What am I eating? An exploration of PFAS in food and food contact materials

Karl Oetjen, PhD | Senior Scientist, Technical Marketing, SCIEX

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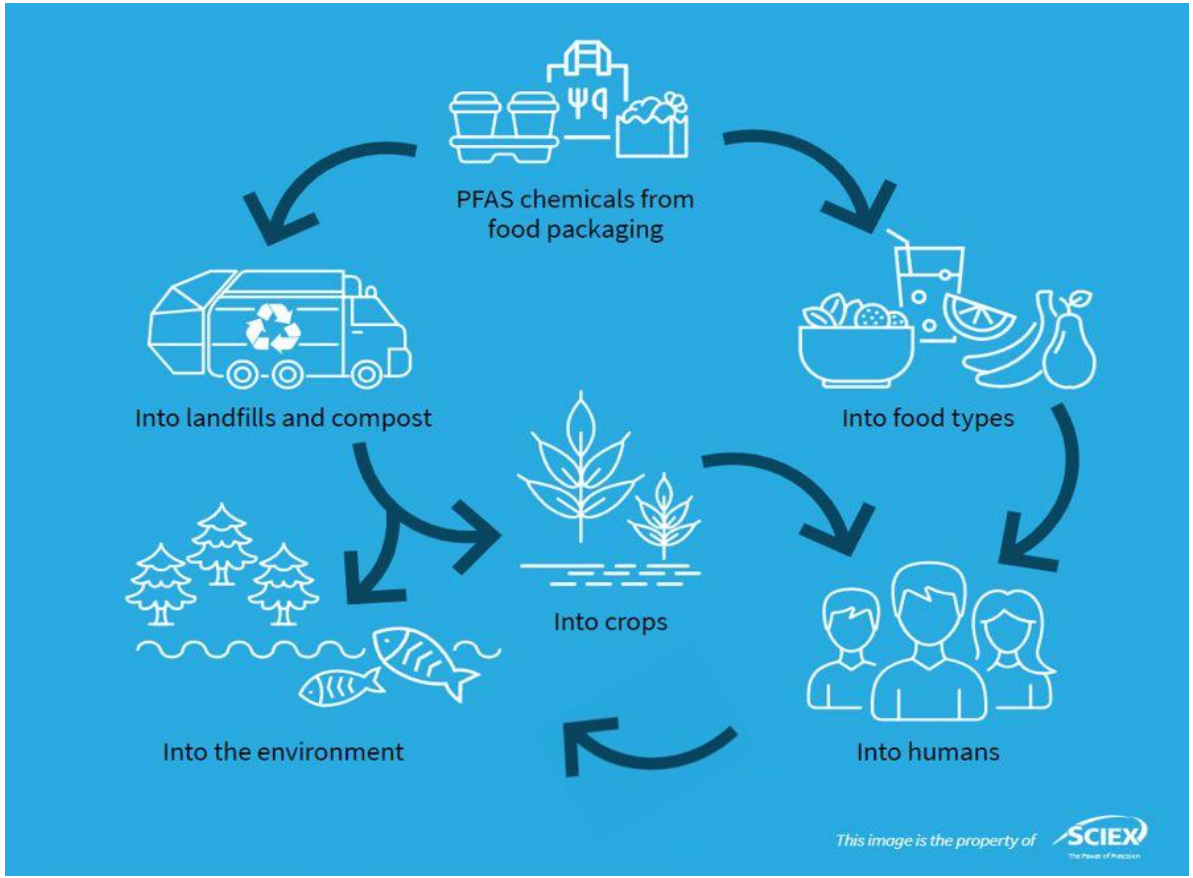
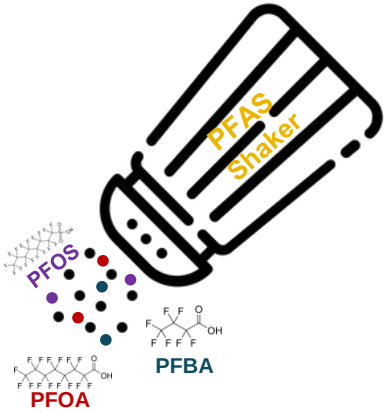
The Power of Precision

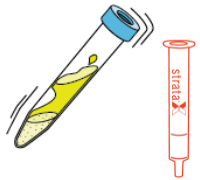
PFAS life cycle



Does inexpensive food contain more PFAS?



The typical LC-MS/MS PFAS workflow

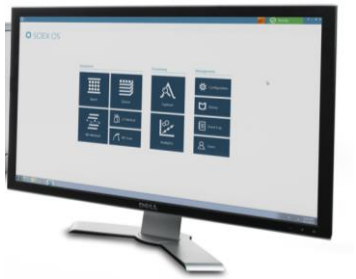


Sample preparation

HPLC system

Column

Mass spectrometer



Data processing

RUO-MKT-11-14298-A

Food analysis: QuEChERS procedure

- Add 5.0 g homogenized sample to 50 mL tube and 10 µL of 1,000 ng/mL isotopically labeled surrogate standard

- Add LC-MS grade water
  - 5 mL fruit or vegetable
  - 15 mL dry foods
  - 25 mL powders (protein, milk, etc.)
- Add 10 mL LC-MS grade ACN
- Add extraction surrogates

