Application Brief

Cosmetics

Determination of 41 Per- and Polyfluoroalkyl Substances (PFAS) in Cosmetics

Using Captiva EMR PFAS Food II passthrough cleanup and LC/MS/MS detection

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Introduction

Per- and polyfluoroalkyl substances (PFAS) are synthetic compounds composed of highly stable carbon–fluorine (C–F) bonds, giving them exceptional thermal and chemical stability, leading to poor environmental degradability. PFAS have been used in cosmetics to provide water resistance, oil repellency, or application smoothness. The unintentionally presented PFAS residues in raw materials may remain in finished cosmetic products, and can be absorbed through the skin. Lip products and spray type products can also lead to inhalation or oral exposure to residues.[1,2]

Agilent Captiva EMR PFAS Food cartridges are designed to provide streamlined and comprehensive matrix removal after QuEChERS extraction for complex sample matrices, demonstrating effective matrix cleanup efficiency and excellent performance for quantitative PFAS analysis.[3,4]

In this study, we performed QuEChERS extraction followed by cleanup with Captiva EMR PFAS Food II cartridges for PFAS analysis in five types of cosmetics.

Experimental

Chemicals and reagents

Native PFAS and isotopically labeled internal standard (ISTD) solutions were from Agilent Technologies and Chemidas (Korea). A set of eight-level calibration standards (0.05 to 10 ng/mL) was prepared in acetonitrile (ACN) to match the extraction solvent.

Equipment and materials

The study was performed using an Agilent 1290 Infinity III LC system consisting of an Agilent 1290 Infinity III high-speed pump (G7120A), an Agilent 1290 Infinity III multisampler (G7167B), and an Agilent 1290 Infinity III multicolumn thermostat (G7116B). The LC system was coupled to an Agilent 6495D triple quadrupole LC/MS system (G6495D) equipped with an Agilent Jet Stream Electrospray ion source. Agilent MassHunter Workstation software was used for data acquisition and analysis.

The 1290 Infinity III LC system was modified using an Agilent InfinityLab PFAS Analysis HPLC conversion kit (part number 5004-0006), including an Agilent InfinityLab PFC delay column, 4.6 × 30 mm (part number 5062-8100). Chromatographic separation was performed using an Agilent ZORBAX RRHD Eclipse Plus C18, 95 Å, 2.1 × 100 mm, 1.8 µm (part number 959758-902).

Agilent consumables used for sample preparation included:

- Bond Elut QuEChERS EN extraction kit, EN 15662 method, buffered salts, ceramic homogenizers (part number 5982-5650CH)
- Captiva EMR PFAS Food II cartridges, 6 mL, 750 mg (part number 5610-2232)
- Captiva filter vial, 0.2 µm, nylon, 100/pk (part number 5610-5936)
- Polypropylene screw cap vials and caps, 2 mL (part number 5191-8121 and 5191-8151)
- Tubes and caps, 50 mL, 50/pk (part number 5610-2049)
- Tubes and caps, 15 mL, 100/pk (part number 5610-2039)

All consumables used in the study were tested and verified for acceptable PFAS cleanliness.

LC/MS/MS instrument conditions

Pump conditions:

Table 1 lists the LC pump conditions.

Table 1. LC pump conditions for LC/MS/MS.

Parameter	Setting
Mobile Phase A	2 mM NH_4OAc in water
Mobile Phase B	2 mM NH_4OAc in methanol (MeOH)
Gradient	Time (min) / A% / B% / Flow (mL/min) 0.0 / 95 / 5 / 0.3 1.0 / 75 / 25 / 0.3 6.0 / 50 / 50 / 0.3 13.0 / 15 / 85 / 0.3 14.0 / 5 / 95 / 0.3 17.0 / 5 / 95 / 0.3 17.1 / 95 / 5 / 0.3 18.0 / 95 / 5 / 0.3 Stop time: 18.0 min Post time: 2.0 min
Column Temperature	40 °C
Injection Volume	0.5 µL

MS acquisition parameters

The MS acquisition parameters were negative ion mode with constant fragmentor setting at 166 V and iFunnel standard mode.

