Submissions to the State of North Carolina and Cape Fear River Watch

The following table identifies submissions made by Chemours pursuant to the Consent Order and Addendum ("COA") for the period of July 1, 2020 through the end of the third quarter on September 30, 2020.[1]

CO Section	Title	Submitted Date
11c	PFAS Characterization Quarterly Report	07/31/2020
12/COA 04d	Stormwater Pollution Prevention Plan	07/01/2020
12/COA 01a	Cape Fear River PFAS Mass Loading Protocol	08/31/2020
12/COA 01b	Files for Current PFAS Loading Model	08/31/2020
12/COA 01c	Outfall 002 PFAS Mass Loading Protocol	08/31/2020
12/COA 02a	Interim Seep Remediation System Plan	08/31/2020
12/COA 01b	Cape Fear River PFAS Mass Loading Assessment - 2020 Q2	09/30/2020
12/COA 04b	Stormwater Treatment System Sampling Plan	09/30/2020
28	Quarterly Progress Report	07/21/2020

[1] Consent Order submissions by Chemours from lodging of the Proposed Consent Order in November 2018 through March 31, 2019 were presented in the 2019 1st quarter report, April 1, 2019 through June 30, 2019 in the 2019 2nd quarter report, July 1, 2019 through September 30, 2019 in the 2019 3rd quarter report, October 1, 2019 through December 31, 2019 in the 2019 4th quarter report, January 1, 2020 through March 31, 2020 in the 2020 1st quarter report, and April 1, 2020 through June 30, 2020 in the 2020 2nd quarter report.

2020 Third Quarter Residential Summary

Item	Cumberland County (East of River)	Cumberland County (West of River)	Bladen County (East of River)	Bladen County (West of River)	Robeson County	Total
Total Number of Residences Sampled	239	430	4	9	37	719
Residences Exceeding GAC Criteria (GenX >= 140 ng/L)	3	3	0	2	0	8
Residences Exceeding RO Criteria (∑PFAS >= 70 ng/L)	68	80	0	5	11	164
Residences Exceeding RO Criteria (PFAS >= 10 ng/L)	94	135	0	0	11	240
Residences Drinking Water Well Detections (Results < 10 ng/L)	22	77	0	1	5	105
Residences Drinking Water Well Non-Detections	52	135	4	1	10	202

Replacement Drinking Water Actions

(Replacement drinking water actions from November 2018[2] - September 30, 2020)

Summary		Number of residents on bottled water	GAC Systems On-line & Confirmation Sampling Complete	Number of Homes Where RO Systems Installed
	Total	2486	89	1139

Bottled Water		Residences Eligible for Bottled Water	Already connected to Public Water	Eligible Residences Receiving Bottled Water
	Q3 2020	550	0	504
	Total	2486	411	2486

GAC		Residences Eligible for GAC	Already connected to Public Water	Public Water Readily Available	Public Water Feasible	Residents Declined GAC System	GAC Systems to Install	Number of Residences Responded to GAC Offer (Interview Conducted or Declined Offer)
	Q3 2020	10	Data Not Available	0	0	0	10	4
	Total	253	22	12	35	4	133	104

		Number of GAC Systems to Install but Resident has Not Responded to Offer	System On-line	Confirmation Sampling Complete	GAC Offer Letters Sent to Residents	Call Log Interactions with GAC Residents	GAC Residence Response Rate
	Q3 2020	Not Applicable	7	7	10	429	Not Applicable
	Total	106	89	89	253	2698	41%

RO		Residences Eligible for RO (includes homes with shared wells)	Number of Residences Responded to RO Offer	Residents Declined RO	Homes/Buildings where RO Systems to be Installed but Resident has Not Responded	RO Residence Response Rate
	Q3 2020	555	897	3	321	Not Applicable
	Total	3754	1993	62	1603	53%

		Number of Homes where RO Systems Installed	Homes/Buildings where RO Systems are to be Installed	Number of RO Offer Letters Sent to Residences	Call Log Interactions with RO Residents
	Q3 2020	370	555	555	2472
	Total	1139	2368	3754	11891

[2] The date the proposed Consent Order was lodged.

Consent Order Progress Details

This section summarizes the activities that have been undertaken by Chemours pursuant to the Consent Order Compliance Measures for the period from July 1, 2020 through the end of the third quarter of 2020 (September 30, 2020). On August 13, 2020, Chemours signed the Addendum to Consent Order Paragraph 12, and the Addendum was entered by the Bladen County Superior Court on October 12, 2020.

Section 7 Control Technology Improvements

The thermal oxidizer (see photo at right) continues to control process emissions at an average PFAS destruction efficiency exceeding 99.99%.

Section 10 No Discharge of Process Wastewater from Chemours' Manufacturing Areas

Chemours continues to not discharge its process wastewater and to ship its process wastewater offsite for disposal. Chemours is recycling treated water internally within several manufacturing processes.

