Method Development for Evaluation of Perfluorinated Alkyl Substances (PFAS) from Mussel Tissue

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Introduction

- Perfluorinated alkyl substances (PFAS) are a large family of synthetic chemicals
- Uses include aqueous firefighting foams (AFFF), cookware, clothing, and more (U.S Environmental Protection Agency 2026, Evich et al. 2022)
- Used for their oil and water repellant properties
- Characterized by long nonpolar fluorinated carbon chains with varied polar functional groups
- Widespread contaminants in soil, water, air, and biota (Evich et al. 2022)
- Known to bioaccumulate (Evich et al. 2022)
- Linked to potential health effects in humans (U.S Environmental Protection Agency 2026)
- Health effects increase the necessity of finding a reliable method to isolate and quantify PFAS in a variety of matrices

Study Objective

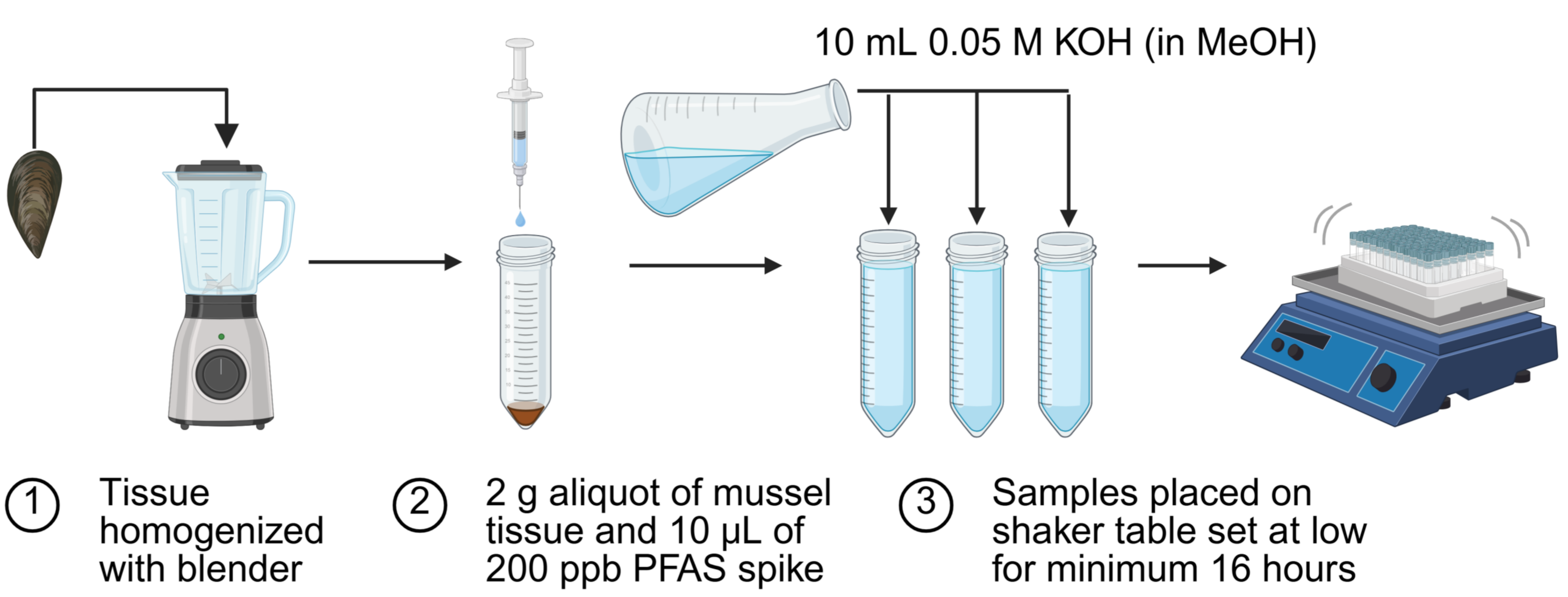
- We sought to modify and validate two extraction and cleanup methods for various PFAS compounds with a recovery of >70% (Table 1)
  - **Method 1:** Quick, Easy, Cheap, Effective, Rugged, and Safe (QuEChERS) is a fast high-salt-based extraction (Restek Corporation 2012)
  - **Method 2:** Environmental Protection Agency (EPA) standardized method 1633, is a time intensive alkaline digestion coupled with solid phase extraction cleanup (SPE) (Fig. 1) (U.S Environmental Protection Agency 2024)
- Both methods were coupled with analysis via liquid chromatography tandem mass spectrometry (LCMSMS)

