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

PFAS NON-TARGETED ANALYSIS AND METHODS INTERIM REPORT #10

Process and Non-Process Wastewater and Stormwater

Prepared by

The Chemours Company FC, LLC

Fayetteville Works

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December 23, 2024

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LIST OF ATTACHMENTS

ACRONYMS AND ABBREVIATIONS

Chemours	The Chemours Company FC, LLC
Facility	Chemours Fayetteville Works, North Carolina
HFPO-TA	hexafluoropropylene oxide trimer acid
LC	liquid chromatography
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

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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 10th interim report. Further details are provided in the references (Chemours, 2020a, 2020b, 2021a, 2021b, 2022a, 2022b, 2023a, 2023b, 2024).

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), and not carried forward further in the non-targeted analysis program. This left 19 potentially unknown PFAS present in General Facility

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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 determined 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
    - 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 10th 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.

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2 GENERAL FACILITY DISCHARGE SAMPLES

Of the 19 potentially unknown PFAS present in General Facility Discharge 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 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.

PFO_6TeA (GFD-6)

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

1) The assessment of PFO_6TeA analysis by an existing analytical method (EPA Method 537Mod Max);
2) The execution of a method detection limit study to establish a reporting limit; and
3) The execution of a matrix interference study to assess the quantification of PFO_6TeA an environmental matrix related to the Facility.

Results achieved since June 2024 are:

a) Analysis of PFO_6TeA analysis can be accomplished by the existing analytical method EPA Method 537Mod Max;
b) The method detection limit for PFO_6TeA was determined to be 1.07 ng/L, which is consistent with a reporting limit of 2 ng/L. Additionally, Precision and Accuracy were demonstrated to be consistent with targeted control limits (L (Attachment 1); and
c) The matrix interference study indicated that Cape Fear River water from River Mile 84 did not cause significant matrix interference for the analysis of PFO_6TeA (Attachment 2).

Next steps for PFO_6TeA will be:

i. 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
ii. Formal addition of PFO_6TeA to the Facility's target analyte list.

GFD-11 Through GFD-15

In the previous interim report (June 2024), additional next steps for potentially unknown PFAS present in General Facility Discharge samples were stated to be:

1) Re-analysis of samples from Chemours Process Wastewater Location 16 (Monomers IXM Area combined processes) using an Thermo Scientific Orbitrap Exploris 240 mass spectrometer (Orbitrap) as the additional mass resolution of the Orbitrap (versus the QToF mass spectrometer used in the initial analysis) may allow the identification of additional ionization fragments from GFD-11 through -15; and

2) Proposal of potential structures for GFD-11 through -15, if additional ionization fragments can be identified and used for proposed structures.

Results achieved since June 2024 are:

a) Re-analysis of samples from Chemours Process Wastewater Location 16 with the Orbitrap was conducted;
b) Higher mass accuracy was achieved, but overall, the results were similar compared to the QToF, with the exception of some additional information for CPWW-5 (see below).

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

i. Continued development of proposed molecular structures 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 samples can be assessed.

3 CHEMOURS PROCESS WASTEWATER SAMPLES

Of the 248 potentially unknown PFAS present in Chemours Process Wastewater samples, the 5 most abundant (CPWW-1 through CPWW-5) 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

CPWW-1 was identified as RSU/HFPO Diadduct (RHDA) by comparison to a standard purified from production samples. In the previous interim report (June 2024), the next steps for RHDA were stated to be:

1) assessment of RHDA analysis by an existing analytical method (EPA Method 537Mod Max);
2) The execution of a method detection limit study to establish a reporting limit; and
3) The execution of a matrix interference study to assess the quantification of RHDA in an environmental matrix related to the Facility.

Results achieved since June 2024 are:

a) Analysis of RHDA analysis can be accomplished by the existing analytical method EPA Method 537Mod Max;
b) The method detection limit for RHDA was determined to be 0.62 ng/L, which is consistent with a reporting limit of 2 ng/L (Attachment 1). Additionally, Precision and Accuracy were demonstrated to be consistent with targeted control limits (L (Attachment 1); and

c) The matrix interference study indicated that Cape Fear River water from River Mile 84 did cause 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 (Attachment 2).

