensure that it remains closed when the control device is in operation.

Observation: **3 RCRA**

Citation:

401 KAR 39:090. Hazardous waste permit program.

Section 1. Owners and Operators of Hazardous Waste Treatment, Storage, and Disposal Facilities. Except as established in subsections (1) through (7) of this section and Sections 5 through 9 of this administrative regulation, standards for owners and operators of hazardous waste treatment, storage, and disposal facilities shall be as established in 40 C.F.R. Part 264, except 40 C.F.R. 264.1(f), 40 C.F.R. 264.1(g)(12), 40 C.F.R. 264.15(b)(5), 40 C.F.R. 264.149, 40 C.F.R. 264.150, 40 C.F.R. 264.301(l), 40 C.F.R. 264.1030(d), 40 C.F.R. 264.1050(g), and 40 C.F.R. 264.1080(e) through (g).

Hazardous Waste Permit No. KYD-005-009-923
Part VII.C.1.3, Table VII.1: Hazardous Waste Management Units and Control Devices Subject to 40 CFR Part 264 – Subpart CC:

Hazardous Waste Management Unit	***Unit Type***	***Brief Description***
Tank Farm Area B *Thirteen (13) storage and process tanks* *Including covers, closure devices, closed-vent systems, and control devices*	*40 CFR Part 264 - Subpart J Tank System*	*Hazardous Waste Storage Tanks: Spent activated carbon/motive water, carbon fines.* *Design capacities from individual tanks vary from 37 to 20,000 Gallons.* *Total design capacity all units: 192,077 Gallons*

VII.C.7.3 *All Tank Farm Area B tanks shall meet the Level 2 requirements discussed in 40 CFR 264.1084(d)(3). The tanks shall be vented directly through a closed-vent system to a control device in accordance with the requirements specified in 40 CFR 264.1084(g).*

> ***VII.C.7.3.1*** *All Tank Farm Area B tanks shall be in compliance with 40 CFR 264.1084(g)(1). Tanks shall be covered by a fixed roof; the fixed roof and its covers and closure devices shall be designed to form a continuous barrier over the entire surface area of the hazardous waste; closure devices shall be designed to operate with no detectable organic emissions; fixed roof and closure devices shall be made of suitable materials that will minimize exposure of the hazardous waste to the atmosphere and maintain their integrity; and the control-vent system and closure device shall be designed and operated in accordance with the requirements of 40 CFR 264.1087. The fixed roof shall be installed with each closure devices secured in the closed position and the vapor headspace underneath the fixed roof vented to the control device except as provided in 40 CFR 264.1084(g)(2).*

40 CFR 264 Subpart CC: Emission Standards for Tanks, Surface Impoundments and Containers.
401 KAR 39:090, Section 1 [40 CFR §264.1081] Definitions.

Observation: 3 RCRA

As used in this subpart, all terms shall have the meaning given them in 40 CFR 265.1081, the Act, and parts 260 through 266 of this chapter.

401 KAR 39:090, Section 1 [§264.1031] Definitions.

Open-ended valve or line means any valve, except pressure relief valves, having one side of the valve seat in contact with hazardous waste and one side open to the atmosphere, either directly or through open piping.

401 KAR 39:090, Section 1 [40 CFR §264.1087] Standards: Closed-vent systems and control devices.

(a) This section applies to each closed-vent system and control device installed and operated by the owner or operator to control air emissions in accordance with standards of this subpart.

(b) The closed-vent system shall meet the following requirements:

(3) In the case when the closed-vent system includes bypass devices that could be used to divert the gas or vapor stream to the atmosphere before entering the control device, each bypass device shall be equipped with either a flow indicator as specified in paragraph (b)(3)(i) of this section or a seal or locking device as specified in paragraph (b)(3)(ii) of this section. For the purpose of complying with this paragraph, low leg drains, high point bleeds, analyzer vents, open-ended valves or lines, spring loaded pressure relief valves, and other fittings used for safety purposes are not considered to be bypass devices.

