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GCMS-TQ™8040 NX Triple Quadrupole Mass Spectrometer

Application News

What's in Your Brew? Detecting Volatile PFAS with Headspace SPME GC/MS/MS

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User Benefits

- Employing the highly sensitive and selective Shimadzu GCMS-TQ8040 NX triple quadrupole mass spectrometer allows for low limits of quantitation for volatile PFAS while minimizing matrix interferences.
- Using the multifunctional AOC™-6000 Plus autosampler, the automated SPME method and simplified sample preparation help to reduce operation errors in PFAS analysis.
- The Shimadzu HS-SPME GC/MS/MS system can quantify volatile PFAS in beer matrices with minimal sample preparation.
- HS-SPME GC/MS/MS is used as a complementary technique to LC/MS in providing a total solution for beverage safety.

■ Introduction

PFAS, or per- and polyfluoroalkyl substances, are a group of synthetic organic chemicals that are known for their persistence, bioaccumulation, and toxicity in the environment.[1-4] The global concern surrounding PFAS pollution continues to grow, as many of their long-term effects on health and the environment are still not fully known.[1,3] Some major human exposure routes to these harmful chemicals include inhalation of contaminated dust, breathing air containing PFAS, transfer to infants through breast milk, and ingestion of contaminated food, such as seafood, drinking water, and even commercial beverages.[1,5-7]

Beer is one of the most consumed beverages worldwide. It is estimated that humans consume more than 49.6 billion gallons of beer in just one year.[7] Beer can be both alcoholic and non-alcoholic and varies in flavor, color, and aroma. Its complex composition, resulting from diverse ingredients and brewing processes, presents analytical challenges for detecting trace-level contaminants. As public awareness and regulatory attention increase, there is a growing need for reliable methods to screen beer for PFAS contamination and ensure product safety.[8] Developing an effective analytical workflow for PFAS analysis in beer matrices is therefore essential to identify contaminated batches and prevent PFAS consumption. This study aims to establish a precise and accurate quantitation method to analyze volatile PFAS in beer. Given the diverse samples analyzed in this study, this method may also be applicable to other alcoholic, non-alcoholic, and carbonated beverages.

This study presents a simple approach for analyzing volatile PFAS including fluorotelomer alcohols and acrylates in beer using Head-Space Solid Phase Microextraction-Triple Quadrupole Gas Chromatography/Mass Spectrometry (HS-SPME GC/MS/MS). This GC/MS method addresses volatile PFAS compounds that are impractical to analyze by LC/MS. The HS-SPME technique, with its minimal sample preparation procedure and fast workflow, offers additional benefits for volatile PFAS analysis in complex matrices.

HS-SPME allows a pre-concentration step as well as higher selectivity compared to GC/MS liquid injection, thus allowing lower detection limits. While previous PFAS HS-SPME GC/MS/MS methods have been developed for simple matrices such as drinking and bottled water, [9-11] the complex composition of beer matrices requires additional isotopically labeled internal standards to effectively compensate for matrix effects.

■ Method

Instrumentation: The instrument system configuration for the application consisted of a Shimadzu GC/MS triple quadrupole mass spectrometer, model GCMS-TQ8040 NX, a multifunctional autosampler (AOC-6000 Plus) equipped with a SPME module and a split/spitless inlet. (**Figure 1**)

Figure 1. Shimadzu GCMS-TQ™8040 NX configured with an AOC™-6000 Plus

Standards and Reagents: The target list consists of ten PFAS in the following chemical classes: (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 were FTOHs, FASAs, FTMAC, and FTAC mass-labelled compounds. A working solution for each analyte at 10 mg/L was prepared in methanol. This standard was stored at 4 °C. LC/MS grade water and methanol were purchased from Honeywell.

An internal calibration curve was prepared in 10 mL of water at concentrations of 2000, 1000, 500, 100, 50, 25, 10, 2.5, and 1 ng/L. The mass labelled internal standard compounds 8:2 FTOH-$^{13}C_2$-d_4, 6:2 FTAC-d_3, 10:2 FTOH-$^{13}C_2$-d_4 and n-ethyl-d_5-perfluoro-1-octanesulfobamide (EtFOSA-d_5)were spiked in each calibrator at 100 ng/L, while 6:2 FTMAC-d_5, 8:2 FTAC-d_3 and 8:2 FTMAC-d_5 were spiked at 10 ng/L. n-methylperfluorooctanesulfonamide-d_3 (N-MeFOSA-d_3) was spiked at 50 ng/L.

