The Chemours Company
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PFAS NON-TARGETED ANALYSIS AND METHODS INTERIM REPORT #13

Process and Non-Process Wastewater and Stormwater

Prepared by

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June 29, 2026

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TABLE OF CONTENTS

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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
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
PMEPA	perfluoromethoxyethylpropionic acid
RHDA	RSU/HFPO Diadduct
QToF	quadrupole time-of-flight

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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 13th 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 (GFD) 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 (CPWW) 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 (GFD and CPWW) 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 not detectable in samples of groundwater adjacent to the Cape Fear River or of the Cape Fear River itself (per the last step above), less abundant PFAS can also be expected to be undetectable. Consequently, the non-targeted program will then be considered complete.

The remainder of this 13th interim report consists of:

- Section 2: General Facility Discharge Samples;
- Section 3: Chemours Process Wastewater Samples;
- Section 4: Additional Progress
- Section 5: Summary; and
- Section 6: References.

2 GENERAL FACILITY DISCHARGE SAMPLES

Chemours submitted in the previous interim report (Chemours 2025b) that the non-targeted assessment of GFD samples is complete.

3 CHEMOURS PROCESS WASTEWATER SAMPLES

The 5 most abundant (CPWW-1 through CPWW-5) PFAS in 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.

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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.

RHDA was added to the suite of diprotic PFAS that is under investigation for improved analytical methodology due to matrix interference-induced over-recovery. A method meant to analyze diprotic and short-chain PFAS (the DSC Method) began development at Eurofins-Sacramento in 2025. Initial work with RHDA by the preliminary DSC Method showed that there was still some matrix enhancement present. Further development of the DSC Method showed that the matrix enhancement of RHDA had been resolved.

In the previous interim report (Chemours, 2025b), the next steps for RHDA were stated to 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.

Results achieved since December 2025 for RHDA are:

- A method detection limit study was conducted.
- A reporting limit of 2.0 nanograms per liter (ng/L) for RHDA was established.

Next steps for RHDA will be:

- Continuing to develop the DSC Method to see how broad the method's application may be, so that it can be assessed as a replacement method for Method 537M.

CPWW-3

A chemical formula of $C_8H_5F_{13}O_6S$ was initially proposed for CPWW-3. The molecular structure of $HO_3SCF_2CF_2OCF(CF_3)CF_2OCHFCF_2OCH$ 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 an Thermo Scientific Orbitrap Exploris 240 mass spectrometer (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.

The higher resolution fragmentation pattern obtained with the Orbitrap was sufficient to propose a structure for the revised empirical formula for CPWW-3. However, there is not a clear synthetic pathway to the proposed structure. Synthesis of the proposed structure is therefore not straightforward.

During the initial non-targeted 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 $HOOCCF(CF_3)OCF_2CF(CF_3)OCF_2CF_2COOH$ has been assigned to CPWW-4 ($C_9H_2F_{14}O_6$) via the synthesis of an authentic standard and comparison of the MS/MS spectrum of the standard to that of CPWW-4. CPWW-4 was named Diadduct Ester Acid (DAE Acid). DAE Acid is a diprotic PFAS and therefore is expected to exhibit significant matrix enhancement by Method 537M as with other diprotic PFAS.

A standard solution of DAE Acid was prepared by Chemours and sent to Eurofins-Sacramento. 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. DAE Acid was assessed for matrix enhancement during the refinement of the DSC method. As with RHDA, the refined DSC Method showed that there was no matrix enhancement of DAE Acid.

In the previous interim report (Chemours, 2025b), the next steps for DAE Acid were stated to 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.

Results achieved since December 2025 for DAE Acid are:

- A method detection limit study was conducted.
- A reporting limit of 2.0 ng/L for DAE Acid was established.

Next steps for DAE Acid will be:

- Continuing to develop the DSC Method to see how broad the method's application may be, so that it can be assessed as a replacement method for Method 537M.

