Application Note

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Volatile PFAS in Cosmetics Using PAL3 Coupled with Triple Quadrupole GC/MS

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Abstract

This application note describes a fully automated workflow for the quantification of over 30 volatile PFAS analytes in cosmetics, including foundations, lipsticks, and mascaras. The method combines a PAL3 Series 2 RTC autosampler with an Agilent 7010D triple quadrupole GC/MS. The analytical performance of the workflow was evaluated in terms of sensitivity, accuracy, and precision.

Introduction

Per- and polyfluoroalkyl substances (PFAS) are a large family of synthetic organic chemicals containing carbon chains where most or all the hydrogen atoms are replaced by fluorine. Due to the strength and stability of the carbon-fluorine bond, PFAS tend to be very stable and chemically inert, which makes them both very useful in various applications and resistant to degradation. This persistence causes environmental contamination and bioaccumulation in humans and wildlife; as a result, PFAS are often labeled "forever chemicals".[1]

In cosmetics, PFAS are valued for their functional properties, including emulsification, stabilization, and water or sweat resistance. Despite these benefits, concerns have grown over their safety due to direct exposure from repeated use through skin, eyes, and mouth. The OECD's 2024 report underscores the risks associated with PFAS in cosmetic formulations, particularly given their potential for long-term health and environmental impacts.[2,3] Regulators around the world are taking decisive steps to ban or restrict PFAS in cosmetics. For example, in the EU, various types of PFAS have been regulated under REACH and POPs regulations that span most industries, including cosmetics.[4,5] EU Cosmetics Regulation (EC) No. 1223/2009 has listed the five types of PFAS as the substances prohibited.[6] A broader EU-wide ban is underway, with France having passed its own ban on PFAS in cosmetics effective 1 January 2026, as part of a larger proposal to ban PFAS in consumer products.[7,8] The US has not banned the use of PFAS in cosmetics at the federal level yet. The USFDA has been evaluating the use of PFAS in cosmetics under the authority of the Cosmetic Regulatory Modernization Act (MoCRA), and is expected to release a report on safety and risk by the end of 2025.[9] Although the federal government is still on the sidelines, many states in the US have been rapidly enacting or implementing the ban and reporting requirements for PFAS in cosmetics, with effective dates ranging from 2025 to 2032.[10-13] Canada has proposed a ban on the use of PFAS in various consumer products, including cosmetics.[14] South Korea has added 12 PFAS into a restricted substances list for cosmetic products in 2024.[15] The Environmental Protection Authority (EPA) of New Zealand has officially announced a ban on the use of PFAS in cosmetic products, effective from 31 December 2026.[16]

As regulations surrounding PFAS continue to evolve, developing a sensitive and robust analytical method is crucial to meet stringent guidelines. The objective of this study was to demonstrate a fully automated workflow using PAL3 Series 2 RTC autosampler coupled with an Agilent 7010D triple quadrupole GC/MS to quantify more than 30 volatile PFAS analytes in cosmetics. The automation method performance includes sensitivity, accuracy, and precision studies.

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 stock solutions and in powdered form. Ethyl acetate (EA) and hexane used for this study were purchased from Sigma and tested for suitability in PFAS analysis.

Instrumentation

An Agilent 8890 GC with a 7010D GC/TQ equipped with HES 2.0 ion source, coupled with a PAL3 Series 2 RTC autosampler, was used for this analysis. An MMI inlet and splitless liner (part number 5190-2293) were used and chromatographic separation was performed using an Agilent J&W DB-624, 30 m × 0.25 mm, 1.40 µm column (part number 122-1334UI). The instrument setup is shown in Figure 1. The acquisition method for 34 native PFAS and four ISTDs, including GC condition, TQ parameters were described in a previous application note.[17]

Figure 1. Agilent 8890 GC with an Agilent 7010D GC/TQ coupled with a PAL3 Series 2 RTC autosampler.

Automated preparation of calibration standards

The calibration solutions were automatically prepared with the PAL3 autosampler. A stock mix standard solution and internal mix standard (ISTD) were manually prepared at a concentration of 1.0 and 0.1 µg/mL (ppm) in a solvent mixture ratio of 80:20 (EA:hexane), respectively. Subsequently, the PAL3 prepared intermediate standard solutions, followed by eight levels of calibration standards ranging from 0.5 to 100 ng/mL (ppb) with a constant amount of ISTD spiked into each level. The standards were mixed well and ready for GC injection.