(i) If a flow indicator is used to comply with paragraph (b)(3) of this section, the indicator shall be installed at the inlet to the bypass line used to divert gases and vapors from the closed-vent system to the atmosphere at a point upstream of the control device inlet. For this paragraph, a flow indicator means a device which indicates the presence of either gas or vapor flow in the bypass line.

(ii) If a seal or locking device is used to comply with paragraph (b)(3) of this section, the device shall be placed on the mechanism by which the bypass device position is controlled (e.g., valve handle, damper lever) when the bypass device is in the closed position such that the bypass device cannot be opened without breaking the seal or removing the lock. Examples of such devices include, but are not limited to car-seal or a lock-and-key configuration valve. The owner or operator shall visually inspect the seal or closure mechanism at least once every month to verify that the bypass mechanism is maintained in the closed position.

Evidence:

Appendix C – Calgon Carbon RCRA Permit

Appendix H – VP1508 Calgon Carbon- Big Sandy Plant – Photographs

Description of Observation:

As required by Section VII.C.G of the hazardous waste permit (**Appendix C**, page 107), vapors from the tanks in Calgon's Tank Farm Area B are vented through a closed vent system to activated carbon control devices. NEIC inspectors observed one open-ended valve on the closed vent system (**Appendix H,** Photo 83). The valve was located in the vicinity of white LDAR tags 58-63. Tag 58 is visible on the right-hand side of photo 83. The open-ended valve is a bypass device that could divert the gas or vapor stream to the atmosphere before the

Observation: **3 RCRA**
control device. The bypass device is required to be equipped with a flow indicator or a seal or locking device. This valve was not equipped with one of the required devices.

Observation: **4 RCRA**
Observation Summary: Calgon has not made a hazardous waste determination for the partially activated carbon that is removed from the baker furnaces during process upsets and placed in lagoon 1.
Citation: ***401 KAR 39:080. Hazardous waste handlers.*** ***Section 1. Generators of Hazardous Waste.*** *(1) Except as established in subsections (2) through (11) of this section and Section 5 of this administrative regulation, the requirements for standards applicable to generators of hazardous waste shall be as established in 40 C.F.R. Part 262, except 40 C.F.R. 262.10(k).* ***401 KAR 39.080, Section 1(1) [40 CFR §262.11] Hazardous waste determination and recordkeeping.*** *A person who generates a solid waste, as defined in 40 CFR 261.2, must make an accurate determination as to whether that waste is a hazardous waste in order to ensure wastes are properly managed according to applicable RCRA regulations. A hazardous waste determination is made using the following steps:* *(a) The hazardous waste determination for each solid waste must be made at the point of waste generation, before any dilution, mixing, or other alteration of the waste occurs, and at any time in the course of its management that it has, or may have, changed its properties as a result of exposure to the environment or other factors that may change the properties of the waste such that the RCRA classification of the waste may change.*
Evidence: Interviews
Description of Observation: When manufacturing virgin activated carbon in the prime lines, coal is placed in the baker furnaces and heated to a high temperature to convert all the hydrocarbons into activated carbon. Process upsets can occur that require the contents of the baker furnaces to be dumped on the pad below the furnace before the coal is completely converted to activated carbon. When this occurs, Calgon sprays the material with water to cool it and then discharges the mixture into the lagoon 1. During the carbon activation process, the high temperatures in the baker furnaces have the potential to crack long hydrocarbons in the coal into shorter hydrocarbon chains, including benzene. Calgon has not made a hazardous waste determination for the partially activated carbon material that is removed from the baker furnaces during process upsets to determine if the benzene levels in the material are high enough to be classified as a hazardous waste. If the material is a hazardous waste, the act of washing the material into lagoon 1, which is an earthen impoundment, would constitute hazardous waste land disposal. Lagoon 1 is not permitted to receive hazardous waste.

Observation: **5 RCRA**

Observation Summary: Calgon had open-ended lines on the 700-series tanks in Tank Farm Area B.