CPWW-5 (PMEPA)

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 CPPW-5. In the previous interim report (Chemours, 2025b), synthesis of an authentic standard corresponding to the proposed molecular structure was underway. The next steps for CPWW-5 were stated to be:

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

Results achieved since December 2025 for CPWW-5 are:

- The proposed 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-5.
- An alternative molecular structure of CF_3-O-CF_2-CF_2-O-$CF(CF_3)$-COOH was proposed
- After multiple attempts, the alternative proposed molecular structure was synthesized.
- The retention time and the MS/MS spectrum of the authentic standard was compared to the MS/MS spectrum of CPWW-5.
    - The retention times of the peaks on the liquid chromatograph were identical (Figure 1).
    - The fragmentation patterns of the largest ion (with a mass-to-charge ratio of 344.9620) in the mass spectra were identical (Figure 2).
- CPWW-5 has therefore been identified as 2,3,3,3-tetrafluoro-2-[1,1,2,2-tetrafluoro-2-(trifluoromethoxy)ethoxy]propanoic acid.
    - This PFAS can also be identified as perfluoromethoxyethylpropanoic acid (PMEPA) and has a CAS number of 1026231-58-2.

Next steps for PMEPA (CPWW-5) will be:

- Analysis of the purity of the authentic standard and generation of a certificate of analysis.
- Assessment of PMEPA analysis by an existing analytical method (EPA Method 537Mod Max).
- Execution of a method detection limit study to establish a reporting limit.

- Execution of a matrix interference study to assess the quantification of PMEPA in an environmental matrix related to the Facility.

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 (Chemours, 2025b), the next steps for CPWW-6 through 10 were stated to be:

- A synthetic pathway will be developed for CPWW-7.
  - 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.

Results achieved since December 2025 for CPWW-6 through 10 are:

- Structures have been proposed for CPWW-8, -9 and -10
  - CPWW-8: CF_3-CF_2-CF_2-O-CF(CF_3)-CF_2-CF_2-COOH
  - CPWW-9: CF_3-CF_2-CF_2-O-CFH-COOH
  - CPWW-10: HOOC-CF_2-CF_2-O-CF(CF_3)-CF_2-O-CFH-COOH
- Synthesis of the proposed structure for CPWW-9 was completed
  - The MS/MS spectrum of the synthesized standard did not match the MS/MS spectrum of CPWW-9
  - Work is underway to propose a revised structure for CPWW-9
- Synthesis of the proposed structures for CPWW-8 and -10 has commenced

Next steps for CPWW-6 through 10 will be:

- Continued synthesis of standards for the proposed structures for CPWW-8 and -10
- Continued development of a proposed molecular structure for CPWW-6
- Development of a revised proposed molecular structure for CPWW-9
- Continued development of a synthetic pathway for CPWW-7

4 ADDITIONAL PROGRESS

As noted above, the fragmentation of many potential unknown PFAS during the initial analysis by the QToF mass spectrometer resulted in very few detectable fragments, making the proposal of structures difficult. Re-analysis was conducted using the Orbitrap mass spectrometer, which provides a higher mass resolution than that provided by the QToF mass spectrometer used in the initial analysis. While the results for some potential unknown PFAS was improved with the Orbitrap (e.g., the Orbitrap results for CPWW-3 resulted in a revised empirical formula and consequently a proposed structure), the Orbitrap results did not add significant information for other potential unknown PFAS.

In order to see if more information could be obtained from new samples, 4 new samples were collected from Location 16 (Chemours Process Wastewater) on August 11 and 28 and October 1 and 3, 2025 and analyzed with the Orbitrap mass spectrometer. Results were broadly similar to the results from the original set of samples. Fragmentation data of the new samples did not add significant information that might lead to the proposal of structures for potential unknown PFAS.

5 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 were assigned tentative empirical formulas and prioritized by abundance (peak height) for further analysis.

<u>Summary of General Facility Discharge (GFD) Samples</u>

Chemours submitted in the previous interim report (Chemours 2025b) 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) have been or are being assessed:

- 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-2: Identified as EVE Acid (a known PFAS).
- 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 (PMEPA): Identified by comparison to authentic standard. Next steps include assessment of analysis by EPA Method 537M.
- CPWW-6 through CPWW-10:
    - Fragmentation of CPWW-6 by the Orbitrap (as well as by the QToF) resulted in very few detectable fragments, and a proposed structure is still under development.
    - 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 a synthetic pathway is not clear.
    - A structure was proposed for CPWW-9 and the standard was synthesized. The MS/MS spectrum of the synthesized standard did not match the MS/MS spectrum of CPWW-9; therefore, work is underway to propose a revised structure.
    - Structures have been proposed for CPWW-8 and -10 and synthesis of the proposed structures has commenced.

