Application Note

Material

Quantitative Volatile PFAS Analysis in Textiles

Using GC-triple quadrupole mass spectrometry

Authors

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Introduction

Per- and polyfluoroalkyl substances (PFAS) are a group of manmade chemicals widely used in textiles for their water, stain, and oil-repellent properties. For decades, the textile industry has been using PFAS for enhancing fabric durability and performance.[1,2] Scientific studies have raised concerns about its potential risks to health.[3-5]

As awareness of these risks has intensified, regulations like REACH/POPs have introduced stringent limits and, in many cases, initiated phase-outs of PFAS usage.[6] Industry-driven standards, such as the ZDHC Manufacturing Restricted Substances List, OEKO-TEX Standard 100, AFIRM Restricted Substance List, and Bluesign standards and criteria complement regulations and reflect a global shift toward stricter compliance.[7-10] Analytical standards like EN 17681-1:2025 provide test methods for PFAS in textiles.[11] To keep pace with evolving regulations and industry initiatives, robust and highly sensitive analytical methods are essential for accurate PFAS detection and quantification in complex textile matrices.

This study demonstrates the ability of the Agilent 8890 GC coupled with the Agilent 7010D GC/TQ system to detect and quantify more than 30 volatile PFAS analytes across various textile matrices, with comprehensive evaluation of sensitivity, accuracy, and precision.

Experimental

Chemicals and consumables

Native and isotopically labeled PFAS standards were sourced from Wellington Laboratories Inc. (Guelph, ON, Canada), AccuStandard, Apollo Scientific, Santa Cruz Biotechnology, and Cambridge Isotope as both stock solutions and in powdered form. Ethyl acetate (EA) and methanol (MeOH) used for this study were purchased from Sigma and tested for suitability in PFAS analysis. All consumables used were from Agilent and were verified to deliver ultratrace levels of PFAS background.

Instrumentation

An 8890 GC with a 7010D GC/TQ equipped with an HES 2.0 ion source was used for this analysis. An Agilent MMI inlet and splitless liner (part number 5190-2293) were used, and chromatographic separation was performed using an Agilent J&W DB-624 column, 30 m × 0.25 mm, 1.40 µm (part number 122-1334UI). The instrument setup is shown in Figure 1. The acquisition method for 34 native PFAS and four internal mix standards (ISTD), including GC condition and TQ parameters, were described in a previous application note.[12]

Figure 1. Agilent 8890 GC with an Agilent 7010D GC/TQ.

Preparation of calibration standards

A stock mix standard solution and ISTD were prepared at a concentration of 1.0 µg/mL (ppm) and 0.1 µg/mL (ppm) in EA, respectively. Eight levels of calibration standards, ranging from 0.5 to 100 ng/mL (ppb), were prepared from intermediate standard solutions. Each calibration level contained a constant ISTD concentration of 5 ng/mL (ppb). All standards were thoroughly mixed prior to GC injection.

Sample extraction

Methanol was used as an extraction solvent for textile samples in accordance with Annex E in EN 17681-1:2025. The extraction procedure is illustrated in Figure 2. Three apparel products—T-shirts, water-proof shorts, and socks—were locally sourced for analysis. To assess PFAS in apparel, the T-shirt was designated as the quality control (QC) sample due to trace-level PFAS detected during preliminary screening. The QC sample was spiked in duplicate at three concentration levels: 10 µg/kg (LSQ), 25 µg/kg (MSQ), and 200 µg/kg (HSQ). These levels were used to evaluate method performance in terms of method detection limits (MDLs), limits of quantification (LOQ), recovery, and precision. Procedural blanks and matrix blanks were included to monitor PFAS background from the extraction process and the QC sample. The remaining apparel products (shorts and socks) were analyzed as unknown samples to demonstrate method applicability across different textile types.

1. Weigh a 1.0 ± 0.05 g sample into a 50 mL sample tube.
2. Spike the appropriate amount of ISTD and target PFAS mix (where QCs required) into the sample tube.
3. Add extraction solvent and sonicate at 60 °C for 1 hour.
4. Cool down and centrifuge.
5. Filter, dilute with EA, and inject onto the GC.

Figure 2. Sample extraction procedure (note that the process included a 20-fold dilution).

Results and discussion

Calibration performance and method sensitivity

The calibration performance was assessed across eight calibration levels for analyte concentration, ranging from 0.5 to 100 ppb. Linear regression was applied, ignoring the origin and using a 1/x weighting. The calibration curve linearity for all 34 targets exceed $R^2 > 0.99$ with a minimum of five calibration points. Method analytical performance is summarized in Table 1, including MDLs, LOQs, and recovery. The certification requirement, in terms of formulation limit for mandatory compounds from ZDHC, is displayed in Table 1.

Table 1. Method analytical performance summary.