MS electrospray ion source settings

The electrospray ion source settings include drying gas at 240 °C, 16 L/min; sheath gas at 370 °C, 11 L/min; nebulizer gas at 25 psi; capillary voltage at 3,200 V (negative) and 3,000 V (positive); and nozzle voltage at 0 V (negative) and 0 V (positive). The MS acquisition conditions for PFAS targets and internal standards were drawn from the Agilent PFAS MRM Database for LC/TQ (part number G1736AA).

Sample information

Five types of cosmetics (sunscreen, lotion, foundation, lipstick, and eyeshadow) were purchased from local stores. For each type, three different commercial products were purchased. To control the potential contamination from external sources, sample pretreatment was limited. However, solid cosmetic samples in powder form were gently tapped to facilitate slight pulverization prior to use.

Table 2. Cosmetic sample details.

Sample Name	Cosmetic Type	Feature
S-A	Sunscreen	Thin, lotion-type formulation
S-B		Thin, lotion-type formulation
S-C		Thick formulation for sports
L-A	Lotion	All-in-one lotion with a thin consistency
L-B		Hand cream
L-C		Functional cream, declared free of harmful chemicals
F-A	Foundation	Matte foundation
F-B		Moisturizing foundation
F-C		Foundation focusing on long-lasting power
Lip-A	Lipstick	A moisturizing lipstick with a glossy feel
Lip-B		Handmade products with a red tint
Lip-C		Lip balm that is free of tar pigments and is certified as nonirritating
E-A	Eyeshadow	Common product
E-B		Common product
E-C		Common product

Sample preparation

An aliquot of 1 g sample powder was extracted using QuEChERS extraction. The crude extract was then diluted with extraction solvent and water to generate a final mixture in 90:10 ACN/water. This sample mixture was then loaded into Captiva EMR PFAS Food II cartridges for matrix removal. Figure 1 shows the sample preparation procedure in detail.