Sodium Chloride (NaCl) was added to each vial to achieve a final salinity concentration of 2% NaCl (w/v). All samples were vortexed for 30 seconds and then placed on the AOC-6000 Plus autosampler rack for HS-SPME analysis.

HS-SPME GC/MS/MS Analysis: In this study, an HS-SPME method was used to improve method performance when analyzing complex aqueous samples. A Multiple Reaction Monitoring (MRM) GC/MS method was used in tandem with the SPME method to enhance selectivity and sensitivity of the targeted PFAS compounds, this was needed since exposure risk starts as low as ng/L levels. The optimized parameters of the instrument method for the targeted PFAS are listed in **Table 1**. A quantifier and qualifiers for each PFAS target are listed in **Table 2**. Quantitation was performed by an internal standard method using an isotope dilution approach. The associated internal standards used for each compound are also listed in **Table 2**.

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

Gas Chromatography	**Nexis^{TM} GC-2030**
Injection port mode	Splitless
Carrier gas	Helium
Injection port temperature (°C)	240
Sampling time (min)	1
Column	SH-I-624Sil MS Capillary, 30 m x 0.25 mmID x 1.40 µm
Flow control mode (cm/sec)	Linear velocity: 45
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	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, quantifier, qualifiers, and internal standard group information for each of the targeted PFAS compounds.

Compound	Ret. Time (min)	Quantifier (*m/z*)	CE	Qualifier #1 (*m/z*)	CE	Qualifier #2 (*m/z*)	CE
8:2 FTOH	22.4	95.0>69.0	15	127.1>77.1	15	95.0>45.1	27
8:2 FTOH-$^{13}C_2$-d_4	22.3	98.0>69.0	15	131.1>81.1	15	98.0>48.1	27
6:2 FTAC	23.1	418.1>99.1	15	99.1>43.1	9	99.1>57.1	12
6:2 FTAC-d_3	23.0	101.1>57.1	12	101.1>45.0	9	102.0>45.0	9
10:2 FTOH	25.6	95.0>69.0	15	127.1>77.1	15	95.0>45.1	27
10:2 FTOH-$^{13}C_2$-d_4	25.5	98.0>69.0	12	131.1>81.1	12	98.0>48.1	27
6:2 FTMAC	25.6	86.1>68.1	6	432.1>113.1	12	432.1>86.1	18
6:2 FTMAC-d_5	25.5	91.1>73.1	6	437.1>118.2	12	437.1>91.1	18
8:2 FTAC	26.4	518.0>99.1	15	99.1>57.1	12	99.1>71.1	6
8:2 FTAC-d_3	26.3	521.1>102.1	15	102.1>58.1	12	102.1>74.1	6
8:2 FTMAC	28.7	532.0>113.1	21	532.0>86.1	21	86.0>68.1	6
8:2 FTMAC-d_5	28.6	537.1>118.1	21	537.1>91.1	21	91.1>73.1	6
MeFOSA	33.5	430.0>111.1	24	430.0>91.1	33	94.0>91.8	57
N-MeFOSA-d_3	33.4	433.1>114.0	24	433.1>94.3	33	97.1>94.1	57
EtFOSA	34.1	108.1>80.0	6	448.0>69.1	27	108.1>44.1	3
EtFOSA-d_5	34.0	113.1>81.0	6	81.0>64.0	24	450.1>69.0	27

Sample Preparation: Five commercially available beer samples and reagent water were analyzed in this study. LC/MS-grade water was used as reagent water, which served as a laboratory control sample (LCS) to assess the general performance of the method in a clean matrix. The samples analyzed included five beer samples (wheat, IPA, lager, IPA non-alcoholic, and lager non-alcoholic beers). These beer samples were analyzed to evaluate the effect of the matrix on method performance.

Ten milliliters of reagent water and beer samples were prepared for instrument analysis. Four replicate aliquots of the laboratory control sample (LCS) were analyzed, while beer samples were analyzed in triplicates for both spiked and unspiked aliquots. The LCS and spiked beer samples were fortified with all analytes at 100 ng/L, representing the midrange concentration of the initial calibration (ICAL). Mass-labeled internal standard compounds, at varying concentrations as described in the standards and reagent section above, were also spiked into these beer samples. Unspiked samples were only fortified with mass-labeled internal standards.

