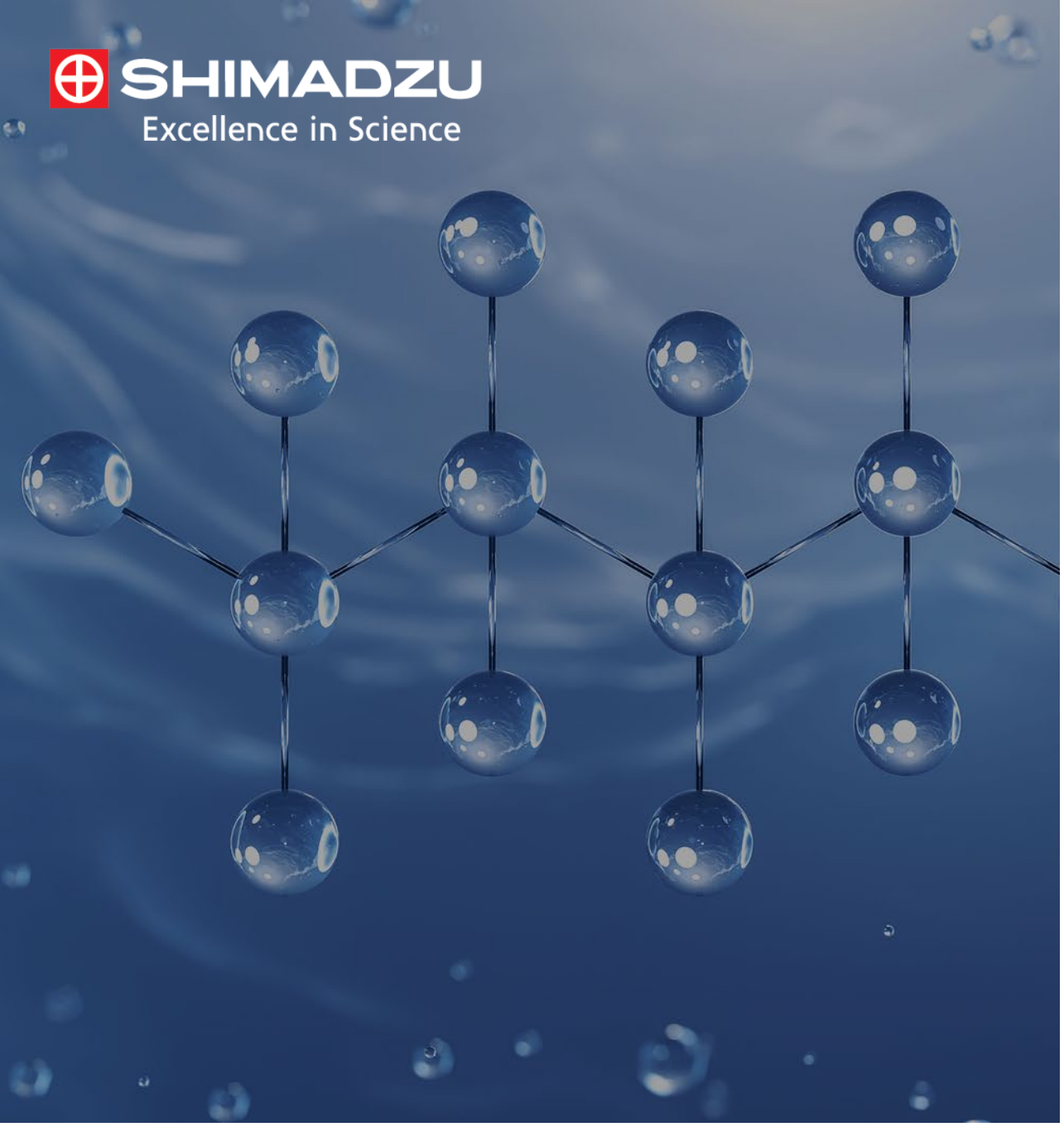
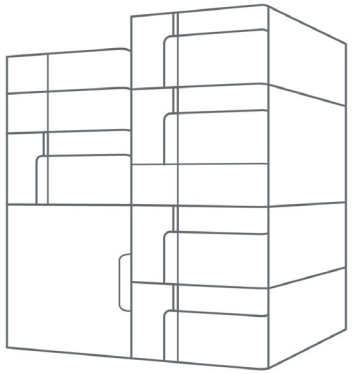


