The Chemours Company
Fayetteville Works
22828 NC Highway 87 W
Fayetteville, NC 28306

PFAS NON-TARGETED ANALYSIS AND METHODS INTERIM REPORT #11

Process and Non-Process Wastewater and Stormwater

Prepared by

The Chemours Company FC, LLC

Fayetteville Works

22828 NC Highway 87 W

Fayetteville, NC 28306

June 26, 2025

The Chemours Company
Fayetteville Works
22828 NC Highway 87 W
Fayetteville, NC 28306

TABLE OF CONTENTS

The Chemours Company
Fayetteville Works
22828 NC Highway 87 W
Fayetteville, NC 28306

LIST OF FIGURES

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
HFPO-TA	hexafluoropropylene oxide trimer acid
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_6TeA	pentadecafluoro-2,4,6,8,10,12-hexaoxatetradecan-14-oic acid
RHDA	RSU/HFPO Diadduct
QToF	quadrupole time-of-flight
TFA	trifluoroacetate

The Chemours Company
Fayetteville Works
22828 NC Highway 87 W
Fayetteville, NC 28306

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 11th interim report. Further details are provided in the references (Chemours, 2020a, 2020b, 2021a, 2021b, 2022a, 2022b, 2023a, 2023b, 2024a, 2024b).

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; and
- Chemours Process Wastewater Samples - samples of process wastewater from Chemours manufacturing areas. These samples were collected at two locations.

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. An atomic mass defect refers to the phenomenon that the mass of an atom is not exactly equal to the number of subatomic particles (protons and neutrons) or the atomic mass number (except for carbon-12 by definition) due to differences in mass lost (as energy) when the atomic nucleus is formed for each isotope. Fluorine is well-known to have a negative mass defect, where the exact mass is slightly less than the mass number. When the QToF mass spectrometer is operated in the negative mode, one can select fluorine-containing features and empirical formulas using available software provided by the instrument vendor.

The initial analysis identified 21 potentially unknown PFAS present in General Facility Discharge samples and 250 potentially unknown PFAS present in Chemours Process Wastewater samples, with a total of 257 potential unique unknown PFAS (14 unknown PFAS were present in both types of samples). Two of the unknown PFAS were later identified as trifluoroacetic acid (TFA) and hexafluoropropylene oxide trimer acid (HFPO-TA), which were not carried forward further in the

non-targeted analysis program. This left 19 potentially unknown PFAS present in General Facility Discharge samples and 248 potentially unknown PFAS present in Chemours Process Wastewater samples, with a total of 255 potential unique unknown PFAS (12 unknown PFAS were present in both types of samples).

Empirical formulas were proposed for all unknown PFAS. This work represented the first part of the Initial Assessment step in the Development Plan. The second part of the Development Plan, the Enhanced Assessment is to develop tentative molecular structures and subsequently, for the highest priority identified PFAS, develop authentic standards (i.e., synthesize samples of the compounds to facilitate traditional targeted analysis). Once an authentic standard is available, the following steps will be 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.

To prioritize developing authentic standards for the most abundant unknown PFAS for each grouping of samples (General Facility Discharge and Chemours Process Wastewater), the 5 most abundant unknown PFAS from each group were advanced to the Enhanced Assessment step. As each group of 5 unknown PFAS from each group is resolved, the next group of 5 will be advanced to the Initial Assessment step. 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 11th interim report consists of:

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

The Chemours Company
Fayetteville Works
22828 NC Highway 87 W
Fayetteville, NC 28306

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 assessed. Nine were shown to not be PFAS or to be previously known PFAS. The tenth (GFD-6) has been identified as pentadecafluoro-2,4,6,8,10,12-hexaoxatetradecan-14-oic acid (PFO_6TeA), for which an authentic standard is commercially available. Progress on PFO_6TeA (GFD-6) and on GFD-11 through GFD-15 is described below.

