

Is Your Next Sip Safe? A Simple, Rapid Method for Measuring volatile PFAS in Juices.

Andy L. Sandy; Dominika Gruszecka; Evelyn Wang
Shimadzu Scientific Instruments, Columbia, MD

1. Introduction

Per- and polyfluoroalkyl substances (PFAS) pollution is a growing global concern due to links with adverse health effects, many of which remain poorly understood.¹ Accurate detection and quantification are therefore essential. While PFAS analysis has traditionally focused on water, attention is rapidly expanding to food and beverages, where testing remains limited despite high stakes and increasing regulations.² A recent PFAS lawsuit against a juice manufacturer underscored the heightened public scrutiny of PFAS in juices, placing food safety and brand reputation at risk.³ This study presents a Headspace Solid-Phase Microextraction–Triple Quadrupole Gas Chromatography/Mass Spectrometry (HS-SPME GC/MS/MS) method for PFAS quantitation in juices. GC/MS enables analysis of volatile PFAS unsuitable for LC/MS, while HS-SPME offers rapid analysis with minimal sample preparation in complex matrices.

2. Methods

A volatile PFAS analysis method was developed on a Shimadzu GCMS-TQ8040 NX with an AOC-6000 Plus multifunctional autosampler equipped with a solid phase microextraction (SPME) module (Figure 1).

Ten PFAS target compounds were included in the Multiple Reaction Monitoring (MRM) method. The PFAS chemical classes were perfluoroalkyl iodides (PFIs), (n:2) fluorotelomer iodides (FTIs), (n:2) fluorotelomer acrylates (FTACs), (n:2) fluorotelomer methacrylates (FTMACs), (n:2) fluorotelomer alcohols (FTOHs), and perfluoroalkane sulfonamides (FASAs). Internal standards (IS) FTOHs, FASAs, FTMAC and FTAC mass-labelled compounds were added to each vial prior to extraction. Concentrations of the target compounds were calculated using isotope dilution.

An eight-point internal calibration curve (1–2000 ng/L) was prepared in 10 mL of reagent water with the targeted compounds, isotopically labelled internal standards and 2% NaCl (w/v) to enhance extraction efficiency. The IS were spiked at 100 ng/L to each calibrator. These calibrators were vortexed for 30 seconds and then placed on the AOC-6000 Plus autosampler rack for HS-SPME analysis.

The optimized parameters of the HS-SPME GC/MS method for the targeted PFAS are listed in Table 1. Quantifier and qualifier ions for each PFAS target are listed in Table 2. The associated internal standard used for each compound is also listed in Table 2.

This study analyzed both laboratory control samples (LCS) and four commercial juice samples. Reagent water was used as the LCS to evaluate method performance in a clean matrix. Juice samples included two carton apple juices (Brands X and Y), one plastic-bottled apple juice (Brand X), and one berry blend carton juice (Brand X). The juice samples were analyzed to evaluate the effect of the matrix on method performance.



Figure 1. Shimadzu GCMS-TQ8040 NX configured with an AOC-6000 Plus

Table 1. GC/MS and HS-SPME method conditions

Gas Chromatography	Nexis GC-2030
Injection mode	Splitless
Carrier gas	Helium
Injection port temperature (°C)	240
Column	SH-I-624Sil MS Capillary, 30 m x 0.25 mmID x 1.40 µm
Flow control mode (cm/sec)	Linear velocity, 45
Total flow (mL/min)	50
Oven temperature	40°C (7 min.), 5°C/min. to 190°C (0 min.), 40°C/min. to 300°C, (5 min.)
Mass Spectrometer	GCMS-TQ8040 NX
Interface temperature (°C)	280
Ion source temperature (°C)	200
Detector voltage (kV)	Relative to Tune 0.4
Threshold	0
Acquisition mode	MRM, Loop time: 0.5 sec
Tuning mode	Normal mode
SPME analysis	AOC-6000 Plus
SPME Fiber	50/30 µm DVB/CAR/PDMS
Incubation time (min)	5
Extraction time (min)	30
Desorption time (min)	7
Agitation speed (rpm)	300
Extraction temperature (°C)	50
Sample volume (mL)	10
Desorption temperature (°C)	240
Sampling salinity	2 % NaCl (w/v)

