



Development of an NMR-Based Method for the Analysis of PFAS

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Why NMR, why this topic?

PFAS are persistent, bioaccumulative and potentially toxic substances widely used in industrial and consumer products. Current standard methods (e.g. LC-MS) face limitations regarding available standards, structural insights and quantification.

This work evaluates NMR spectroscopy as a complementary analytical tool for PFAS analysis.

Objectives



Build a ¹⁹F-NMR spectral library of selected PFAS



Develop a method for PFAS analysis by ¹⁹F-NMR

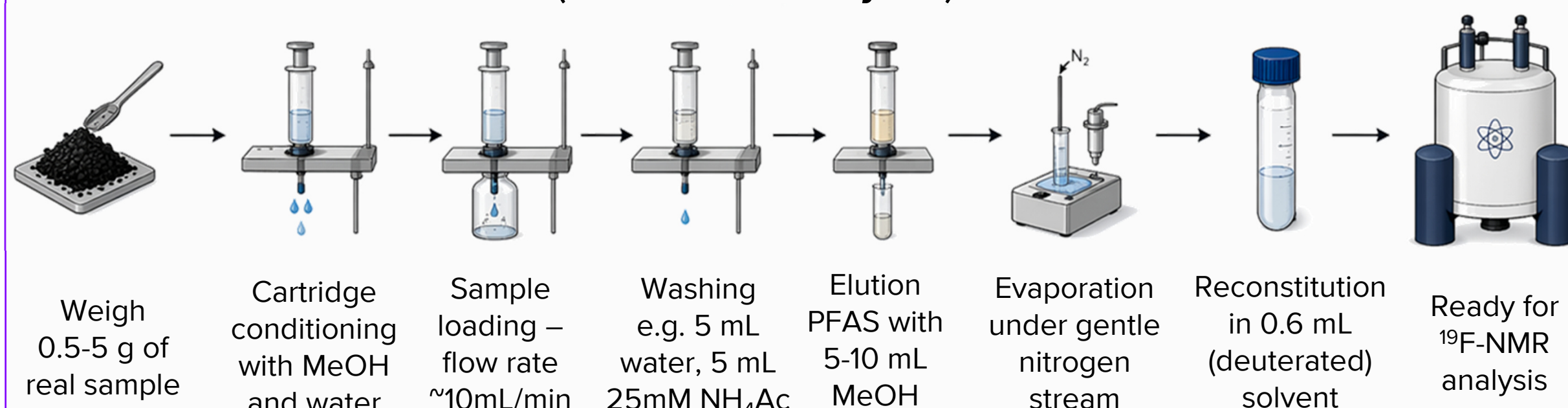


Investigate quantification and detection limits

Sample Preparation

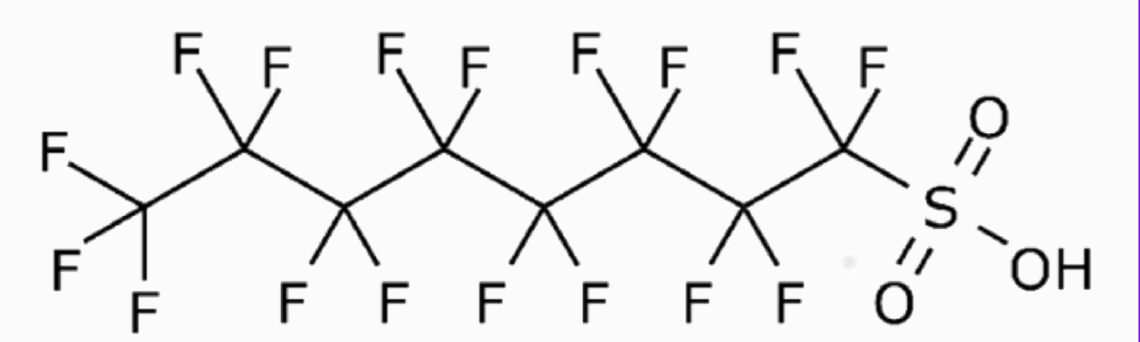
Direct dissolution in suitable solvents (for higher concentrations)

Solid-Phase-Extraction (for trace analysis)