Section 11 Characterization of PFAS in Process and Non-Process Wastewater and Stormwater at the Facility

During the third quarter of 2020, Chemours' consultant Geosyntec prepared the 2020 second quarter report describing and analyzing characterization sampling of process water, non-process wastewater and stormwater that occurred in April and June 2020. Chemours submitted the report to NCDEQ on July 31, 2020. During the third quarter, sampling occurred on August 26, 2020. Geosyntec is preparing the 2020 third quarter report for submission under separate cover by December 19, 2020 to include data from this sampling event. This report will be the final quarterly report under Paragraph 11(c) for the initial characterization period, with ongoing sampling to continue pursuant to Paragraph 11(d). Paragraph 11(c) requires the final quarterly report to be submitted 18 months after the sampling workplan was approved. December 19, 2020 is 18 months after June 19, 2019, when the sampling workplan was approved by NCDEQ.

Section 11.2 Characterization of PFAS Contamination in River Sediment

During the third quarter of 2020, Chemours' contractors began preparing the Sediment Characterization Report. The report was submitted to NCDEQ, Cape Fear River Watch, and downstream water utilities on October 23, 2020.

Section 12 Accelerated Reduction of PFAS Contamination in the Cape Fear River and Downstream Water Intakes, and Addendum to Consent Order Paragraph 12

On September 30, 2020, Chemours started operation of the treatment system for the Old Outfall pursuant to Consent Order Paragraph 12(e) and a NPDES permit issued by NCDEQ.

As noted above, during the third quarter of 2020, Chemours signed the Addendum to Consent Order Paragraph 12. Chemours' Addendum implementation activities during the third quarter included:

Consent Order Addendum Paragraph 1

On August 31, 2020, Chemours submitted to DEQ three items pursuant to Consent Order Addendum Paragraph 1:

- Cape Fear River PFAS Mass Loading Calculation Protocol (01(a));
- The files used to prepare the most recent version of the Mass Loading Model (01(b)); and
- Site Conveyance Network and Outfall 002 PFAS Mass Loading Calculation Protocol (01(c)(ii)).

On August 31, 2020, Chemours also began collecting the following samples:

- Twice weekly, 24-hour composite samples from the Cape Fear River at the Tar Heel Ferry Road bridge, including samples up to twice per month when more than 1.5" of rainfall occurred, and recording flows at W.O. Huske Dam (01(a)(i)(1-3)); and
- Once weekly, 24-hour composite samples from Outfall 002, including samples when more than 1" of rain occurred (01(c)(i)).

On September 30th, Chemours submitted to DEQ the Cape Fear River PFAS Mass Loading Assessment – Second Quarter 2020 Report pursuant to Consent Order Addendum paragraph 01(b). This submission was also pursuant to quarterly reporting of mass loading sampling outlined in the Corrective Action Plan (Paragraph 16).

Consent Order Addendum Paragraph 2

On August 31st, Chemours submitted to DEQ the Interim Seep Remediation System Plan. In addition to submitting this plan, Chemours' contractors advanced the design and required permit applications for installing interim seep remediation systems.

Consent Order Addendum Paragraph 4

On July 1st, Chemours submitted to DEQ the Stormwater Pollution Prevention Plan.

On September 30th, Chemours submitted to DEQ the Stormwater Treatment System Sampling Plan. Chemours' internal engineering team and Chemours' contractors also developed a plan during the third quarter of 2020 to separate stormwater and non-contact cooling water flows in order to facilitate the future capture and treatment of stormwater in the Monomers/IXM area during storms producing rainfall volumes and intensities up to 1" within a 24-hour period.

Section 14 Toxicity Studies

Chemours has all five Consent Order Attachment B substances synthesized. Four of the five test substances have been delivered to the contract laboratory and the fifth is going through final purification.

Mammalian Toxicology

There is little or no mammalian toxicology data available for these substances, therefore it was necessary to perform range finder studies prior to beginning the Consent Order studies. In these studies, rats and mice are dosed for two weeks and a number of toxicologically important endpoints are measured. This data is necessary to set appropriate dose levels for the Consent Order studies. As of September 30th, eight range finder studies (one rat study and one mouse study for each of the four available test substances) have begun. When the final test substance is available, the range finder studies for that substance can begin. Concurrently, the method for dose analysis is being developed. This method will be submitted for approval by NCDEQ prior to the start of the 28-day Consent Order studies. The same method will be used for both the 28-day and 90-day studies. The protocols for the 28-day immunotoxicity studies are being finalized while Chemours is waiting for the range finder data. The dose levels for the 90-day study depend on results obtained in the 28-day study; therefore the current focus is on the 28-day study protocols.

Aquatic Toxicology

Protocols for the aquatic toxicity studies are being developed by the contract laboratory and will be submitted to NCDEQ when available. Analytical method development for the analysis method is beginning in October 2020; the final method will be submitted for approval by NCDEQ prior to the beginning of the aquatic toxicology work.