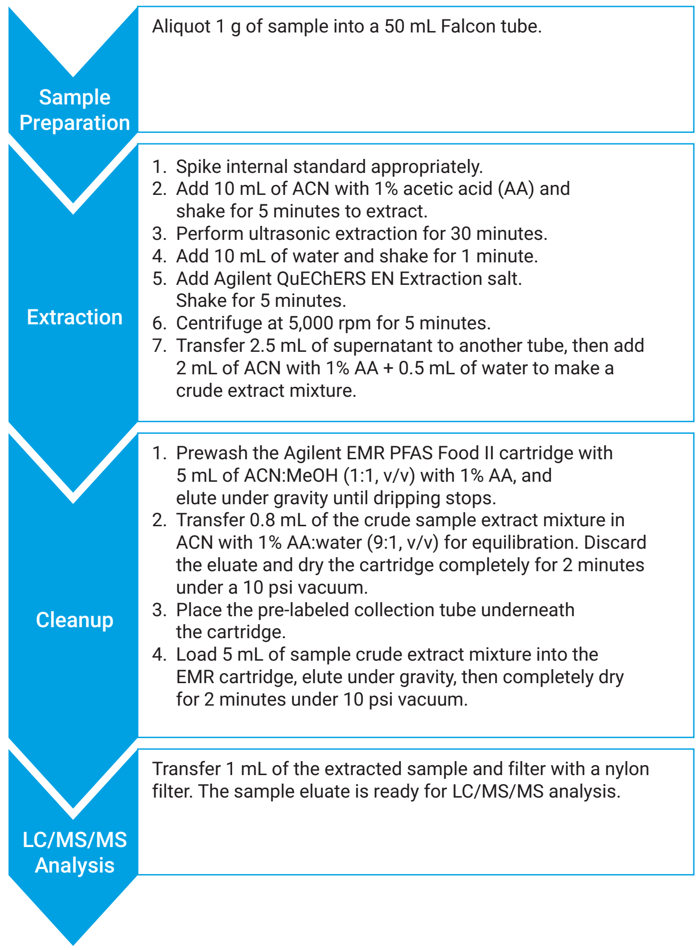
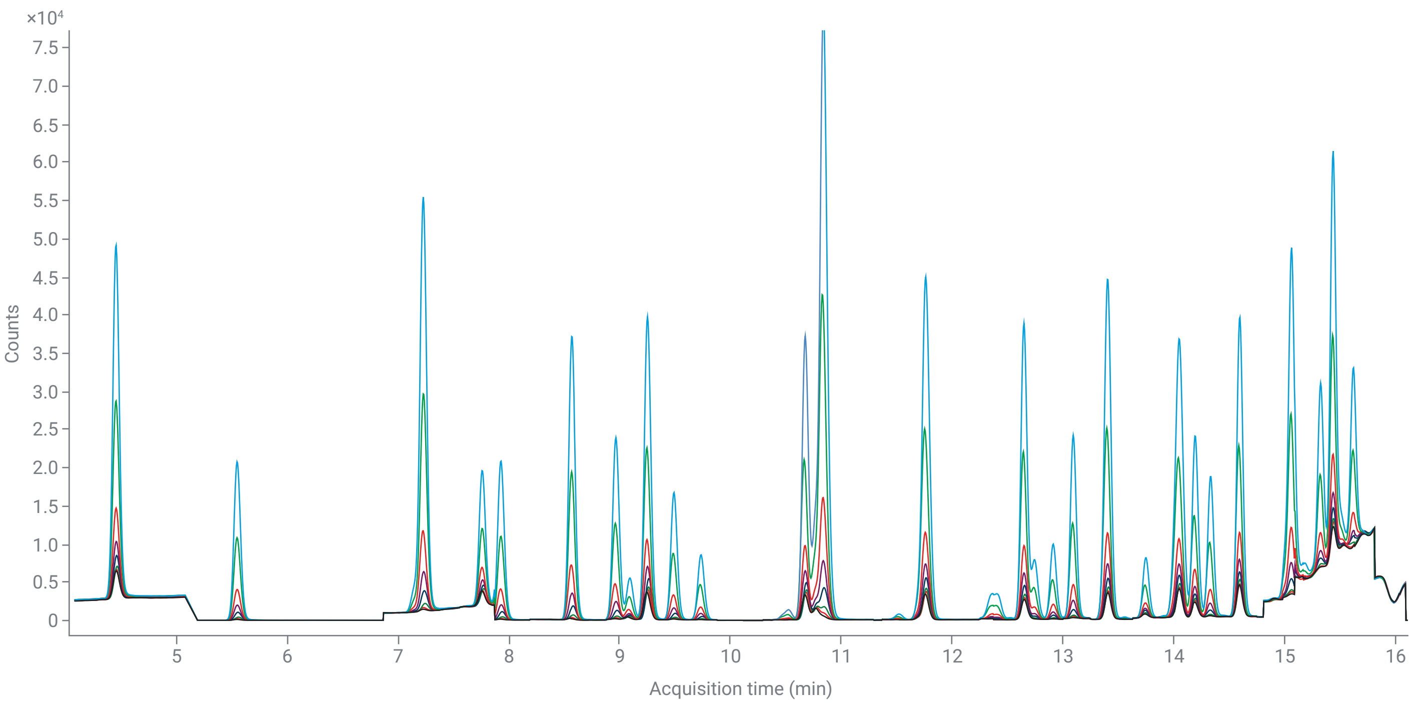


Figure 1. Sample preparation procedure.

Results and discussion

Chromatography and calibration curve linearity

Figure 2 shows the overlaid chromatograms for 41 PFAS calibration standards, including 0.05, 0.1, 0.2, 0.5, 1.0, 2.0, 5.0, and 10.0 ng/mL in ACN. The table on the right shows the peaks identification and corresponding retention time. Across 41 PFAS compounds, R^2 values ranged from 0.9940 to 0.9998, indicating excellent linearity for all analytes' calibration curves across the calibration range of 0.05 to 10.0 ng/mL.