PFAS QUESTIONS: ANSWERED

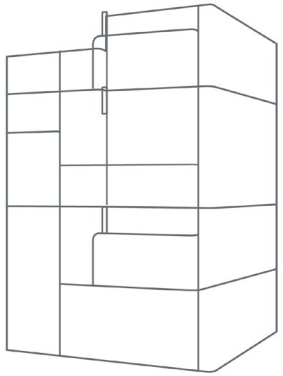
Dominika Gruszecka
Food & Consumer Products
Shimadzu Scientific Instruments

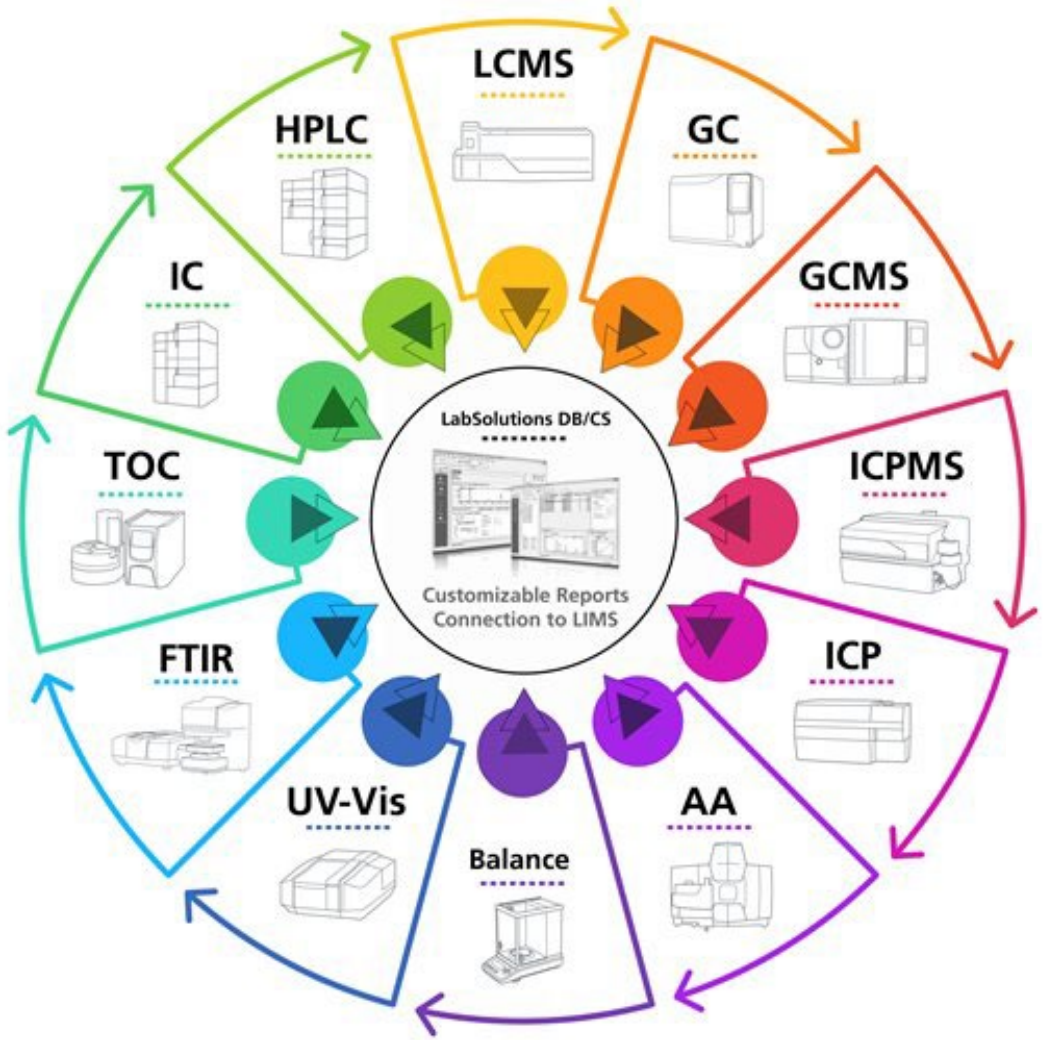
Instruments used for PFAS analysis

Liquid Chromatography & Supercritical Fluid Chromatography

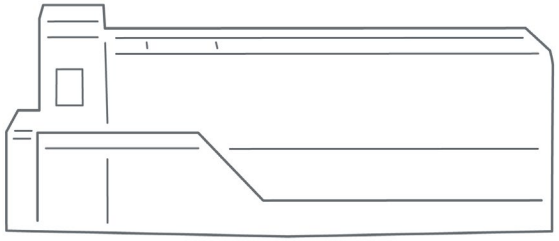


Ion Chromatography

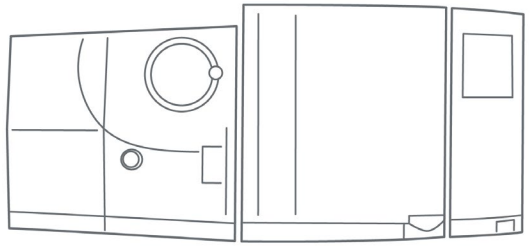




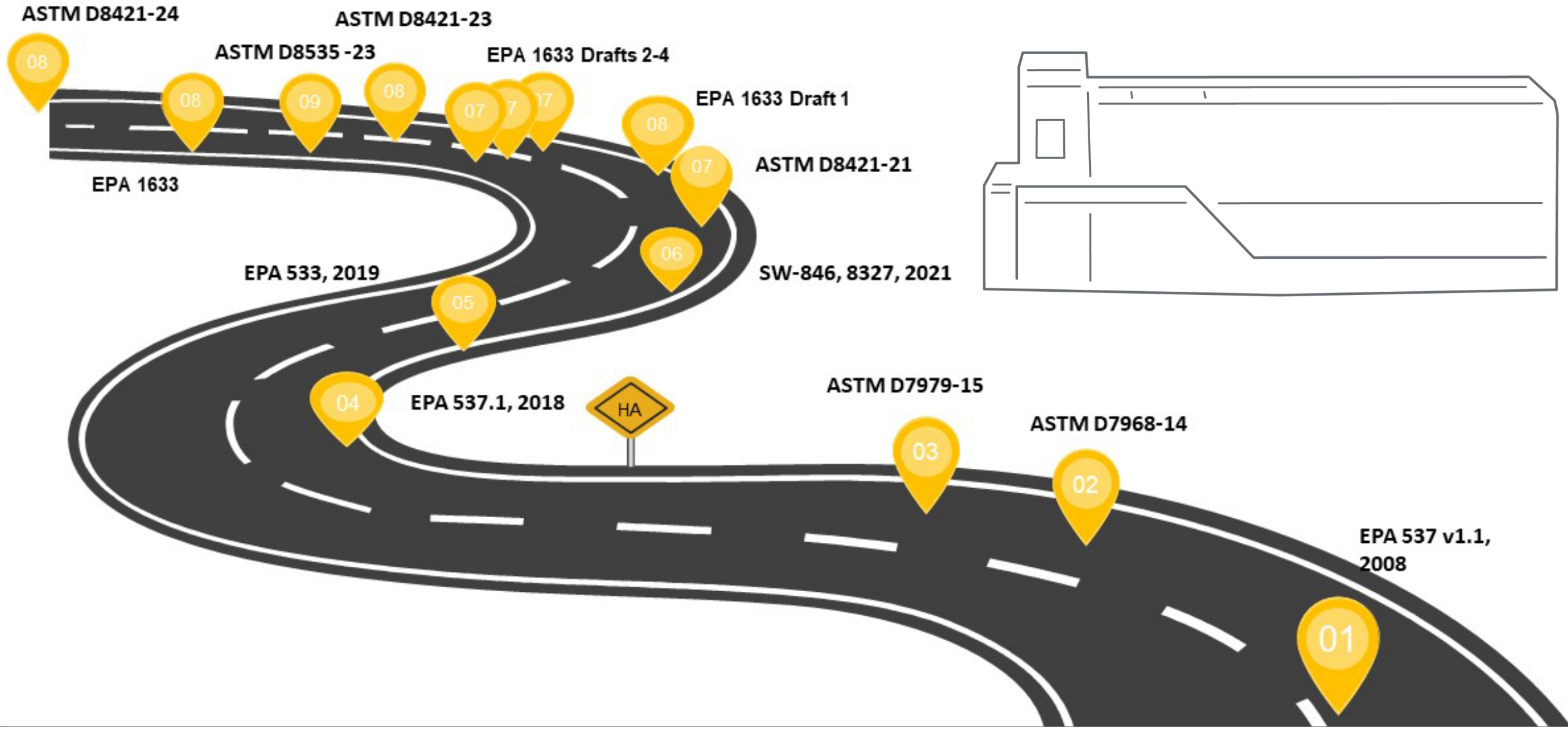
Liquid Chromatography-Mass Spectrometry



Gas Chromatography – Mass Spectrometry



Where methods and practices are coming from



