SHIMADZU
Excellence in Science

Application News

Gas Chromatograph Mass Spectrometer

Analysis of Volatile PFAS in Textiles Using GCMS-TQ8040 RX with MMI

Yui Higashi, Masataka Furuta

User Benefits

- Using the GCMS-TQ8040 RX and the Multi-Mode Injection Unit (MMI), 12 volatile PFAS including compounds listed in EN 17681-2, can be analyzed at the ppb level without preconcentration.
- The spike recovery test shows good percentage recoveries of all compounds (80–120 %) that are at or below the EU Persistent Organic Pollutants (POPs) threshold limit (25 ppb).

■ Introduction

Per- and polyfluoroalkyl substances (PFAS) are used in textile products because of their useful properties, such as heat resistance and water repellency. However, PFAS are also extremely stable substances that resist degradation and accumulate in the environment, which has led to concerns about their effects on humans. Since eliminating sources of PFAS is essential to prevent them from accumulating in the environment and in living organisms, many countries are passing laws that restrict the production, distribution, and use of PFAS in products. For example, the EU POPs Regulation sets a permitted threshold of up to 0.025 mg/kg (25 ppb) for certain PFAS (including PFOA, PFOS, and PFHxS) in substances, mixtures, or articles[1]. EN 17681 defines standard test methods for PFAS analysis in textiles[2),3)].

In a previous Application News, No. 01-01108, an example of analyzing volatile and nonvolatile PFAS in textiles using LC/MS/MS and GC/MS/MS based on this standard was introduced. This study shows an example of trace analysis of volatile PFAS (Table1) in textiles using the newly developed GCMS-TQ RX series equipped with the Multi-Mode Injection Unit (MMI).

Table 1 Target Compounds

#	Compounds	Sample Type	IS
1	Me-PFOA	Target	-
2	4:2FTOH*1	Target	1
3	6:2FTOH*1	Target	1
4	8:2FTOH*1	Target	1
5	10:2FTOH*1	Target	1
6	6:2 FTA	Target	2
7	8:2 FTA	Target	2
8	10:2 FTA	Target	2
9	6:2 FTMA	Target	2
10	8:2 FTMA	Target	2
11	NMeFOSE	Target	2
12	NEtFOSE	Target	2
13	9Me 8:2 FTOH	Surrogate	1
14	D7-N-MeFOSE	Surrogate	2

*1: Not targeted by EN 17681-2 but measured by GC/MS/MS

■ GCMS-TQ8040 RX

Delivering highly sensitive multi-component simultaneous analysis, the system improves operational efficiency through Analytical Intelligence™, while also providing performance as a single GC-MS. The GCMS-TQ RX Series is a versatile triple quadrupole GC-MS that demonstrates high performance across a wide range of application fields. By combining it with the newly developed Multi-Mode Injection Unit (MMI), multiple injection methods are supported and analytical capability is further expanded.

Fig. 1 GCMS-TQ™8040 RX

■ Multi-Mode Injection Unit (MMI)

The Multi-Mode Injection Unit (MMI) supports a wide range of injection modes in a single unit, including split/splitless (SPL), programmable temperature vaporization (PTV), large-volume injection (LVI), direct injection, and thermal desorption/extraction (TD/TE). Dedicated software with an intuitive user interface enables easy method creation and seamless method transfer, simplifying analytical workflows. In this Application News, use of the MMI enables a threefold increase the injection volume compared with conventional injection methods, while maintaining peak shape.

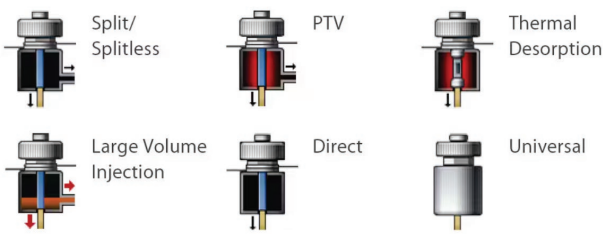


Fig. 2 Multi-Mode Injection Unit

■ Optimization of MMI Injection Conditions

With conventional SPL units, the maximum injection volume that could be used while maintaining peak shape and reproducibility was about 2 µL because of the limited vaporization chamber volume. In this study, using the programmed temperature vaporization injection mode of the MMI, the injection volume was increased from 2 µL to 6 µL. As a result, peak intensity increased for all compounds.