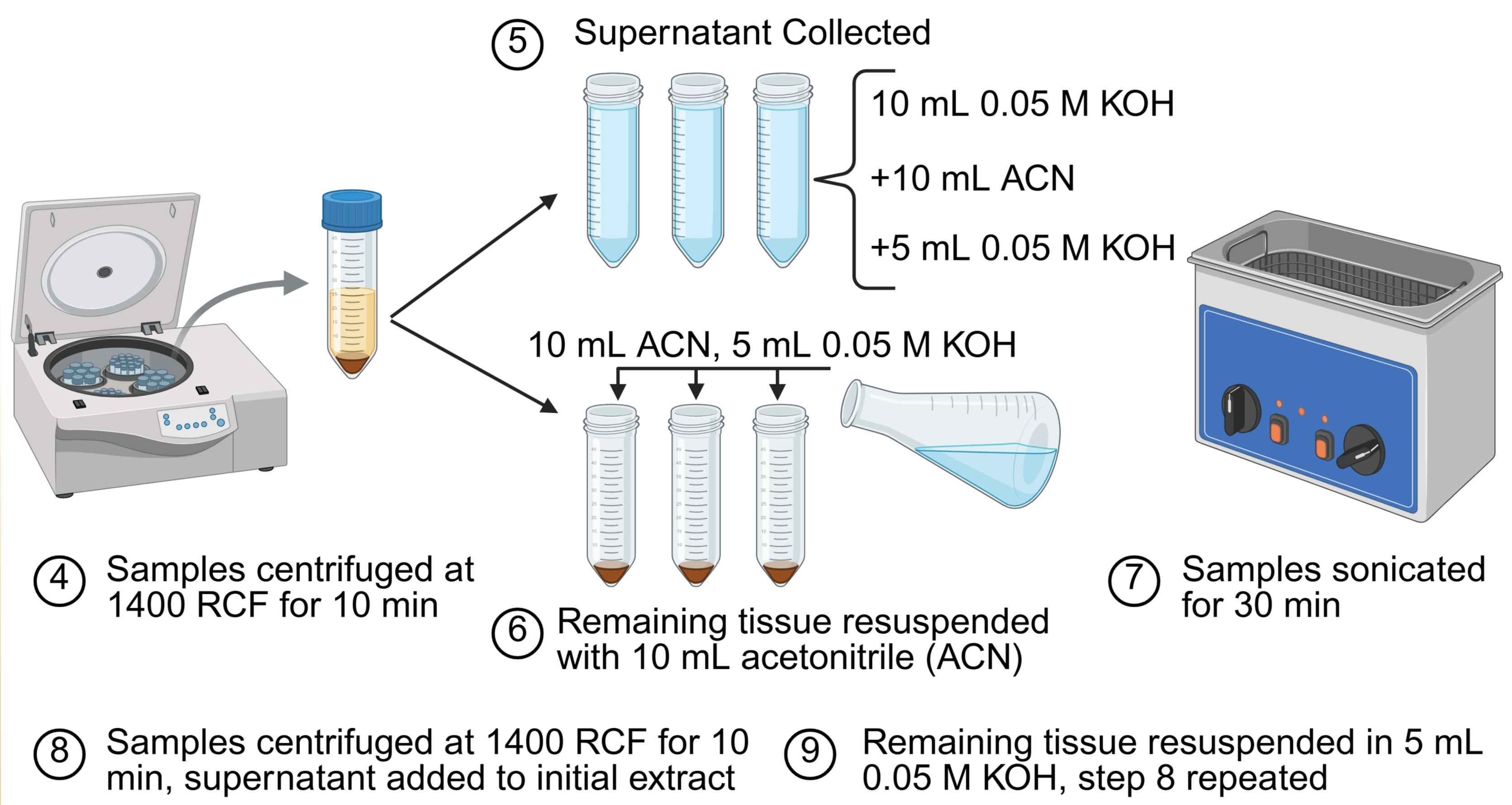
Table 1. List of PFAS analyzed in this study.

