

# Putting the (Atmospheric) Pressure on Forever Chemicals: A Targeted Analysis of GC Amenable PFAS using APGC–MS/MS

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## Sample Analysis

A variety of sample extracts were run using the APGC-MS/MS method. The food packaging samples that were tested did not contain any of the targeted PFAS, but the environmental samples (water, soil and fish tissues collected at various sites) did have detectable and quantifiable amounts of GC amenable PFAS. Fluorotelomer alcohols (FTOHs) were the most predominantly detected amongst the samples, with wastewater and ski wax impacted soil containing the highest levels. Figure 3 demonstrates 6:2 FTOH detections in wastewater, soil and shrimp.

Sample extract post spikes were quantified to determine the accuracy of the method, with an example being shown in Figure 4 of a brown paper food wrapper. For most compounds, the calculated concentration matches the sample spike accurately. The acrylates (FTAc) and acetates (FTOAc) are known to undergo atmospheric degradation which can explain why these compounds quantified lower than expected.

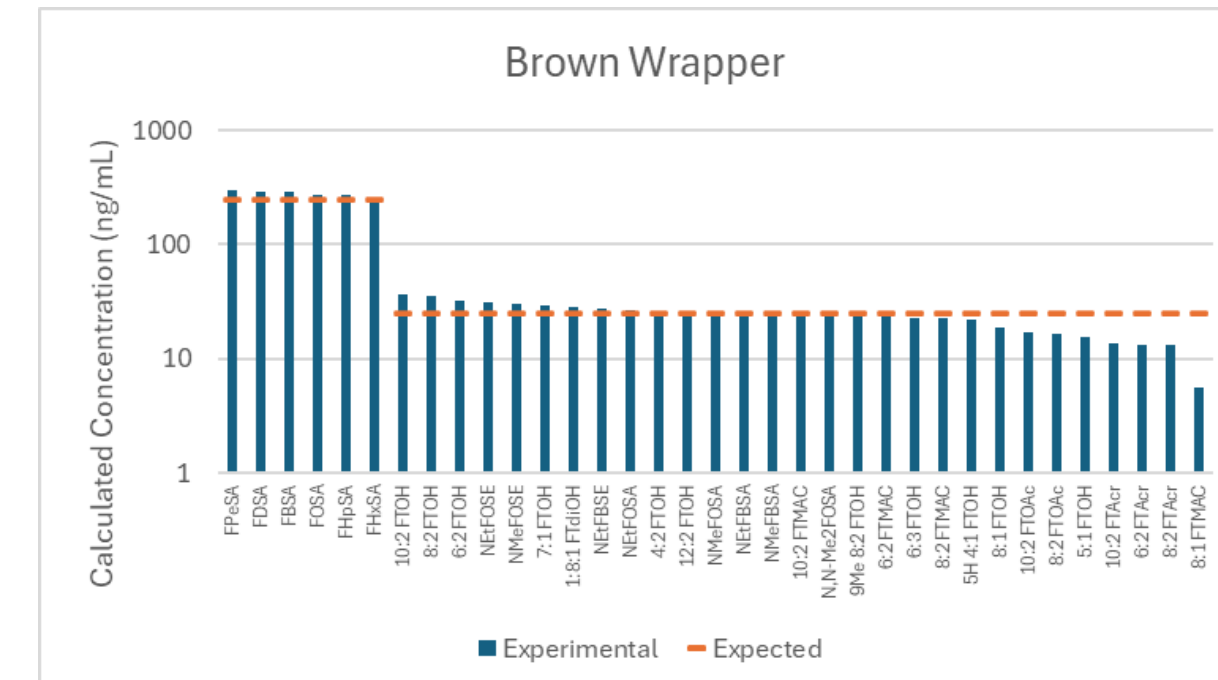


Figure 4. Quantitation of PFAS spiked into a brown wrapper food packaging extract. Blue bars indicate the calculated concentration and orange dashes indicate the expected concentration spiked into the sample.