Section 16 Groundwater Remediation

Chemours conducted a baseline mass loading monitoring event described in the Corrective Action Plan (CAP) by collecting groundwater, surface water, and river samples and measuring flows in surface water bodies at and around the Site in July 2020. These results will be described in the third quarterly report for this program to be submitted by December 29, 2020 pursuant to Addendum to Consent Order Paragraph 12, Paragraph 1(b).

During the third quarter, Chemours also collected groundwater samples from sitewide monitoring wells and from Chemours' installed offsite groundwater monitoring wells. These samples are presently undergoing laboratory analysis and are planned to be reported to NCDEQ during the fourth quarter of 2020.

On July 31st, Chemours submitted to DEQ the Cape Fear River Table 3+ PFAS Mass Loading Assessment – First Quarter 2020 Report as outlined in the CAP for quarterly reporting of mass loading sampling.

On September 30th, Chemours submitted to DEQ the Cape Fear River PFAS Mass Loading Assessment – Second Quarter 2020 Report as outlined in the CAP for quarterly reporting of mass loading sampling. This submission was also pursuant to reporting requirements outlined in Consent Order Addendum Paragraph 1(b).

Sections 19 and 20 Provision of Public Water Supplies, Whole Building Filtration Systems, and Reverse Osmosis Drinking Water Systems

As shown in the summary tables above, Chemours continues to make significant progress in implementing the Consent Order requirements of Paragraphs 19 and 20. Since resuming RO installations in June 2020, following the COVID-19 postponement period, the pace of RO acceptance

rates and installations has been on the rise. O&M activities for installed GAC systems continues uninterrupted. Bottled water delivery also continues uninterrupted, with three vendors serving 2,486 homes.

Section 21 Private Well Testing

The Adaptive Step Out and Infill Sampling Program has been ongoing since the 3rd quarter of 2019 (other than during the COVID-19 postponement period between March and May of 2020). Three to eight stages of step out sampling have occurred across the sectors and the current step out distance intervals range from 5.5-6.5 miles to 13.5-14.5 miles from the Site. Results for some of the current stage of Step Out sampling are still pending. Four of the sectors, 3, 6, 8, and 9, have been delineated (i.e., no further step outs for these sectors are needed) and an additional four, 5, 7, 14 and 15, are near delineated (results pending). Distance intervals for current infill sampling for all sectors range from 2.5-7.5 miles to 5.5-14.5 miles from the Site.

Section 22 Provision of Sampling Results

Chemours provided (and continues to provide) sampling results to NCDEQ and residents as required under the Consent Order. Chemours has provided sampling results to NCDEQ by sending a courtesy email notification and by uploading sampling results to the state Equis database. Chemours has also provided final lab reports to NCDEQ. Chemours has provided sampling results to residents by including preliminary results with water filtration system initial offer letters and sending the final lab reports to residents within the following 30 days. Chemours has also provided non-detect sampling results to residents.

Section 23 Interim Replacement of Private Drinking Water Supplies

All residents eligible to receive the interim replacement drinking water supplies have received the supplies (i.e., bottled water or voucher card for bottled water). As of September 30, 2020, there are 2,486 residences receiving bottled water.

Section 26 Total Organic Fluorine

Please see Appendix A for the quarterly progress report from Dr. Susan D. Richardson.

Section 28 Reporting

Chemours submitted the Consent Order 2nd quarter 2020 progress report on July 21, 2020.

Sections 29 and 30 Public Information

Chemours has continued to post its Consent Order submissions at https://www.chemours.com/Fayetteville-Works/en-us/c3-dimer-acid/compliance-testing/.

Appendix A

4th Progress Report
Development of a Total Organic Fluorine (TOF) Method for the Analysis of Process Wastewater Streams and Air from Fayetteville Works (NC)
Susan D. Richardson, Danielle C. Westerman, Alexandria L. Forster, and Ying Zhang
University of South Carolina
September 29, 2020

Since the last report on June 22, 2020 there have been several positive updates to our lab's instrumentation capabilities and working conditions. Because of the Covid-19 virus, the University of South Carolina limited research hours in phases up until August 1, but since August 1, the University has allowed a full return to research without limitations. During the week of August 17-21, a Thermo Fisher Scientific field engineer was able to come install a new ion chromatography (IC) instrument, the Dionex Integrion HPIC System, and this engineer conducted familiarization training for this instrument. During this training, it came to our attention that the analytical/guard column set that was sent to our lab was not the correct column for our needed applications, so a new set of columns were ordered, but did not arrive until September 15.

