

Extending PFAS Analytical Workflows with APGC-MS/MS Analysis: Analysis of Environmental and Food Packaging Samples

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Abstract

An atmospheric pressure gas chromatography tandem quadrupole mass spectrometry (APGC-MS/MS) method was evaluated for the analysis of 35 GC-amenable per- and polyfluoroalkyl substances (PFAS) in environmental and food packaging matrices. The method successfully identified fluorotelomer alcohols (FTOHs) in wastewater and soil samples, while comparison with LC-MS/MS data provided evidence of precursor transformation to perfluoroalkyl carboxylic acids (PFCAs) during wastewater treatment. Analysis of food packaging materials detected none of the targeted PFAS, and post-spike experiments demonstrated accurate quantification at concentrations relevant to current and emerging regulatory limits. Overall, the method proved to be a sensitive and complementary tool to LC-MS/MS for PFAS characterization, enabling more comprehensive assessment of samples.

Benefits

- An APGC-MS/MS method for the analysis of 35 GC-amenable PFAS allowing for characterization of PFAS

samples beyond the suite of commonly targeted LC-MS/MS compounds, enabling a more complete assessment of PFAS burden

- Sensitive and robust analysis to detect GC-amenable PFAS in complex sample matrices, supporting reliable data generation across diverse sample types
- Provides complementary information to LC-MS/MS workflows by identifying PFAS precursor compounds and transformation pathways, helping laboratories better understand contamination sources, environmental fate, and treatment outcomes
- Enables confident monitoring of food contact materials at concentrations well below emerging European regulatory limits, supporting compliance testing and regulatory preparedness
- Expands laboratory capabilities beyond routine targeted PFAS analysis, allowing customers to deliver comprehensive investigations, differentiate their analytical services, and generate deeper insight into PFAS occurrence in environmental samples

Introduction

While liquid chromatography coupled with mass spectrometry (LC-MS) remains the dominant approach for routine PFAS analysis, it is not well suited for all PFAS classes. A subset of PFAS compounds, particularly volatile and semi-volatile species, exhibit chromatographic and analytical characteristics that favor gas chromatography (GC)-based methods. These include fluorotelomer alcohols (FTOHs), fluorotelomer acrylates (FTAcres), fluorotelomer methacrylates (FTMACs), fluorotelomer acetates (FTOAcS), and several related precursor compounds. Other PFAS classes, such as sulfonamides and sulfonamidoethanols, can be successfully analyzed using either LC or GC platforms.

Many GC-amenable PFAS are recognized as precursor compounds because they can undergo environmental and biological transformation to form PFCAs and perfluoroalkyl sulfonates (PFSA)s, the terminal degradation products that have been the focus of most PFAS monitoring and toxicological studies. In addition to their role in PFAS manufacturing processes, these compounds have historically been incorporated into commercial products, including food contact materials and textile treatments, where they provide oil, stain, and water-resistant properties.

The environmental behavior of these precursor compounds differs from that of many ionic PFAS due to their

increased volatility, which enables atmospheric transport and deposition over long distances. As a result, they can contribute to PFAS contamination through pathways that are not fully captured by conventional LC-based analytical approaches. Although toxicological information for many of these compounds remains limited, their potential health impacts and their ability to generate persistent PFCA and PFSA transformation products have increased interest in their measurement and environmental occurrence.

FTOH, FTAcr, FTMAC, PFSA, N-xFOSA, and N-xFOSE compounds are known to have been used as raw materials in the synthesis of surface protectants, such as those used to grease proof paper and paperboard food packaging materials.¹ In recent years, global efforts have been made to phase out and ban their use in food contact materials. In 2020, the US Food and Drug Administration (FDA) announced the voluntary removal of 6:2 FTOH for use in food contact materials with complete removal of all packaging containing this compound in 2025.^{2,3} Furthermore, in 2025, 35 Food Contact Notifications (FCNs) for PFAS compounds that declare the use of a substance safe in food contact materials were revoked by the FDA.⁴ Concurrently, from August 12, 2026, Regulation (EU) 2025/40 restricted PFAS in food contact packaging enforcing strict limits for individual targeted PFAS, the sum of targeted PFAS, and total PFAS content.⁵ In accordance with this regulation, a limit of 25 ppb was set for individual targeted PFAS in any food packaging material.