## Results and Discussion

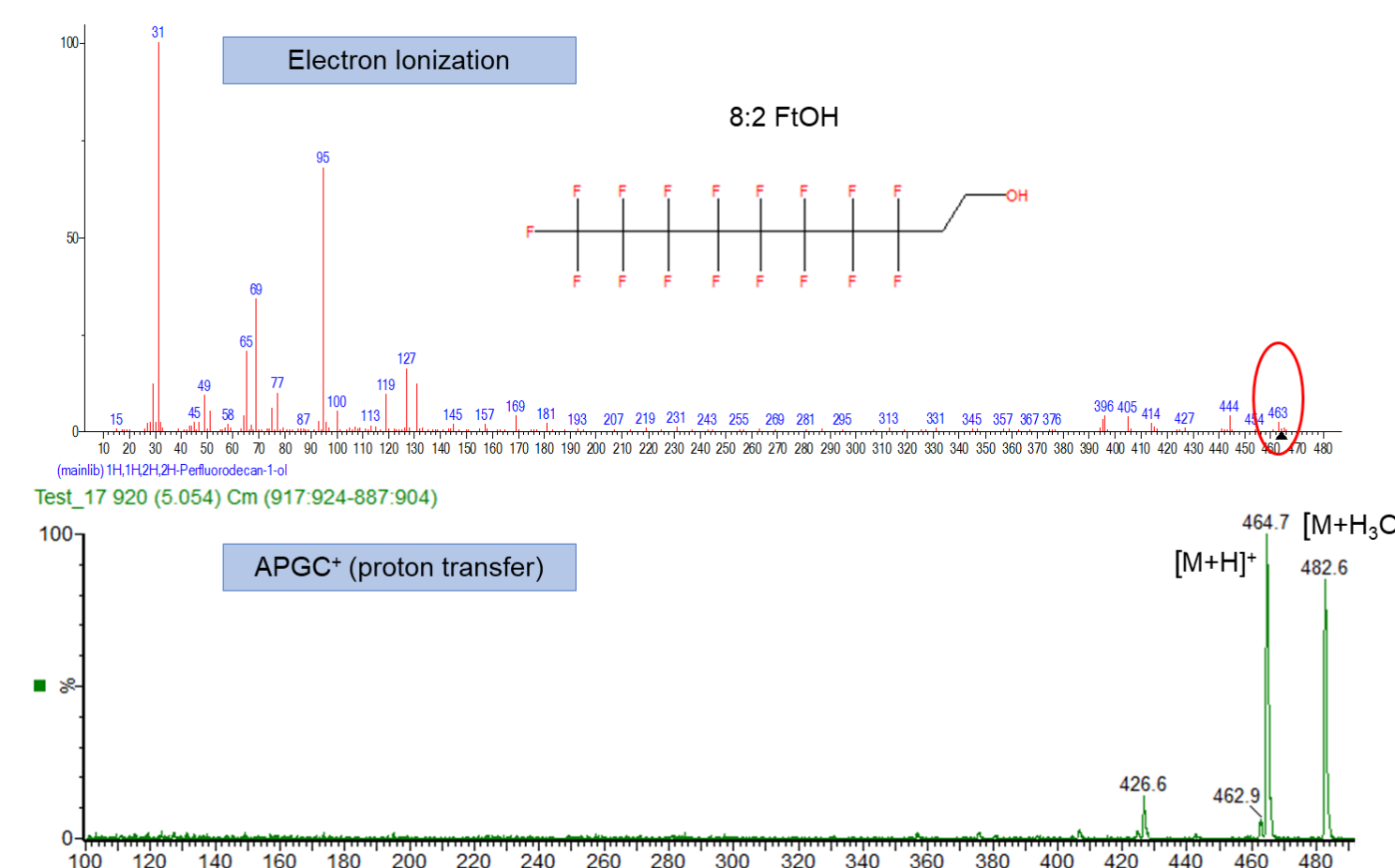


Figure 1. Mass spectrum of 8:2 FTOH using electron ionization (top) and APGC ionization (bottom).