Classification	Compounds	Acronyms	Molecular Weights	Molecular Weights (transition measured)	General Structure	Analysis
Perfluorinated Sulfonic Acids (PFSAs)	Perfluorobutane Sulfonic Acid	PFBS (n=3)	300	299>99; 299>80	$CF_3(CF_2)_n$–S(=O)(=O)–OH	LCMSMS
	Perfluorohexane Sulfonic Acid	PFHXS (n=5)	400	399>99; 399>80		
	Perfluorooctane Sulfonic Acid	PFOS (n=7)	500	499>99; 499>80		
	Perfluorodecane Sulfonic Acid	PFDS (n=9)	600	599>99; 599>80		
Perfluorinated Carboxylic Acids (PFCAs)	Perfluorobutanoic Acid	PFBA (n=2)	214	213>169	$CF_3(CF_2)_n$–C(=O)–OH	LCMSMS
	Perfluorohexanoic Acid	PFHxA (n=4)	314	313>269		
	Perfluoroheptanoic Acid	PFHpA (n=5)	364	363>319; 363>169		
	Perfluorooctanoic Acid	PFOA (n=6)	414	413>369; 413>169		
	Perfluorononanoic Acid	PFNA (n=7)	464	463>419; 463>169		
	Perfluorodecanoic Acid	PFDA (n=8)	514	513>469		
	Perfluoroundecanoic Acid	PFUnDA (n=9)	564	563>519; 563>169		
	Perfluorotridecanoic Acid	PFTrDA (n=11)	664	663>619		
	Perfluorooctadecanoic Acid	PFODA (n=16)	914	913>869		