Analyte	RT (min)
PFBA	4.5
PFMPA	5.6
3-3 FTCA	7.2
PFPeA	7.2
PFBS	7.8
PFMBA	7.9
PFEESA	8.6
NFDHA	9.0
4-2 FTSA	9.1
PFHeA	9.3
PFPeS	9.5

Analyte	RT (min)
HFPO-DA	9.7
PFHpA	10.7
PFHxS	10.8
DONA	10.8
5-3 FTCA	10.9
6-2 FTSA	11.7
PFHpS	11.8
PFOA	11.8
PFOS	12.6
PFNA	12.6
Tetraconazole	12.7

Analyte	RT (min)
7-3 FTCA	12.9
9Cl-PF3ONS	13.1
8-2 FTSA	13.4
PFDA	13.4
PFNS	13.4
N-MeFOSAA	13.7
PFUnDA	14.0
PFDS	14.0
N-EtFOSAA	14.1
FOSA	14.2
11Cl-PF3OUdS	14.3

Analyte	RT (min)
PFDoDA	14.6
PFDoS	15.0
PFTrDA	15.1
MeFOSE	15.3
N-MeFOSA	15.3
EtFOSE	15.6
N-EtFOSA	15.6

Figure 2. The overlaid chromatograms of calibration standards from 0.05 to 10 ng/mL in ACN. Peak identification is shown in the table.

Recovery of analytes in cosmetics

Analyte recoveries were evaluated by fortifying PFAS standard to all five types of cosmetics matrices at 20 ng/g. The fortified samples were then extracted using the QuEChERS method followed by matrix cleanup on Captiva EMR PFAS Food II cartridges. Since the total dilution factor being introduced through sample preparation procedure was 20-fold, the final concentration of sample eluate after EMR cleanup corresponded to 1 ng/mL. The final sample extract was injected into LC/MS/MS for analysis and quantified against the established calibration curve for assessment of analyte recovery.

Figure 3 exhibits the recoveries of the 41 analytes in the five different types of cosmetic samples, where three different samples from each type were spiked. Overall, 97.6% of 41 analytes in all five types of cosmetics delivered acceptable recoveries within 70 to 120%. The five outliers were generated for five analytes in lipstick matrix, resulting from matrix effect or interferences.