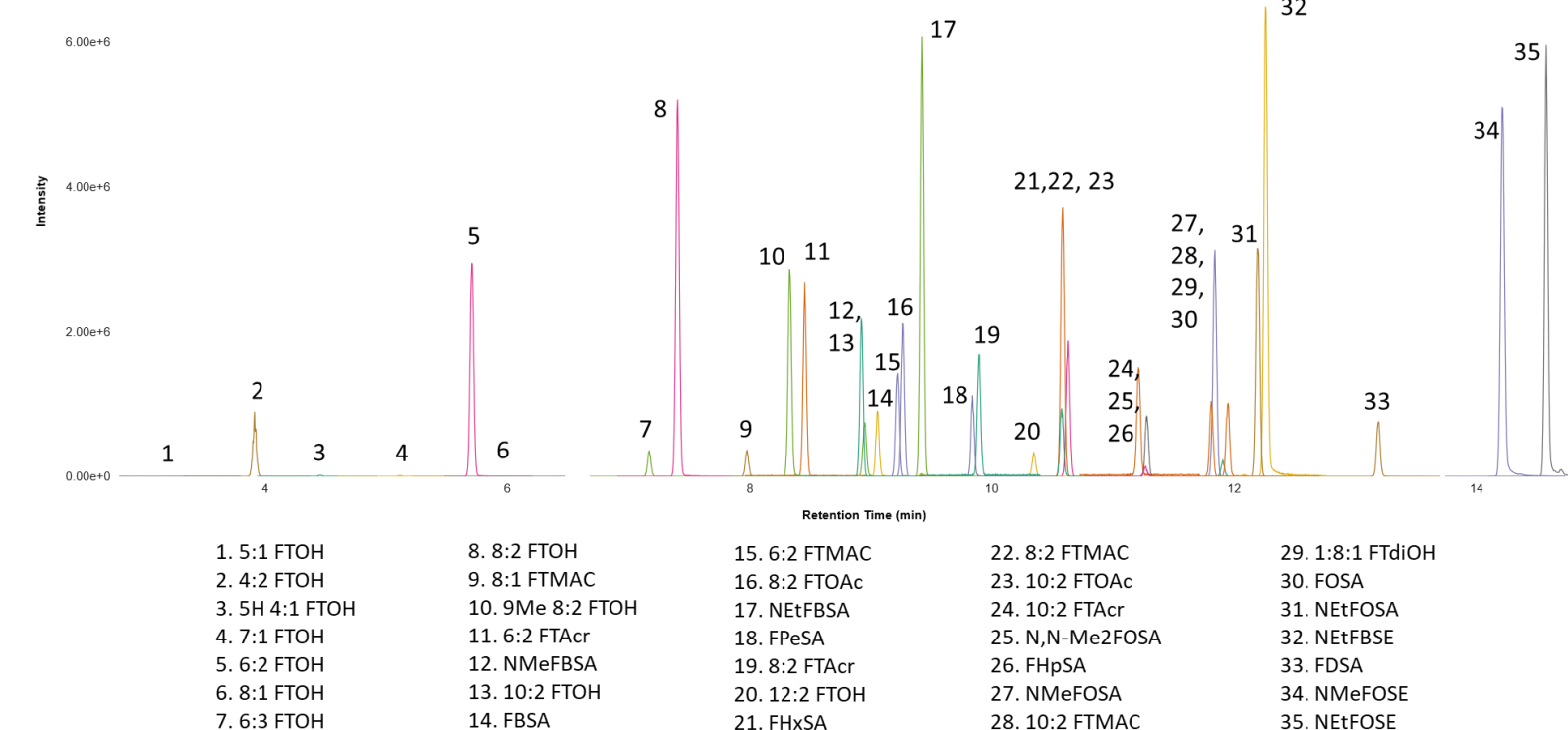


Figure 2. Chromatogram of extracted paper board french fry box post spiked with 25 ng/mL PFAS.

Compound	LLOQ (ng/mL)	LLOQ S/N	Calibration Range (ng/mL)
4:2 FTOH	0.1	64	0.1 - 50
6:2 FTOH	0.05	104	0.1 - 50
8:2 FTOH	0.05	182	0.05 - 50
10:2 FTOH	0.05	19	0.05 - 50
12:2 FTOH	0.1	49	0.1 - 50
6:3 FTOH	0.1	55	0.1 - 50
1:8:1 FtDiOH	1	30	1 - 50
9Me 8:2 FTOH	0.05	583	0.05 - 50
5:1 FTOH	10	27	N/A
5H 4:1 FTOH	2.5	18	2.5 - 50
7:1 FTOH	5	30	N/A
8:1 FTOH	5	16	2.5 - 50
N-MeFBSA	0.025	11	0.05 - 50
N-EtFBSA	0.005	26	0.05 - 50
NMeFOSA	0.01	20	0.05 - 50
NEtFOSA	0.01	45	0.05 - 50
N,N-Me2FOSA	0.5	409	0.5 - 50
N-EtFBSE	0.025	20	0.05 - 50
N-MeFOSE	0.01	34	0.05 - 50
N-EtFOSE	0.01	25	0.05 - 50
8:2 FTAc	0.05	20	0.05 - 50
8:2 FTAc	0.05	13	0.05 - 50
10:2 FTAc	0.05	11	0.05 - 50
6:2 FTMAC	0.025	112	0.05 - 50
8:2 FTMAC	0.005	27	0.05 - 50
10:2 FTMAC	0.005	52	0.05 - 50
8:1 FTMAC	0.025	241	0.1 - 50
8:2 FTOAc	0.01	48	0.05 - 50
10:2 FTOAc	0.01	90	0.05 - 50
FBSA	5	23	5 - 500
FPeSA	1	31	1 - 500
FHxSA	1	90	1 - 500
FHpSA	1	42	1 - 500
FOSA	1	68	1 - 500
FOSA	2.5	318	2.5 - 500

Table 1. LLOQ for each PFAS included in the APGC-MS/MS method.