GFD-6 (PFO_6TeA)

In the previous interim report (December 2024), the next steps for PFO_6TeA were stated to be:

1) Analysis of PFO_6TeA in samples of groundwater adjacent to the Cape Fear River or of the Cape Fear River itself to assess the potential presence of PFO_6TeA; and
2) Formal addition of PFO_6TeA to the Facility's target analyte list.

Results achieved since December 2024 are:

a) Samples were collected from 6 groundwater wells - 2 wells on-site and 4 wells adjacent to the Cape Fear River - and from the Cape Fear River at Bladen Bluffs (Figure 1) and analyzed for PFO_6TeA (see Table below). PFO_6TeA was only detected in perched groundwater beneath the facility. It was not detected in 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.

Description	Location	Sample Date	Concentration of PFO6TeA (ng/L)
On-site *(perched zone)*	NAF-03	10-Oct-24	32,000
	MW-24	10-Oct-24	1,100
Adjacent to the Cape Fear River *(Black Creek Aquifer)*	PIW-1D	8-Oct-24	< 140
	PIW-3D	8-Oct-24	< 140
Adjacent to the Cape Fear River *(flood plain)*	LTW-01	10-Oct-24	< 57
	LTW-03	10-Oct-24	< 140
Cape Fear River	Bladen Bluffs	12-Sep-24	< 1.1

These results were provided to DEQ via email on March 21, 2025. Laboratory reports are available at 90 - NTA Lab Reports.

Because PFO_6TeA was not detected in groundwater adjacent to the Cape Fear River, or in the river itself, Chemours will analyze and report on this compound under future Paragraph 11 required sampling. No further action on PFO_6TeA is required under Consent Order Paragraph 11 subpart (a).

GFD-11 Through GFD-15

In the previous interim report (December 2024), additional next steps for GFD-11 through -15 (the next five potentially unknown PFAS present in General Facility Discharge samples) were stated to be:

1) Continued development of proposed molecular structures with subsequent steps of synthesizing an authentic standard so the potential presence of GFD-11 through -15 in samples of groundwater adjacent to the Cape Fear River or of the Cape Fear River itself samples can be assessed.

Results achieved since December 2024 are:

a) Re-analysis of General Facility Discharge samples was conducted using a Thermo Scientific Orbitrap Exploris 240 mass spectrometer (Orbitrap) for mass resolution that is higher than that provided by the QToF mass spectrometer used in the initial analysis. It was thought 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.

Next steps for GFD-11 through GFD-15 will be:

i. Review the area counts of target PFAS to the area counts of unknown PFAS in the Orbitrap analysis of the General Facility Discharge samples to gain an understanding of the remaining mass of unknown potential PFAS.

3 CHEMOURS PROCESS WASTEWATER SAMPLES

Of the 248 potentially unknown PFAS present in Chemours Process Wastewater (CPWW) samples, the 5 most abundant (CPWW-1 through CPWW-5) have been assessed (note: Chemours process wastewater is no longer discharged to the environment). 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 was identified as RSU/HFPO Diadduct (RHDA) by comparison to a standard purified from production samples. In the previous interim report (December 2024), the next steps for RHDA were stated to be:

1) Analysis of RHDA in samples of groundwater adjacent to the Cape Fear River or of the Cape Fear River itself to assess the potential presence of RHDA; and

2) Addition of RHDA to the suite of diprotic PFAS being evaluated for analytical improvement.

Results achieved since December 2024 are:

a) Samples were collected from 6 groundwater wells – 2 wells on-site and 4 wells adjacent to the Cape Fear River - and from the Cape Fear River and analyzed for RHDA (see Table below). RHDA was detected in all samples. However, the matrix interference study (Chemours, 2024b) indicated that Cape Fear River water from River Mile 84 caused significant matrix interference for the analysis of RHDA. RHDA is a diprotic PFAS and exhibits the significant over-recovery in Cape Fear River water that has been observed with other diprotic PFAS. This over-recovery has been demonstrated in groundwater for other diprotic PFAS; therefore, over-recovery of RHDA in groundwater, as well as in river water, is expected.