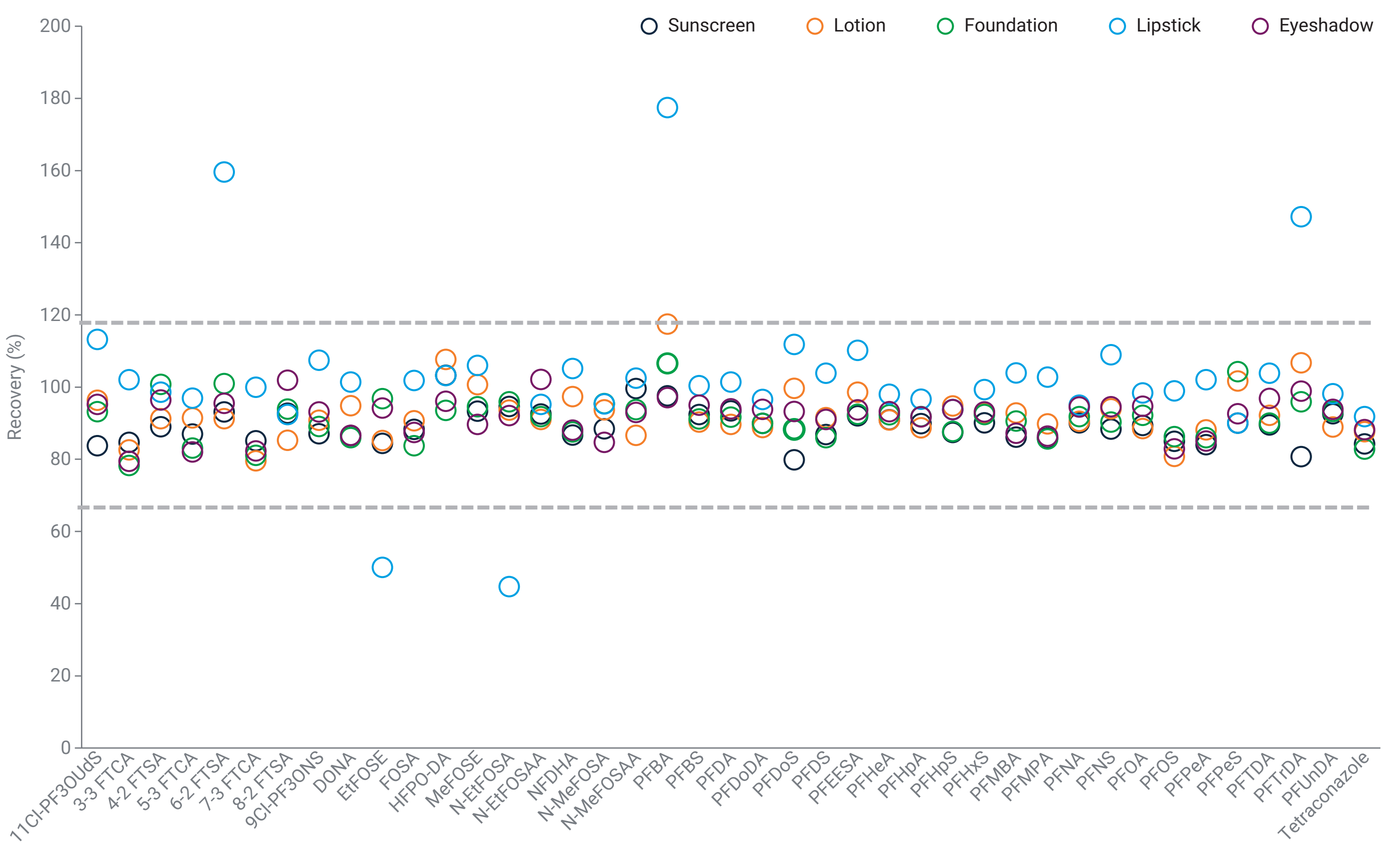


Figure 3. Analyte recoveries in five types of cosmetics.

Instrument method detection limit in cosmetics

The instrument method detection limit (IMDL) was determined by analyzing seven replicates of matrix blank extracts spiked at 0.1 to 0.5 ng/mL. The average calculated IMDL was 0.032 ng/mL in cosmetic extracts, corresponding to 0.64 ng/g in cosmetic samples. Figure 4 presents the range of calculated IMDL for 41 analytes in five types of cosmetic matrices.

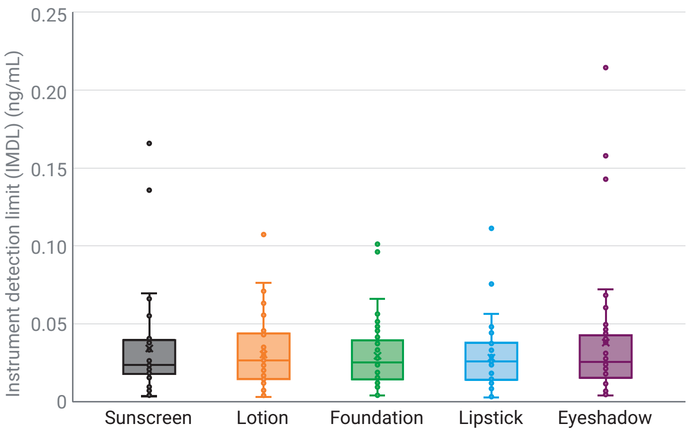


Figure 4. Calculated IMDL for 41 analytes in five types of cosmetics.