Automated sample preparation

The entire automated sample preparation workflow is illustrated in Figure 2. Mixed organic solvents with different polarities were recommended for extracting PFAS from high-oil content matrix, such as cosmetics.[18,19] The mixture of EA and hexane was used as extraction solvent in this study to maximize the extraction efficiency for a wide range of PFAS.

Seven cosmetic products from various brands—including foundations, lipsticks, and mascaras—were locally sourced and selected for analysis. These product types have previously been reported to contain PFAS.[2,19] Among them, a liquid makeup foundation with trace-level PFAS background (based on initial screening) was designated as the quality control (QC) sample for this study. Three QC levels were prepared by spiking the foundation matrix at target concentrations of 50 µg/kg (low spike quality, LSQ), 125 µg/kg (medium spike quality, MSQ), and 250 µg/kg (high spike quality, HSQ). Each level was prepared in duplicate to assess method performance in terms of sensitivity, recovery, and reproducibility. An unspiked portion of QC sample was used as a matrix blank (MB). The remaining six cosmetic products were analyzed as unknown samples to evaluate the applicability of the method across different types of cosmetic products (**Note:** The complete procedure in .xml format is available upon request).

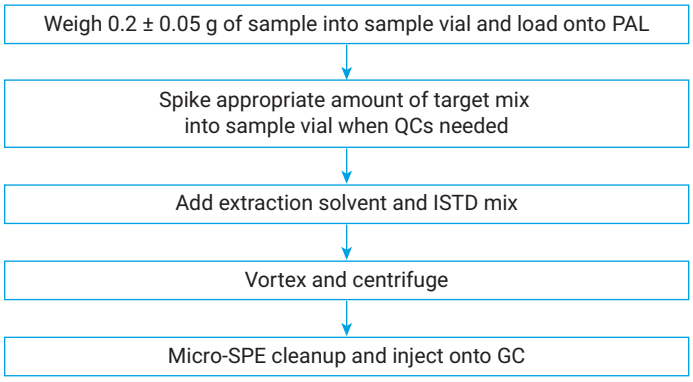

Figure 2. Automated sample preparation procedure (**Note:** 25 times dilution was involved due to the process).

Results and discussion

Method linearity

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. Figure 3 shows the linearity of 4:2 FTOH (A), 6:2 FTOH (B), 8:2 FTOH (C), and 10:2 FTOH (D) across the entire calibration level 1 to 8, demonstrating the excellent performance of the PAL3 autosampler for calibration preparation.