For Me-PFOA, peak broadening occurred as the injection volume increased because of the methanol used as the solvent. However, by using the co-injection mode of the AOC-30i Autosampler and co-injecting 2 µL of ethyl acetate into 6 µL of sample, the peak shape was improved. Through optimization of the injection conditions, the injection volume could be increased while maintaining peak shape (Fig. 3).

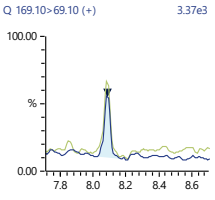
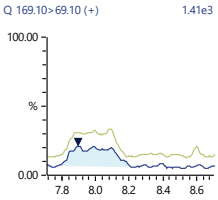
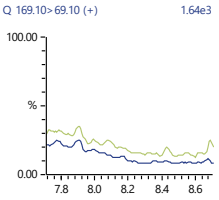
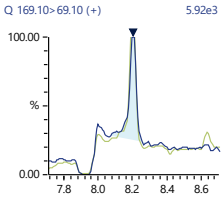
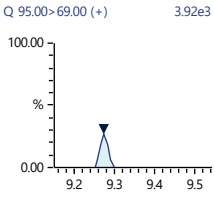
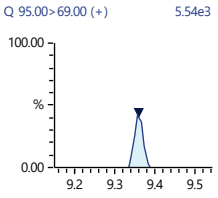
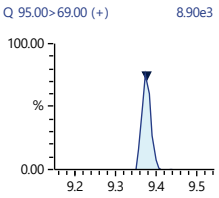
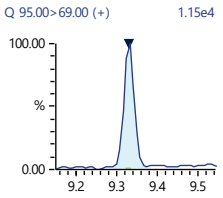
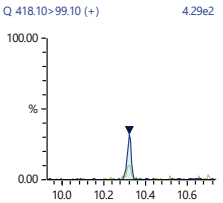
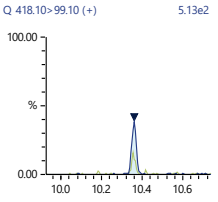
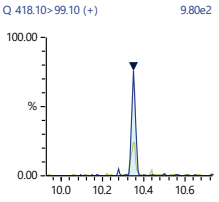
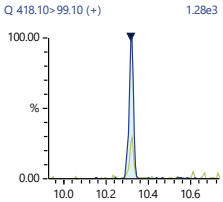
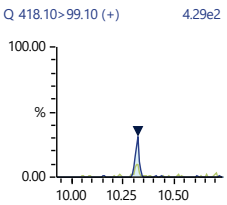
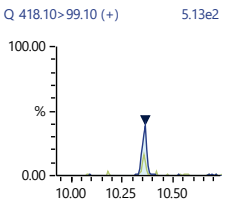
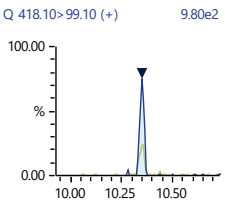
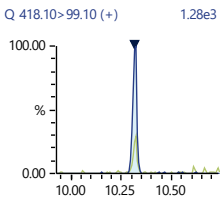
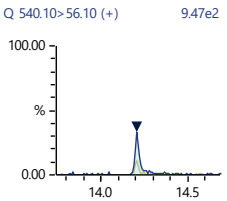
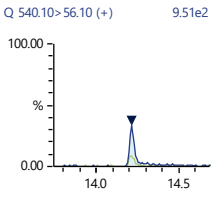
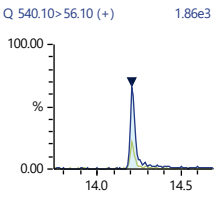
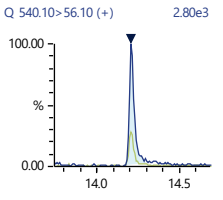
Injection volume	Sample 2 µL	Sample 4 µL	Sample 6 µL	Sample 6 µL + Ethyl Acetate 2 µL
Me-PFOA				
6:2 FTOH				
6:2 FTA				
6:2 FTMA				
N-EtFOSE				

Fig. 3 MRM Chromatograms of Standard Samples at 1 µg/L for Compounds with Changed Injection Volumes.