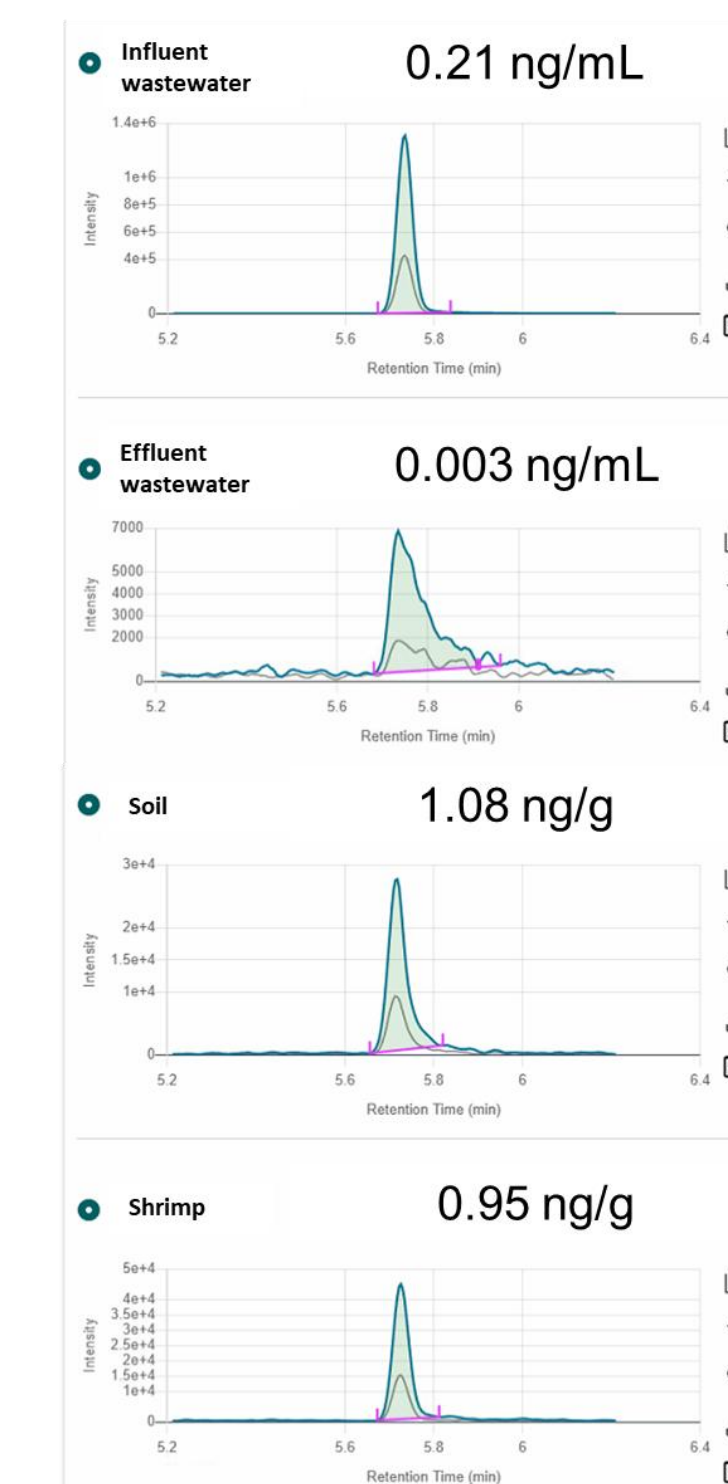


Figure 3. Examples of 6:2 FTOH detected in influent wastewater, effluent wastewater, soil and shrimp sample extracts.

## Method Development and Performance

A method was developed and optimized for the analysis of 35 PFAS using APGC+ ionization. The benefit of using APGC for the analysis of these types of compounds is demonstrated in Figure 1, showing the intact [M+H]+ molecular ion for 8:2 FTOH (m/z 465), compared to the completely fragmented spectra resulting from EI. This allows for increased sensitivity and specificity of the APGC technique.