As discussed in a previous application note, atmospheric pressure gas chromatography coupled with tandem quadrupole mass spectrometry (APGC-MS/MS) provides a sensitive approach for the determination of volatile and semi-volatile PFAS.⁶ The development of the method and the overall method performance are discussed there. The work presented here extends that methodology to the analysis of authentic environmental and food packaging samples, demonstrating the value of GC-MS/MS techniques for more comprehensive PFAS characterization and improved understanding of PFAS sources and transformation pathways.

Experimental

Sample Preparation

Native PFAS standards were purchased from Wellington Laboratories and AccuStandard. Isotope labeled internal standards were purchased from Wellington Laboratories.

Environmental samples were previously extracted using EPA Method 1633A⁷ and analyzed using LC-MS/MS.^{8,9} Full details of the sample preparation can be found in the referenced application notes and followed the EPA

1633A protocol. Final sample extracts were estimated to be in a solution of 94% methanol, 4% water, 1% ammonium hydroxide, and 0.6% acetic acid. Calibration standards were prepared from 0.01 to 50 ng/mL for all compounds, except the sulfonamides at 0.10 to 500 ng/mL, in the same solvent composition.

Food packaging was collected from several locations and was either paper or paperboard material. Examples of packaging tested include a french fry box, paper sandwich wrappers, paper plates, paper coffee cups, and a bakery bag. A 10 x 10 cm portion of each packaging material was cut into small pieces and weighed to determine extracted mass. 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.

Proton transfer conditions were established in the APGC source using three vials of water in the source door tray. Under proton transfer conditions, water vapor present in the APGC source facilitates the formation of protonated analyte ions, improving sensitivity for MS/MS detection. Each vial was capped with a small piece of capillary tubing pierced through the vial cap.

Isopropyl alcohol (IPA) and methanol were used as wash A and B, respectively, in the GC autosampler. Both pre and post injection washes were performed. IPA was found to help reduce carryover of the sulfonamide compounds.

Method Conditions

GC Conditions

GC system:	Agilent® 8890 Gas Chromatography System
Autosampler:	7693A Automatic Liquid Sampler
Column:	Restek® Rtx-200 30 m x 0.25 mm ID, 0.50 µm Column
Inlet liner:	Restek Siltek Deactivated Straight with Wool (4.0 x 6.5 x 78.5 mm)

Carrier gas:	Nitrogen
Flow rate:	2 mL/min
Injection:	Pulsed Split (40 psi until 0.6 minutes)
Split ratio:	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 Conditions

MS system:	Xevo™ TQ Absolute Mass Spectrometer
Ionization mode:	API+
Ionization mechanism:	Proton transfer (water)

Collision gas:	Argon
Source temperature:	150 °C
Corona current:	1.0 µA
Cone gas flow:	250 L/hr
Auxiliary gas flow:	150 L/hr
MRM method:	See Appendix for Full MRM Method details

Data Management

Software:	waters_connect™ for Quantitation Software
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Results and Discussion

The applicability of the method developed for GC amenable PFAS⁶ was evaluated in a variety of environmental and food packaging sample matrices. An example chromatogram of the full panel of PFAS targeted in all samples is shown in Figure 1, demonstrating a wastewater sample extract post spiked with 25 ng/mL (in vial concentration). This sample contained a significant amount of 6:2 FTOH (peak 5), therefore, the peak response shown in this example is elevated, relative to other compounds shown. Otherwise, this example demonstrates that the presence of sample matrix did not affect the peak shape or resolution.

Environmental Sample Analysis:

Of the environmental samples tested, only wastewater and soil samples contained detectable levels of the targeted GC-amenable PFAS, consisting predominantly of the fluorotelomer alcohols.

Wastewater treatment plants are known to be a significant source of volatile and semi-volatile PFAS emissions.¹⁰

Figure 2A demonstrates the results of the analysis of influent and effluent wastewater samples. 6:2, 8:2, 10:2 and 12:2 FTOH were quantified in the influent wastewater in the range of 6.3–219 ng/L (sample concentration). Only 6:2 and 8:2 FTOH were observed above the detection limit in the effluent and were quantified at 2.9 and 2.3 ng/L, respectively. This suggests the substantial removal of the fluorotelomer alcohols during the treatment process. When considering the previous analysis of the same samples using LC-MS/MS (Figure 2B), it appears as though the FTOH compounds in the influent water are transforming into their PFCA end products. In the LC-MS/MS analysis, the concentration of the C4–C10 PFCAs either increased or appear to remain relatively unchanged in the effluent water when compared to the influent water. A typical pathway of degradation for 6:2 FTOH is shown in Figure 2C, with the end products being C4–C6 PFCAs. These observations suggest that precursor transformation occurring during treatment may contribute to PFCA concentrations observed in the effluent. Without the ability to fully characterize the samples using both LC-MS/MS and GC-MS/MS, it would not be possible to identify the incoming precursor PFAS contributing to the PFCA effluent levels.