S/N	Compound Name	CAS No.	MDL (µg/kg)	LOQ (µg/kg)	Recovery (%)	Certification Requirement from ZDHC (µg/kg)
1	10:2 FTUCA	70887-94-4	0.051	10	117	
2	4:2 FTOH	2043-47-2	0.115	10	113	1000
3	1H,1H-Perfluoro-3,6,9-trioxadecan-1-ol	147492-57-7	0.144	25	108	
4	3-(Perfluorohexyl)-1,2-epoxypropane	38565-52-5	0.078	10	96	
5	3:3 FTOH	679-02-7	0.128	10	110	
6	6:1 FTOH	375-82-6	0.077	10	97	
7	5H 4:1 FTOH	355-80-6	0.113	10	84	
8	6:2 FTOH	647-42-7	0.099	10	101	1000
9	PFODA	16517-11-6	0.557	N.D.	< 60 (42)	
10	1H,1H-Perfluorooctyl acrylate	307-98-2	0.990	N.D.	< 60 (53)	
11	(Heptafluorobutanoyl)pivaloylmethane	17587-22-3	0.348	10	86	
12	((2,2,3,3-Tetrafluoropropoxy)methyl)oxirane	19932-26-4	0.114	10	100	
13	Nonafluoropentanamide	13485-61-5	0.093	10	99	
14	7H 6:1 FTOH	335-99-9	0.176	10	92	
15	8:2 FTOH	678-39-7	0.170	10	95	1000
16	10:1 FTOH	307-46-0	0.158	10	94	
17	7:3 FTOH	25600-66-2	0.173	10	115	
18	11:1 FTOH	423-65-4	0.254	10	120	
19	10:2 FTOH	865-86-1	0.667	10	71	1000
20	Perfluoropentanamide	355-81-7	0.108	10	61	
21	8:2 FTAC	2790545-9	0.074	10	83	
22	Perfluorooctanamide	423-54-1	0.296	10	91	
23	1H,1H,9H-Perfluorononyl acrylate	4180-26-1	0.637	25	98	
24	MeFBSA	68298-12-4	0.106	10	109	
25	Triethoxy((perfluorohexyl)ethyl)silane	51851-37-7	0.070	10	71	
26	1H,1H,8H,8H-Perfluoro-3,6-dioxaoctane-1,8-diol	129301-42-4	0.126	200	138	
27	MeFHxSA	68259-15-4	0.044	10	110	
28	FBSA	30334-69-1	0.058	10	119	
29	N-MeFOSA	31506-32-8	0.026	10	107	
30	FHxSA	41997-13-1	0.098	10	111	
31	N-EtFOSA	4151-50-2	0.127	10	115	
32	PFOSA	754-91-6	0.135	25	113	
33	MeFOSE	24448-09-7	0.050	10	105	
34	N-EtFOSE	1691-99-2	0.075	10	103	

N.D. = Not detected

MDLs obtained from nine continuous injections of QC samples were used to evaluate instrument sensitivity in sample matrix and calculated using Agilent MassHunter Quantitative Analysis software, version 12. A sub-ppb level of MDLs was achieved for all analytes, demonstrating the superior sensitivity of the Agilent 7010D for trace levels of volatile PFAS analysis in sample matrix.

LOQs were used to evaluate the overall workflow performance against standard requirements. In this study, LOQs were determined based on matrix-spiked QC levels while meeting compound identification criteria and achieving recoveries between 60% and 140%, with %RSD ≤ 20%. Overall, 28 compounds reached an LOQ of 10 µg/kg and three compounds achieved 25 µg/kg (Table 1), demonstrating that the developed workflow delivers high extraction efficiency and excellent sensitivity for most volatile PFAS in textile matrices. LOQs were not determined for PFODA and 1H,1H-Perfluorooctyl acrylate due to poor QC recoveries (40% to 53%) as indicated in Table 1. For four specific compounds—4:2 FTOH, 6:2 FTOH, 8:2 FTOH, and 10:2 FTOH—an LOQ of 10 µg/kg was achieved, surpassing the formulation limits set by ZDHC. These findings confirm that the workflow is suitable for PFAS screening in textiles and ensures compliance with standard methods and regulatory requirements.

Method recovery and repeatability

Sample preparation performance was evaluated based on matrix-spiked QC recoveries and %RSD across three QC levels. For each level, two technical replicates with three injections were analyzed to calculate mean recoveries and %RSD (n = 6). More than 80% of analytes achieved recoveries within the acceptable range of 60% to 140% across LSQ, MSQ, and HSQ. Figure 3 shows recovery values at the LOQ level for all analytes. Notably, FTOHs exhibited recoveries between 70% and 120% (highlighted in dark blue), indicating high extraction efficiency of the workflow for volatile PFAS in textile matrices.