Initial calibration verification (ICV) and continuing calibration verification (CCV) quality control (QC) samples were prepared for instrument analysis using 10 mL of reagent water. These QC samples were fortified with all analytes at 100 ng/L, representing the midrange concentration of the ICAL. The mass-labeled internal standards were spiked into the QC samples at varying concentrations, as outlined in the standards and reagent section.

Sodium chloride (NaCl) was added to all water and QC samples to achieve a final salinity of 2% NaCl (w/v). Each sample vial was vortexed for 30 seconds and then placed on the AOC-6000 Plus rack for HS-SPME GC/MS/MS analysis.

PFAS contamination can occur during analysis from various sources, such as consumables or solvents. In this study, all consumable, solvents, standards and reagent water were analyzed and no PFAS were detected under the method conditions.

Instrumental analysis: A demonstration of proficiency study of the instrumentation system capability to conduct PFAS analysis on beer samples was performed. Prior to the analysis of samples, the system background was evaluated by analyzing method blanks to confirm that the instrument and reagents were free of contaminants and interferences. Subsequently, an initial calibration (ICAL) was analyzed. Prior to analyzing the samples, an ICV was performed to verify the accuracy of the calibration curve. In addition, a CCV was analyzed within the batch to ensure the accuracy of the calibration curve was maintained and no major drift was observed. Both ICV and CCV data are used to validate the integrity of the calibration curve, which is used to quantitate targeted compounds in the samples. In this study, the ICV and CCV accuracy should be within 70-130 % for the calibration curve to be considered valid.

A demonstration of precision and accuracy was first performed on the LCS. After the evaluation of method performance in this clean matrix, precision and accuracy tests were carried out on the beer samples. For beer samples, the spiked samples were analyzed for accuracy and precision evaluation. It is important to evaluate the amount of target PFAS in the unspiked matrix so that accurate adjustment can be made to the expected concentration of the spiked matrix.

■ Results and Discussion

Prior to calibration and sample analysis, system background was evaluated as a quality control measure. Method blanks were analyzed, confirming that the system was free of contaminants and interferences. None of the target PFAS in the method blanks were detected at quantifiable concentrations.

In this study, a calibration curve for all analytes was prepared over a range of 1 to 2000 ng/L. The calibration curve results demonstrated a strong linear relationship for all compounds, with a coefficient of determination (R^2) ≥ 0.996. The linear range and R^2 values for each target PFAS are provided in **Table 3**.

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

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

An ICV standard was run prior to sample analysis. When compared to the initial calibration curve, the ICV recoveries for all compounds fell within the 70-130% range, which meets the established method criteria. A CCV standard was run after the ICV and after an average of 16 samples to assess the stability of the calibration curve and its ability to quantify the targeted compounds in the samples. The CCV recoveries for all compounds were within the 70-130% range, as compared to the initial calibration curve, satisfying the method criteria.

For the LCS, the concentration of each analyte in the replicate analyses was calculated using the initial calibration (ICAL). The mean percent recovery (mean % recovery) and the percent relative standard deviation (%RSD) were then determined for each analyte of interest. The mean percent recovery ranged from 91 to 101, while the % RSD for the analytes in these replicates ranged from 0.9 to 6.5 (**Table 4**). The LCS results met the mean % recovery and %RSD method criteria, which were established respectively at 70-130% and ≤ 20%.

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

Compound	LCS 100 ng/L	
	Mean % Recovery	% RSD
8:2 FTOH	99	1.3
6:2 FTAC	92	3.7
6:2 FTMAC	95	1.8
10:2 FTOH	92	0.9
8:2 FTAC	91	6.5
8:2 FTMAC	96	6.3
MeFOSA	101	5.5
EtFOSA	96	1.1

Prior to this study, preliminary analyses were conducted on other complex matrices, specifically juice samples. The results demonstrated significant matrix effects for most targeted volatile PFAS compounds.[12] Additionally, accurate quantification proved challenging for many compounds that lacked corresponding isotopically labeled internal standards within these complex juice matrices. Given the wide variability in the behavior of targeted compounds across complex matrices, when the corresponding isotopically labeled internal standard was unavailable, it is essential to evaluate each compound using a range of internal standards. This approach helps identify the most suitable internal standard that closely mirrors the behavior of the target compound within the matrix.