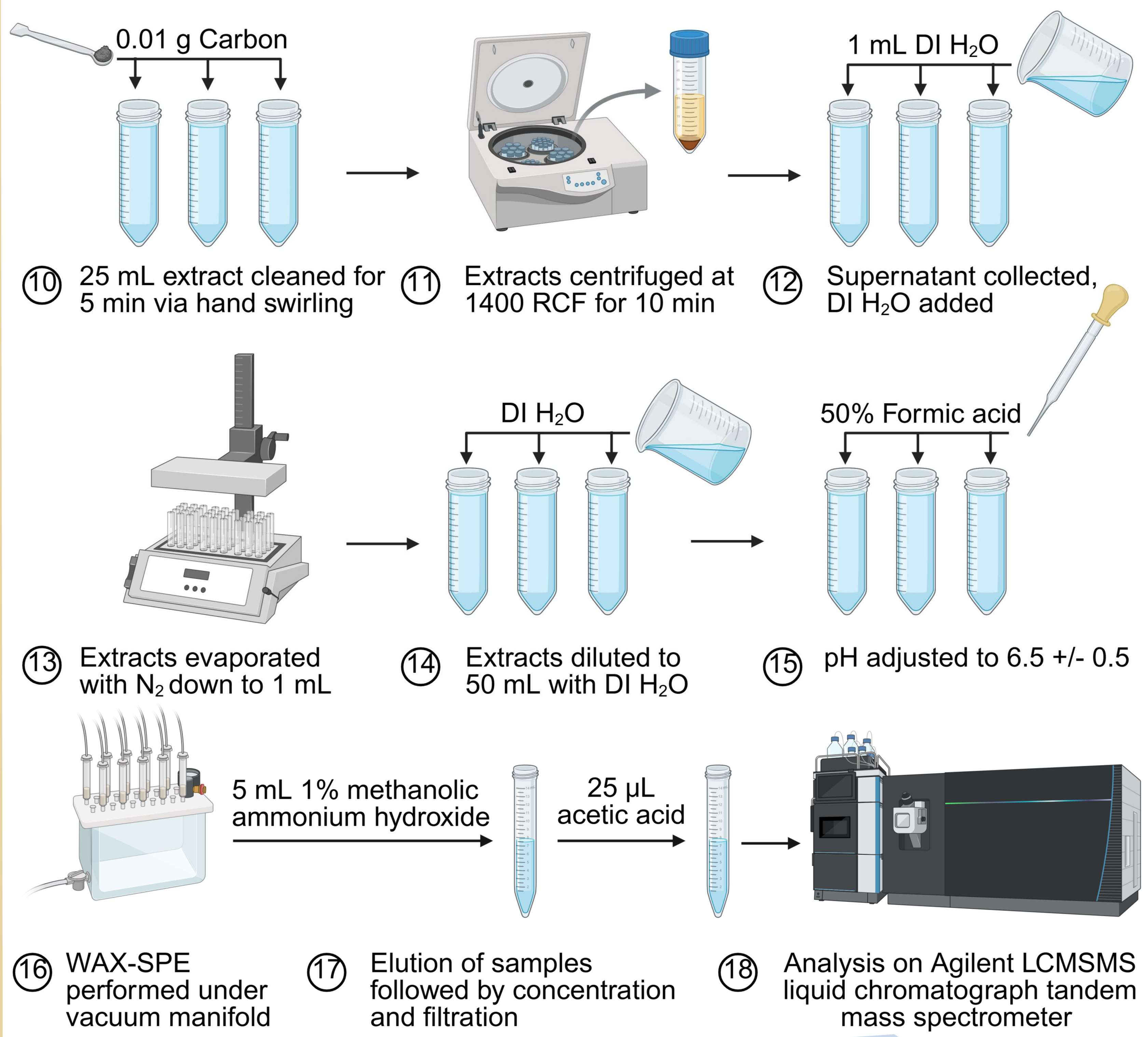
Methods

Sample Extraction





Cleanup and Concentration