Description	Location	Sample Date	Concentration of RHDA (ng/L)
On-site *(perched zone)*	NAF-03	10-Oct-24	19,100 J
	MW-24	10-Oct-24	1,000 J
Adjacent to the Cape Fear River *(Black Creek Aquifer)*	PIW-1D	8-Oct-24	600 J
	PIW-3D	8-Oct-24	2,600 J
Adjacent to the Cape Fear River *(flood plain)*	LTW-01	10-Oct-24	1,500 J
	LTW-03	10-Oct-24	1,400 J
Cape Fear River	Bladen Bluffs	12-Sep-24	6.3 J

Laboratory reports are available at 90 - NTA Lab Reports.

Next steps for RHDA will be:

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

CPWW-3

As reported in the previous interim report (December 2024), a structure for CPWW-3 was proposed ($HO_3SCF_2CF_2OCF(CF_3)CF_2OCHFCF_2OCH$) and a standard corresponding to that structure was synthesized. However, the tandem mass spectrometry (MS/MS) spectrum of the synthesized standard did not match the MS/MS spectrum of CPWW-3; therefore, the proposed structure was rejected. The next steps for CPWW-3 ($C_8H_5F_{13}O_6S$) were stated to be:

1) Continued development of a proposed molecular structure, with subsequent steps of 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 December 2024 are:

a) The analysis of CPWW-3 via the Orbitrap generated a revised empirical formula from $C_8H_5F_{13}O_6S$ to $C_8H_2F_{14}O_7$.
b) The fragmentation pattern of CPWW-3 via the Orbitrap was identical to the fragmentation pattern of Compound 37 found by McCord and Strynar (2019), which they identified as a polyfluorinated ether acid with a single hydrogen substitution. McCord and Strynar did not propose a structure for $C_8H_2F_{14}O_7$.

These results illustrate an intrinsic difficulty that may be encountered during non-targeted analysis, namely, that empirical formulas generated by computer software may be incorrect, leading to time spent attempting to derive a structure for the incorrect chemical formula. When an alternative candidate chemical formula is generated later, the process of structure derivation is required to be repeated.

Next steps for CPWW-3 will be:

i. Development of a proposed molecular structure for the revised empirical formula, with subsequent steps of developing a synthetic pathway to 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.

CPWW-4 (DAE Acid)

In the previous interim report (December 2024), an authentic standard for CPWW-4 (Diadduct Ester Acid (DAE Acid), $C_9H_2F_{14}O_6$), $HOOCCF(CF_3)OCF_2CF(CF_3)OCF_2CF_2COOH$) had been synthesized, and the next steps were stated to be:

1) Assessment of the ability of Method 537Mod Max to analyze DAE Acid;
2) The execution of a method detection limit study to establish a reporting limit for DAE Acid;
3) The execution of a matrix interference study to assess the quantification of DAE Acid in an environmental matrix related to the Facility; and
4) Analysis of samples of groundwater adjacent to the Cape Fear River and of the Cape Fear River itself to see if DAE Acid is detectable.

Results achieved since December 2024 are:

a) The purity of the authentic standard was evaluated via nuclear magnetic resonance (NMR) – this proved more difficult than expected because the compound is not a solid that is easy to handle but rather a sticky, glue-like material. The standard was found to be 96% pure.

b) A secondary species was identified in the standard, and was assessed by LC-MS/MS to confirm that it does not interfere with the analysis of DAE Acid.

Next steps for DAE Acid will be:

i. Prepare standard solutions of DAE Acid to send to Eurofins-Sacramento for an assessment of the ability of Method 537Mod to analyze DAE Acid.
ii. 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.