■ EN 17681-2 Sample Preparation

The EN 17681-2 sample preparation method is shown in Fig. 4. Samples were prepared by cutting a 1 g sample into pieces no larger than 1 cm square, adding methanol and a surrogate, and then performing ultrasonic extraction at 60 °C for 2 hours. The resulting extracted solution was centrifuged, and the supernatant analyzed by GC/MS/MS. EN 17681-2 states that samples can be concentrated by nitrogen purging if necessary, but this step was omitted.

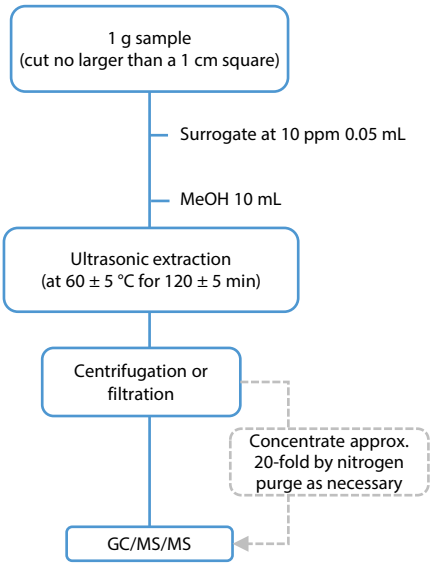


Fig. 4 EN 17681-2 Sample Preparation

■ Measurement of Calibration Standards

Calibration standards were prepared at concentrations between 1 and 100 µg/L to construct internal standard calibration curves, except for Me-PFOA. Table 3 shows calibration standard measurements. 9Me 8:2 FTOH and D7-N-MeFOSE were used as the internal standard. The four FTOH compounds in the Table 3 are not targeted by the EN 17681-2 method. Typically, they are analyzed more often by GC/MS(/MS) than by LC/MS/MS. For this reason, these four FTOH compounds were also measured by the GC/MS/MS. The coefficient of correlation (R) was greater than 0.998 for all calibration curves, showing good linearity. The repeatability (n = 5) of the lowest calibration curve concentration was also good for all compounds at %RSD below 12. Fig. 5 shows MRM chromatograms for 12 target compounds at 1 µg/L. These MRM chromatograms show good peak profiles even at the lowest calibration concentration.

Table 3 Results for Calibration Standards

#	Compound	Quantifier Ion	Calibration Curve Range (µg/L)	Correlation Coefficient (R)	Area %RSD (n = 5) at 1 µg/L
1	Me-PFOA	169.10>69.10	1-100	0.9990	7.3 %
2	4:2FTOH	196.10>127.10	1-100	0.9996	6.2 %
3	6:2FTOH	95.00>69.00	1-100	0.9983	2.8 %
4	8:2FTOH	95.00>69.00	1-100	0.9985	11.4 %
5	10:2FTOH	95.00>69.00	1-100	0.9981	8.6 %
6	6:2 FTA	418.10>99.10	1-100	0.9995	7.3 %
7	8:2 FTA	518.10>99.00	1-100	0.9993	4.2 %
8	10:2 FTA	618.10>99.10	1-100	0.9989	8.3 %
9	6:2 FTMA	432.10>113.20	1-100	0.9994	5.4 %
10	8:2 FTMA	532.10>113.10	1-100	0.9991	7.0 %
11	NMeFOSE	526.10>461.70	1-100	0.9991	2.1 %
12	NEtFOSE	540.10>56.10	1-100	0.9993	4.4 %
13	9Me 8:2 FTOH	95.00>69.00	50	-	1.6 %
14	D7-N-MeFOSE	531.10>169.10	50	-	3.1 %

■ EN 17681-2 (GC/MS(/MS)) Analysis Conditions

Analysis was performed using a GCMS-TQ8040 RX triple-quadrupole mass spectrometer (Fig. 1) combined with an (MMI) (Fig. 2). The analytical conditions are shown in Table 2.

Table 2 GCMS-TQ8040 RX Analysis Conditions

GC (Nexis GC-2060)		
Autosampler:		AOC-30i
MMI Mode:		Programmable temperature vaporization
Injection Mode:		Pulsed splitless mode (high pressure injection 250 kPa, 2.5 min)
Sampling Time:		2min
Injection Temp.:		40 °C (0.1 min) ⇒ 150 °C/min ⇒ 280 °C (17 min)
Carrier Gas:		He
Carrier Gas Control:		Linear velocity (40 cm)
Column:		SH-I-624Sil MS (P/N 221-75963-60) (60 m × 0.32 mm I.D., 1.8 µm)
Guard Column:		SH-I (P/N 227-36305-01) (5 m × 0.32 mm)
Column Temp.:		40 °C (3 min) – 25 °C/min – 250 °C – 15 °C/min – 280 °C (5 min)
Injection Volume	L1:	6 µL (Sample)
	L2:	1 µL (Ethyl acetate)
	L3:	1 µL (Ethyl acetate)
MS (GCMS-TQ8040 RX):		
Ion Source Temp.:		200 °C
Interface Temp.:		250 °C
Acquisition Mode:		MRM
m/z:		See Table 3