Next steps for RHDA will be:

i. 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
ii. Addition of RHDA to the suite of diprotic PFAS being evaluated for analytical improvement.

CPWW-3

For CPWW-3 ($C_8H_5F_{13}O_6S$), a molecular structure had not yet been proposed. In the previous interim report (June 2024), the next steps for CPWW-3 were stated to be:

1) Continued development of a molecular structure for CPWW-3.

Results achieved since June 2024 are:

a) A structure was proposed ($HO_3SCF_2CF_2OCF(CF_3)CF_2OCHFCF_2OCH$) and a standard corresponding to that structure was synthesized. The MS/MS spectrum of the synthesized standard did not match the MS/MS spectrum of CPWW-3; therefore, the proposed structure was rejected.

Next steps for CPWW-3 will be:

i. 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.

CPWW-4

For CPWW-4 ($C_9H_2F_{14}O_6$), a molecular structure was proposed (HOOC-CF_2-CF_2-O-CF(CF_3)-CF_2-O-CF(CF_3)-COOH), a process for synthesizing an authentic standard for the proposed structure for CPWW-4 was identified, and synthesis of an authentic standard was initiated. In the previous interim report (June 2024), the next steps for CPWW-4 were stated to be:

1) Completion of the synthesis of an authentic standard; and
2) Comparison of the CPWW-4 to the authentic standard.

Results achieved since June 2024 are:

a) The authentic standard was synthesized;
b) The MS/MS spectrum of the authentic standard was compared to the MS/MS spectrum of CPWW-4 and was found to match. Therefore, CPWW-4 has been identified as HOOC-CF_2-CF_2-O-CF(CF_3)-CF_2-O-CF(CF_3)-COOH, and has been nicknamed DAE Acid (diadduct ester acid)

Next steps for CPWW-4 will be:

i. Assessment of the ability of Method 537Mod Max to analyze CPWW-4;
ii. The execution of a method detection limit study to establish a reporting limit for CPWW-4; and
iii. The execution of a matrix interference study to assess the quantification of CPWW-4 in an environmental matrix related to the Facility; and
iv. Analysis of samples of groundwater adjacent to the Cape Fear River and of the Cape Fear River itself to see if CPWW-4 is detectable.

CPWW-5

For CPWW-5 ($C_6HF_{11}O_4$), a molecular structure was proposed (CF_3-O-CF_2-O-CF_2-CF_2-CF_2-COOH). In the previous interim report (June 2024), the next steps for CPWW-5 were stated to be:

1) Development of a synthetic pathway for an authentic standard of the proposed structure for CPWW-5

Results achieved since June 2024 are:

a) A synthetic pathway for an authentic standard was developed; and
b) Synthesis of the authentic standard was begun.

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.
iii. 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.

CPWW-6 Through CPWW-10

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

1) Beginning work on identifying the molecular structures of the second set of five most abundant potential unknown PFAS (CPWW-6 through -10) in the Chemours Process Wastewater samples using the Orbitrap mass spectrometer.

Results achieved since June 2024 are:

a) Work has begun on assessing the results from the Orbitrap mass spectrometer, but no structures have yet been proposed.

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

i. 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.

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. One issue that will become significant as the non-targeted program proceeds is that the abundance of the unidentified potential PFAS becomes smaller, and 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 proposed to potentially provide insight into the mass of potential PFAS that remains unknown.

In the previous interim report (June 2024), steps to assess mass balance from a sample from Location 16 were stated to be:

1) Conduct a targeted analysis using Method 537 Mod Max to quantify as many known PFAS as possible; and
2) Conduct an Adsorbable Organic Fluorine analysis via EPA Method 1621 to assess the total mass of organic fluorine present.

Results achieved since June 2024 are:

a) Targeted analysis using Method 537 Mod Max 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 ng/L. In STW-LOC16-042820, the total mass of fluorine in target compounds was 24,000 ng/L (Attachment 3)
b) Adsorbable Organic Fluorine analysis via EPA Method 1621 was conducted on the same two samples from Location 16. Six replicates of each Location 16 sample were analyzed (Attachment 3). The total amount of adsorbable organic fluorine in STW-LOC16-012920 was 12,000 ng/L. The total amount of adsorbable organic fluorine in STW-LOC16-042920 was non-detect, with a reporting limit of 10,000 ng/L.