6 REFERENCES

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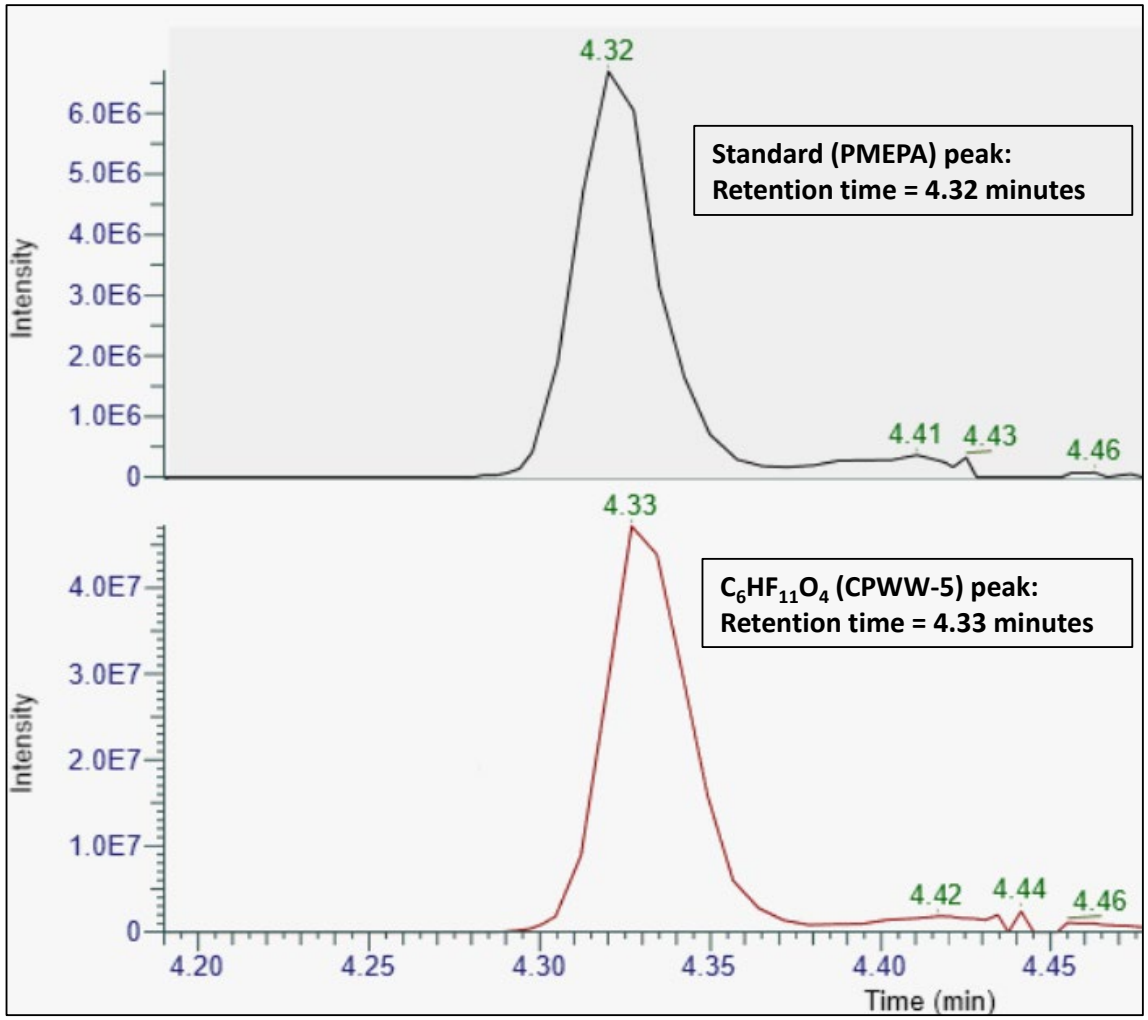