Plastics

Extractables & Leachables

- Tested samples from 7 materials:
  - Glass, carton, metal, new plastics (3 varieties), recycled plastic
- Water extract from food-grade packaging
- 50% methanol co-solvation, 30ul liquid injection
- Based on EPA method 8327
- Run with mid-sensitivity instrument: LCMS-8050
  - Now recommending LCMS-8060NX

Unknown samples: packaging

- Highest level of PFAS was measured in the bottle made from recycled plastic
- The bottle labeled “Plastic 3” contained the highest amount of PFBA
- The water source was identified as “Spring Water”: it could indicate local PFAS contamination near the source of that water

PFAS	Blank	Glass	Cardboard	Metal	Plastic 1	Plastic 2	Plastic 3	Recycled Plastic
PFBA	NQ	NQ	NQ	23.8	NQ	15.3	104.3	18.1
PFPeA	ND	ND	ND	ND	ND	ND	ND	ND
4-2 FTS	ND	NQ	NQ	NQ	NQ	NQ	NQ	NQ
PFHxA	ND	ND	ND	ND	ND	ND	ND	ND
PFBS	NQ	ND	ND	NQ	ND	ND	ND	ND
PFHpA	ND	NQ	NQ	ND	NQ	NQ	ND	ND
PFHxS	ND	ND	ND	ND	ND	ND	ND	ND
PFPeS	ND	ND	ND	ND	ND	ND	ND	ND
6-2 FTS	NQ	NQ	ND	NQ	81.5	NQ	ND	253.9
PFOA	ND	NQ	NQ	NQ	NQ	NQ	NQ	NQ

Concentrations shown in ppt.