The Adsorbable Organic Fluorine analysis via EPA Method 1621 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 for the mass balance work will be comparing the area counts of target PFAS to the area counts of unknown 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. This step will provide insight into the mass of potential PFAS that remains unknown.

As PFAS are identified and assessed, the remaining mass of unknown potential PFAS will become smaller, and may become a “de minimus” mass that, 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), will provide evidence that the non-targeted analysis program has identified and assessed relevant PFAS.

5 REFERENCES

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

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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.

ATTACHMENT 1

Method Detection Limit Study Results and Precision and Accuracy Results for RHDA and PFO6TeA

Method	PFC_IDA_Chemours/3535_PFC
Matrix	Water

Reviewer	Z8QU
Review Date	10/07/2024

Analyte	Spike Conc	Unit	RL	MDL	Calc MDL	MDLb Option	MDLs	MDLb (99th)	MDLb (t-stat)	MDLb (Max)	CalcMDL/MDL	RL/CalcMDL	Spike/MDL	Blank Hits > MDL (%)	Comment
RSU-HFPO Diadduct	2.000	ng/L	2.000	0.010	0.619993	MDLb (Max)	0.619993		0.000000	0.000000	62.06	3.23	200.20	0.0	set MDL to calc MDL
pentadecafluoro-2,4,6,8,10,12-hexaoxatetradecan-14-oic acid	1.898	ng/L	2.000	0.010	1.078653	MDLb (Max)	1.078653		0.000000	0.000000	107.97	1.85	189.99	0.0	set MDL to calc MDL

Analyst Demonstration of Capability

Eurofins Sacramento

10/8/2024

Preparation Method(s):	3535
Analytical Method(s):	537 (modified)
Matrix:	Water
Method Description:	Fluorinated Alkyl Substances
Preparation SOP No:	WS-LC-0025 R 4.0 TK 10/9/24
Analytical SOP No:	WS-LC-0025 R 4.0 TK 10/9/24

We, the undersigned, CERTIFY that:

1. The analyst identified above, using the cited test method with the specifications in the cited SOP, which is in use at this facility for the analysis of samples under the laboratory's Quality Assurance Plan, has completed the Demonstration of Capability (DOC).
2. The test method(s) was performed by the analyst identified on this certificate.
3. A copy of test method(s) and laboratory SOPs are available for all personnel on-site.
4. The data associated with the demonstration of capability are true, accurate, complete and self-explanatory.
5. All raw data necessary to reconstruct and validate these analyses have been retained at the facility. The associated information is organized and available for review.

Technical Director Chris Williams	**Signature**	**Date** 10/9/24
QA Manager Robert Hrabak	**Signature**	**Date** 10/8/24

Analyst Demonstration of Capability

ANALYST DEMONSTRATION OF CAPABILITY

Method 537 (modified)
Method Desc: Fluorinated Alkyl Substances

Laboratory: Eurofins Sacramento

Limit Group: LC - PFC Chem - Water - QC

13C4 PFBA

Analysis Dates: 10/1/2024 to 10/1/2024

All values within Control limits

Current Limits Recovery LCL	Current Limits Recovery UCL	Current Limits Precision Std Dev	Current Limits Precision Units
25	150		%

Demonstration of Capability Recovery Mean	Demonstration of Capability Precision Std Dev	Units		Amount
101.5	3.36854(	%	Pass	50.0

Laboratory ID	Anal Date	Batch	Smp	Analyst	Prep Analyst	Result	Units	Spike Amount	% Rec	In Rec Limits?
LCS 320-803508/2-A	10/01/2024	803849	13	Phomsopha, Thep	Gachet, Melody 1	48.47[illegible]	ng/L	50.0	97	Pass
LCS 320-803508/3-A	10/01/2024	803849	14	Phomsopha, Thep	Gachet, Melody 1	50.98[illegible]	ng/L	50.0	102	Pass
LCS 320-803508/4-A	10/01/2024	803849	15	Phomsopha, Thep	Gachet, Melody 1	52.53[illegible]	ng/L	50.0	105	Pass
LCS 320-803508/5-A	10/01/2024	803849	16	Phomsopha, Thep	Gachet, Melody 1	51.05[illegible]	ng/L	50.0	102	Pass

