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PFAS NON-TARGETED ANALYSIS AND METHODS INTERIM REPORT #12

Process and Non-Process Wastewater and Stormwater

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ACRONYMS AND ABBREVIATIONS

Chemours	The Chemours Company FC, LLC
CPWW	Chemours Process Wastewater
DAE Acid	Diadduct Ester Acid
Facility	Chemours Fayetteville Works, North Carolina
GFD	General Facility Discharge
LC	liquid chromatography
MS/MS	tandem mass spectrometry
ng/L	nanograms per liter
Orbitrap	Thermo Scientific Orbitrap Exploris 240 mass spectrometer
PFAS	per- and polyfluoroalkyl substances
PFO ₆ TeA	pentadecafluoro-2,4,6,8,10,12-hexaoxatetradecan-14-oic acid
RHDA	RSU/HFPO Diadduct
QToF	quadrupole time-of-flight

1 INTRODUCTION

This interim report has been prepared by The Chemours Company FC, LLC (Chemours) to provide an update on the characterization of previously unidentified per- and polyfluoroalkyl substances (PFAS) in aqueous samples collected from process wastewater, non-process wastewater (i.e., non-contact cooling water) and stormwater at the Chemours Fayetteville Works, North Carolina site (the Facility). This work is being conducted pursuant to Paragraph 11 subpart (a) in the Consent Order executed 25 February 2019 between Chemours and the North Carolina Department of Environmental Quality with the Cape Fear River Watch as intervenor. The overall purpose of this program is to identify previously unknown PFAS that may be present in samples of collected water and to develop standards and methods to facilitate the quantitative analysis of these PFAS, as described in the PFAS Non-Targeted Analysis and Methods Development Plan, Version 2 (Chemours and Geosyntec, 2019). This is the 12th interim report. Further details are provided in the references.

The samples assessed via the non-targeted program were divided into two categories:

- General Facility Discharge Samples - samples of stormwater, treated non-Chemours process wastewater and/or non-contact cooling water discharging to the Cape Fear River. These samples were collected at five locations. Samples were collected in 2019; and
- Chemours Process Wastewater Samples - samples of process wastewater from Chemours manufacturing areas. These samples were collected at two locations. Samples were collected in 2019 and 2020. Chemours process wastewater has not been discharged to the Cape Fear River since November 2017.

Samples were analyzed by liquid chromatography (LC) coupled to high-resolution quadrupole time-of-flight (QToF) mass spectrometry (Chemours, 2020a). Potential unknown PFAS were assigned a tentative empirical formula (defined as the number of atoms present in a compound but not the arrangement of the atoms) from unidentified chromatographic peaks with a signal-to-noise level greater than six and using the atomic mass defect of fluorine as the molecular feature.

The next steps are to develop tentative molecular structures and develop authentic standards (i.e., synthesize samples of the compounds to facilitate traditional targeted analysis). These steps are time-consuming; consequently, the unknown PFAS in each group (General Facility Discharge and Chemours Process Wastewater) were organized in decreasing order of abundance (by peak height) and the most abundant unknown PFAS were prioritized.

Once an authentic standard is available, the following steps are taken:

- The addition of the PFAS to an existing analytical method (e.g., Method 537M) will be assessed:
 - If the PFAS can be added to an existing method, a method detection limit study will be conducted; and

- If the PFAS cannot be added to an existing method, new method development will be evaluated.
- A matrix interference study will be conducted so that the ability to reliably quantify the PFAS in environmentally relevant matrices can be assessed.
- The PFAS will be analyzed in samples of groundwater adjacent to the Cape Fear River or of the Cape Fear River itself to see if it is detectable.

Once PFAS that have been identified are no longer detectable in samples of groundwater adjacent to the Cape Fear River or of the Cape Fear River itself, the non-targeted program will be considered complete as no further PFAS detections are expected to occur.

The remainder of this 12th interim report consists of:

- Section 2: General Facility Discharge Samples;
- Section 3: Chemours Process Wastewater Samples;
- Section 4: Summary; and
- Section 5: References.