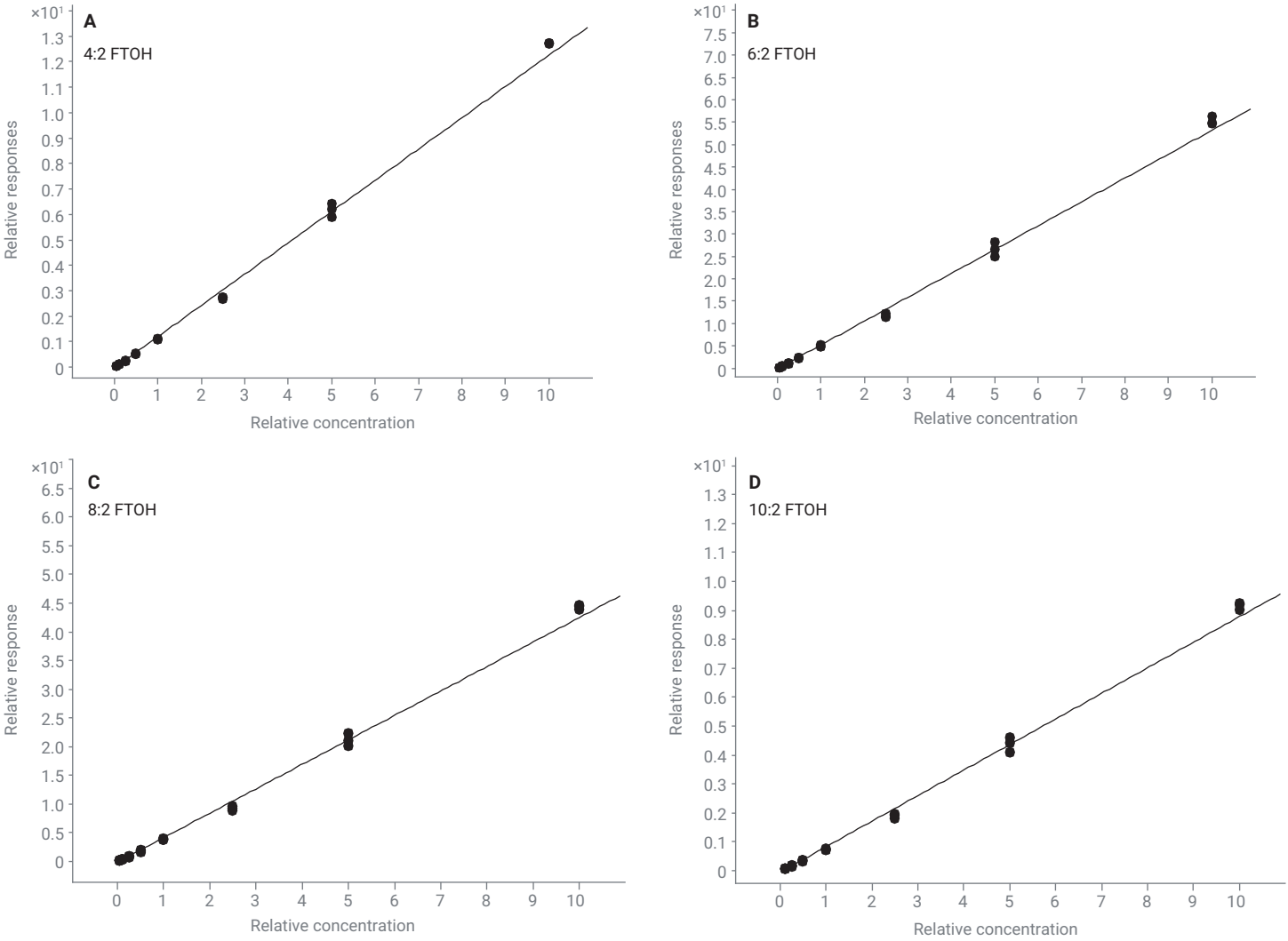

Figure 3. Linearity of representative FTOH compounds: 4:2 FTOH (A), 6:2 FTOH (B), 8:2 FTOH (C), and 10:2 FTOH (D) across the entire calibration range.

Method sensitivity: MDL and LOQ

The method detection limit (MDL) was evaluated based on nine continuous injections of QC samples. MDL values, calculated using Agilent MassHunter Quantitative Analysis software version 12, are summarized in Table 1.

Table 1. Analytical results summary.