Although both juice and beer are considered complex matrices, the findings from juice sample analyses cannot be directly applied to beer due to differences in matrix composition. One key distinction is the presence of carbonation in beer, which is absent in juice. The alcohol content in beer can also influence the partition coefficient of extraction and add complexities to the matrix effect.[13] To assess the influence of the beer matrix on analytical performance, precision and accuracy experiments were conducted.

To demonstrate the importance of choosing appropriate internal standards for quantifying volatile PFAS, **Figure 2** presents the quantification of 8:2 FTMAC as an example across various beer matrices using each internal standard included in the method. The results showed that accurate quantitation of 8:2 FTMAC was achieved when using its specific isotopically labeled internal standard, 8:2 FTMAC-d_5. In contrast, even internal standards from the same PFAS chemical class, such as 6:2 FTMAC-d_5, were unable to consistently correct for matrix effects across the five beer samples. When using 8:2 FTMAC-d_5 for quantitation, the %RSD for 8:2 FTMAC recoveries ranged from 1.1% to 3.6%, demonstrating reliable performance.

Although isotopically labeled internal standards are essential for accurate PFAS analysis in complex matrices, many PFAS compounds still lack commercially available labeled standards, as the development and availability of these compounds are still limited. For any complex matrices analyzed beyond the scope of this study, it is strongly recommended that, in cases where labeled internal standards for a target compound are unavailable, suitable alternative internal standards be carefully evaluated within the specific matrix. This evaluation is crucial to ensure that the internal standard closely mimics the analyte's behavior, thereby enabling reliable quantitation.

After assigning internal standards to each of the target compounds, the beer samples were analyzed. The concentration of each targeted PFAS in the replicate analyses for both spiked and unspiked samples was calculated using the ICAL. None of the targeted PFAS were detected at quantifiable concentrations in the unspiked sample; therefore, no adjustment to the recovery concentrations was necessary.

Despite the analysis of a wide variety of beer samples with different complex matrices, the quantitation of targeted compounds with their own internal standards resulted in accurate and reproducible recoveries (**Table 5**). The mean percent recoveries for all PFAS compounds across the five beer samples ranged from 78% to 126%, with relative standard deviations (%RSD) below 8%. These results highlight the feasibility and reliability of the method for PFAS quantitation across diverse beer types.