2 GENERAL FACILITY DISCHARGE SAMPLES

Of the 19 potentially unknown PFAS present in General Facility Discharge (GFD) samples, the 10 most abundant (GFD-1 through GFD-10) have been fully assessed. Nine were shown to not be PFAS or to be previously known PFAS. The tenth (GFD-6), pentadecafluoro-2,4,6,8,10,12-hexaaxatetradecan-14-oic acid (PFO₆TeA) will be analyzed and reported under future Paragraph 11 required sampling.

GFD-11 Through GFD-15

Fragmentation of GFD-11 through GFD-15 during the initial analysis showed very few detectable fragments for each analyte, making the proposal of structures difficult. Re-analysis of GFD-11 through -15 was conducted using a Thermo Scientific Orbitrap Exploris 240 mass spectrometer (Orbitrap), which provides a higher mass resolution than that provided by the QToF mass spectrometer used in the initial analysis. It was hoped that the higher resolution might allow the identification of additional ionization fragments from GFD-11 through -15 to aid in proposing molecular structures. However, the results from the Orbitrap did not add significant information that could be employed in proposing molecular structures for GFD-11 through GFD-15.

In the previous interim report (June 2025), next steps for GFD-11 through -15 (the next five potentially unknown PFAS present in General Facility Discharge samples) were stated to be:

- Compare the area counts of target PFAS to the area counts of unknown PFAS in the Orbitrap analysis in the General Facility Discharge samples to gain an understanding of the remaining mass of unknown potential PFAS (GFD-11 through -15) relative to the mass of known PFAS in the General Facility Discharge samples.

Results achieved since June 2025 are:

- The total area of peaks that could be identified (i.e., by comparison to known standards) and the total area of peaks that could not be identified (i.e., GFD-11 through -15) were compared.
- About 96% of the total PFAS mass could be identified.

Chemours submits that the non-targeted assessment of GFD samples is now complete:

- Potential unidentified PFAS in GFD samples were assessed in descending order of peak height.
- As previously described, once potential unidentified PFAS that have been identified are no longer detectable in samples of groundwater adjacent to the Cape Fear River or of the Cape Fear River itself, the non-targeted program will be considered complete as PFAS detections of less abundant PFAS are not expected to occur in the environment beyond the Facility boundaries. The least abundant peak that has been identified, GFD-6 (PFO₆TeA), was not detected in groundwater samples from 2 flood plain wells and 2 Black Creek Aquifer wells

adjacent to the Cape Fear River, nor was it detected in the Cape Fear River a few miles downstream at Bladen Bluffs.

- About 96% of the total PFAS mass from the GFD samples has been identified. Additionally, based upon the analysis performed by both the QToF and the Orbitrap, peak sizes for GFD-11 through -15 are very small, and no further identifiable fragments are expected. It is therefore unlikely that molecular structures can be proposed for GFD-11 through -15.

3 CHEMOURS PROCESS WASTEWATER SAMPLES

The 5 most abundant (CPWW-1 through CPWW-5) PFAS in Chemours Process Wastewater (CPWW) have been assessed. One (CPWW-2) was identified as EVE Acid, which is a known PFAS associated with the Facility, and does not need to be further investigated. Progress on CPWW-1, CPWW-3, CPWW-4, CPWW-5 and CPWW-6 through CPWW-10 is described below.

CPWW-1 (RHDA)

CPWW-1 has been identified as RSU/HFPO Diadduct (RHDA) by comparison to a standard purified from production samples. RHDA was detected in samples collected from 6 groundwater wells – 2 wells on-site and 4 wells adjacent to the Cape Fear River - and from the Cape Fear River. However, RHDA is a diprotic PFAS and, in a matrix interference study using Cape Fear River water from River Mile 84, RHDA exhibited the significant over-recovery that has been observed with other diprotic PFAS with EPA Method 537M. Consequently, there is currently no analytical method that can accurately quantify RHDA.

In the previous interim report (June 2025), the next steps for RHDA were stated to be:

- Add RHDA to the suite of diprotic PFAS that is under investigation for improved analytical methodology due to matrix interference-induced over-recovery.

Results achieved since June 2025 are:

- A method meant to analyze diprotic and short-chain PFAS (the DSC Method) began development at Eurofins-Sacramento.
- Initial work with RHDA by the preliminary DSC Method showed that there was still some matrix enhancement present.
- The DSC Method continues to be developed and now shows that the matrix enhancement of RHDA has been resolved.