In addition to the FTOHs, the GC analysis of the wastewater samples identified N-MeFOSE and N-EtFOSE in both the influent and effluent water. Figure 3 compares the detected concentrations using both APGC-MS/MS and LC-MS/MS analysis. Calculated concentrations in the influent water samples were consistent from both analysis techniques, confirming that APGC-MS/MS and LC-MS/MS provide complementary results for compounds measurable by both methods. Due to the sensitivity of the APGC technique being higher for these compounds, both were detected in the effluent water. When using LC-MS/MS, the levels were below the detection limit, and therefore not detected.

Soil samples were collected at two different ski facilities at locations where ski wax application is known to have occurred. One site was a downhill ski slope (D samples), and the second site was a cross-country ski track (C samples). Studies on the impact of air quality during application of fluorinated ski waxes have identified predominantly 6:2 and 8:2 FTOH.¹¹ The APGC-MS/MS analysis of these samples identified 6:2, 8:2 and 10:2 FTOH in the soil, as detailed in Table 1, with known locations of wax application indicated. The downhill site had approximately an order of magnitude higher levels of 6:2 FTOH than the cross-country site. At the downhill site, levels were highest at the race start line (D2) and the wax application area located right before the start line (D4). The finish line (D3) had the third highest levels, which could correlate to wax abrading off the skis as the skier begins to stop. At the cross-country site, there are elevated levels at one of the known wax application sites (C1), but the levels are fairly consistent at all sites, even those farthest from the wax application sites (C8). This could indicate that the presence of FTOHs at this location may be more attributed to atmospheric deposition than ski wax use.

To assess the accuracy of the method, soil and surface water extracts were post spiked with the equivalent of 2.5–25 ng/mL (in vial concentration) of the targeted PFAS. Figure 4 compares the average calculated concentration of the post spiked samples compared to the expected concentration for each compound. The FTOH, N-xFOSA, and N-xFOSE compounds quantified within good accuracy to the expected concentration. The FTAc, FTMAC, and FTOAc compounds had lower than expected concentrations when post spiked into the samples. These compounds are intermediate compounds in the oxidative pathway that eventually terminates in PFCAs and therefore most likely experienced some oxidative degradation while sitting in the GC autosampler. The sulfonamides (FSA) had a high calculated concentration which was due to inlet carryover in these samples.

Food Packaging Sample Analysis

Food packaging materials for this study were obtained from various sources in the United States and consisted of paper and paperboard materials. Upon analysis using the APGC-MS/MS method, none of the samples had any detectable levels of the 35 targeted PFAS. Although this was a limited sampling scope confined to seven different packaging materials, the findings are compliant with current FDA restrictions, at least for the targeted PFAS in this study.

The extracted packaging samples were post spiked at 25 ng/mL (ppb) to evaluate the method's use for compliance testing in accordance with the EU food contact packaging regulation. Chromatograms of selected PFAS at 25 ng/mL in different paper and paperboard packaging materials can be seen in Figure 5. All PFAS had excellent response at this level demonstrating that this method would confidently be able to monitor food packaging materials at well below the required limit of 25 ng/mL.

The accuracy of the method was also tested by comparing the calculated concentration of the post spiked packaging samples with the expected 25 ng/mL (250 ng/mL for sulfonamides) concentration. Figure 6 demonstrates this comparison in the brown paper wrapper sample, with all compounds showing excellent accuracy, except for the FTAc, FTMAC and FTOAc compounds which, like the environmental samples, quantified at lower concentrations than expected, potentially due to oxidation.