Table 2. Retention time, quantitative ion, reference ions, and internal standard group for each target PFAS compound

Compound Type	Name	Ret. Time (min)	Quantifier (m/z)	Qualifier #1 (m/z)	Qualifier #2 (m/z)	Internal standard group
Targets	6:2 FTI	19.6	473.9>326.9	69.0>50.0	473.9>263.0	1
	8:2 FTOH	22.5	95.0>69.0	127.1>77.1	95.0>45.1	1
	6:2 FTAC	23.1	418.1>99.1	99.1>43.1	99.1>57.1	2
	8:2 FTI	23.5	574.0>426.9	169.0>69.0	574.0>65.1	2
	10:2 FTOH	25.7	95.0>69.0	127.1>77.1	95.0>45.1	3
	6:2 FTMAC	25.6	86.1>68.1	432.1>113.1	432.1>86.1	5
	8:2 FTAC	26.4	518.0>99.1	99.1>57.1	99.1>43.1	6
	8:2 FTMAC	28.7	86.0>68.1	86.0>41.1	532.00>113.1	7
	MeFOSA	33.6	131.1>69.1	169.0>69.0	94.00>91.8	8
	EiFOSA	34.2	108.1>80.0	448.0>69.1	108.10>44.1	4
Internal Standards	8:2 FTOH ¹³ C2	22.4	98.0>69.0	131.1>81.1	98.00>48.1	1
	6:2 FTAC d3	23.1	101.1>57.1	101.1>45.0	102.00>45.0	2
	10:2 FTOH ¹³ C2	25.6	98.0>69.0	131.1>81.1	98.00>48.1	3
	EiFOSA d5	34.1	113.1>81.0	81.0>64.0	450.10>69.0	4
	6:2 FTMAC-d5	25.5	91.1>73.1	437.1>118.2	437.1>91.1	5
	8:2 FTAC-d3	26.3	521.1>102.1	102.1>58.1	102.1>74.1	6
	8:2 FTMAC-d5	28.6	91.1>73.1	537.1>91.1	537.1>118.1	7
	N-MeFOSA-d3	33.5	433.1>114.0	433.1>94.3	97.1>94.1	8

Prior to samples analysis, the system background was evaluated by analyzing method blanks to confirm that the instrument and reagents were free of interferences. An initial calibration verification (ICV) was performed to verify the accuracy of the calibration curve. Continuing calibration verifications (CCV) were performed to ensure the accuracy of the calibration curve was maintained.

A demonstration of precision and accuracy were evaluated using the LCS as well as spiked and unspiked juice samples. The LCS and spiked samples were fortified with all analytes at 100 ng/L and mass-labeled internal standards at varying concentrations, while unspiked samples were spiked only with mass-labeled internal standards at varying concentrations.

3. Results

The system was deemed free of contaminants and inferences. None of the target PFAS in the method blank were found in quantifiable concentration. In the study, the calibration curve for each target included at least seven calibrators. Calibration curve results showed a good linear fit for all compounds with coefficient of determination (R^2) \geq 0.993. The linear range and R^2 of each PFAS target are shown in Table 3.

Table 3. Summary of PFAS calibration range and coefficient of determination.

Compound	Calibration range (ng/L)	R^2
6:2 FTI	1.0-2000	0.993
8:2 FTOH	1.0-2000	0.999
6:2 FTAC	2.5-2000	0.996
8:2 FTI	1.0-2000	0.998
6:2 FTMAC	2.5-2000	0.994
10:2 FTOH	1.0-2000	>0.999
8:2 FTAC	2.5-2000	0.994
8:2 FTMAC	2.5-1000	0.996
MeFOSA	10.0-2000	>0.999
EiFOSA	2.5-2000	>0.999

When compared to the initial calibration curve, the ICV accuracy for all compounds was within 70 – 130%, meeting the method criteria. A CCV standard was ran after the ICV and after an average of 14 samples. In comparison to the initial calibration curve, the CCV accuracy for all compounds was within 70 – 130%.