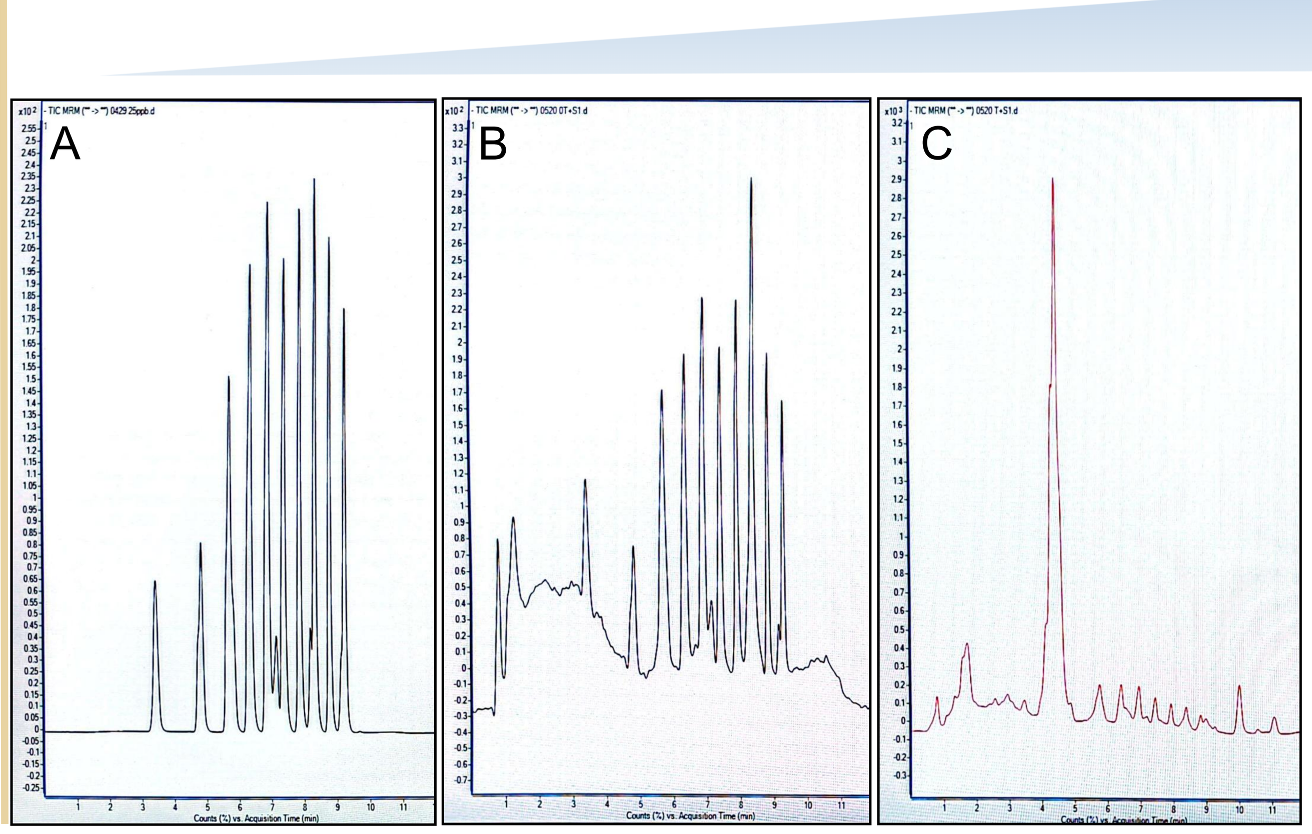
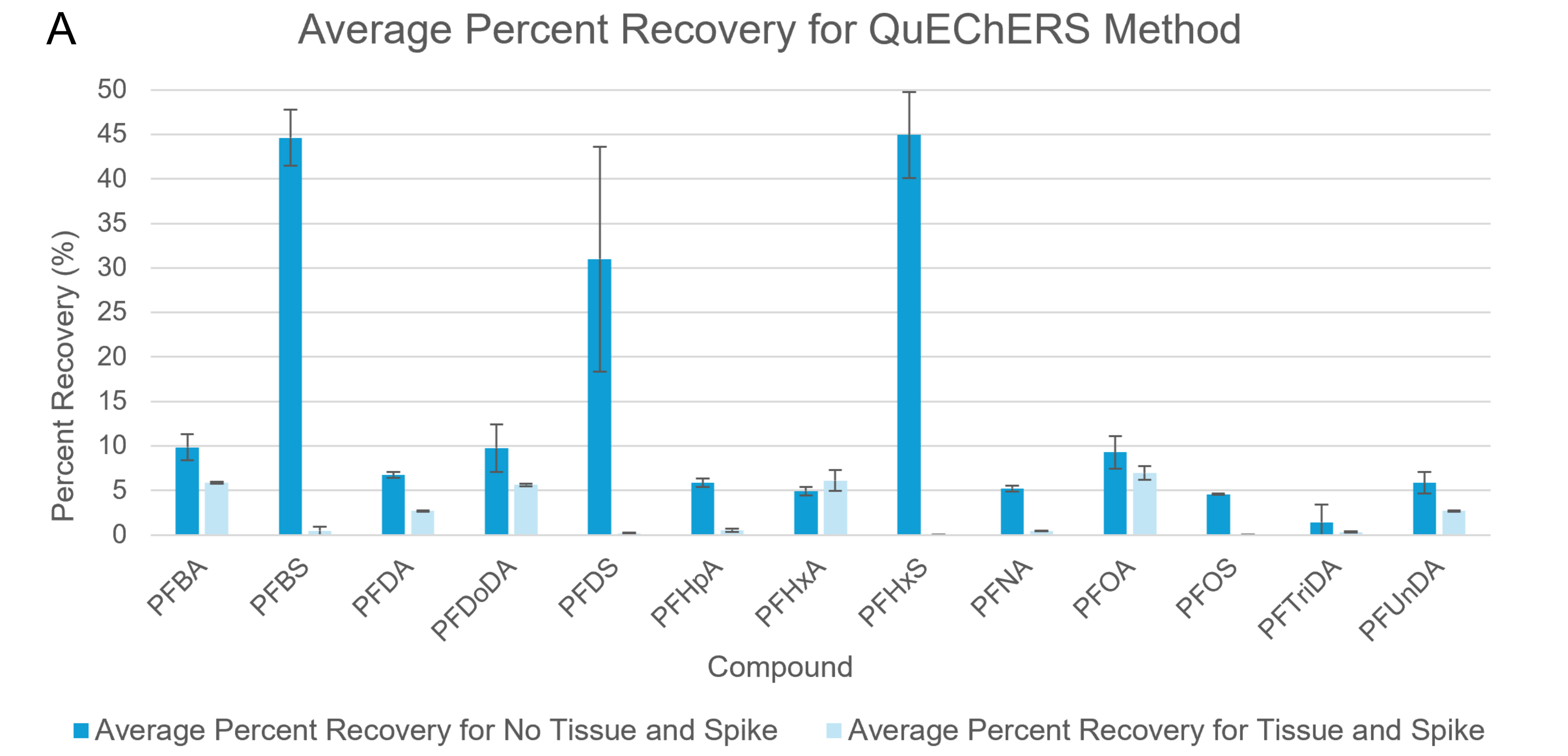