Workflow repeatability was assessed using %RSD of recoveries at MSQ. As shown in Figure 4, all analytes demonstrated %RSD below 20%, confirming that the workflow is highly reproducible and reliable for routine PFAS analysis in textiles.

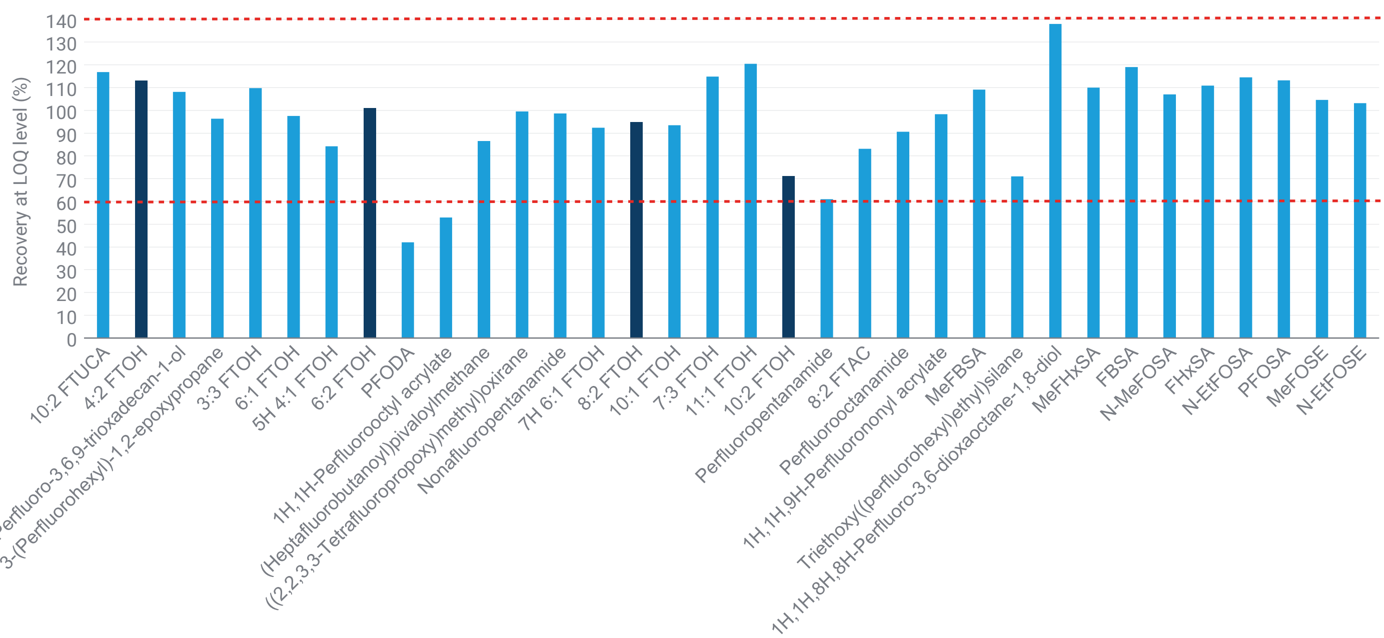

Figure 3. Recovery values at LOQ level for all analytes.