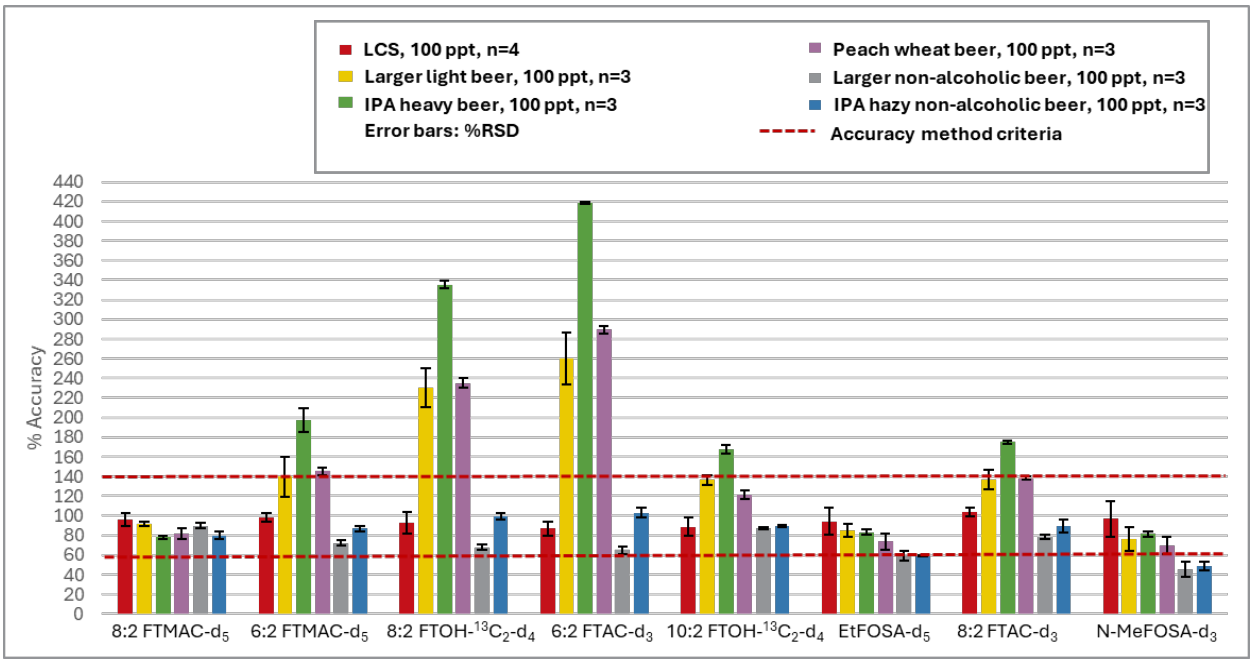

Figure 2. 8:2 FTMAC accuracy results using multiple isotopic labelled internal standards.

Table 5. Precision and Accuracy results of PFAS in beer samples.

	Larger beer		IPA beer		Wheat beer		Larger non-alcoholic beer		IPA non-alcoholic beer	
	Mean % Recovery	%RSD	Mean % Recovery	%RSD	Mean % Recovery	%RSD	Mean % Recovery	%RSD	Mean % Recovery	%RSD
8:2 FTMAC	92	2.0	78	1.5	82	5.2	90	2.3	80	3.5
6:2 FTMAC	92	2.8	97	7.7	83	2.0	90	3.5	86	1.0
8:2 FTOH	99	0.7	99	1.3	93	1.1	93	0.3	90	1.0
6:2 FTAC	115	1.8	124	3.1	126	1.0	113	3.4	114	1.2
10:2 FTOH	88	1.7	89	1.4	81	1.3	87	0.5	86	1.5
EtFOSA	100	1.0	111	0.7	101	1.4	103	1.2	118	2.5
8:2 FTAC	88	3.0	87	1.4	82	1.8	82	1.8	83	5.1
N-MeFOSA	85	4.9	95	4.0	92	5.2	82	1.9	90	5.1

■ Conclusion

A simple and innovative approach was developed to measure PFAS in complex beer samples. A Shimadzu GCMS-TQ8040 NX triple quadrupole mass spectrometer, configured with an AOC-6000 Plus solid-phase microextraction (SPME) unit was used for the analysis.

Method blanks showed no detectable PFAS, and the calibration curve demonstrated excellent linearity ($R^2 \geq 0.996$). ICV and CCV recoveries were all within 70–130%, established as the method criteria. For general method performance a LCS was evaluated. The mean PFAS recovery in the LCS was 91 to 101%, while the % RSD for the analytes in these replicates ranged from 0.9 to 6.5%. LCS results met the mean % recovery and %RSD method criteria, which were established respectively at 70-130% and ≤ 20%. An isotope dilution approach was used for all compounds to achieve accurate quantitation in complex beer matrices. Overall, the mean percent recovery for the five beer samples ranged from 78 - 126% and % RSD < 8 for all compounds. The overall results satisfied the method criteria.

The workflow presented in the study offers key advantages in terms of simplicity, speed, precision, and accuracy that are critical for routine monitoring of volatile PFAS in challenging matrices.

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■ Consumables

Item Name	Item Description	Part Number
Capillary column	GC, SH-I-624Sil MS Capillary, 30 m x 0.25 mmID x 1.40 um	221-75962-30
SPME Inlet liner	SPME liner 0.75mm x 5.0 x 95 for Shimadzu GCs Deact., 5pk (Restek)	REST-22279
Head-Space sample vials	20ml magnetic screw-cap clear headspace vial kit	220-97331-16
Liquid injection sample vials	1.5 mL Amber glass vial w/Cap & septa	220-97331-31
Methanol	Methanol, LCMS Honeywell Chromasolv(R); 99.9%	220-91545-11
Ultra-pure water	Water, LCMS Honeywell Chromasolv(R); 99.9%	220-91545-12
SPME fiber [a]	Smart Fiber, SPME, 80 µm DVB/C-WR/PDMS 1pc,	227-35345-01
SPME fiber [b]	SPME fiber assembly 50/30 µm(DVB/CAR/PDMS) (Millipore Sigma)	57298-U
Methylene Chloride	Methylene Chloride (GC Resolv ™) Fisher Chemical (Fisher Scientific)	D154-4

[a] Configure with an AOC-6000 Plus multifunctional autosampler.
[b] Configure with an AOC-6000 multifunctional autosampler.

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