13C2 PFDA

Analysis Dates: 10/1/2024 to 10/1/2024

All values within Control limits

Current Limits Recovery LCL	Current Limits Recovery UCL	Current Limits Precision Std Dev	Current Limits Precision Units
25	150		%

Demonstration of Capability Recovery Mean	Demonstration of Capability Precision Std Dev	Units		Amount
108.4	2.99074(	%	Pass	50.0

Laboratory ID	Anal Date	Batch	Smp	Analyst	Prep Analyst	Result	Units	Spike Amount	% Rec	In Rec Limits?
LCS 320-803508/2-A	10/01/2024	803849	13	Phomsopha, Thep	Gachet, Melody 1	55.10[illegible]	ng/L	50.0	110	Pass
LCS 320-803508/3-A	10/01/2024	803849	14	Phomsopha, Thep	Gachet, Melody 1	52.17(	ng/L	50.0	104	Pass
LCS 320-803508/4-A	10/01/2024	803849	15	Phomsopha, Thep	Gachet, Melody 1	55.52[illegible]	ng/L	50.0	111	Pass
LCS 320-803508/5-A	10/01/2024	803849	16	Phomsopha, Thep	Gachet, Melody 1	54.04[illegible]	ng/L	50.0	108	Pass

RSU-HFPO Diadduct

Analysis Dates: 10/1/2024 to 10/1/2024

All values within Control limits

Current Limits Recovery LCL	Current Limits Recovery UCL	Current Limits Precision Std Dev	Current Limits Precision RPD	Current Limits Precision Units
40	160		40	%

Demonstration of Capability Recovery Mean	Demonstration of Capability Precision Std Dev	Units		Amount	Amount/RL
90.65	3.43660	%	Pass	40.0	20

Laboratory ID	Anal Date	Batch	Smp	Analyst	Prep Analyst	Result	Units	Spike Amount	RL	% Rec	% D	In Rec Limits?
LCS 320-803508/2-A	10/01/2024	803849	13	Phomsopha, Thep	Gachet, Melody 1	37.93[illegible]	ng/L	40.0	2.00	95		Pass
LCS 320-803508/3-A	10/01/2024	803849	14	Phomsopha, Thep	Gachet, Melody 1	34.73(	ng/L	40.0	2.00	87		Pass
LCS 320-803508/4-A	10/01/2024	803849	15	Phomsopha, Thep	Gachet, Melody 1	36.70[illegible]	ng/L	40.0	2.00	92		Pass
LCS 320-803508/5-A	10/01/2024	803849	16	Phomsopha, Thep	Gachet, Melody 1	35.67[illegible]	ng/L	40.0	2.00	89		Pass

pentadecafluoro-2,4,6,8,10,12-hexaoxatetradecan-14-oic acid

Analysis Dates: 10/1/2024 to 10/1/2024

All values within Control limits

Current Limits Recovery LCL	Current Limits Recovery UCL	Current Limits Precision Std Dev	Current Limits Precision RPD	Current Limits Precision Units
40	160		40	%

Demonstration of Capability Recovery Mean	Demonstration of Capability Precision Std Dev	Units		Amount	Amount/RL
102.9	3.34413(	%	Pass	37.960[illegible]	19

Laboratory ID	Anal Date	Batch	Smp	Analyst	Prep Analyst	Result	Units	Spike Amount	RL	% Rec	% D	In Rec Limits?
LCS 320-803508/2-A	10/01/2024	803849	13	Phomsopha, Thep	Gachet, Melody 1	38.45(	ng/L	37.96056	2.00	101		Pass
LCS 320-803508/3-A	10/01/2024	803849	14	Phomsopha, Thep	Gachet, Melody 1	37.69(	ng/L	37.96056	2.00	99		Pass
LCS 320-803508/4-A	10/01/2024	803849	15	Phomsopha, Thep	Gachet, Melody 1	39.48[illegible]	ng/L	37.96056	2.00	104		Pass
LCS 320-803508/5-A	10/01/2024	803849	16	Phomsopha, Thep	Gachet, Melody 1	40.61[illegible]	ng/L	37.96056	2.00	107		Pass

Precision = standard deviation of percent recoveries of spiked control samples.