Background of „forever chemicals“

- PFAS are characterised by highly stable C-F bonds
→ extreme persistence (so called “forever chemicals”)
- Wide applications: textiles, cosmetics, firefighting foams, ski wax, food packaging and more
- Adverse effects on environment and human health
- Analysis is challenging due to:
 - low concentrations (ng/mL range)
 - large structural diversity
 - complex sample matrices



Example: Perfluorooctanesulfonic acid (PFOS)

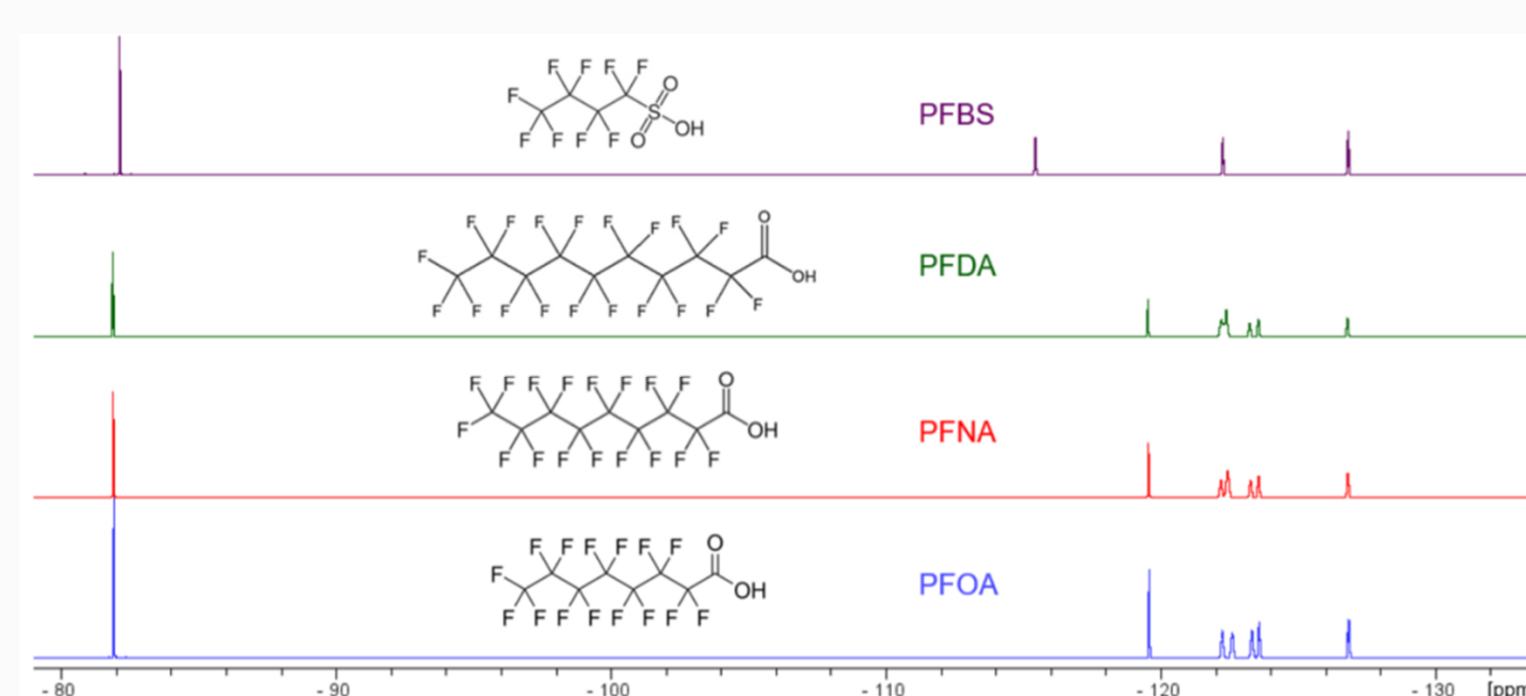
Key experiments

- Spectral characterisation of six selected PFAS compounds
- Influence of solvents on ¹⁹F-NMR chemical shifts
- Analysis of real PFAS-containing samples (fluorinated ski wax, fishing line)
- Quantitative ¹⁹F-NMR experiments
- Determination of T₁-relaxation times
- Determination of LOD/LOQ
- Evaluation of SPE methods (EPA 1633 vs. EPA 533)