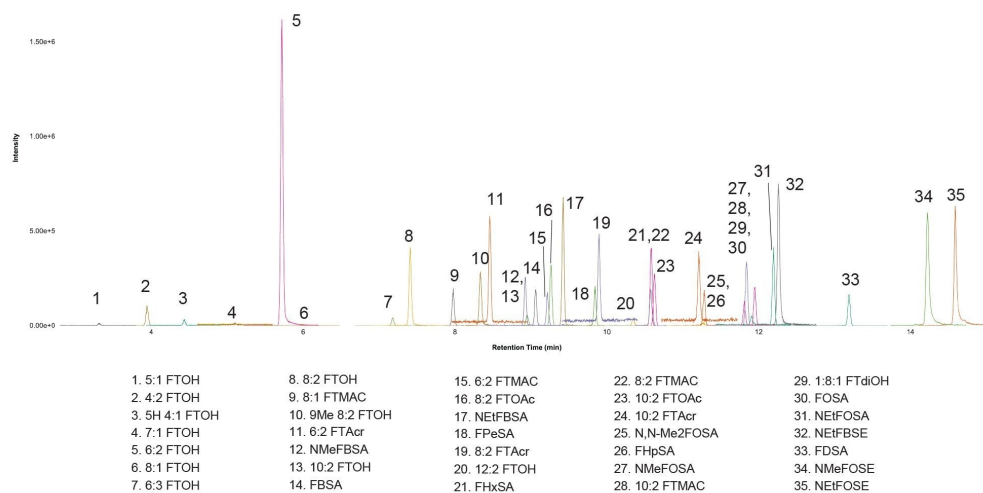
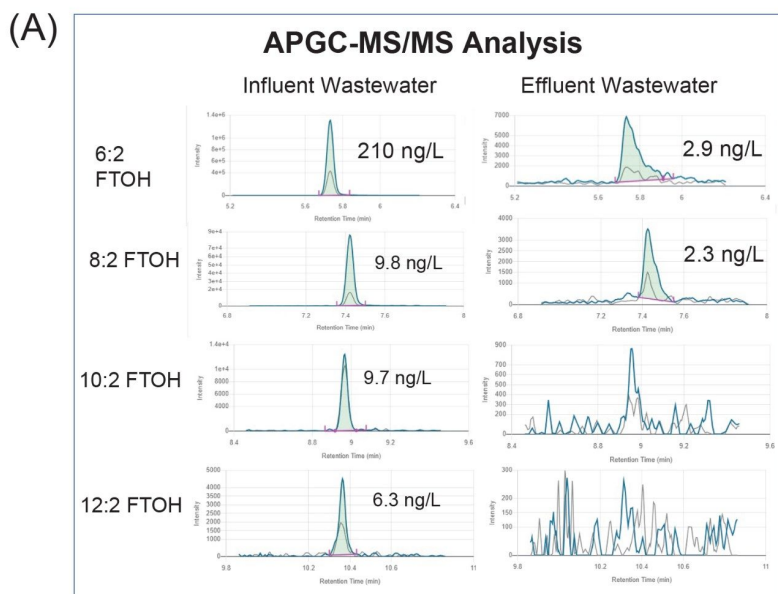


Figure 1. Influent wastewater extract spiked with 25 ng/mL (250 ng/mL FBSA, FPeSA, FHxSA, FHpSA, FOSA, FDSA) GC amenable PFAS compounds.



(B) **LC-MS/MS Analysis**

	Influent	Effluent
PFBA	34.0	26.5
PFPeA	14.3	27.3
PFHxA	21.1	84.3
PFHpA	4.9	5.48
PFOA	11.3	14.6
PFNA	1.1	1.35
PFDA	1.5	3.51
PFDoDA	0.49	-
PFTreDA	BLQ	-
PFBS	47.9	50.0
PFPeS	0.53	0.26
PFHxS	41.6	8.92
PFOS	7.4	5.85
GenX	0.77	0.63
6:2 FTS	11.7	5.32
FOSA	0.31	0.21
N-MeFOSAA	2.59	-
N-EtFOSAA	1.31	-
N-MeFOSE	3.6	-
N-EtFOSE	3.3	-
3:3 FTCA	11.0	5:3 FTCA 4.18
5:3 FTCA	68.4	-
7:3 FTCA	6.5	-
Total PFAS	295.7	Total PFAS 238.4