Next steps for RHDA will be:

- The DSC Method will be further assessed to see if it can be developed into a formal analytical method.
- This will include steps such as method detection limit studies and the assessment of other PFAS to see how broad the method's application may be.

CPWW-3

A chemical formula of $C_8H_5F_{13}O_6S$ was initially proposed for CPWW-3. The molecular structure of $HO_3SCF_2CF_2OCF(CF_3)CF_2OCHF_2OCH$ was proposed for this chemical formula. This molecular structure was synthesized, but the tandem mass spectrometry (MS/MS) spectrum of the synthesized standard did not match the MS/MS spectrum of CPWW-3. Subsequent assessment of

CPWW-3 with the Orbitrap revised the proposed empirical formula to $C_8H_2F_{14}O_7$. These results illustrate an intrinsic difficulty that may be encountered during non-targeted analysis, namely, that a proposed structure, once synthesized, does not match the non-targeted analyte when analyzed by MS/MS. At this point, the process of proposing an empirical formula and/or a structure must be repeated.

In the previous interim report (June 2025), next steps for CPWW-3 were stated to be:

- Develop a proposed molecular structure for the revised empirical formula, with subsequent steps of developing a synthetic pathway to the structure and synthesizing an authentic standard so the potential presence of CPWW-3 in samples of groundwater adjacent to the Cape Fear River or of the Cape Fear River itself can be assessed.

Results achieved since June 2025 are:

- CPWW-3 was assessed using the Orbitrap mass spectrometer
- The higher resolution fragmentation pattern obtained with the Orbitrap was sufficient to propose a revised structure for CPWW-3.
- Synthesis of the proposed revised structure is not straightforward; there is not a clear synthetic pathway to the structure.

During the initial assessment, CPWW-3 was found in process water samples from Location 16 that were collected in 2019 and 2020. Four new samples were collected from Location 16 (August 11, 2025, August 28, 2025, October 1, 2025, and October 3, 2025). Analysis of these samples showed that CPWW-3 was either not present or present in much lower relative amounts than in 2019 and 2020. Given the difficulty in creating a synthetic pathway and the decreased presence in recent samples from Location 16, further work on CPWW-3 will be paused.

CPWW-4 (DAE Acid)

A structure of $HOCCF(CF_3)OCF_2CF(CF_3)OCF_2CF_2COOH$ has been assigned to CPWW-4 ($C_9H_2F_{14}O_6$) via the synthesis of an authentic standard and comparison of the tandem mass spectrometry (MS/MS) spectrum of the standard to that of CPWW-4. CPWW-4 was named Diadduct Ester Acid (DAE Acid).

In the previous interim report (June 2025), next steps for CPWW-4 were stated to be:

- Prepare standard solutions of DAE Acid to send to Eurofins-Sacramento for an assessment of the ability of Method 537Mod to analyze DAE Acid.
- If DAE Acid can be analyzed by Method 537Mod, a matrix interference study will be conducted so that the ability to reliably quantify DAE Acid in environmentally relevant matrices can be assessed.

Results achieved since June 2025 are:

- A standard solution of DAE Acid was prepared by Chemours and sent to Eurofins-Sacramento.
- DAE Acid is a diprotic PFAS and therefore is expected to exhibit significant matrix enhancement by Method 537M as with other diprotic PFAS.
- Since the DSC Method development was already underway (see above for RHDA), DAE Acid was added to this method for assessment, rather than assessing it by Method 537M.
- During the refinement of the DSC Method (described above for RHDA), DAE Acid was added to the method to assess its matrix enhancement.
- As with RHDA, the refined DSC Method showed that there was no matrix enhancement of DAE Acid.

Next steps for DAE Acid will be:

- The DSC Method will be further assessed to see if it can be developed into a formal analytical method.
- This will include steps such as method detection limit studies and the assessment of other PFAS to see how broad the method's application may be.

CPWW-5

A chemical formula of $C_6HF_{11}O_4$ and a molecular structure of $CF_3-O-CF_2-O-CF_2-CF_2-CF_2-COOH$ has been proposed for CPWW-5. By the previous interim report (June 2025), synthesis of an authentic standard corresponding to the proposed molecular structure has begun.