1. Ion Chromatography Updates

1.1. IC Capability Improvements

The previous IC used for this TOF research project was the Dionex ICS-1600, which utilizes a carbonate eluent. Due to the high conductivity baseline with this eluent and the water dip (Figure 1), quantifying fluoride was problematic. After repair of a leak on the ICS-1600 system, the fluoride peak moved closer to the water dip, which could hinder accurate quantification of fluoride. As a result, we purchased the new Integrion IC system, which uses sodium hydroxide as the eluent and eliminates the water dip (Figure 2) and lowers the background, allowing improved detection and quantification of fluoride.

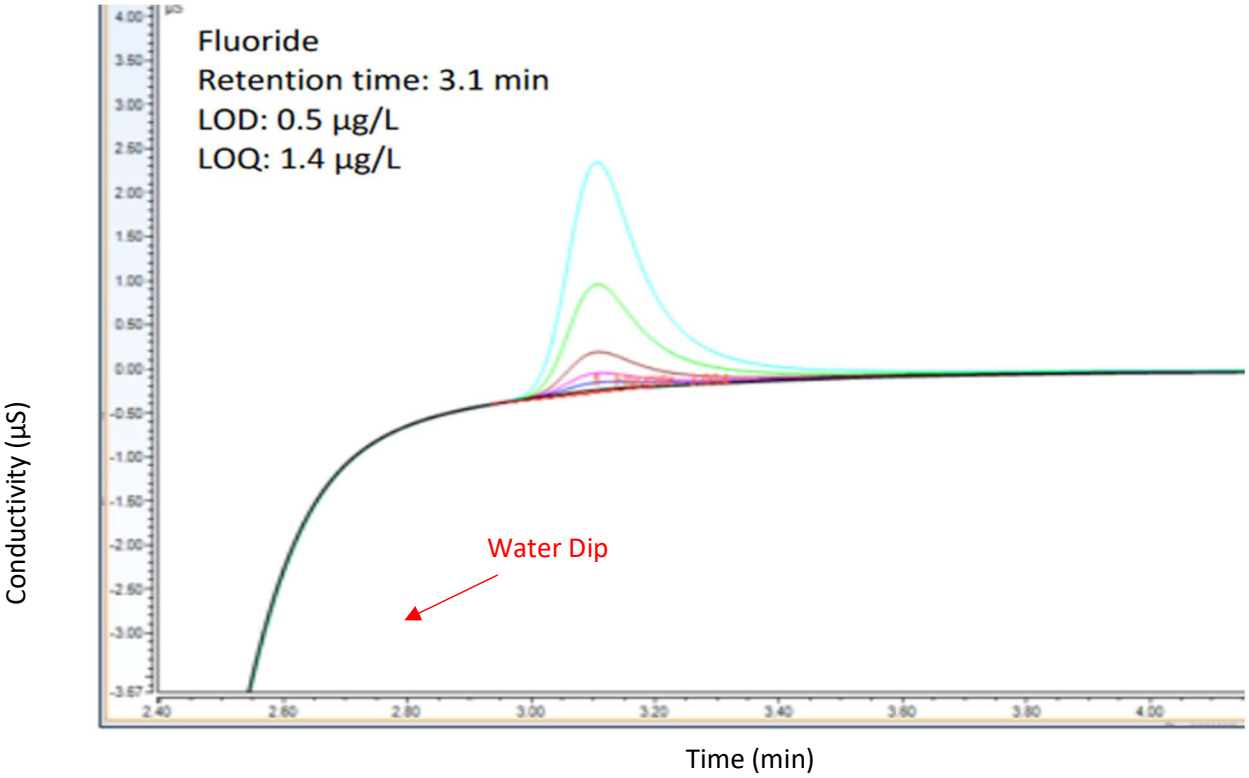

Figure 1. Elution of fluoride by IC (IonPac AS9-HC carbonate eluent column).

