



Simple and fast automated SPE clean-up for determination of perfluoroalkyl substances in food matrices



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Results

70

Recovery

%

Recovery

60

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■ Extraction with 1% NH4OH in acetonitrile

Introduction

Perfluoroalkyl substances (PFASs) are widely used in consumer products and industrial applications and can be found ubiquitously e.g. in the environment, in food and in human tissues. Due to their ubiquitous presence also in laboratory equipment (i.e. plastic tubes, fittings etc.) analysis of these compounds is quite challenging and good control of procedural blank contamination is essential. Quantitative analysis of PFASs requires an efficient sample preparation method

employing sample extraction and clean-up usually by using solid-phase extraction (SPE). Manual solid-phase extraction is, however, time consuming and thus unfavorable in routine analysis where a high number of samples has to be analysed. The purpose of this project is to develop a simple and fast method using automated SPE. Low concentrations of different types of PFASs should be reliably detected in matrices of animal and plant origin. As a starting point the development is primarily based on determination of perflourocarboxylic and perfluorosulfonic acids in beef spiked with analytes of interest.

■ Extraction with acetonitrile

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■ 1% NH4OH in methanol ■ 3 % NH4OH in methanol ■ 3 % NH4OH in methanol after alkaline extraction

Figure 3: Recovery of detected PFAS in samples of beef (1 µg/kg) after extraction with acetonitrile and washing step

with 25 mMol ammonium acetate or 1 % formic acid in acetonitrile.

Figure 2: Recovery of detected PFAS in samples of beef (1 μg/kg) after extraction with acetonitrile or 1 % NH₄OH in acetonitrile.

Experimental

• 5 g sample (+ internal standards)

Sample weight

Extraction

10 mL solvent

- A) Acetonitrile
- B) 1 % NH₄OH in acetonitrile

Shaking and ultrasound (20 min)

Addition of ethylene glycol; 60 °C, 2 h to 1 mL

Evaporation

Automated SPE (anion exchanger)

- 1. Conditioning
- H₂O
- Methanol
- 2. Loading (evaporated raw extract + 9 mL
- 3. Washing
- C) 25 mMol ammonium acetate in acetonitrile
- D) 1 % formic acid in acetonitrile
- 4. Elution
 - NH₃
 - E) 1 % in methanol
 - F) 3 %
- F₁) in methanol

F₂) in methanol after B)

Evaporation

Addition of the keeper ethylene glycol; 60 °C, 2 h evaporation to dryness

Extract preparation &

Measurement

Dissolved in 500 µl methanol/ 1% formic acid in water (2+1/v+v)

→ LC-MS/MS (external calibration)

Figure 1: Analytical procedure for the determination of perfluoroalkyl substances in food of animal origin.









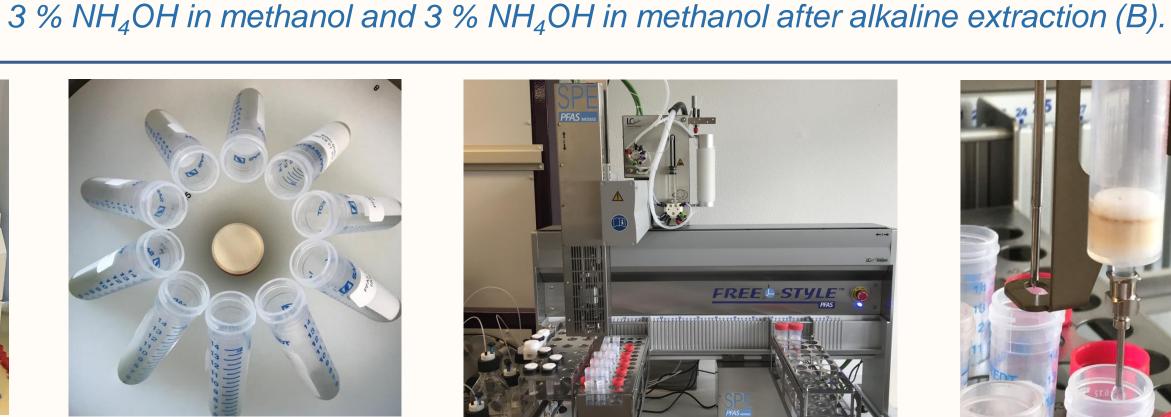


Figure 4: Recovery of detected PFAS in samples of beef (1 μg/kg) after elution with 1 % NH₄OH in methanol (purple),



■ wash solution: 25 mMol ammonium acetate in acetonitrile

wash solution: 1 % formic acid in acetonitrile



Conclusion

- →Better recovery and reproducibility by extraction with alkalized solvent, preferably acetonitrile
- → Keeper is necessary to avoid loss of analyte during evaporation
- → Comparable recovery by using different washing solutions, best reproducibility with 25 mMol ammonium acetate in acetonitrile
- →Best recovery and reproducibility by elution with 3 % NH4OH in methanol

Outlook

- Improvement of extraction efficiency
- Integration of a cleaning step to reduce matrix effects
- Using of internal standards for recovery correction
- Method validation
- Expansion of the spectrum of analytes as well as matrices