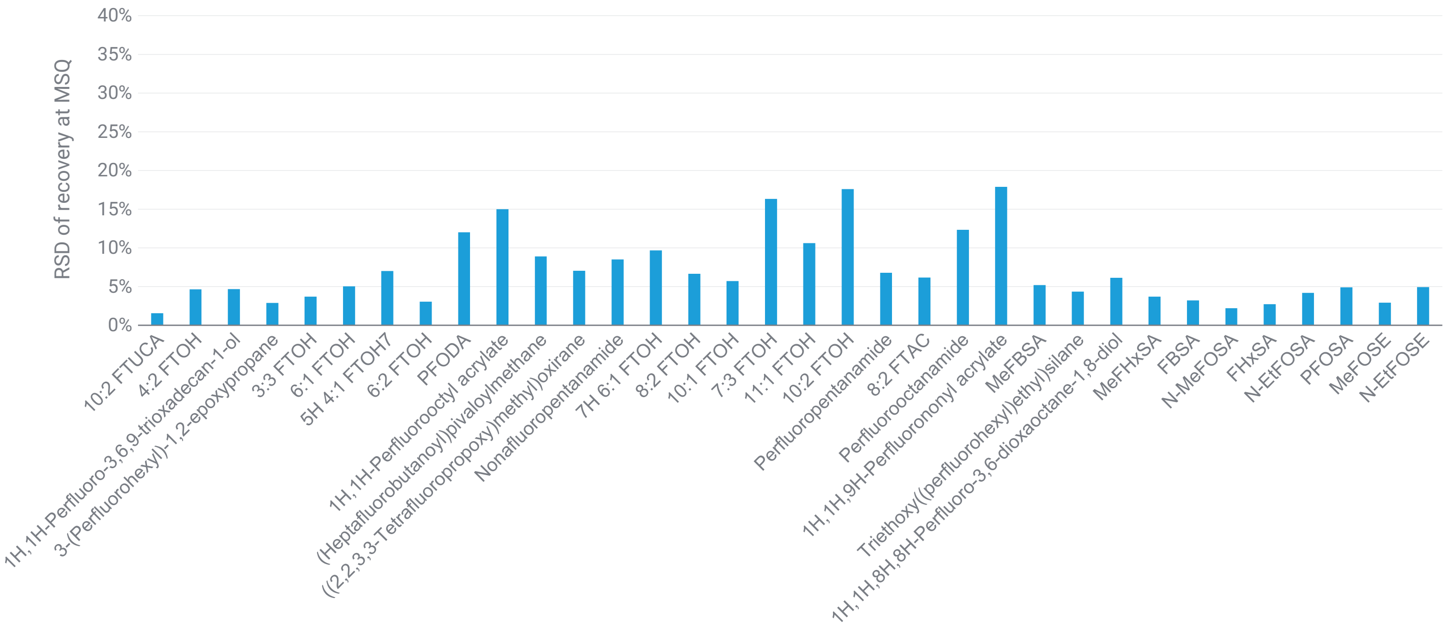

Figure 4. %RSD of recoveries at MSQ for all analytes.

Sample analysis

PFAS are widely used in textile materials due to their exceptional resistance to heat, water, and oil.[1,2,13] In this study, PFAS compounds—specifically 8:2 FTOH and 10:2 FTOH, were detected above MDLs in sample waterproof shorts, as shown in Figure 5. However, the concentrations of these two compounds remained within the formulation limits set by ZDHC. Although FTOHs are not explicitly listed as individual compounds under the EU POPs regulation, they fall under restrictions applied to related substances such as PFOA, PFCA, and PFHxA. These results demonstrate that the developed method enables accurate and reliable detection of PFAS in apparel products, providing valuable data to support regulatory bodies in risk management and the establishment of appropriate limits.

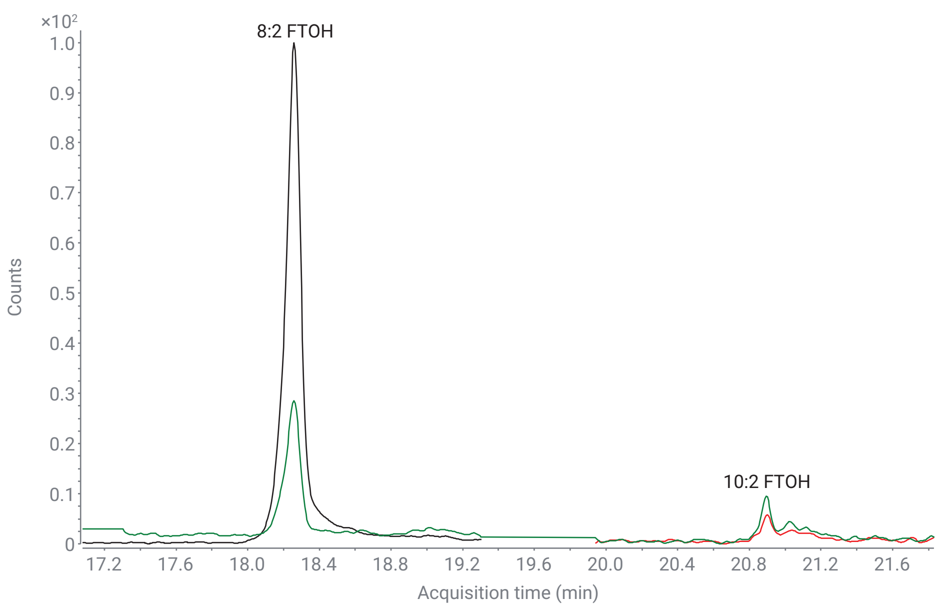

Figure 5. The MRM chromatogram of waterproof shorts with 8:2 FTOH and 10:2 FTOH determined above MDLs.

Conclusion

An accurate and robust method was developed using an Agilent 8890 GC coupled with an Agilent 7010D GC/TQ system for the analysis of volatile PFAS in textiles, following the established analytical guidelines. The workflow delivered excellent performance, achieving sub-ppb MDLs for all targets and LOQs of 10 µg/kg for more than 80% of compounds. These results confirm high extraction efficiency and exceptional sensitivity for PFAS in textile matrices. Recovery precision (%RSD) remained within 20% across sample preparations, underscoring method reproducibility and suitability for routine, high-throughput PFAS analysis in materials such as textiles.

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