S/N	Compound Name	CAS Number	ISTD	MDL (µg/kg)	LOQ (µg/kg)	Recovery at LOQ Level
1	10:2 FTUCA	70887-94-4	$^{13}C_2$-6:2 FTOH	66.43	250	96%
2	4:2 FTOH	2043-47-2	$^{13}C_2$-6:2 FTOH	0.62	50	101%
3	1H,1H-Perfluoro-3,6,9-trioxadecan-1-ol	147492-57-7	$^{13}C_2$-6:2 FTOH	0.53	50	106%
4	PFHxDA	67905-19-5	$^{13}C_2$-6:2 FTOH	N.D.	N.D.	< 20%
5	3-(Perfluorohexyl)-1,2-epoxypropane	38565-52-5	$^{13}C_2$-6:2 FTOH	0.46	50	89%
6	3:3 FTOH	679-02-7	$^{13}C_2$-6:2 FTOH	0.49	50	84%
7	6:1 FTOH	375-82-6	$^{13}C_2$-6:2 FTOH	0.84	50	76%
8	5H 4:1 FTOH	355-80-6	$^{13}C_2$-6:2 FTOH	0.45	50	82%
9	6:2 FTOH	647-42-7	$^{13}C_2$-6:2 FTOH	0.48	50	69%
10	PFODA	16517-11-6	$^{13}C_2$-6:2 FTOH	2.91	125	74%
11	1H,1H-Perfluorooctyl acrylate	307-98-2	$^{13}C_2$-6:2 FTOH	0.51	50	95%
12	((2,2,3,3-Tetrafluoropropoxy)methyl)oxirane	19932-26-4	$^{13}C_2$-6:2 FTOH	0.45	50	87%
13	Nonafluoropentanamide	13485-61-5	$^{13}C_2$-6:2 FTOH	0.33	50	86%
14	7H 6:1 FTOH	335-99-9	$^{13}C_2$-6:2 FTOH	0.61	50	68%
15	8:2 FTOH	678-39-7	$^{13}C_2$-6:2 FTOH	0.65	50	82%
16	10:1 FTOH	307-46-0	$^{13}C_2$-6:2 FTOH	0.59	50	85%
17	7:3 FTOH	25600-66-2	$^{13}C_2$-6:2 FTOH	0.66	50	85%
18	11:1 FTOH	423-65-4	$^{13}C_2$-6:2 FTOH	0.79	50	89%
19	10:2 FTOH	865-86-1	$^{13}C_2$-6:2 FTOH	1.01	125	104%
20	Perfluoropentanamide	355-81-7	$^{13}C_2$-6:2 FTOH	0.45	50	84%
21	8:2 FTAC	2790545-9	$^{13}C_2$-6:2 FTOH	1.46	50	105%
22	Perfluorooctanamide	423-54-1	$^{13}C_2$-6:2 FTOH	0.36	50	83%
23	1H,1H,9H-Perfluorononyl acrylate	4180-26-1	$^{13}C_2$-6:2 FTOH	0.76	50	84%
24	MeFBSA	68298-12-4	$^{2}H_3$-N-MeFOSA	N.D.	N.D.	< 20%
25	Triethoxy((perfluorohexyl)ethyl)silane	51851-37-7	d_7-N-MeFOSE-M	0.05	50	44%
26	1H,1H,8H,8H-Perfluoro-3,6-dioxaoctane-1,8-diol	129301-42-4	$^{2}H_3$-N-MeFOSA	0.19	50	98%
27	MeFHxSA	68259-15-4	$^{2}H_3$-N-MeFOSA	0.09	50	48%
28	FBSA	30334-69-1	$^{2}H_3$-N-MeFOSA	0.27	50	73%
29	N-MeFOSA	31506-32-8	$^{2}H_3$-N-MeFOSA	0.09	50	78%
30	FHxSA	41997-13-1	$^{2}H_3$-N-MeFOSA	0.26	50	74%
31	N-EtFOSA	4151-50-2	$^{2}H_3$-N-MeFOSA	0.26	50	84%
32	PFOSA	754-91-6	$^{2}H_3$-N-MeFOSA	0.23	50	68%
33	N-MeFOSE	24448-09-7	d_7-N-MeFOSE-M	0.09	50	88%
34	N-EtFOSE	1691-99-2	$^{2}H_9$-EtFOSE	0.14	50	82%

N.D. = Not determined

The method LOQs presented in Table 1 were established based on matrix-spiked QC levels, with recoveries ranging from 60% to 110% and %RSD ≤ 20%. Figure 4 illustrates the LOQ distribution across all analytes. Notably, 29 out of 34 compounds achieved an LOQ of 50 µg/kg, highlighting the high extraction efficiency and superior sensitivity of the automated workflow for the majority of volatile PFAS in the cosmetic matrix. An LOQ of 50 µg/kg was also assigned to triethoxy((perfluorohexyl)ethyl)silane and MeFHxSA, based on consistent recoveries of 40 to 50% across all QC levels with %RSD < 5%.

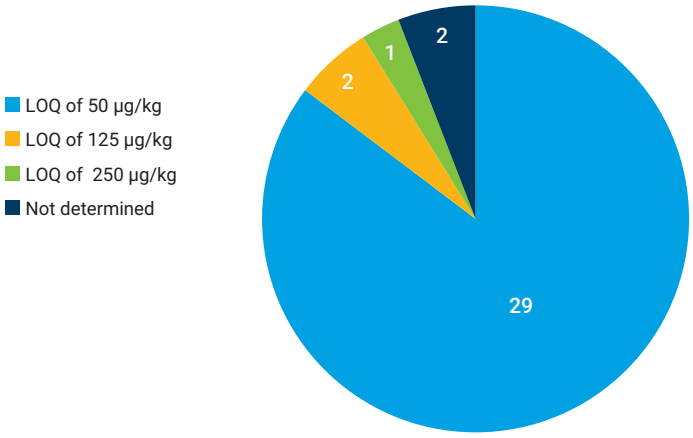

Figure 4. Method LOQ distribution for all analytes.