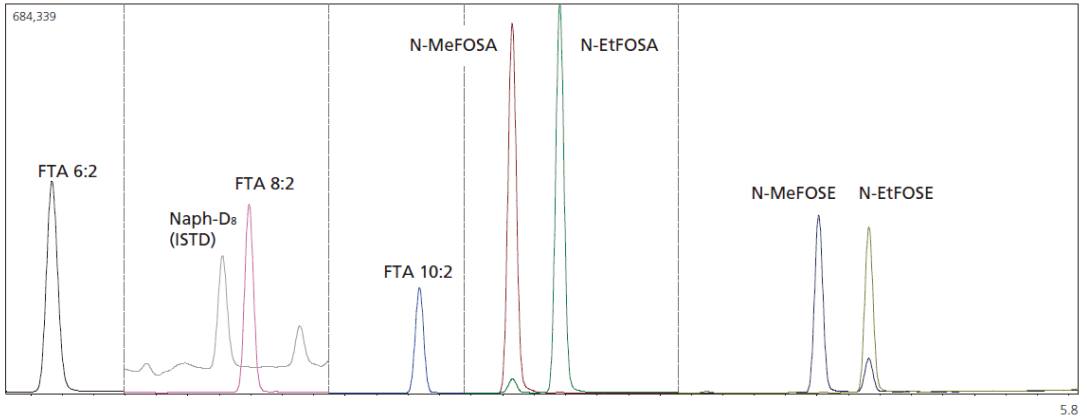
Textiles

Stain resistance & water barriers

GCMS QP2020NX

Chemical Ionization GCMS

Separation of PFAS by CI-GCMS after solvent extraction from treated textile samples



Summary of accuracy % at low, mid, and high concentration levels

Compound	Accuracy%		
	7.50 ng/mL	30.0 ng/mL	150 ng/mL
FTA 6:2	104	88.6	88.1
FTA 8:2	76.6	83.7	83.1
FTA 10:2	95.8	87.4	82.1
N-MeFOSA	89.8	78.8	79.7
N-EtFOSA	97.7	80.0	82.7
N-MeFOSE	84.5	81.0	85.9
N-EtFOSE	92.1	83.4	86.2

