Analysis of ***55 PFAS in Food Contact Material: Baking Paper*** Using EluCLEAN® PFAS Universal SPE Cartridges

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Content

Key Features

- EU sets strict limits on PFAS in food packaging from August 2026
- Excellent recovery rates and low standard deviations for 55 PFAS analytes (extraction and clean-up method according to US EPA 1633)
- No detectable PFAS background contamination from the SPE cartridge
- EluCLEAN® PFAS – Universal SPE cartridge is a special combination phase that improves the recovery rates of PFAS analytes through numerous interactions. It can be used in a wide variety of matrices. It is also available as HP version with an improved matrix reduction e.g. for highly pigmented/coloured matrices

LCTech Products

SPE cartridges

Part No.: 20841, 20842, 20843 (Universal)

EluCLEAN® PFAS – Universal

Sorbent: special combination phase

Other Relevant LCTech Application Notes and Product Information

https://www.lctech.de/application-notes

https://www.lctech.de/en/products/pfas-workflow

Figure 1. EluCLEAN® PFAS Univesral SPE cartridge

1. Introduction

Per- and polyfluorinated alkyl substances (PFAS), a large group of synthetic chemicals have been widely used in various industrial and consumer products since last 60 years. They are often referred to as "forever chemicals" because of their extreme environmental persistence. PFAS are resistant to heat, water, and oil which makes them ideal for use in food contact materials. This includes applications in fast food wrappers, microwave popcorn bags, bakery bags, baking papers, pizza boxes, paperboard containers, and non-stick cookware coatings. Their presence in these materials helps improve product performance and shelf life.

However, research has revealed the high toxicity of PFAS compounds and thus the EU sets PFAS limits in food packagaing. The regulations (EU 2025/40, Packaging and Packaging Waste Regulation (PPWR)) sets strict limits on PFAS in food packaging from August 2026 [1]. All EU Member States must strictly follow the regulation. From 12 August 2026, food-contact packaging shall not be placed on the market if it contains per- and polyfluorinated alkyl substances (PFAS) in a concentration equal to or above the following limit values to the extent that the placing on the market of packaging containing such a concentration of PFAS is not prohibited pursuant to another Union legal act:

- 25 ppb for any PFAS as measured with targeted PFAS analysis.
- 250 ppb for the sum of PFAS measured with targeted analysis.
- 50 ppm for PFASs (polymeric PFAS included). If the total fluorine content exceeds 50 mg/kg, additional documentation will be required.

Analytical methods to measure PFAS from these materials require solid phase extraction (SPE) prior to liquid chromatography-tandem mass spectrometry (LC-MS/MS) analysis.
In this application note EluCLEAN® PFAS– Universal SPE has been used for SPE to cleanup extract baking paper prior LC-MS/MS analysis.

Figure 2: Workflow for sample preparation of food samples

2. Experimental

2.1 Sample Preparation

1 g of baking paper was cut into tiny pieces and spiked with 55 Native PFAS and 24 isotope dilution standard as mentioned in table below in a falcon tubes.

Table 1. 55 Native PFAS and 24 isotope dilution standard spiked concentrations

Compounds	ng
Labelled compounds	0.84– 16.67
11Cl-PF3OUdS, 9Cl-PF3ONS, ADONA, HFPO-DA, NFDHA, PFEESA, PFMB, PFMPA, PFPeA	4
PFBA, 4:2FTS, 6:2FTS, 8:2FTS	8
N-MeFOSE, N-EtFOSE	20
5:3 FTCA, 7:3 FTCA	40
FBSA-I, PFECHS, FHXSA-I, P37DMOA, FOUEA, 6:2 diPAP, 8:2 diPAP	3.35
PFHxDA, PFODA	1.67
L-PFUdS, L-PFTrDS	3.67
6:2 PAP, 8:2 PAP, PFDPA	33.35
All other PFAS*	2

*All PFAS are visible in Fig 4 below

The method was designed to comply with the SPE procedure described in US EPA 1633 method.

2.1.1 Sample Extraction

For extraction 10 mL 0.3% NH_4OH in MeOH was added to the falcon tubes with baking paper. The sample was then vertically vortexed for 5 minutes at 800 rpm and then centrifuged at 2700 rpm for 10 minutes. The extracts is then transferred to another falcon tubes. The process was repeated 2 times with different volume of 0.3% NH_4OH in MeOH; 1x with 15 mL and another time with 5 mL. The total 30 mL volume of extracts was collected. The extraction method was similar to US EPA 1633 method [2] (also to comply with the regulation method). However, other extraction procedure like pressuriuzed fluid extraction (as mentioned in Fig. 1) can also be implemented based on their performance.

2.1.2 Evaporation of extract

The collected extract is then evaporated using vacuum centrifuge automated evaporation device called D-EVA to around 5-7 mL end volume. Then the extract is again diluted with reagent water to 50 mL final volume and acidified with 50% formic acid to pH 4-6.