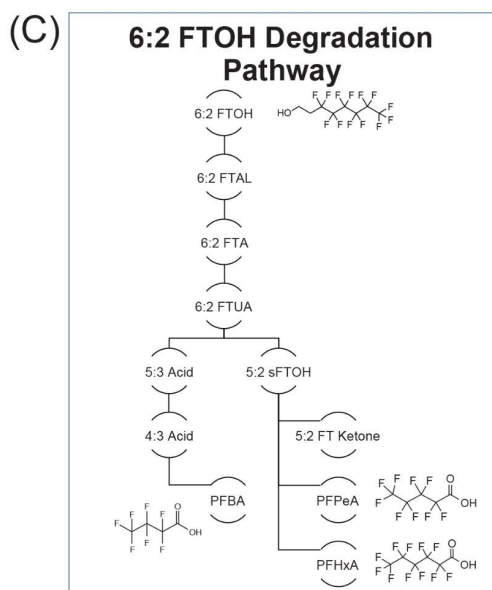


Figure 2. FTOH compounds detected in wastewater samples using APGC-MS/MS analysis. Comparing the previous LC-MS/MS analysis results and known degradation pathway of the FTOH compounds indicate the increased PFCA results may be due to the degradation of the FTOHs detected in the samples.

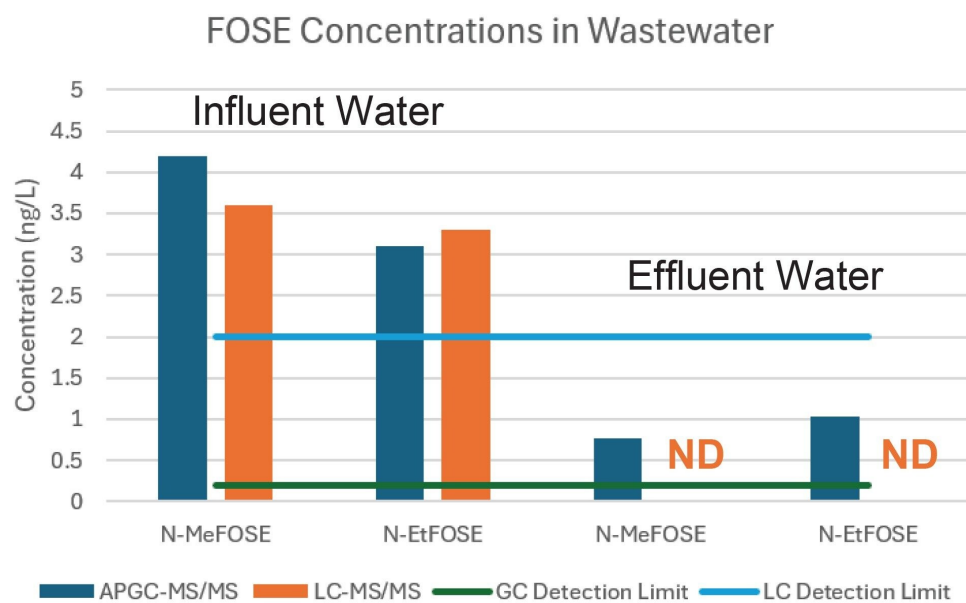


Figure 3. Comparison of N-MeFOSE and N-EtFOSE quantified in wastewater samples using both LC-MS/MS and APGC-MS/MS.

Sample	6:2 FTOH	8:2 FTOH	10:2 FTOH
D2	6.87	0.54	ND
D4*	5.07	0.79	0.34
D3	4.27	0.70	0.30
D1	2.34	0.28	ND
C1*	1.08	0.26	0.24
C2	0.89	0.32	ND
C9*	0.85	0.40	0.27
C8	0.83	0.23	ND
C7*	0.81	0.42	0.24
C4	0.75	0.23	ND
C5	0.72	0.24	ND
C6	0.69	0.24	0.24
C3	0.57	0.23	ND

Table 1. GC amenable PFAS detected in ski wax impacted soil, reported in ng/g. () indicates known area of wax application. Results are sorted by highest to lowest concentration of 6:2 FTOH.*