Bruker Avance III HD 500 MHz (with BBFO probe)

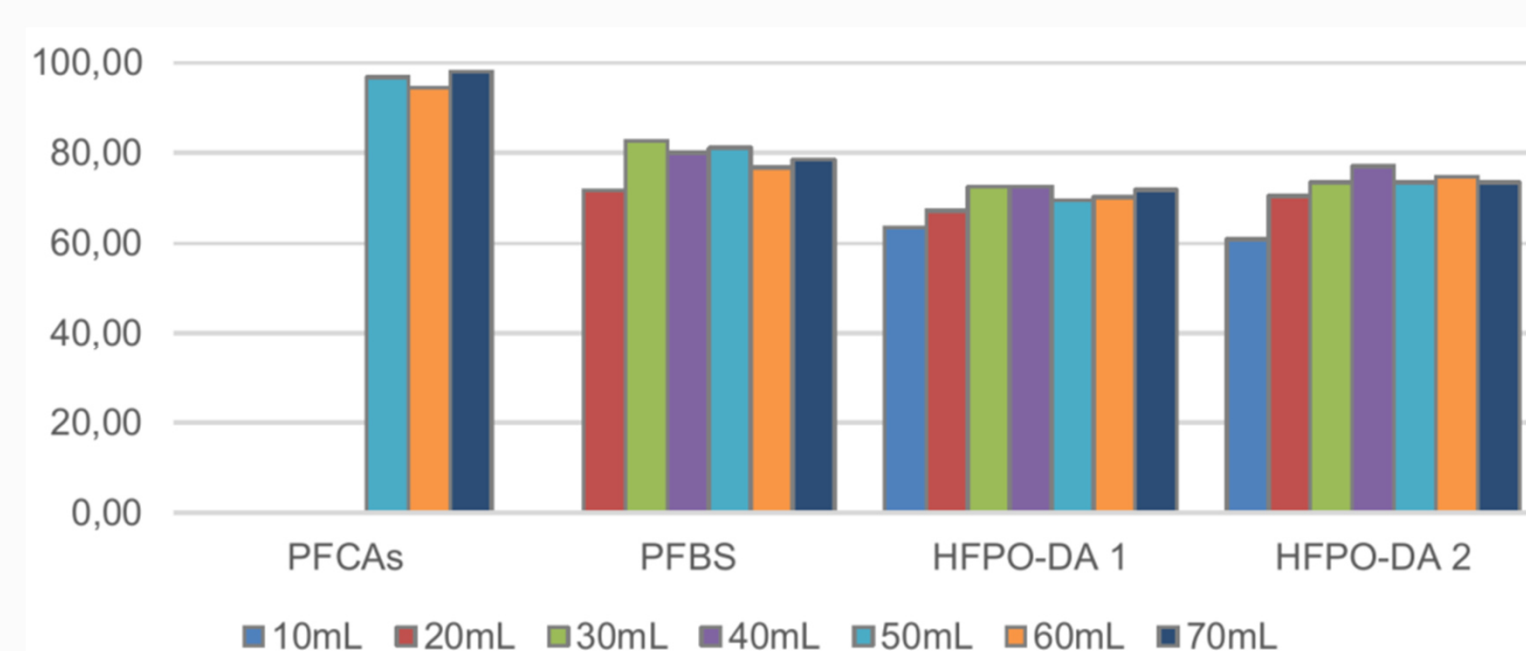
Results



¹⁹F-NMR spectra of PFAS in MeOH/MeOD 9:1

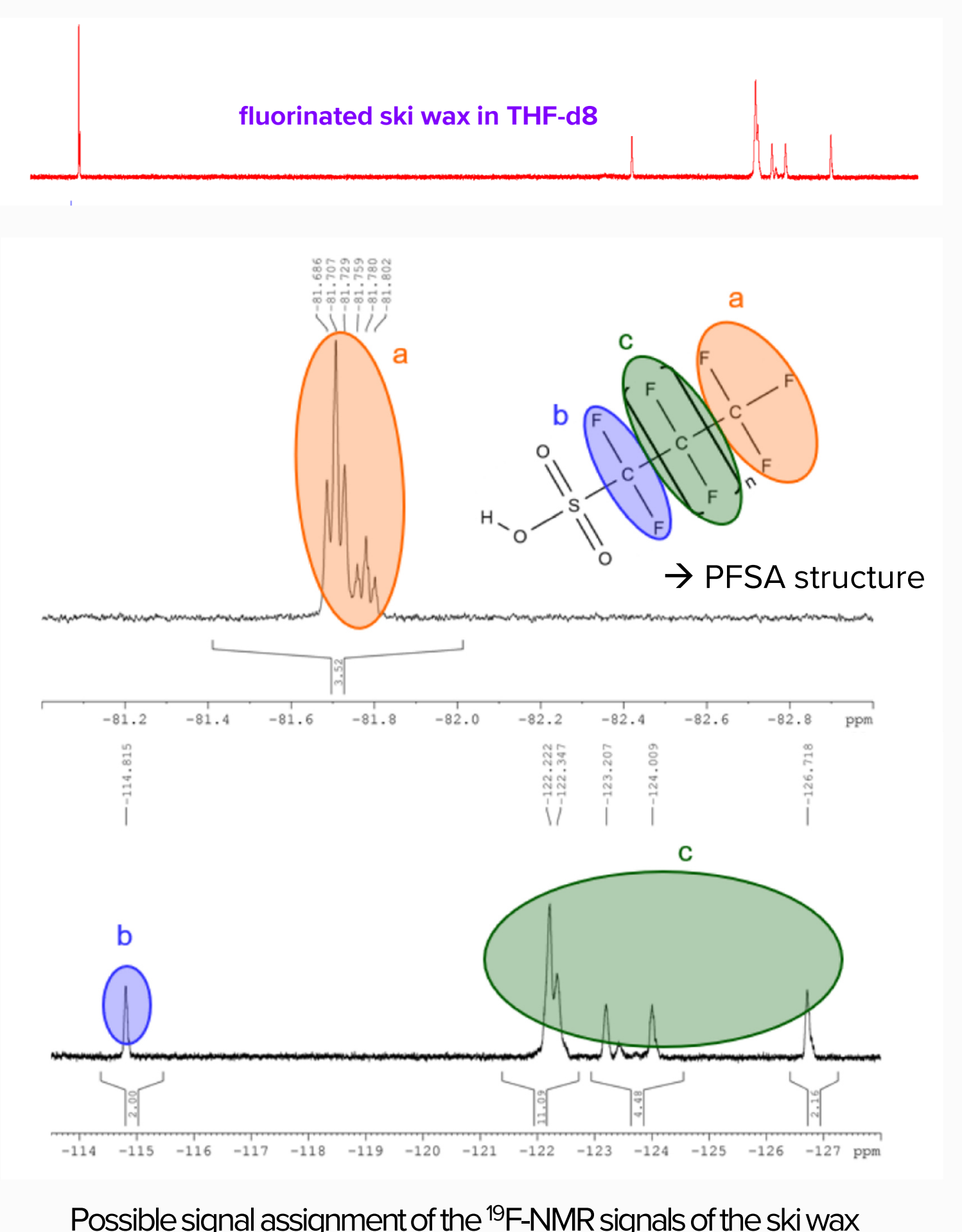
Detection Limits (¹⁹F-qNMR)

LOD: 0.002-0.007 mg/mL LOQ: 0.006-0.020 mg/mL
LOD: approx. 0.0002 mg/mL for SPE extraction



Extraction coefficient of SPE extraction with different sample volumina (low concentration test approx. 0.25 µg/mL)

Real PFAS-samples



Possible signal assignment of the ¹⁹F-NMR signals of the ski wax

Take-Home Message and Outlook

A functional NMR-based method for the analysis of PFAS was developed. ¹⁹F-NMR provides high selectivity and structural information as well as reliable quantification (qNMR). Although the sensitivity is lower compared to LC-MS, **NMR is a powerful complementary technique**, especially for screening, structural elucidation and quantification of reference materials and method development.

- Improve sensitivity for trace analysis
→ NEW NEO-600
- Combine with sample enrichment techniques
- Expand spectral databases
- Develop standardized NMR workflows for PFAS analysis

Comment: results presented here are part of Minh Quan Nguyen bachelor's thesis "Methodenentwicklung für die Analyse per- und polyfluorierter Alkylsubstanzen (PFAS) mittels NMR-Spektroskopie"