PFAS detection in real cosmetic samples

Of the 15 cosmetic products assessed, one eyeshadow sample was detected with a trace level of positive PFAS analytes, including PFHeA, PFHpA, PFNA, and PFOA, at about 0.1 ng/g in the sample. Figure 5 shows the method blanks and positive detection of PFHpA and PFNA in this eyeshadow product.

Conclusion

A simplified, rapid, and reliable method using QuEChERS extraction followed by EMR mixed-mode passthrough cleanup using the Agilent Captiva EMR PFAS Food II cartridge and LC/MS/MS detection was developed for 41 PFAS analysis in cosmetics.

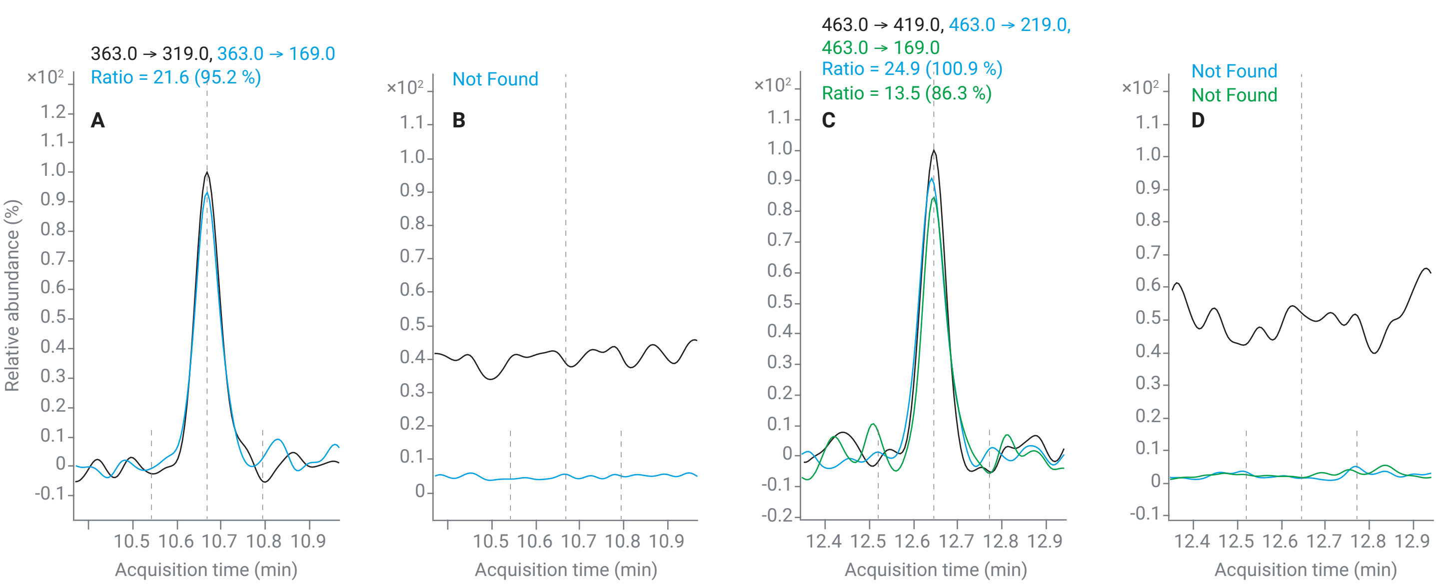


Figure 5. Detection of PFAS in an eyeshadow sample. (A) Positive detection of PFHpA in the sample. (B) Method blank in the PFHpA acquisition window. (C) Positive detection of PFNA in the sample. (D) Method blank in the PFNA acquisition window.

References

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3. Zhao, L.; Parry, E.; Determination of 40 PFAS in Tilapia Tissue Following EPA 1633 Method Guidance, *Agilent Technologies application note*, publication number 5994-8232EN, **2025**.
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