The sensitivity achieved using this method is highlighted in Table 1, showing the lower limit of quantitation (LLOQ) that was established for each compound. Most PFAS included in the method had an LLOQ of 0.025 – 0.1 ng/mL. The sulfonamides ranged from 1 – 5 ng/mL LLOQ, whereas the x:1 FTOH compounds were in the range of 5 – 10 ng/mL.

An example of the chromatographic separation is demonstrated in Figure 2, highlighting all PFAS post-spiked into a food packaging extract (paper board French fry box). PFAS were spiked at 25 ng/mL to demonstrate the individual PFAS limit to be enacted in food packaging in Europe beginning August 2026.

## Introduction

Analysis of per- and polyfluoroalkyl substances (PFAS) is routinely performed by laboratories around the globe using liquid chromatography coupled to mass spectrometry. This technique is sufficient to cover an extensive list of compounds but is not well suited for analysis of more volatile classes of PFAS, such as fluorotelomer alcohols and acrylates, among others. As focus continues to expand beyond the routinely monitored compounds and matrices, the addition of gas chromatography coupled to mass spectrometry is becoming an important tool to provide a more comprehensive overview of the PFAS composition of a sample. Many of the GC amenable PFAS can be present in a variety of sample types from environmental to food and food packaging.

## Methods

### Sample Preparation

Water (surface, ground, influent and effluent), soil and fish samples were extracted using EPA Method 1633A<sup>1</sup> and previously analyzed using LC-MS/MS.<sup>2,3</sup> Full details of the sample preparation can be found in the referenced application notes and followed the EPA 1633A protocol. Final samples were in a solution of approximately 94% methanol, 4% water, 1% ammonium hydroxide and 0.6% acetic acid. Calibration standards were made in the same sample composition.

Food packaging was collected from several locations and was either a paper or paper board material. A 10cm x 10 cm portion of each packaging material was cut into small pieces. The sample was placed into a 50 mL centrifuge tube and 20 mL of methanol containing 1% ammonium hydroxide (v/v) was added. The samples were sonicated for 1 hour and an aliquot was removed for analysis.

### APGC-MS/MS Conditions

**GC System:** Agilent 8890 with ALS Autosampler  
**Column:** Rtx-200 30m x 0.25mm x 0.50 μm  
**Inlet Liner:** Siltek Deactivated Straight with Wool, 4.0 mm x 6.5 x 78.5  
**Carrier Gas:** Nitrogen  
**Flow Rate:** 2 mL/min  
**Injection:** Pulsed Split (10:1)  
**Injection Port Temperature:** 240° C  
**Injection Volume:** 1 μL  
**Makeup Gas:** Nitrogen at 300 mL/min  
**Transfer Line Temperature:** 310° C  
**Oven Program:**

Rate (°C/min)	Temperature (°C)	Hold (min)	Total Time (min)
Initial	40	1	1
12	200	0	14.3
50	310	3.5	20

**MS System:** Xevo™ TQ Absolute Mass Spectrometer  
**Ionization:** API+, Proton Transfer using water  
**Corona Current:** 1.0 μA  
**Cone Gas Flow:** 250 L/hr  
**Auxiliary Gas Flow:** 150 L/hr  
**Software:** waters\_connect™ for Quantitation

## Conclusions

- A sensitive and accurate method was created and optimized to analyze GC amenable PFAS using APGC-MS/MS, an atmospheric pressure ionization technique.
- Sample extraction and final sample composition was adapted from EPA 1633 to simplify the analysis of samples already prepared and characterized for LC-MS/MS analysis and provide a complimentary analysis using gas chromatography.
- GC amenable PFAS were detected in environmental samples, a class of PFAS compounds that would not be characterized using only LC-MS/MS analysis.

### References

- US Environmental Protection Agency. EPA 1633A: Analysis of Per- and Polyfluoroalkyl Substances (PFAS) in Aqueous, Solid, Biosolids, and Tissue Samples by LC-MS/MS. January 2024.
- K Organtini, K Rosnack, C Plummer, P Hancock, O Burt. Analysis of Per- and Polyfluoroalkyl Substances (PFAS) in Accordance with EPA 1633 Part 2: Analysis of Aqueous Matrices. Waters Application Note 720008143. 2024.
- K Organtini, K Rosnack, C Plummer, P Hancock, O Burt. Analysis of Per- and Polyfluoroalkyl Substances (PFAS) in Accordance with EPA 1633 Part 2: Analysis of Soil and Tissue. Waters Application Note 720008230. 2024.