CPWW-5

In the previous interim report (December 2024), a synthetic pathway for an authentic standard had been developed and synthesis of the authentic standard had begun. The next steps for CPWW-5 ($C_6HF_{11}O_4$) were stated to be:

1) Completion of the synthesis of the authentic standard; and
2) Comparison of the MS/MS spectrum of the authentic standard to the MS/MS spectrum of CPWW-5.
3) If the MS/MS spectra match, assessment of the ability of Method 537Mod Max to analyze CPWW-5, execution of a method detection limit study and a matrix interference study and analysis of samples of groundwater adjacent to the Cape Fear River and of the Cape Fear River itself to see if CPWW-5 is detectable.

Results achieved since December 2024 are:

a) Synthesis of the authentic standard has progressed and is pending completion.

Next steps for CPWW-5 will be:

i. Completion of the synthesis of the authentic standard; and
ii. Comparison of the MS/MS spectrum of the authentic standard to the MS/MS spectrum of CPWW-5.

CPWW-6 Through CPWW-10

In the previous interim report (December 2024), the next steps for CPWW-6 through CPWW-10 were stated to be:

1) Continued development of proposed molecular structures, with subsequent steps of synthesizing an authentic standard so the potential presence of CPWW-6 through CPWW-10 in samples of groundwater adjacent to the Cape Fear River or of the Cape Fear River itself can be assessed.

Results achieved since December 2024 are:

a) A structure was proposed for CPWW-7 ($C_3H_2F_6O_4S$): CF_3O-CFH-CF_2-SO_3H;
b) The proposed structure was synthesized by Chemours; and
c) The retention time and MS/MS spectrum of CPWW-7 were compared to those of the synthesized compound and were found not to match.

These results illustrate another 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 structure derivation is required to be repeated.

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

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

4 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; and

- 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.

In the previous interim report (December 2024), the next steps were stated to be:

1) 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 can be achieved than was obtained from the Total Organic Fluorine analysis and potentially providing insight into the mass of potential PFAS that remains unknown.

Results achieved since December 2024 are:

a) STW-LOC16-012920 and STW-LOC16-042820 have been analyzed using the Orbitrap mass spectrometer; and
b) The identification of target PFAS versus unknown potential PFAS is underway.

Next steps will be:

i. Finalize the identification of the target PFAS in the samples from STW-LOC16-012920 and STW-LOC16-042820; and
ii. Compare the area counts of target PFAS to the area counts of unknown potential PFAS.

5 REFERENCES

Chemours, 2024a. PFAS Non-Targeted Analysis and Methods Interim Report #9. June 28, 2024.

Chemours, 2024b. PFAS Non-Targeted Analysis and Methods Interim Report #10. December 23, 2024.

Chemours, 2023a. PFAS Non-Targeted Analysis and Methods Interim Report #7. June 30, 2023.

Chemours, 2023b. PFAS Non-Targeted Analysis and Methods Interim Report #8. December 29, 2023

Chemours, 2022a. PFAS Non-Targeted Analysis and Methods Interim Report #5. June 30, 2022.

Chemours, 2022b. PFAS Non-Targeted Analysis and Methods Interim Report #6. December 30, 2022.

Chemours, 2021a. PFAS Non-Targeted Analysis and Methods Interim Report #3. July 30, 2021.