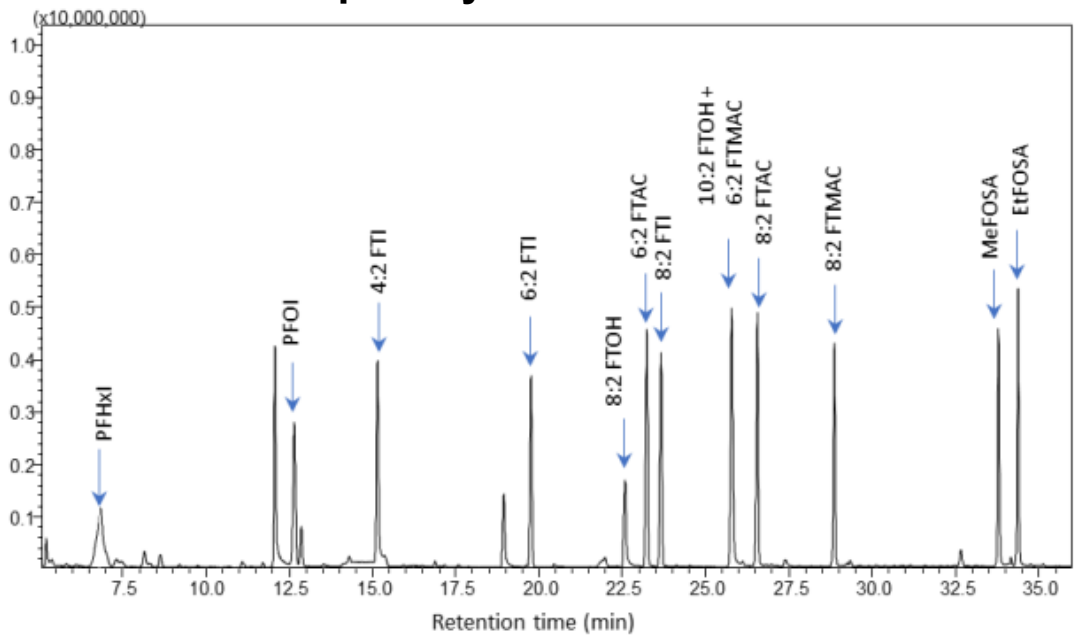
Research:
Beyond EPA regulated PFAS

GCMS QP2020NX

Volatile PFAS

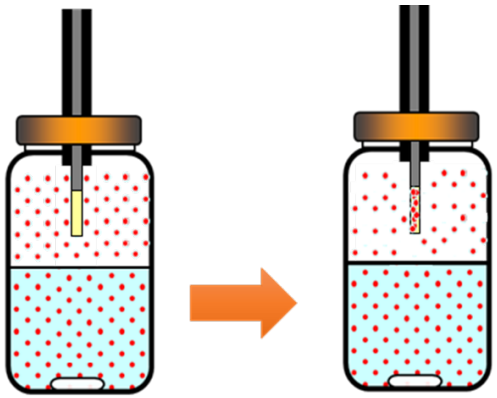
- 13 PFAS including fluorotelomer alcohols and iodides not in EPA 1633
- Automated extraction from sample headspace

Separation of PFAS extracted from contaminated water samples by SPME-HS-GCMS



Volatile PFAS

- Solid phase microextraction
  - Collects off-gassing compounds
  - Pre-concentrates
  - Leaves matrix behind
  - Collects analyte from solid or liquid samples



Compound	Calibration Range (ng/L)	R^2	RF %RSD
PFHxI	2.5-2000	0.993	10.89
PFOI	2.5-2000	0.997	10.26
4:2 FTI	2.5-800	0.993	8.28
6:2 FTI	25-800	0.994	13.53
8:2 FTOH	25-2000	0.997	5.37
6:2 FTAC	25-2000	0.998	19.87
8:2 FTI	2.5-800	0.996	13.59
10:2 FTOH	2.5-2000	0.999	10.38
6:2 FTMAC	2.5-800	0.995	12.43
8:2 FTAC	5-250	0.995	14.81
8:2 FTMAC	2.5-250	0.998	19.51
MeFOSA	5-2000	>0.999	17.79
EtFOSA	10-2000	0.999	11.40

AOAC

- Food testing
- Plastics and food packaging

LCMS-8060NX

Call for Methods

- 30 PFAS, 11 matrices
- Required limits of detection range 0.01-5 µg/kg depending on matrix category
- PFOS, PFOA, PFNA, and PFHxS are regulated in some foods in the EU
- Met conditions and submitted recommended methods for review

- New working group for PFAS in food contact materials
  - Developing requirements for methods

Extraction

Sample cleanup

Supercritical Fluid Extraction

Benefits over solvent extraction

Automation:

- Reduces analyst errors

Green chemistry:

- Reduces organic solvent use & overall waste

Selectivity:

- Pressures and temperatures can be varied for step-wise extraction
- 100% CO_2 for nonpolar compounds, add solvent for polar compound extraction

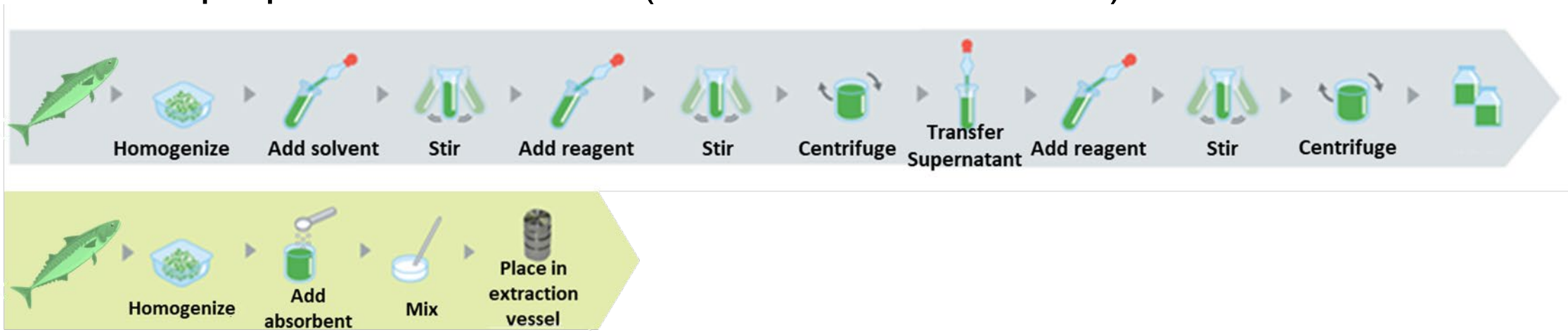
Speed:

- Faster diffusion into sample

Automated Extraction

SFE prep for fatty matrices: 18 PFAS extracted from fish

- Alternate prep methods to SPE (Solid Phase Extraction)



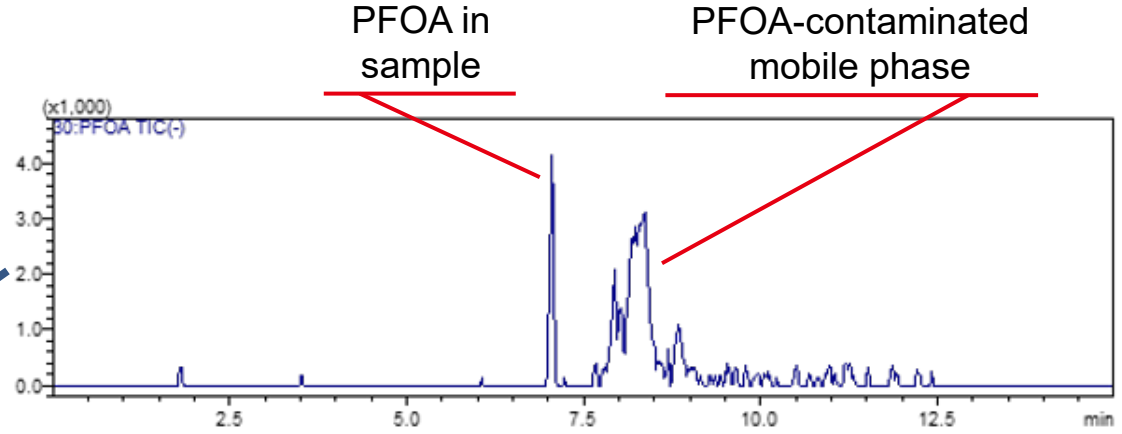
- LCMS conditions: EPA method 533
- Recovery reproducibility at lowest point (2 µg/kg) was below 25% for 89% of compounds
- At 20 µg/kg, all compounds recovered with RSD below 20%

What have we **Discovered** along the way?

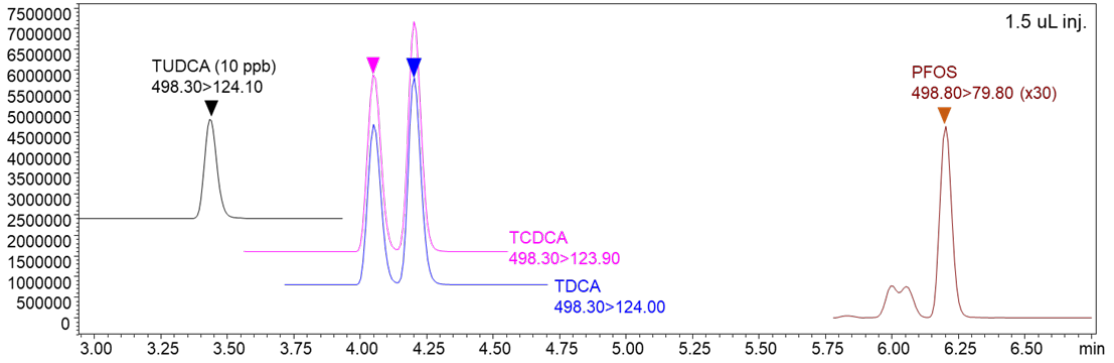
- Testing environment contamination
- Delay columns
- Cholic acid interferences

Lessons Learned

Delay column separates environmental and solvent contamination from sample targets



Separation of PFOS from Cholic Acids and baseline resolution between PFOS branched and linear isomers



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