2.1.3 Solid phase extraction

PFAS analytes were enriched in EluCLEAN® PFAS – Universal SPE cartridges. SPE method was similar to US EPA 1633 method. Then, 50 µL of concentrated acetic acid and 10 µL NIS (MPFAC-HIF-IS) solution were

added to each sample eluate and vortexed. (In case eluate looks turbid or with particles: the eluates can be filtered through a syringe filter (25 mm filter, 0.2 µm nylon membrane) into another clean 15 mL polypropylene centrifuge tube). 20 µL of concentrated acetic acid and 10 µL NIS (MPFAC-HIF-IS) solution were added to each sample eluate and vortexed and evaporation was done in D-EVA (automated vacuum centrifuge with cold trap for solvents). The samples were subsequently analyzed in LC-MS/MS.

2.1.4 Evaporation/Concentration of Eluate

All samples were evaporated to around 1 mL using D-EVA Rotational Vacuum Concentrator (temperature: 45°C, vacuum: 20 mbar) and transferred into a 1.5 mL polypropylene vial and kept at 0 – 4 °C for LC-MS/MS analysis.

2.2 Instrumentation

2.2.1 MS Conditions

Table 2. MS Conditions

Parameter	Value
MS	TSQ Quantis (Thermo)
Polarity	Negative
Spray voltage	2500 V
Sheath Gas	50 Arb
Aux Gas	10
CID Gas	2 mTorr
Ion transfer tube temp	325 °C
Vaporizer Temp	300 °C
Q1 resolution	0.7 FWHM
Q3 resolution	1.2 FWHM
Cycle time	0.5 sec
Chromatographic peak width	6 sec

2.2.2 LC Instrument Conditions

Table 3. LC Conditions

Parameter	Value	
LC	Thermo Scientific Vanquish Flex UHPLC system	
Analytical column	Accucore RP-MS, 2.1*100 mM, 2.6 µm	
Delay column	Agilent ZOBRAX Eclipse plus C18, 4.6*50 mm; 3.5 µm	
Column temperature	45 °C	
Injection volume	5 µL	
Mobile Phase	A) 20 mM ammonium acetate H2O with 2 % MeOH and 0.1 % acetic acid B) 20 mM ammonium acetate MeOH with 2 % H2O and 0.1 % acetic acid	
Gradient Flow rate	0.5 mL/min	
Gradient	Time (min) 0 1 6 13 14 17 20 22 25	% 0 30 45 80 95 95 95 0 0

3. Results

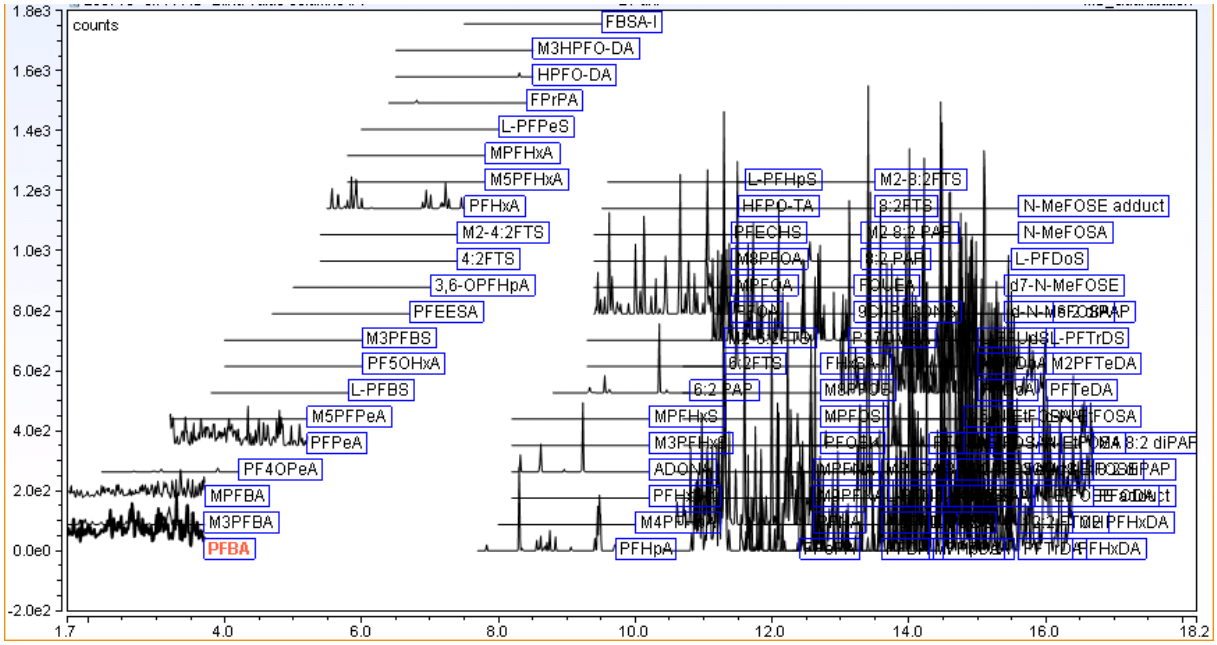

Figure 3. Chromatogram demonstrating EluCLEAN® PFAS –Universal cartridges show no PFAS background contamination when screened for 55 PFAS analytes