Method recovery and repeatability

Three matrix-spiked QC levels were defined and prepared in duplicate at concentrations of 50 µg/kg (LSQ), 125 µg/kg (MSQ), and 250 µg/kg (HSQ). Internal standards (ISTDs) were pre-spiked into the QC samples to compensate for matrix effects. These spiked QCs underwent the full extraction process, automated using the PAL3 autosampler, as illustrated in Figure 2. Target recoveries were calculated based on duplicate technical preparations with triplicate injections (n = 6). Method repeatability was evaluated by calculating the %RSD of recoveries from the two technical preparations at each QC level.

Overall, ≥ 80% of analytes exhibited recoveries within the acceptable range of 60% to 110% across all three QC levels. Recoveries at the LOQ level for all analytes are summarized in Table 1. Notably, %RSD values below 16% were achieved for all analytes at the MSQ level (Figure 5), meeting general performance requirements for PFAS analysis in other matrices.[20]

Figure 6 shows the overlaid chromatograms of 4:2 FTOH (A), 6:2 FTOH (B), and 8:2 FTOH (C) at the LOQ level from two technical preparations, corresponding to a ready-to-inject concentration of 2 ppb. These results underscore the reliability and robustness of the automated workflow for PFAS analysis in cosmetic products.

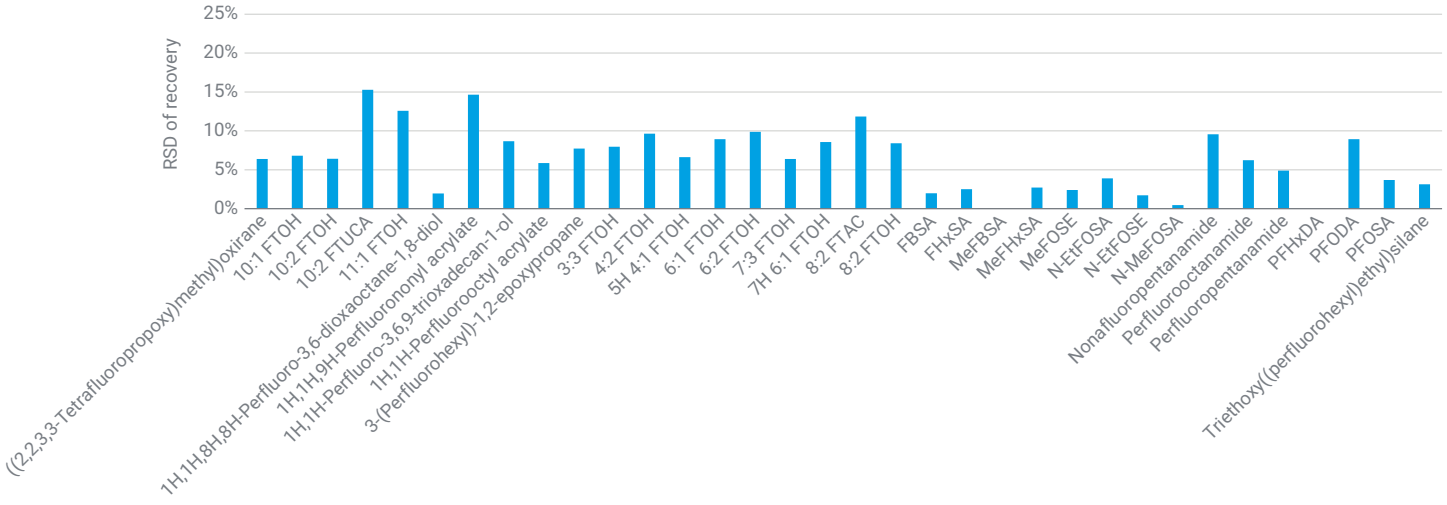

Figure 5. %RSD of recoveries for all targets at MSQ.