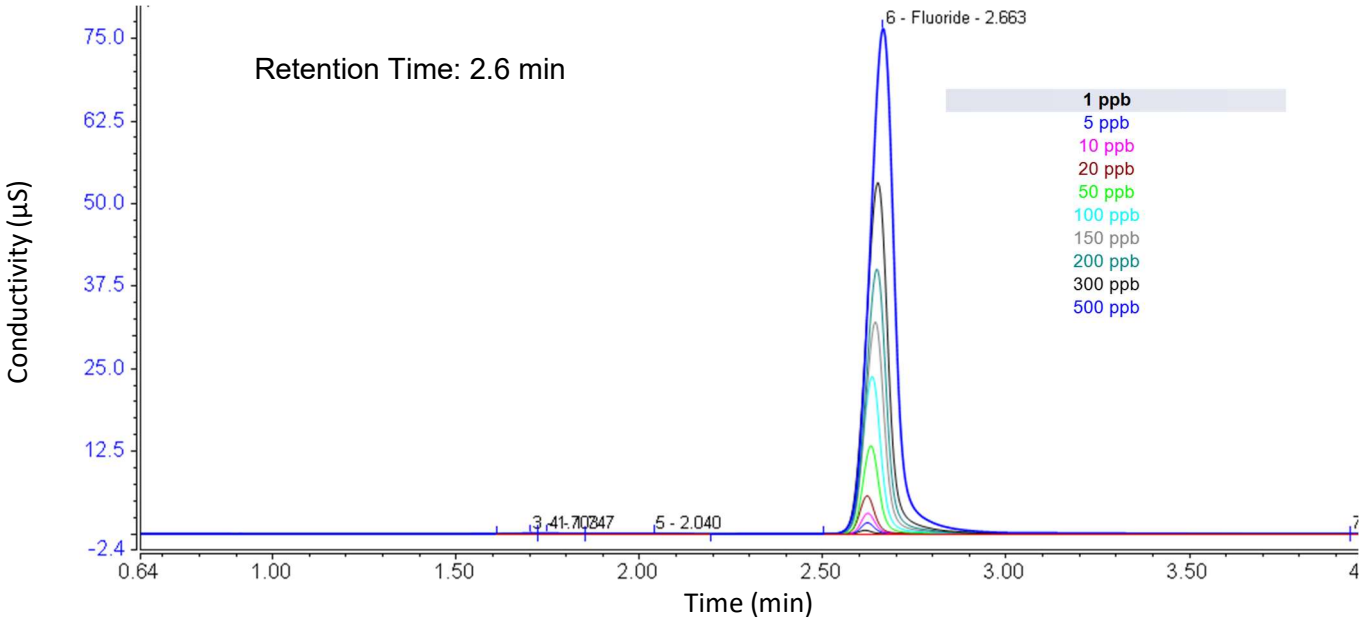

Figure 2. Elution of fluoride by IC (Integrion HPIC AS28 hydroxide eluent column) using a sodium fluoride standard to build a calibration curve for fluoride quantification.

1.2. IC Column Optimization

Experiments for percent recovery of total organic fluorine for several per-and polyfluoroalkyl substances (PFAS) were conducted using an AS16, 4 µm analytical and guard column pair with 35 mM NaOH eluent concentration. The chromatograms from these experiments all showed fluoride peaks coeluting with another anion (Figure 3), but the coeluting peak did not appear in the blanks. Considering several factors, such as the similar affinities of fluoride and acetate with the solid phase of the AS-16 columns, as well as the chemical structures of the standards tested, the coeluting peak could potentially be acetate. Even with a gradient of the hydroxide eluent, the two peaks were unsuccessfully separated. In order to better detect fluoride, the Thermo AS28 4µm analytical and guard columns were installed. The fluoride peaks for recovery experiments have not presented any coeluting peaks thus far.

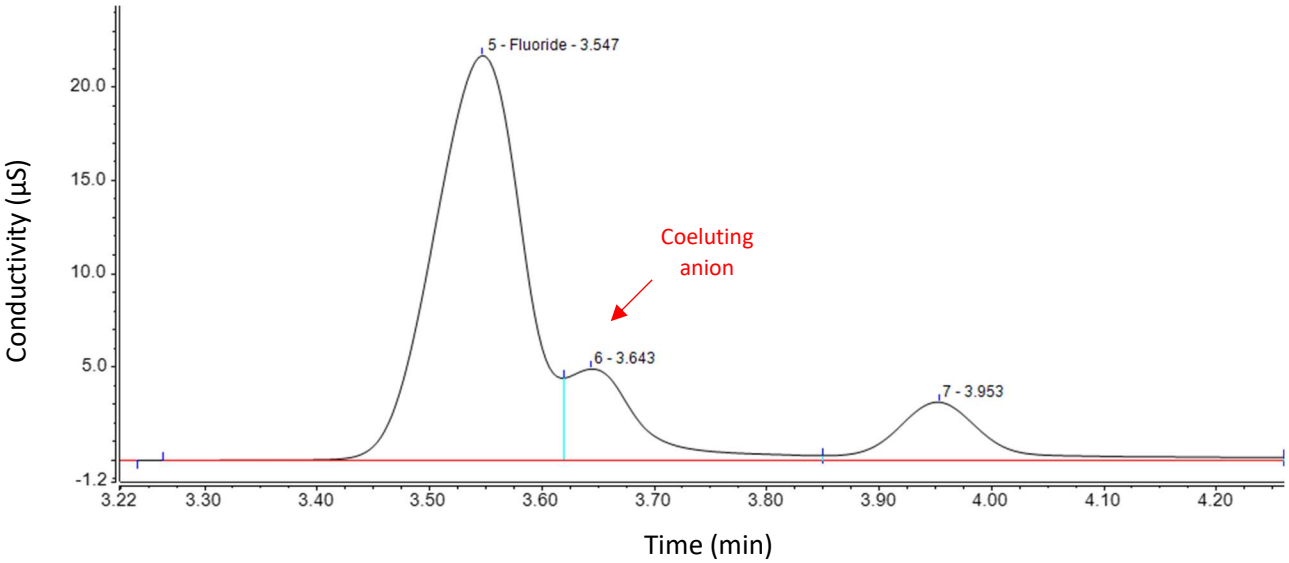

Figure 3. Chromatogram of a fluoride peak resulting from 50 ppb PFOA standard tested for percent recovery of absorbable organic fluorine (AOF) with the Dionex Integrion (AS16 35 mM hydroxide eluent).