In the previous interim report, next steps for CPWW-5 were stated to be:

- Completion of the synthesis of the authentic standard.
- Comparison of the MS/MS spectrum of the authentic standard to the MS/MS spectrum of CPWW-5.

Results achieved since June 2025 are:

- Synthesis of the authentic standard was progressed but was not completed.

Next steps for CPWW-5 will be as they were in the previous interim report:

- Complete synthesis of the authentic standard.
- Compare the MS/MS spectrum of the authentic standard to the MS/MS spectrum of CPWW-5.

CPWW-6 Through CPWW-10

Previously, a chemical formula of $C_3H_2F_6O_4S$ and a molecular structure of $CF_3O-CFH-CF_2-SO_3H$; had been proposed for CPWW-7. The proposed structure was synthesized by Chemours but the retention time and MS/MS spectrum of CPWW-7 were compared to those of the synthesized compound and were found not to match. As with CPWW-3, this demonstrates a difficulty that may be encountered during non-targeted analysis when a proposed structure, once synthesized, does not match the non-targeted analyte when analyzed by MS/MS, requiring a new empirical formula and/or structure to be proposed.

In the previous interim report (June 2025), next steps for CPWW-6 through 10 were stated to be:

- Continued development of proposed molecular structures for CPWW-6, -7, -8, -9 and -10.

Results achieved since June 2025 are:

- The analytes were assessed using the Orbitrap mass spectrometer.
- This resulted in a new proposed structure for CPWW-7 ($CF_3-CFH-O-CF_2-SO_3H$).
- Additionally, an initial proposed structure for CPWW-10 is under development.
- Fragmentation of CPWW-6, -8 and -9 by the Orbitrap (as well as by the QToF) resulted in very few detectable fragments for each analyte, making the proposal of a structure difficult.

Next steps for CPWW-6 through CPWW-10 will be:

- A synthetic pathway will be developed for CPWW-7.
 - o Note that synthesis of the proposed revised structure is not straightforward; there is not a clear synthetic pathway to the structure.
- The initial proposed structure for CPWW-10 will be refined.
- Development of proposed molecular structures for CPWW-6, -8 and -9 will be continued.

Additional Progress

In the first interim report (Chemours 2020a), unidentified potential PFAS with their empirical formulas were listed in order of ion abundance for each of the General Facility Discharge and Chemours Process Wastewater samples, and work began on the most abundant unidentified potential PFAS in each group of samples. As the non-targeted program proceeds (i.e., as non-targeted analytes are identified in approximate order of abundance), the remaining unidentified potential PFAS become less abundant (with smaller peaks). These smaller peaks may not be large enough to undergo the fragmentation needed to further identify the unidentified potential PFAS. Assessment of mass balance of a sample from Chemours Process Wastewater Location 16 (which had the highest number of unidentified potential PFAS of the locations assessed in the first interim report) was therefore proposed to potentially provide insight into the mass of potential PFAS that remains unknown. If the remaining mass of unknown potential PFAS becomes a “de minimus”

mass, this will provide further evidence that the non-targeted analysis program has identified and assessed relevant PFAS (along with results that show that identified PFAS are no longer present in samples of groundwater adjacent to the Cape Fear River or of the Cape Fear River itself, as described in the Introduction).

As reported previously (Chemours, 2024b), targeted analysis using Method 537 Mod Max and Adsorbable Organic Fluorine analysis via EPA Method 1621 was conducted on two samples from Location 16 (STW-LOC16-012920 and STW-LOC16-042820):

- In STW-LOC16-012920
 - the total mass of fluorine in target compounds was 14,000 nanograms per liter (ng/L)
 - the total amount of adsorbable organic fluorine was non-detect, with a reporting limit of 10,000 ng/L
- In STW-LOC16-042820
 - the total mass of fluorine in target compounds was 24,000 ng/L
 - the total amount of adsorbable organic fluorine was 12,000 ng/L.

The Adsorbable Organic Fluorine analysis did not capture all the organic fluorine present in the targeted analysis; therefore, no comparison to any remaining, non-targeted organic fluorine could be made.

The next step was to compare the area counts of target PFAS to the area counts of unknown potential PFAS in the Orbitrap analysis of STW-LOC16-012920 and STW-LOC16-042820, to see if a more accurate mass balance could be achieved than was obtained from the Total Organic Fluorine analysis and therefore provide insight into the mass of potential PFAS that remains unknown. STW-LOC16-012920 and STW-LOC16-042820 were analyzed using the Orbitrap mass spectrometer and identification of target PFAS versus unknown potential PFAS was initiated.