For the LCS, the concentration of each analyte in the replicate analyses (n=4) was calculated. The mean % recovery ranged from 83 to 115%, while the %RSD for analytes in these replicates ranged from 0.6 to 6.8% (Table 4). These results satisfied the established method criteria for clean sample matrices (70–130% mean recovery; \leq 20 for %RSD).

Table 4. Precision and Accuracy (n=4) of PFAS in LCS.

Compound	LCS 100 ng/L	
	Mean % Recovery	%RSD
6:2 FTI	102	1.4
8:2 FTOH	83	2.0
6:2 FTAC	88	6.8
8:2 FTI	97	6.1
6:2 FTMAC	101	2.2
10:2 FTOH	90	0.6
8:2 FTAC	115	3.7
8:2 FTMAC	96	3.0
MeFOSA	94	3.8
EiFOSA	95	0.8

Preliminary analyses from precision and accuracy experiments on the juice matrices revealed significant matrix effects on most target compounds. Moreover, compounds lacking corresponding isotopically labeled internal standards could not be quantified accurately in complex juice matrices. These initial findings highlighted the importance of using an isotopically labeled internal standard for each target volatile PFAS compound to ensure accurate quantitation in complex matrices. Figure 2 illustrates the quantitation of 8:2 FTMAC across various matrices using each of the internal standards included in this method.

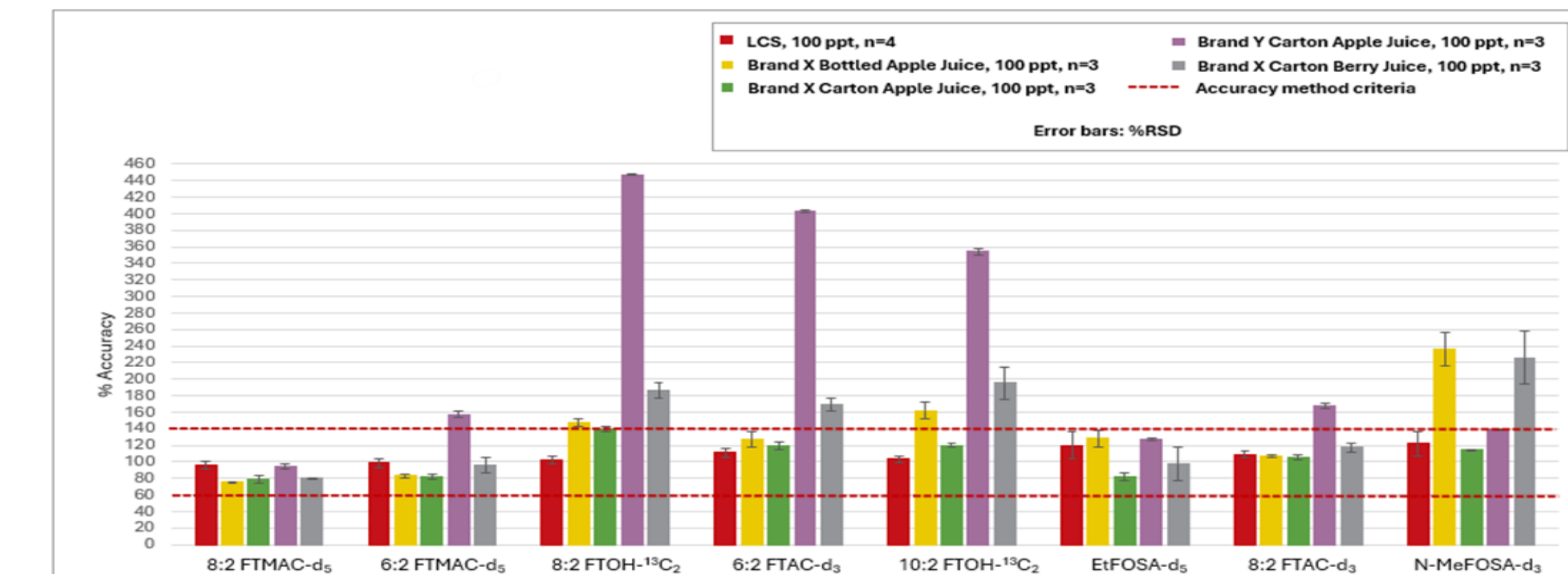


Figure 2. 8:2 FTMAC accuracy results using multiple isotopic labelled internal standards.