3.1 Recovery Rates and RSD % of 55 PFAS

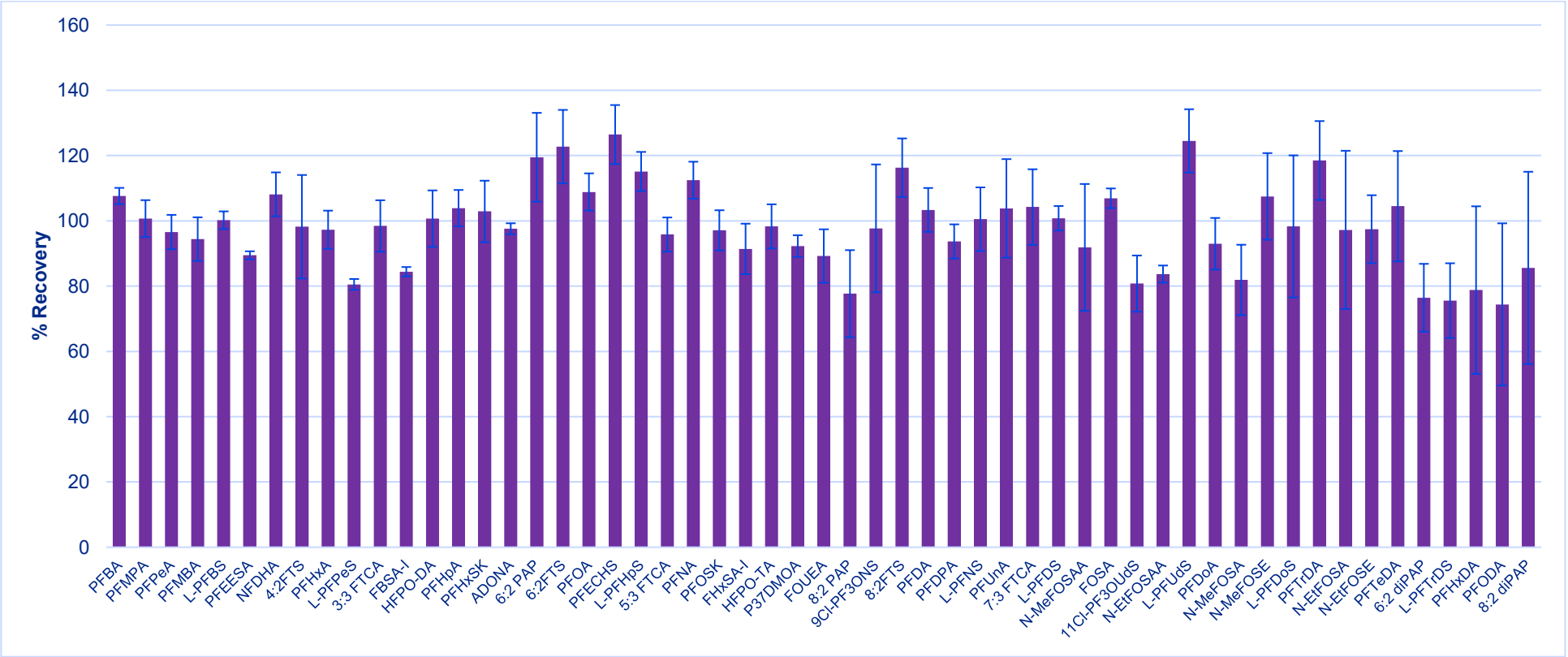

Figure 4. Recovery rates of 55 PFAS (incl. 40 PFAS listed in US EPA method 1633) from baking paper extracted with EluCLEAN® PFAS – Universal SPE cartridges (n=4, spiked concentration=2-40 ng in 1 g baking paper)

4. Conclusion

EluCLEAN® PFAS – Universal SPE cartridge has no detectable PFAS background contamination. This cartridge is ideally suited for clean-up of PFAS when extracting from baking paper. Recoveries of samples are very well in between the acceptable criteria of 70 - 130 % with RSD below 20%. The described method was developed to analyze PFAS in food packaging materials to meet EU regulation.

5. References

[1] European Union. (2025). *Regulation (EU) 2025/40 of the European Parliament and of the Council of [date] amending Regulation (EC) No [XXX] as regards [subject].* Official Journal of the European Union, L [number]/[page]. https://eur-lex.europa.eu/legal-content/EN/TXT/?uri=OJ:L_202500040

[2] **Environmental Protection Agency (EPA). (2024).** *Method 1633A: Determination of per- and polyfluoroalkyl substances in water, soil, and biosolids by liquid chromatography-tandem mass spectrometry (LC-MS/MS).* U.S. Environmental Protection Agency. https://www.epa.gov/system/files/documents/2024-12/method-1633a-december-5-2024-508-compliant.pdf

Any Questions?
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