Notes:
PMEPA - perfluoromethoxyethylpropanoic acid

Retention Times for PMEPA Authentic Standard and Unknown $C_6HF_{11}O_4$ (CPWW-5)
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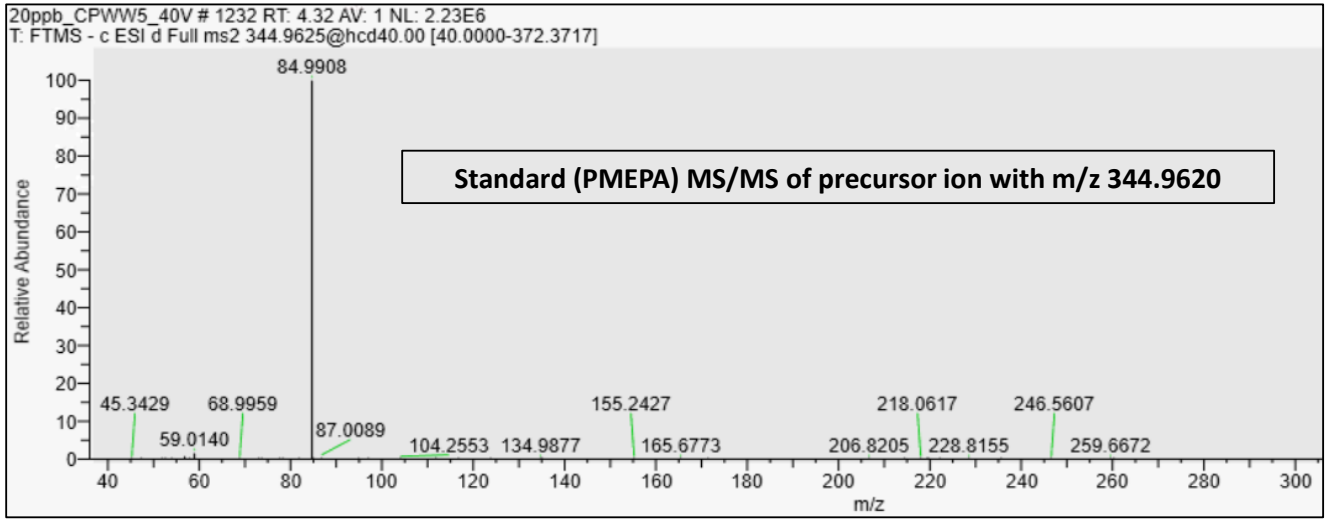
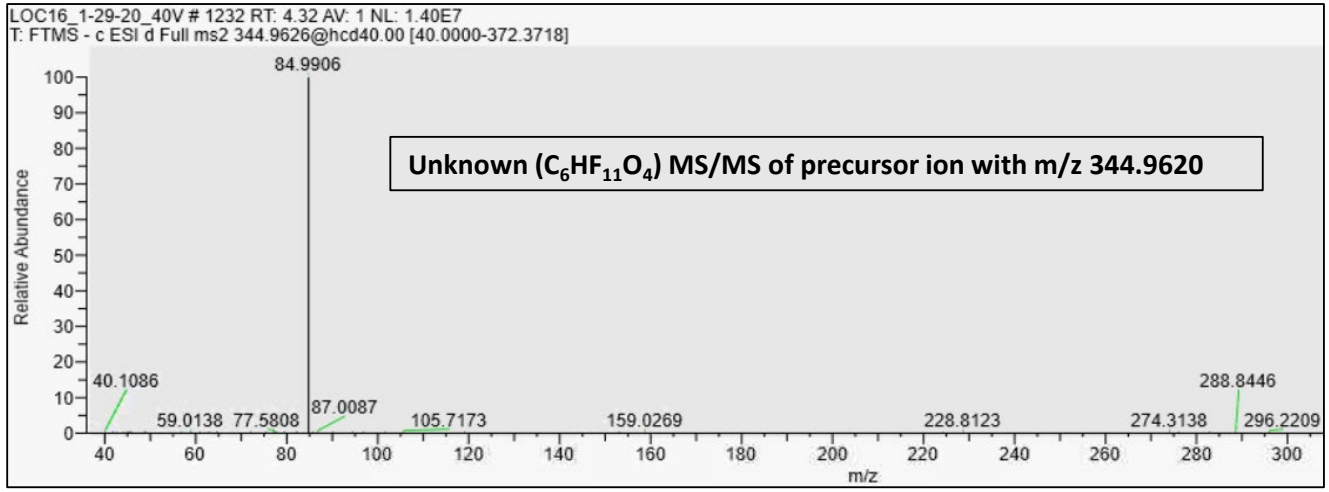
Figure

1





Notes:

EIC - extracted ion chromatogram for mass-to-charge ratio of 344.9620
m/z - mass to charge ratio
PMEPA - perfluoromethoxyethylpropanoic acid

Tandem Mass Spectra for PMEPA Authentic Standard and Unknown $C_6HF_{11}O_4$ (CPWW-5)
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Figure **2**

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