The results indicated that an accurate quantitation of 8:2 FTMAC was achieved using its corresponding isotopically labeled internal standard, 8:2 FTMAC-d5. Even using internal standards, 6:2 FTMAC-d5, within the same PFAS chemical class cannot guarantee to compensate for matrix effect of this analyte.

The %RSD of 8:2 FTMAC, using 8:2 FTMAC-d5 to quantitate its recoveries, ranged from 0.8 to 5.1. Following the assignment of internal standards for each of the target compounds, juice samples were analyzed. None of the target PFAS were detected at quantifiable concentrations in the unspiked sample; therefore, no adjustment to the recovery concentrations was necessary. Across the four complex juice matrices, mean percent recoveries ranged from 69 – 120% with %RSDs below 12. These results met the method performance criteria for complex matrices.

Table 5. Precision and Accuracy (n=3) of PFAS in juice matrices.

Compound	Brand X Bottled AJ		Brand X Carton AJ		Brand Y Carton AJ		Brand X Carton BJ	
	Mean % Recovery	%RSD	Mean % Recovery	%RSD	Mean % Recovery	%RSD	Mean % Recovery	%RSD
6:2 FTI	105	3.3	120	3.8	76	1.4	84	4.2
8:2 FTOH	90	0.6	87	2.1	83	0.9	86	0.7
6:2 FTAC	70	3.0	69	5.8	74	2.9	79	5.5
8:2 FTI	88	7.9	79	10.9	70	2.5	91	11.6
6:2 FTMAC	95	0.3	95	1.4	99	1.7	94	2.2
10:2 FTOH	101	2.5	93	1.9	117	1.9	92	3.2
8:2 FTAC	115	1.9	114	2.7	104	0.4	112	2.6
8:2 FTMAC	75	1.0	79	5.1	95	3.1	80	0.8
MeFOSA	96	0.9	91	2.5	95	4.0	86	3.7
EiFOSA	87	1.3	88	0.5	88	0.3	86	0.2

4. Conclusion

Overall, these results demonstrate the robustness and reliability of the method for PFAS analysis in two carton apple juices (Brands X and Y), one plastic bottled apple juice (Brand X), and a berry blend carton juice (Brand X). Given the diversity of matrices evaluated, the method may also be suitable for other beverages, including sports drinks and vitamin-enhanced waters.

Reference

- Alazaiza, M. Y., Alzghoul, T. M., Ramu, M. B., Amr, S. S. A., & Abushammala, M. F. (2025). PFAS Contamination and Mitigation: A Comprehensive Analysis of Research Trends and Global Contributions. Case Studies in Chemical and Environmental Engineering, 101127. <https://doi.org/10.1016/j.cscee.2025.101127>
- Per- and Polyfluoroalkyl Substances (PFAS) (2024, June 26). US. Food and Drug Administration. Retrieved July 22, 2024, from <https://www.fda.gov/food/environmental-contaminants-food/and-polyfluoroalkyl-substances-pfas>
- Florida's Natural PFAS Lawsuit Disputes "Natural" Claim (2023, September 26). Milberg. Retrieved May 18th, 2026 from [https://milberg.com/news/floridas-natural-pfas-lawsuit/#:~:text=Water,%20orange%20juice,%20and%20per-%20and%20polyfluoroalkyl%20substances%20\(PFAS\),%200ne](https://milberg.com/news/floridas-natural-pfas-lawsuit/#:~:text=Water,%20orange%20juice,%20and%20per-%20and%20polyfluoroalkyl%20substances%20(PFAS),%200ne)