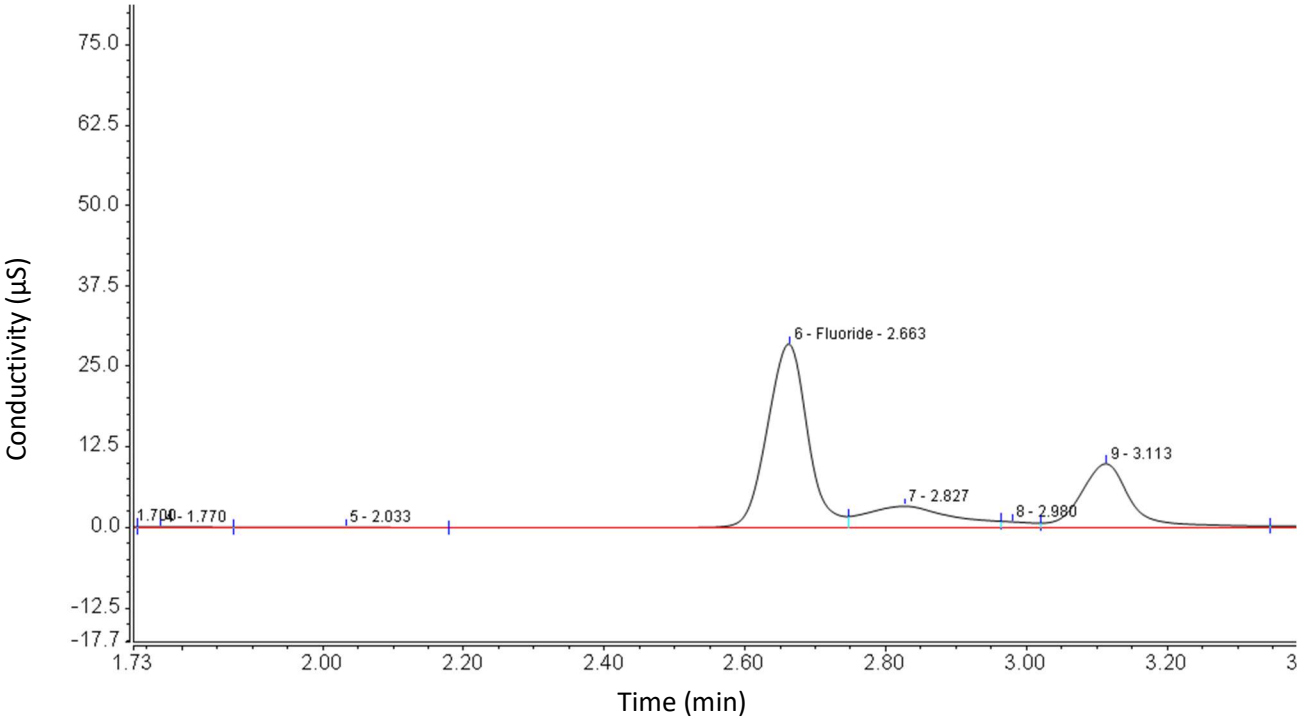

Figure 4. Chromatogram of the fluoride peak resulting from 50 ppb PFOA standard tested for percent recovery of AOF with the Dionex Integrion (AS16 55mM hydroxide eluent).

2. Absorbable Organic Fluorine Optimization

2.1. Absorption Solution Optimization

After the process of combustion, off gasses are bubbled into an aqueous solution referred to as the absorption solution. This solution should have the ability to efficiently cleanse the off-gasses of fluoride, should be compatible with the IC analysis (i.e. does not interfere with the fluoride peak), and potentially contain a reducing agent in case any halides are oxidized to halogens. Currently experiments with varying compositions and concentrations of absorbing solutions are being conducted. At the time of this report, three different buffer solutions (carbonate, phosphate, and ammonium hydroxide) are being tested at 1 mmol with and without hydrogen peroxide as a reducing agent. A standard mix of 10 µL of 100 µg/L as F^- of

three PFAS standards was injected into the ceramic boats containing quartz wool. Future work will include varying the concentration of these buffers and hydrogen peroxide to be used, as well as testing 18MΩ water with and without hydrogen peroxide. Considering the weak bond between two fluorine atoms compared to the strong polar bond in HF, 18MΩ water alone might be sufficient as an absorption solution. Results of this experiment will be included in the next monthly meeting with Chemours on October 8 since they are currently being analyzed by IC.