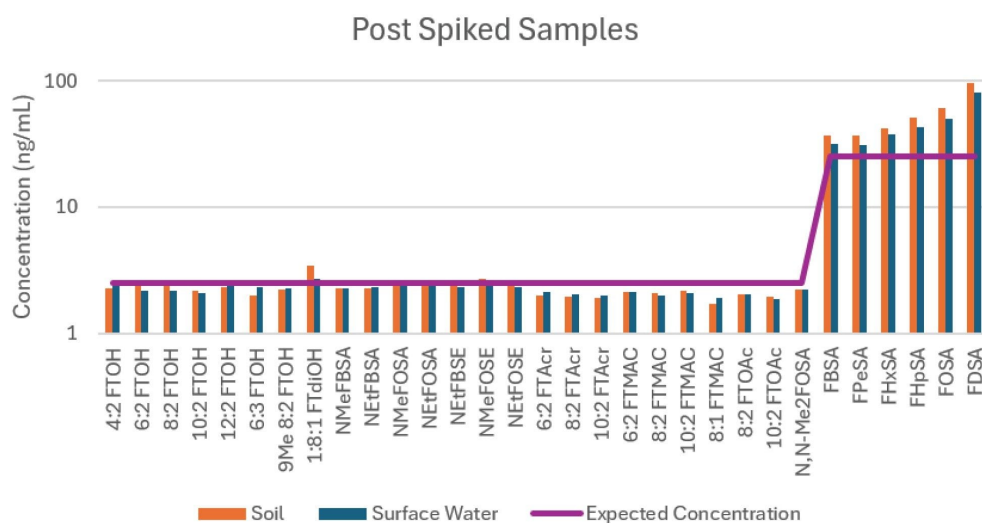


Figure 4. Post spiked soil and surface water samples comparing the calculated concentration to the expected concentration (2.5–25 ng/mL). Y axis is displayed as a log scale.



Figure 5. Example chromatograms showing overlaid quantitative and qualitative MRMs of selected PFAS spiked into food packaging materials at 25 ng/mL.

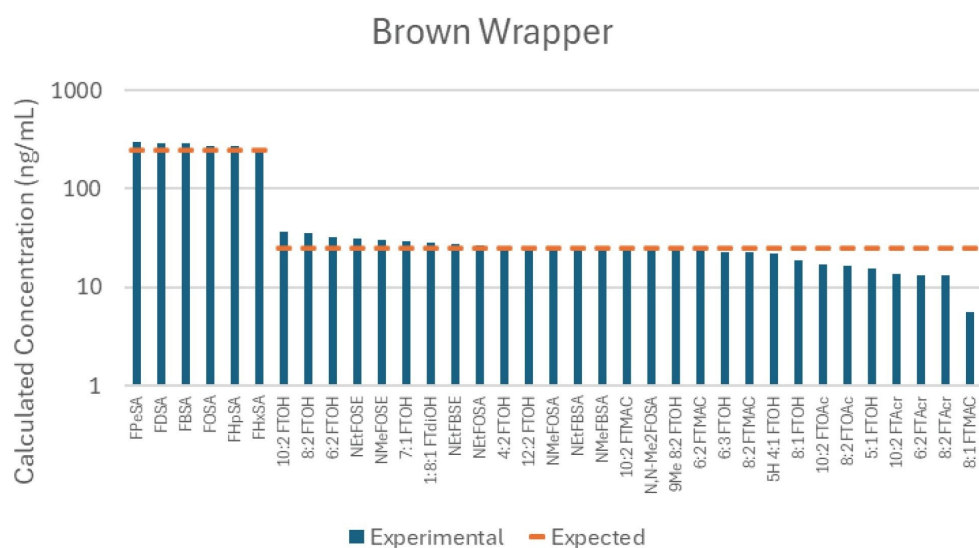


Figure 6. Post spiked paper wrapper sample comparing the calculated concentration to the expected concentration (25–250 ng/mL). Y axis is displayed as a log scale.

Conclusion

The APGC-MS/MS method developed for the analysis of 35 GC-amenable PFAS demonstrated robust performance across diverse environmental and food packaging matrices, providing sensitive and selective detection of volatile and semi-volatile PFAS that are not routinely captured by LC-MS/MS alone. Application of the method identified FTOHs as the predominant GC-amenable PFAS in wastewater and soil samples. This method proved to be complementary to previous LC-MS/MS analysis by identifying precursor compounds that appear to have transformed into terminal PFCAs during the treatment process. The method also successfully characterized PFAS contamination associated with ski wax application and showed good accuracy for most target analytes in environmental matrices. Analysis of paper and paperboard food packaging materials did not detect any of the targeted PFAS, suggesting compliance with current regulatory restrictions for the compounds included in this study. Post-spike experiments confirmed the method's suitability for monitoring PFAS at concentrations well below emerging European regulatory limits. By expanding PFAS characterization beyond routinely monitored analytes, the combined APGC-MS/MS and LC-MS/MS workflow provides laboratories with

greater confidence when investigating PFAS sources, environmental fate, and regulatory compliance. Overall, the method performance was strong, demonstrating that APGC-MS/MS is a valuable complementary tool to LC-MS/MS for PFAS characterization, environmental monitoring, source identification, and regulatory compliance testing.