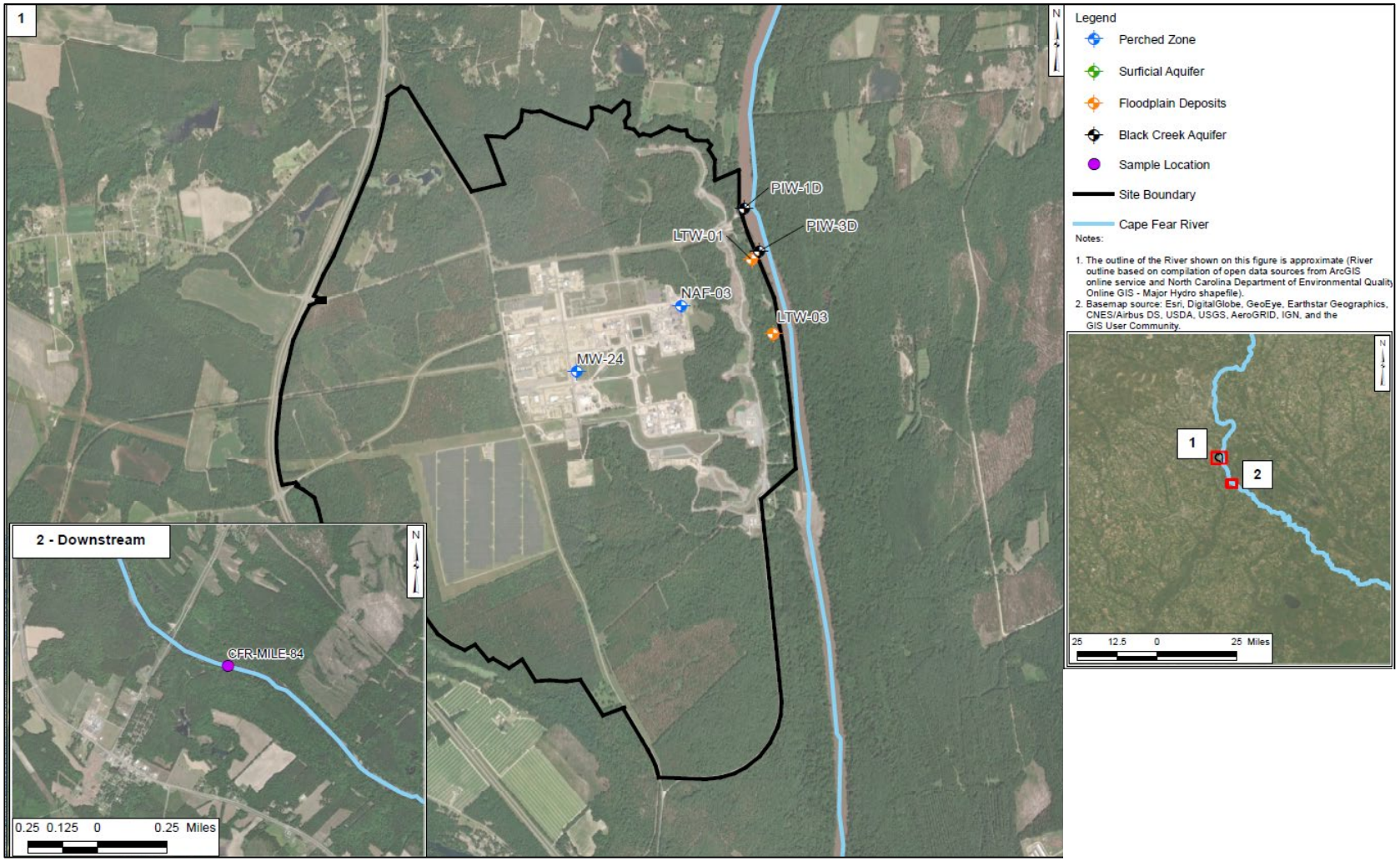
Chemours, 2021b. PFAS Non-Targeted Analysis and Methods Interim Report #4. December 22, 2021.

Chemours, 2020a. PFAS Non-Targeted Analysis and Methods Interim Report. June 30, 2020.

Chemours, 2020b. PFAS Non-Targeted Analysis and Methods Interim Report #2. December 31, 2020.

Chemours and Geosyntec Consultants, 2019. PFAS Non-Targeted Analysis and Methods Development Plan. Version 2. December 5, 2019.

McCord, J. and M. Strynar, 2019. Identification of Per- and Polyfluoroalkyl Substances in the Cape Fear River by High Resolution Mass Spectrometry and Nontargeted Screening. Environ. Sci. Technol., 53 (9).



Notes:
PFO6TeA - pentadecafluoro-2,4,6,8,10,12-hexaoxatetradecan-14-oic acid
RHDA - RSU/HFPO Diadduct

Location of Samples Collected for Analysis of PFO6TeA and RHDA
Chemours Fayetteville Works, North Carolina

Chemours™

June 2025

Figure

1