2.2. Activated Carbon Columns Breakthrough Test

While testing the recovery for several PFAS standards, the two carbons used in line per sample were placed into separate ceramic boats and the off-gas from combustion was collected in separate vials for IC analysis in order to understand the organic fluorine breakthrough potential into the second carbon. This test is to ensure that organic fluorine is not being rinsed through both carbons and out to waste, which would therefore lower percent recoveries. Standard consensus requirements for breakthrough is that the total organic halogen in the second carbon should be less than 10% of the sum of the two carbons in line. For this set of experiments, only two carbons were placed in line and 50 mL of sample was passed through each column, followed by 10 mL of potassium nitrate rinsing solution (Figure 5). The pH of these samples was adjusted to a pH <1 with nitric acid before rinsing through the columns as suggested by previous work.

Table 1. Recoveries and breakthrough of three PFAS standards*

Compound	AOF Percent Recovery (µg/L)	Carbon 2 Breakthrough Percent
Gen X	66%	8.2%
PFOS	72%	10.3%
PFOA	65%	12.9%

*Experiments were done in triplicate and blank subtracted. The pH was lowered to a pH <1 before passing water over carbons.

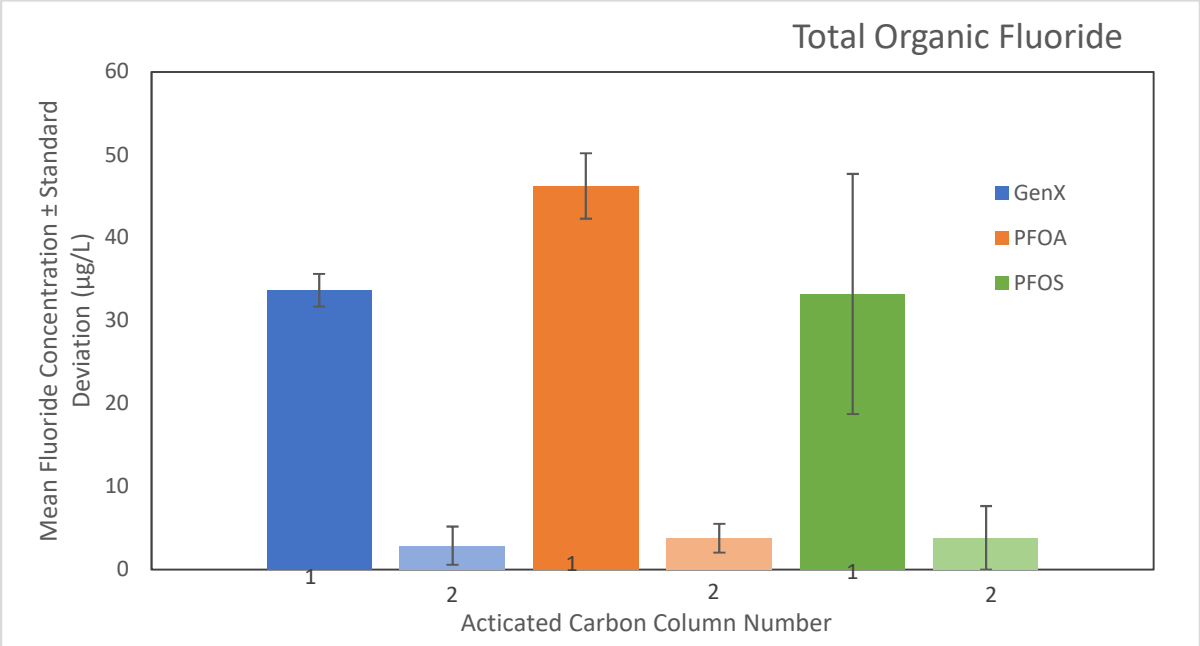

Figure 5. Fluoride breakthrough analysis of two activated carbons in series from three PFAS compounds.

3. Upcoming Absorbable Organic Fluorine Work

Work will continue to be done towards optimizing the percent recoveries for total AOF. Some of these parameters include a further in-depth analysis of the sample pre-treatment including effect the pH of the samples has on fluorine recovery, which acid should be used to drop the pH of samples, and optimal sample holding time and temperature. Experiments will be done to test the composition and pH of the solution rinse that removes inorganics from the carbon columns. Currently, potassium nitrate is used. Several combustion factors, such as carrier gas flow rate, combustion temperature, and time, will also be investigated.

4. Liquid Chromatography-High Resolution Mass Spectrometry Experiments

A mix of 26 standards (some purchased, some provided by Chemours) was analyzed at an initial concentration of 500 ppb in 50:50 MeOH:water, utilizing an ultrahigh performance liquid chromatograph coupled to an Agilent 6545 quadrupole time-of-flight (UHPLC-QTOF) mass spectrometer with electrospray ionization in negative ion mode (ESI-). A full scan-MS analysis was utilized for quantification due to the increased sensitivity of the QTOF instrument in MS mode relative to product ion-MS/MS. The mass spectrometer was operated at a fragmentation voltage of 110 V, capillary voltage 4000 V, gas temperature 300 °C, drying gas 12 L min^{-1}, and nebulizer pressure of 35 psi. The scan range was from m/z 50 to 1300. UHPLC parameters are shown in Table 2.