10/8/2024

ATTACHMENT 2

Matrix Interference for PFO6TeA and RHDA During Analysis by Method 537Mod Max

Geosyntec consultants

Geosyntec Consultants of NC, P.C.
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Memorandum

Date:	December 19, 2024
To:	The Chemours Company FC, LLC
From:	Geosyntec Consultants of NC, PC
Subject:	Matrix Interference for PFO6TeA and RHDA During Analysis by Method 537 Mod Max

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Geosyntec Consultants of NC, PC (Geosyntec) has prepared this memorandum for The Chemours Company FC, LLC (Chemours) to describe matrix interference observed during the analysis of two per- and polyfluorinated alkyl substances (PFAS), pentadecafluoro-2,4,6,8,10,12-hexaoxatetradecan-14-oic acid (PFO6TeA) and RSU/HFPO Diadduct (RHDA), by the EPA Method 537 Modified Max (537 Mod Max; a modified and extended version of EPA Method 537) analytical method. During the analysis of a sample by a given analytical method, the sample matrix refers to all the components of the sample other than the compounds of interest that are being analyzed by the analytical method. The sample matrix can be very complex and can have a considerable effect on the quality of the results obtained, and such effects are called matrix interference. Matrix interference can result in a positive bias (matrix enhancement) or in a negative bias (matrix suppression) of the analytical results.

The remainder of this memorandum is organized into the following sections:

- **Background**: Describing identification of matrix interference issues;
- **Scope and Methods**: Describing how the matrix interference issue was assessed:
- **Results**: Describing the results of the assessment;
- **Discussion**: Interpreting the results of the assessment; and
- **Recommendations**: Guidance for use of Method 537 Mod Max for PFO6TeA and RHDA

BACKGROUND

Matrix interference issues have previously been identified with analytical methods that have been used to analyze samples for PFAS associated with Chemours Fayetteville Works (the Site). These issues result in matrix enhancement of the diprotic Table 3+ PFAS (R-PSDA, R-EVE, and

Hydrolyzed PSDA). The matrix enhancement could lead to overestimation of the concentrations of these three PFAS in environmental samples.

Another method that was used to analyze some Table 3+ PFAS is the Table 6 method, which was found to reliably quantify PMPA and PEPA with a lower reporting limit than the Table 3 method (2 nanograms per liter (ng/L) for Table 6; 10 and 20 ng/L, respectively, for Table 3). The Table 6 method was found to have matrix interference that leads to unreliable quantitation of MMF, DFSA, MTP and PFPrA (formerly known as PPF Acid). Note that R-PSDA, R-EVE, and Hydrolyzed PSDA are not on the Table 6 analyte list.

It was necessary to submit samples for analyses using both the Table 3 and Table 6 methods to analyze a target analyte list of twenty Table 3+ PFAS to a reporting limit of 5 ng/L; therefore, Method 537 Mod Max was developed which included twenty Table 3+ PFAS to a reporting limit of at least 5 ng/L, thereby removing the need for sample analysis by both the Table 3 method and the Table 6 method.

A matrix interference study conducted on Table 3+ PFAS analyzed by Method 537 Mod Max showed that the diprotic Table 3+ PFAS (R-PSDA, R-EVE, and Hydrolyzed PSDA) demonstrated matrix enhancement when analyzed by 537Mod Max, echoing the results observed in the Table 3 and Table 6 methods.

This work reported in this memo is a part of efforts to improve analytical methods available for Table 3+ PFAS by examining matrix interference associated with analysis by Method 537 Mod Max. PFO6TeA and RHDA are two PFAS that have recently been added to Method 537 Mod Max, and a matrix interference study was commissioned to examine potential matrix effects in the analysis of PFO6TeA and RHDA in samples of river water from the Cape Fear River. Note that PFO6TeA is a monoprotic PFAS, while RHDA is a diprotic PFAS.