Figure 1 A to C. Sample chromatograms from LCMSMS analysis using EPA Standardized Method 1633 used for validating PFAS extraction in mussel tissue. A is standard, B is no tissue spike, C is tissue spike.

Results and Discussion



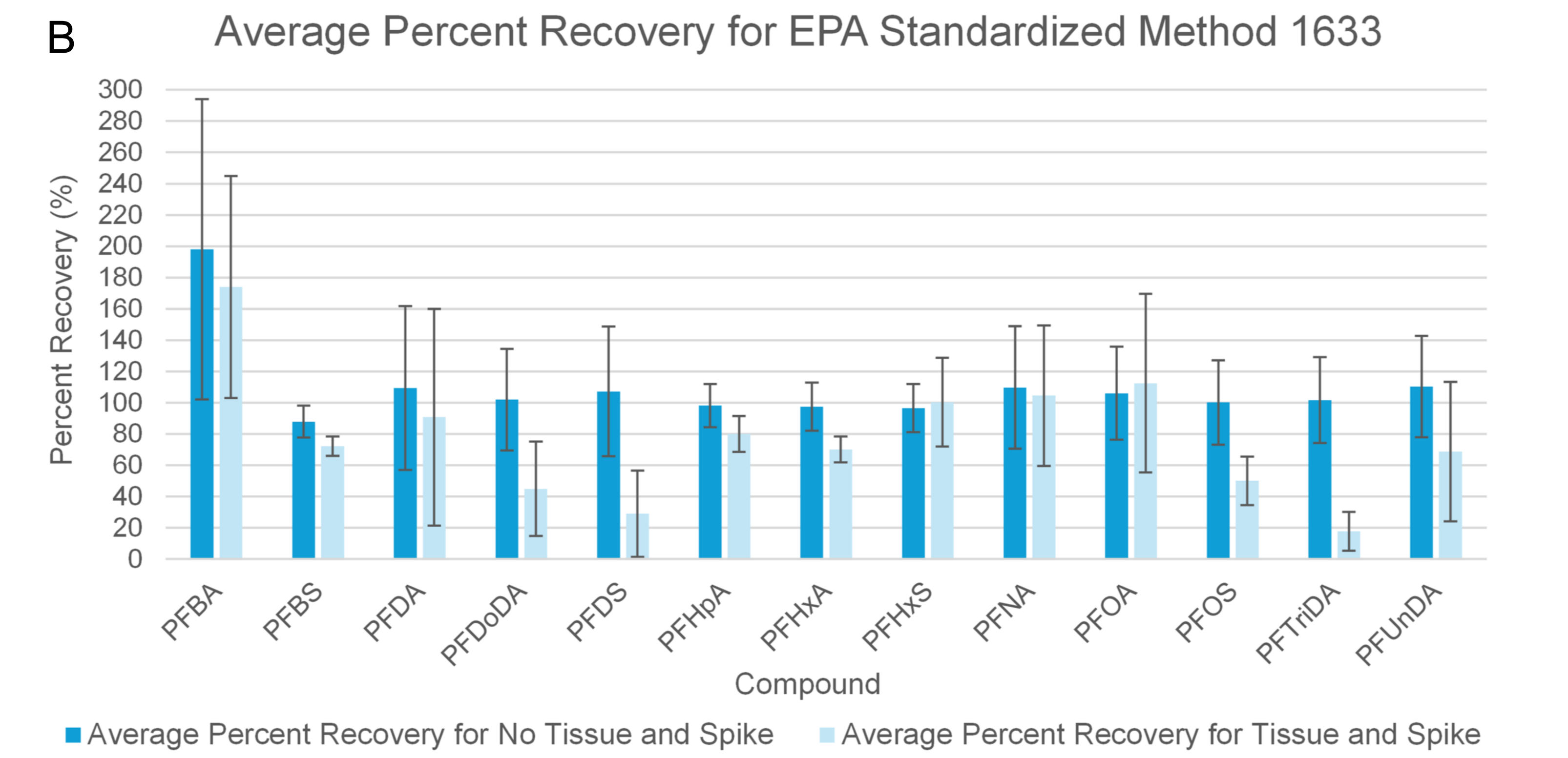


Figure 2 A and B. Average percent recoveries for each method. 2 A shows recoveries for the QuEChERS method for no tissue (turquoise) and tissue (light blue). 2 B shows recoveries for the EPA Standardized Method 1633 for no tissue (turquoise) and tissue (light blue).

- Spike and recoveries without tissue using QuEChERS ranged from 1.42% to 44.95% and with tissue ranged from 0.02% to 6.95% (Fig. 2A)
- Spike and recoveries without tissue using EPA method ranged from 87.92% to 198.16% and with tissue ranged from 17.7% to 173.93% (Fig. 2B)
- Each measured compound saw a percent recovery increase of eight-fold or larger under the EPA method
- EPA method recovered all compounds except for PFDoDA, PFDS, PFOS, PFTriDA, and PFUnDA with a yield of >70%

Conclusion and Future Work

- EPA method poses a more reliable and effective method for the isolation and quantification of PFAS residuals in mussel tissue
- Further exploration of PFDoDA, PFDS, PFOS, PFTriDA, and PFUnDA to increase recovery
- Future work includes utilizing the EPA method to assess regional contamination in mussel samples collected from bays in the Puget Sound Region
- Next phase of the study will provide data to model potential human exposure to PFAS from shellfish consumption

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References