Table 2. UHPLC Parameters

Parameter	Value	
Instrument	1290 Infinity II UHPLC Binary Pump	
Mobile Phase	A) 0.1% acetic acid in water B) 0.1% acetic acid in methanol	
Gradient	Time (min)	%B
	0	5
	14	95
	15	100
	18	100
	18.1	5
	25	5
Flow rate	0.4 mL/min	
Column	Agilent Zorbax RRHD Stable Bond C18 (50 x 2.1 mm, 1.8 µm)	
Temperature	50 °C	
Injection Volume	10 µL	

Once a full scan spectrum is extracted for a detected standard, a mass match score (Table 3) is then calculated based on the accurate mass, isotope distribution and isotope spacing. The score is defined as weighted aggregate of the three individual metrics, therefore, it is based on not only the mass accuracy, but also the isotopic fidelity. Mass spectra with a mass difference of ±5 ppm and a score greater than 75 were considered of interest and reported below.

Table 3. UHPLC-High Resolution-MS Analysis of PFAS Standards

Name	Formula	Observed m/z	Calculated Mass	Theoretical Mass	Diff (ppm)	Score	RT (min)
PFDA	C10 H F19 O2	512.9600	513.9673	513.9673	0.02	99.4	11.55
PFUdA	C11 H F21 O2	562.9577	563.9651	563.9641	1.65	98.2	12.17
PFDoA	C12 H F23 O2	612.9546	613.9618	613.9609	1.35	98.5	12.73
PFTeDA	C14 H F27 O2	712.9475	713.9554	713.9545	1.23	98.9	5.48
PFPrA	C3 H F5 O2	162.9824	163.9897	163.9897	0.34	99.9	11.11
PFBA	C4 H F7 O2	212.9792	213.9865	213.9865	0.09	99.7	9.25
PMPA	C4 H F7 O3	228.9739	229.9811	229.9814	-1.28	87.3	3.09
PFBS	C4 H F9 O3 S	298.9430	299.9502	299.9503	-0.20	99.5	5.87
NVHOS	C4 H2 F8 O4 S	296.9473	297.9540	297.9546	-2.19	99.5	4.26
PFPeA	C5 H F9 O2	262.9756	263.9831	263.9833	-0.69	94.9	5.48
PEPA	C5 H F9 O3	278.9706	279.9779	279.9782	-1.19	87.3	6.10
PFO3OA	C5 H F9 O5	310.9606	311.9680	311.9680	0.02	86.2	7.83
PFHxA	C6 H F11 O2	312.9727	313.9799	313.9801	-0.46	99.8	7.58
GenX	C6 H F11 O3	328.9679	329.9754	329.975	1.25	94.7	10.81
PFHxS	C6 H F13 O3 S	398.9369	399.9440	399.9439	0.39	99.5	9.06
R-PSDCA	C6 H2 F12 O4 S	396.9408	397.9480	397.9482	-0.57	77.2	8.85
PFHpA	C7 H F13 O2	422.9911	363.9772	363.9769	0.86	99.0	7.39
R-PSDA	C7 H2 F12 O6 S	440.9310	441.9383	441.9381	0.48	99.4	3.34
Hydrolized PSDA	C7 H3 F11 O7 S	438.9353	439.9426	439.9424	0.53	99.6	4.21
EVE Acid	C8 H F13 O4	406.9597	407.9670	407.9667	0.72	99.5	9.66
PFOA	C8 H F15 O2	412.9665	413.9738	413.9737	0.15	98.9	9.97
PFOS	C8 H F17 O3 S	498.9312	499.9383	499.9375	1.70	98.1	10.39
R-EVE	C8 H2 F12 O5	404.9637	405.9710	405.9711	-0.12	99.7	3.11
Hydro-EVE Acid	C8 H2 F14 O4	426.9653	427.9727	427.9730	-0.65	97.9	9.12
PFNA	C9 H F17 O2	508.9690	463.9708	463.9705	0.66	99.3	9.13
HFPO-TA	C9 H F17 O4	540.9591	495.9609	495.9603	1.13	98.7	9.49

All standards were detected with high mass accuracy, with all but 4 compounds (PMPA, PEPA, PFO3OA, and Byproduct 6) having a mass match score greater than 94. Compounds PEPA and PFO3OA were found to have the lowest sensitivity in this method, which may explain the relatively low mass accuracy (87.4, and 77.2, respectively). Especially with respect to these two compounds, further method optimization is required before quantifying all compounds via UHPLC-QTOF.