Citation:

Hazardous Waste Permit No. KYD-005-009-923

VII.B AIR EMISSION STANDARDS FOR EQUIPMENT LEAKS

> ***VII.B.1 40 CFR Part 264 - Subpart BB*** *contains air emission standards for equipment leaks and applies to all equipment that contains or contacts hazardous wastes with organic concentrations of at least ten (10) percent by weight.*
>
> ***VII.B.1.2 T****he Permittee, as part of its reactivation process of spent activated carbon through thermal treatment, operates the Area B Tank Farm tanks D-701 through D-710 and D-901 through D-903, and the Carbon Regeneration Unit and Feed System, along with their ancillary equipment. This ancillary equipment is identified in Attachment B - Facility Description: Sub-Section B-8 -Equipment Leak Standards: Table B-3, of the Approved Permit Application ("Attachment B-Table B-3"), and contains or contacts hazardous waste with organic concentrations greater than ten (10) percent and less than twenty (20) percent by weight for greater than 300 hours per calendar year. The hazardous waste streams managed in this equipment are not in the gaseous state and have total concentrations, of the organic components, less than 20 percent by weight, making the equipment managing these hazardous waste streams equipment in heavy liquid service. This equipment is subject to 40 CFR Part 264 - Subpart BB and Permit Condition VII.B.*

401 KAR 39:090, Section 1 [40 CFR §264.1031] Definitions.

> *Open-ended valve or line means any valve, except pressure relief valves, having one side of the valve seat in contact with hazardous waste and one side open to the atmosphere, either directly or through open piping.*

401 KAR 39:090, Section 1 [40 CFR §264.1056] Standards: Open-ended valves or lines.

> *(a)(1) Each open-ended valve or line shall be equipped with a cap, blind flange, plug, or a second valve.*
>
> *(2) The cap, blind flange, plug, or second valve shall seal the open end at all times except during operations requiring hazardous waste stream flow through the open-ended valve or line.*

Evidence:
Appendix H – VP1508 Calgon Carbon- Big Sandy Plant – Photographs
Interviews

Description of Observation:
In Tank Farm Area B Calgon maintains the 700-series of permitted hazardous waste tanks, identified as D-701 through D-710. As stated in the Calgon hazardous waste permit, tanks D-701 through D-710 are subject to the air emissions requirements in 40 CFR Part 264, Subpart BB.

Observation: **5 RCRA**

During the inspection, NEIC inspectors observed open-ended lines associated with the following tanks:

Tank	Component Tag Number
D-701	966
D-702	961
D-703	959
D-704	Not in the inventory
D-705	960
D-706	963

According to Calgon representatives, the components identified above are clean-out ports for the water overflow line on each corresponding tank. A photograph of the clean out port for Tank D-704 is shown in **Appendix H,** photo 82. However, even if the components are clean-out ports, they are also open-ended lines and must be equipped with a cap, plug, blind flange, or second valve to seal the open end.

Observation: **6 RCRA**

Observation Summary: Calgon operates the intensive screener to separate the motive water from the spent carbon before treatment in the carbon regeneration furnace. The intensive screener is not included in the RCRA permit as a hazardous waste management unit.

Citation:

401 KAR 39:005. Definitions for 401 KAR Chapter 39.

RELATES TO: KRS 224.1, 224.10, 224.46, 224.50-545(2)(a), 322.010, 40 C.F.R. Parts 260, 261, 262, 268, 270.

Section 1. Definitions. Except as established in this section, definitions for 401 KAR Chapter 39 shall be as established in 40 C.F.R. 260.10.

401 KAR 39:005, Section 1 [40 CFR Section 260.10] Definitions.

Ancillary equipment means any device including, but not limited to, such devices as piping, fittings, flanges, valves, and pumps, that is used to distribute, meter, or control the flow of hazardous waste from its point of generation to a storage or treatment tank(s), between hazardous waste storage and treatment tanks to a point of disposal onsite, or to a point of shipment for disposal off-site.

Treatment means any method, technique, or process, including neutralization, designed to change the physical, chemical, or biological character or composition of any hazardous waste so as to neutralize such waste, or so as to recover energy or material resources from the waste, or so as to render such waste non-hazardous, or less hazardous; safer to transport, store, or dispose of; or amenable for recovery, amenable for storage, or reduced in volume.