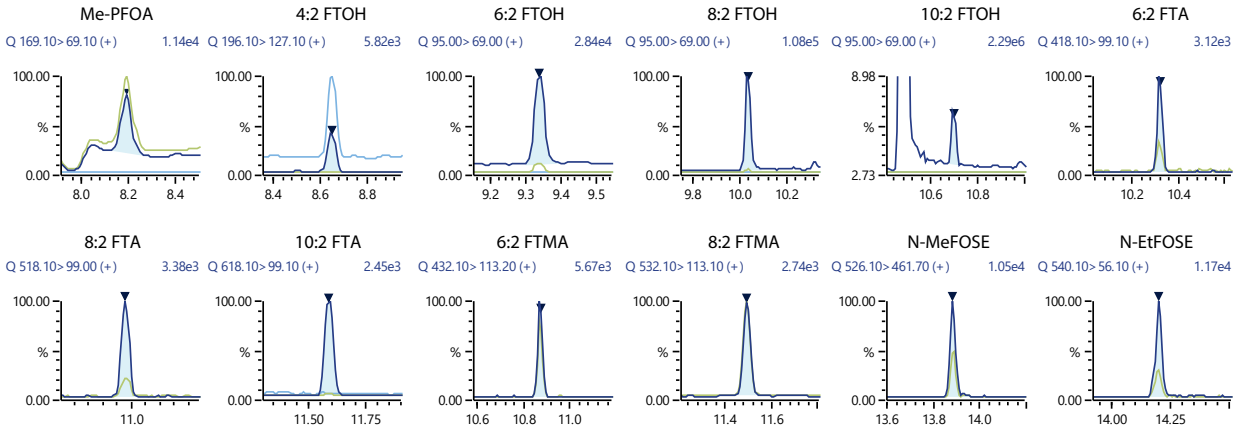


Fig. 5 MRM Chromatograms at 1 µg/L

■ Spike Recovery Test

A 1 g fragment of a 100 % cotton glove was used as a sample, and PFAS were added to achieve a concentration of 25 ppb in the sample. Pretreatment was performed in accordance with Fig. 4. The spike recovery results are shown in Fig. 6. Overall, the method demonstrated good recovery for all spiked compounds, with mean percentage recovery (n = 3) within 80 to 120 % for all compounds and the percentage recovery %RSD (n = 3) within 10 %.

■ Results from Analysis of Ski Wear

Analyzing the sample by GC/MS/MS using the EN 17681-2 method detected 8 out of the 12 compounds targeted in this study. Of these, 8:2 FTOH and 10:2 FTOH were detected at levels above the upper limit of their respective calibration curve ranges (Fig. 7).