SCOPE AND METHODS

The study of matrix interference in 537 Mod Max was conducted by commissioning a laboratory matrix interference study to systematically analyze the potential for matrix interference for PFO6TeA and RHDA. The matrix interference study was commissioned at Eurofins Sacramento (SAC), using a Cape Fear River water sample as the matrix. The river water was collected from Bladen Bluffs (River Mile 84) on September 12, 2024, and shipped to SAC under chain-of-custody.

Three samples were prepared: one unamended and two amended. The amended river water samples were prepared by amending river water with 50 and 500 ng/L PFO6TeA and RHDA. The three samples were each analyzed at four dilutions: undiluted (1x dilution), 2-fold dilution, 5-fold

dilution and 10-fold dilution, for a total of 12 samples. A matrix spike analysis was also conducted on each of the 12 samples (Table 1). Note that the ideal recovery of a compound in a matrix spike is 100%, with a range of 70-130% typically considered acceptable.

RESULTS

PFO6TeA

Table 1 shows that PFO6TeA does not demonstrate matrix interference when analyzed in Cape Fear River water.

The amended concentrations of 50 and 500 ng/L are recovered well (92% to 136%) for all dilutions, with an average recovery of 101%. The matrix spikes are generally also recovered well for all dilutions, with an average recovery of 91%, although there are 2 recoveries of 55% and 185%, which may be outliers and are not considered excessive or systematic.

RHDA

Table 1 shows that RHDA does demonstrate matrix interference when analyzed in Cape Fear River water.

The amended concentrations of 50 and 500 ng/L are over-recovered (up to 460%) when the samples are undiluted or diluted 2-fold or 5-fold, and the over-recovery decreases as dilution increases. When the samples are diluted 10-fold, the amended recovery is acceptable.

Similarly, the matrix spikes are also over-recovered (up to 697%) when the samples are undiluted or diluted 2-fold or 5-fold, and the over-recovery again decreases as dilution increases. When the samples are diluted 10-fold, the matrix spike recovery is acceptable.

DISCUSSION

PFO6TeA did not demonstrate matrix interference in this study using Cape Fear River water. This observation is consistent with previous matrix interference studies which did not show significant matrix interference for monoprotic PFAS, as PFO6TeA is also a monoprotic PFAS.

Significant over-recovery was observed for RHDA both in terms of recovering the amended concentration and of recovering a matrix spike, indicating significant matrix interference by the Cape Fear River water matrix. The matrix interference was less when the sample matrix was diluted. This observation is consistent with previous matrix interference studies which showed

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significant matrix interference for diprotic PFAS, as RHDA is also a diprotic PFAS. The cause of the matrix interference is not fully understood.

RECOMMENDATIONS

Based on this study, Geosyntec recommends that environmental samples related to the Site can be analyzed for PFO6TeA, but not for RHDA. Further work needs to be done to understand and resolve the matrix interference observed for diprotic PFAS in environmental matrices related to the Site.

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Attachments

Table 1: Matrix Interference Study Results for PFO6TeA and RHDA

TABLE 1
MATRIX INTERFERENCE STUDY RESULTS FOR PFO6TeA AND RHDA
Chemours Fayetteville Works, North Carolina

Sample	Sample Amendment	Sample Dilution	PFO6TeA			RHDA		
			Measured Concentration (ng/L)	Recovery of Amendment (%)	Recovery of Matrix Spike (%)	Measured Concentration (ng/L)	Recovery of Amendment (%)	Recovery of Matrix Spike (%)
River water collected from the Cape Fear River at River Mile 84 on September 12, 2024	None	Undiluted	<1.1	--	85%	6.3	--	697%
		2-fold	<2.2	--	90%	4.9	--	411%
		5-fold	<5.4	--	68%	<4.6	--	267%
		10-fold	<27	--	84%	<23	--	128%
	50 ng/L PFO6TeA or RHDA	Undiluted	54	108%	75%	230	460%	*
		2-fold	46	92%	91%	190	380%	417%
		5-fold	68	136%	55%	130	260%	230%
		10-fold	55	110%	73%	40	80%	103%
	500 ng/L PFO6TeA or RHDA	Undiluted	400	80%	*	1,800	360%	*
		2-fold	470	94%	*	1,400	280%	*
		5-fold	470	94%	185%	890	178%	289%
		10-fold	450	90%	100%	410	82%	110%
			Average:	*101%*	*91%*	***Average:***	*260%*	*295%*