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Appendix

Table 1. APGC-MS/MS MRM Method Details

Compound	Precursor (m/z)	Product (m/z)	CV (V)	CE (V)	RT (min)
4:2 FTOH	265	181	15	20	3.88
		227	15	10	3.88
6:2 FTOH	365	281	20	20	5.71
		327	20	15	5.71
8:2 FTOH	465	69	30	40	7.41
		427	30	15	7.41
10:2 FTOH	565	69	35	30	8.96
		145	35	25	8.96
12:2 FTOH	665	69	45	45	10.36
		181	45	40	10.36
6:3 FTOH	379	295	5	20	7.18
		341	5	10	7.18
1:8:1 FTdiOH	463	403	10	20	11.93
		423	10	15	11.93
9Me 8:2 FTOH	515	76.9	35	25	8.34
		477	35	15	8.34
5:1 FTOH	301	281	30	10	3.30
	319	301	10	10	3.30
5H 4:1 FTOH	233	213	10	5	4.43
	251	233	20	5	4.43
7:1 FTOH	401	381	40	10	5.10
	419	401	5	10	5.10
8:1 FTOH	451	431	40	10	5.98
	469	431	5	15	5.98
NMeFBSA	314	69	25	25	8.94
		131	25	25	8.94
NEtFBSA	328	64	15	20	9.43
		300	15	10	9.43
NMeFOSA	514	69	25	40	11.82
		169	25	30	11.82
NEtFOSA	528	69	30	50	12.21
		92	30	15	12.21
NEtFBSE	372	71	25	20	12.27
		354	25	10	12.27
NMeFOSE	558	169	10	35	14.24
		540	10	15	14.24
NEtFOSE	572	462	20	30	14.60
		554	20	15	14.60
N,N-Me2FOSA	528	69	25	45	11.28
		92	25	40	11.28
6:2 FTAc	419	54.9	25	20	8.46
		72.9	25	20	8.46
8:2 FTAc	519	54.9	30	25	9.90
		72.9	30	20	9.90

Compound	Precursor (m/z)	Product (m/z)	CV (V)	CE (V)	RT (min)
10:2 FTAc	619	54.9	50	40	11.22
		73	50	20	11.22
6:2 FTMAC	433	59	35	40	9.22
		87	35	30	9.22
8:2 FTMAC	533	59	35	30	10.60
		87	35	20	10.60
10:2 FTMAC	633	59	35	30	11.85
		87	35	25	11.85
8:1 FTMAC	469	68.9	35	25	7.97
		441	35	20	7.97
8:2 FTOAc	507	61	20	20	9.27
		61	20	45	9.27
10:2 FTOAc	607	61	20	20	10.63
		69	20	40	10.63
FBSA	300	69	5	20	9.07
		216	5	15	9.07
FPeSA	350	69	5	20	9.86
		269	5	10	9.86
FHxSA	400	69	5	20	10.58
		336	5	10	10.58
FHpSA	450	69	5	40	11.29
		386	5	10	11.29
FOSA	500	69	25	50	11.97
		436	25	15	11.97
FDSA	600	69	35	35	13.20
		536	35	15	13.20
D4 4:2 FTOH	269	181	5	20	3.88
		230	5	10	3.88
13C2 6:2 FTOH	367	281	5	25	5.70
		329	5	15	5.70
13C2 8:2 FTOH	467	381	5	20	7.41
		429	5	10	7.41
D2 13C2 10:2 FTOH	569	481	35	25	8.96
		531	35	20	8.96
d3 NMeFOSA	517	169.6	10	25	11.82
		453	10	15	11.82
d5 NEtFOSA	533	65	10	20	12.21
		501	10	15	12.21
d7 NMeFOSE	565	64.3	10	30	14.25
		547	10	15	14.25
d9 NEtFOSE	581	467	10	25	14.60
		563	10	15	14.60

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