Evidence:

Appendix H – VP1508 Calgon Carbon- Big Sandy Plant – Photographs

Appendix O – Calgon Carbon RCRA Permit Application

Observation: **6 RCRA**

Description of Observation:

The intensive screener (**Appendix H,** photo 90) removes motive water from the carbon before the carbon is treated in the regeneration furnace. Calgon's RCRA permit application (**Appendix O,** page 245), lists the intensive screener as ancillary equipment to the carbon regeneration unit. The intensive screener does not meet the definition of ancillary equipment because its primary purpose is not to transport hazardous waste, but rather to remove motive water to make the carbon waste more amenable to treatment by the carbon regeneration unit. Reducing the volume of waste through the removal of motive water constitutes treatment under the definition. Units that treat hazardous waste must be regulated under the permit and have specific permit requirements, either as a tank or a miscellaneous unit.

Observation: **7 RCRA**

Observation Summary: Calgon operates units that manage motive water. Motive water is water that was mixed with spent carbon that is listed hazardous waste. These units, which include the motive water sumps, motive water trenches, and the diester sump, are not included in the Calgon RCRA permit as hazardous waste management units.

Citation:

401 KAR 39:005. Definitions for 401 KAR Chapter 39.

RELATES TO: KRS 224.1, 224.10, 224.46, 224.50-545(2)(a), 322.010, 40 C.F.R. Parts 260, 261, 262, 268, 270.

Section 1. Definitions. Except as established in this section, definitions for 401 KAR Chapter 39 shall be as established in 40 C.F.R. 260.10.

401 KAR 39:005, Section 1 [40 CFR Section 260.10] Definitions.

Storage means the holding of hazardous waste for a temporary period, at the end of which the hazardous waste is treated, disposed of, or stored elsewhere.

Sump means any pit or reservoir that meets the definition of tank and those troughs/trenches connected to it that serve to collect hazardous waste for transport to hazardous waste storage, treatment, or disposal facilities; except that as used in the landfill, surface impoundment, and waste pile rules, "sump" means any lined pit or reservoir that serves to collect liquids drained from a leachate collection and removal system or leak detection system for subsequent removal from the system.

Tank means a stationary device, designed to contain an accumulation of hazardous waste which is constructed primarily of non-earthen materials (e.g., wood, concrete, steel, plastic) which provide structural support.

Evidence:

Appendix O – Calgon Carbon RCRA Permit Application

Appendix H – VP1508 Calgon Carbon- Big Sandy Plant – Photographs

Description of Observation:

Calgon operates units that manage motive water or a mixture of motive water and hazardous waste that are not permitted as RCRA storage units. Motive water is used to move spent carbon from the loading sumps into the hazardous waste tanks and, ultimately, to the regeneration furnace. Calgon mixes non-hazardous and hazardous spent carbon wastes

Observation: **7 RCRA**
(including listed hazardous waste carbon) when received which, per the mixture rule, would make all spent carbon handled by Calgon a listed hazardous waste. A full list of the hazardous waste codes received by Calgon is in the RCRA permit application (**Appendix O,** pages 153-185). The list includes F, K, P and U listed hazardous wastes. Any motive water mixed with the listed spent carbon is also a listed hazardous waste. The units that are managing motive water and are not in the Calgon permit as hazardous waste management units include the motive water main sumps and trench collection system, and the diester sump. The collection and transfer sumps (main sumps) (Appendix H, photo 84) and trench collection system are identified as “other identified equipment” in the RCRA permit application (**Appendix O,** pages 266-267). The main sumps receive water flows from the trench system within the carbon regeneration area. This includes water from the cleanup of spent activated carbon that is washed into the trenches. The water in the main sump is returned to permitted tank D-707, the spent carbon motive water tank (**Appendix O**, page 250). According to Calgon representatives, the hazardous waste stored in the frac tanks in Observation 1 RCRA is also returned to the main sump. The diester sump receives a mixture of motive water and spent carbon generated by the intensive screener (**Appendix H**, photos 87, 88, and 89). The water from the diester is also returned to permitted tank D-707, the spent carbon motive water tank (**Appendix O,** page 250). Units that store hazardous waste must be regulated under the permit and have specific permit requirements.