Notes:
< - result is below reporting limit
% - percent
ng/L - nanograms per liter
* - the analyte present in the sample is greater than 4 times the matrix spike concentration; therefore, control limits are not applicable

ATTACHMENT 3

Adsorbable Organic Fluorine Results

STW-LOC16-012920

Targeted Analysis

Analyte	Analyte Concentration (µg/L)	% of Analyte Mass That is Fluorine	Fluorine Concentration (µg/L)
PFO2HxA	2,974	54.054%	1,608
HFPO-DA	2,966	63.320%	1,878
Hydro-PSDA	629	47.482%	299
PFPrA	584	57.913%	338
PFO3OA	473	54.797%	259
DFSA	249	21.577%	54
PFMOAA	228	52.766%	120
PS Acid	154	55.612%	86
EVE	83	60.526%	50
R-PSDA	67	51.565%	35
PMPA	44	57.814%	25
PFO4DA	39	55.281%	22
NVHOS	37	50.984%	19
Hydro-PS Acid	26	57.308%	15
R-EVE	23	56.144%	13
MMF	21	27.133%	6
Hydro-EVE	20	62.135%	12
PEPA	18	61.058%	11
PFO5DA	15	55.621%	8
R-PSDCA	1	57.265%	1
TFA	17,645	49.987%	8,820
RHDA	306	52.345%	160
HFPO-TA	166	65.108%	108
PFO6TeA	17	55.872%	9
PFPeA	169	64.758%	109
PFHpA	18	67.842%	12
PFBA	8	62.135%	5
PFNA	1	69.597%	1
PFHxA	1	66.546%	1
		Total (µg/L)	**14,000**

Adsorbable Organic Fluorine Analysis

Sample	Measured AOF (µg/L)
STW-LOC16-012920 Sample #1	<10,000
STW-LOC16-012920 Sample #2	<10,000
STW-LOC16-012920 Sample #3	<10,000
STW-LOC16-012920 Sample #4	<10,000
STW-LOC16-012920 Sample #5	<10,000
STW-LOC16-012920 Sample #6	<10,000
Average (µg/L)	**<10,000**

STW-LOC16-042820

Targeted Analysis

Analyte	Analyte Concentration (µg/L)	% of Analyte Mass That is Fluorine	Fluorine Concentration (µg/L)
Hydro-PSDA	1,595	47.482%	757
DFSA	1,255	21.577%	271
HFPO-DA	985	63.320%	624
PFO2HxA	627	54.054%	339
PFPrA	506	57.913%	293
PFMOAA	449	52.766%	237
R-PSDA	342	51.565%	176
PFO5DA	89	55.621%	50
PFO3OA	82	54.797%	45
Hydro-PS Acid	60	57.308%	34
PFO4DA	55	55.281%	30
NVHOS	43	50.984%	22
PMPA	37	57.814%	21
R-EVE	26	56.144%	15
Hydro-EVE	22	62.135%	14
PEPA	14	61.058%	8.5
R-PSDCA	13	57.265%	7.4
EVE	0.4	60.526%	0.2
TFA	32,483	49.987%	16,237
RHDA	7,276	52.345%	3,809
HFPO-TA	790	65.108%	514
PFO6TeA	140	55.872%	78
PFPeA	48	64.758%	31.1
PFBA	43	62.135%	26.7
PFHpA	9	67.842%	5.8
PFHxA	8	66.546%	5.6
PFNA	2	69.597%	1.6
PFOA	1	59.649%	0.7
		Total (µg/L)	**24,000**

Adsorbable Organic Fluorine Analysis

Sample	Measured AOF (µg/L)
STW-LOC16-042820 Sample #1	12,000
STW-LOC16-042820 Sample #2	11,000
STW-LOC16-042820 Sample #3	12,000
STW-LOC16-042820 Sample #4	13,000
STW-LOC16-042820 Sample #5	13,000
STW-LOC16-042820 Sample #6	12,000
Average (µg/L)	**12,000**