In the previous interim report (June 2025), next steps for this task were:

- Finalize the identification of the target PFAS in the samples from STW-LOC16-012920 and STW-LOC16-042820.
- Compare the area counts of target PFAS to the area counts of unknown potential PFAS in the Orbitrap analysis of STW-LOC16-012920 and STW-LOC16-042820 to gain an understanding of the remaining mass of unknown potential PFAS.

Results achieved since June 2025 are:

- The total area of peaks that could be identified (i.e., by comparison to known standards) and the total area of peaks that could not be identified (i.e., potentially unknown PFAS) were compared.
- Less than 95% of the peaks could be identified and consequently work on the CPWW analytes will continue as described above.

4 SUMMARY

This report provides an update on the identification and analysis of previously unknown per- and polyfluoroalkyl substances (PFAS) in water samples from process wastewater, non-process wastewater, and stormwater at the Chemours Fayetteville Works, North Carolina. Samples for non-targeted analysis were divided into General Facility Discharge (stormwater, treated non-Chemours wastewater, and non-contact cooling water (collected in 2019 from five locations) and Chemours Process Wastewater (process wastewater from manufacturing). Samples were analyzed for non-targeted analytes using liquid chromatography (LC) coupled with high-resolution quadrupole time-of-flight (QToF) mass spectrometry. Unknown PFAS assigned tentative empirical formulas and prioritized by abundance (peak height) for further analysis.

Summary of General Facility Discharge (GFD) Samples:

- Of 19 unknown PFAS in GFD samples, the 10 most abundant were fully assessed; 9 were not PFAS or were previously known, and the 10th (GFD-6) has been identified as PFO6TeA.
- For GFD-11 through GFD-15, advanced mass spectrometry using the Orbitrap mass spectrometer did not yield sufficient fragment data for proposal of molecular structures.
- Once potential unidentified PFAS that have been identified are no longer detectable in samples of groundwater adjacent to the Cape Fear River or of the Cape Fear River itself, the non-targeted program will be considered complete as PFAS detections of less abundant PFAS are not expected to occur in the environment beyond the Facility boundaries. The least abundant peak that has been identified, GFD-6 (PFO6TeA), was not detected in groundwater samples from 2 flood plain wells and 2 Black Creek Aquifer wells adjacent to the Cape Fear River, nor was it detected in the Cape Fear River a few miles downstream at Bladen Bluffs.
- Additionally, about 96% of total PFAS mass in GFD samples has been identified and the remaining unidentified PFAS are of very low abundance and unlikely to be structurally characterized.
- Chemours therefore submits that the non-targeted assessment of GFD samples is complete.

Summary of Chemours Process Wastewater (CPWW) Samples:

- The 10 most abundant PFAS (CPWW-1 through CPWW-10) are being assessed.
- CPWW-2: Identified as EVE Acid (a known PFAS).
- CPWW-1 (RHDA): Identified but cannot be accurately quantified by current analytical methods due to matrix interference. A new method (DSC) is under development to potentially address this.
- CPWW-3: The initial proposed structure was synthesized, but the mass spectrum of the synthesized standard did not match CPWW-3. A revised structure was proposed, but

synthesis is challenging and recent samples from the same location (Location 16) show decreased presence of CPWW-3, so work on CPWW-3 is paused.

- CPWW-4 (DAE Acid): Identified but cannot be accurately quantified by current analytical methods due to matrix interference. A new method (DSC) is under development to potentially address this.
- CPWW-5: Synthesis of authentic standard ongoing. Once completed, next steps include spectral comparison of the standard to CPWW-5.
- CPWW-6 through CPWW-10: The initial proposed structure for CPWW-7 was synthesized, but the mass spectrum of the synthesized standard did not match CPWW-7. A revised structure was proposed but synthesis is challenging. An initial proposed structure for CPWW-10 is under development. Fragmentation of CPWW-6, -8 and -9 by the Orbitrap (as well as by the QToF) resulted in very few detectable fragments for each analyte, making the proposal of structures difficult.

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