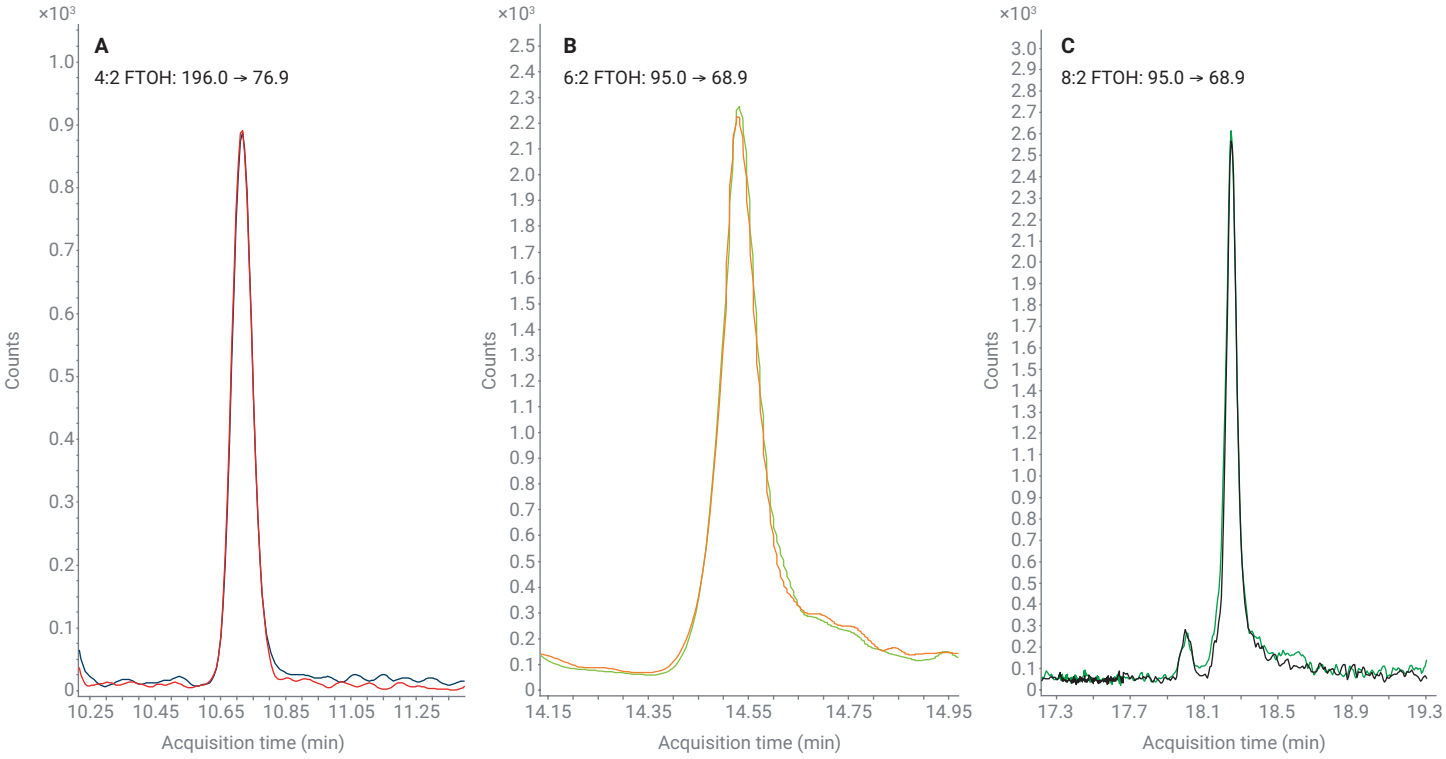

Figure 6. Chromatogram overlaid of 4:2 FTOH (A), 6:2 FTOH (B), and 8:2 FTOH (C) at LOQ level from two technical preparations.

Sample analysis

Seven cosmetic samples from various brands—including foundations, lipsticks, and mascaras—were analyzed. One liquid foundation was designated as a quality control (QC) sample, while the remaining six were treated as unknowns. According to the OECD report published in 2024, cosmetic products such as shampoo, eye makeup, foundation, facial cleansers, lipsticks, and lip glosses may contain PFAS, which serve functional or performance-enhancing roles.[2,19] In this study, several PFAS compounds were detected above the method detection limit (MDL) in the unknown samples, including 10:2 FTOH, MeFHxSA, MeFOSE, EtOSE and 1H,1H-perfluorooctyl acrylate. The results also demonstrated that the automated workflow is effective for PFAS extraction and quantitation in cosmetic matrices.

Conclusion

An accurate and robust automated workflow was successfully developed using the PAL3 Series 2 RTC autosampler coupled with the Agilent 7010D GC/TQ system for the analysis of volatile PFAS in cosmetic products. Most PFAS targets achieved an LOQ of 50 µg/kg and MDL below 1.5 µg/kg, with recoveries falling within the acceptable range of 60% to 110%. These results demonstrate excellent extraction efficiency and reliability of the automated workflow. Furthermore, automation significantly reduces human error and enhances productivity, while maintaining high sensitivity in PFAS analysis.

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