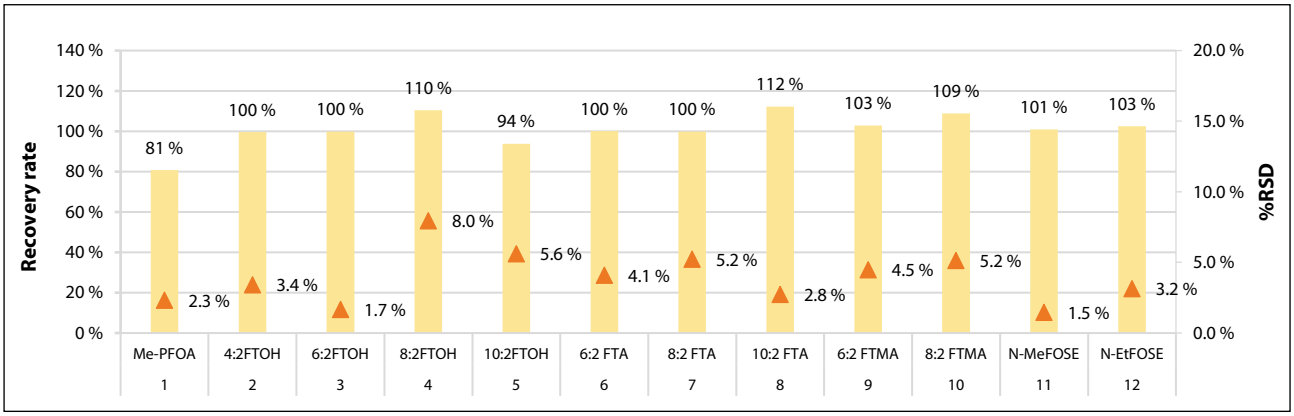


Fig. 6 Percentage Recoveries and Repeatability Data

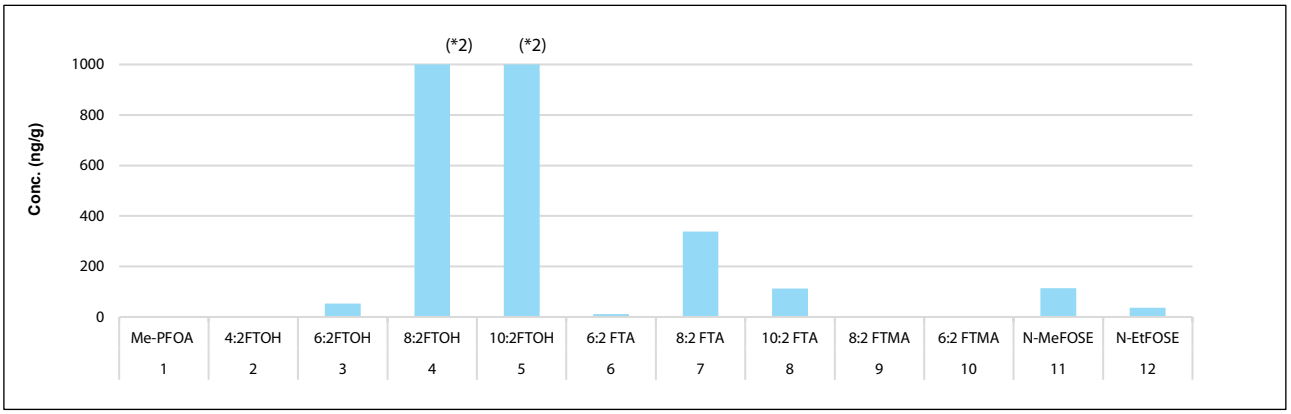


Fig. 7 Sample Measurements

*2: These compounds were detected at levels above the calibration curve concentration range. comparison, the graph in Fig. 7 shows data up to the maximum concentration on the calibration curve.

■ Conclusion

Using the GCMS-TQ8040 RX and the MMI, volatile PFAS in textiles were analyzed. The results showed that all compounds measured in this study could be analyzed at 25 ppb or below. Furthermore, in the spike recovery test at the EU POPs regulatory limit of 25 ppb, recoveries were within 80 to 120 % for all compounds, showing good results. By increasing the injection volume with the MMI, trace levels of volatile PFAS can be measured even with the standard GCMS-TQ8040 RX model.

Acknowledgements
We would like to express our sincere gratitude to Mr. Na and Dr. Kim at KOTITI Testing & Research Institute for their valuable support in this research.

References

1) EUR-Lex - 02019R1021-20251203 - EN - EUR-Lex (accessed July 21, 2026)
2) EN 17681-1:2025 Textiles and textile products - Per- and polyfluoroalkyl substances (PFAS) - Part 1: Analysis of an alkaline extract using liquid chromatography and tandem mass spectrometry (accessed July 21, 2026)
3) EN 17681-2:2022 Textiles and textile products - Organic fluorine - Part 2: Determination of volatile compounds by extraction method using gas chromatography (accessed July 21, 2026)

01-01257-EN First Edition: Aug. 2026

SHIMADZU
Shimadzu Corporation
www.shimadzu.com/an/

› Please fill out the survey

Related Products Some products may be updated to newer models.

› GCMS-TQ RX Series
Triple Quadrupole Gas Chromatograph Mass Spectrome···

› LabSolutions™ GCMS
Analytical Data System for GCMS

Related Solutions

› Environment

› PFAS

› Chemicals

› Textile

› Price Inquiry

› Product Inquiry

› Technical Service / Support Inquiry

› Other Inquiry