Add QuEChERS salt packet

Vortex 3 min, centrifuge 5 min

Transfer 200 µL to conical vial for analysis

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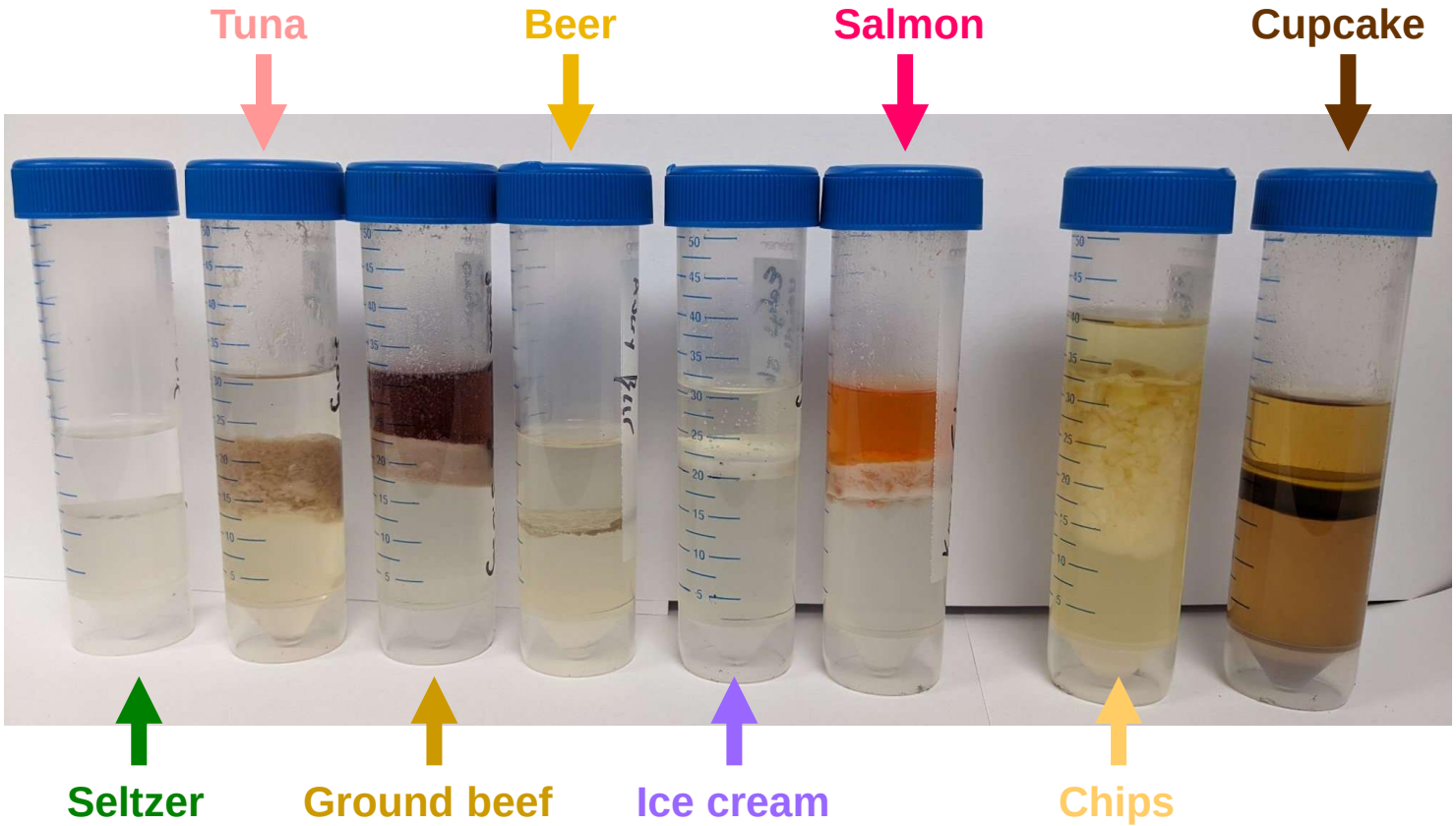
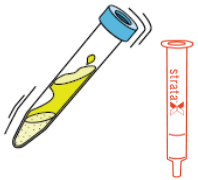
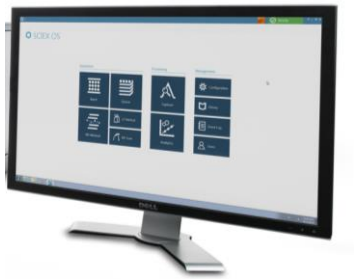
Samples



The typical LC-MS/MS PFAS workflow





Sample preparation

HPLC system

Column

Mass spectrometer

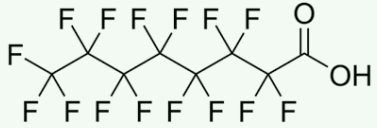
Data processing

RUO-MKT-11-14298-A

Common PFAS target analytes

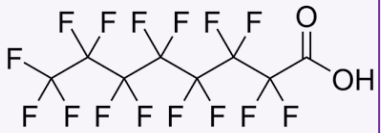
For targeted methods, analytes are typically negatively charged and are amenable to reverse phase LC conditions

Perfluorinated carboxylates



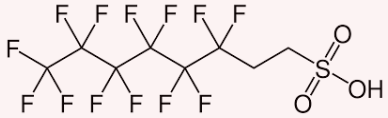
Perfluorooctanoate (PFOA)

Perfluorinated sulfonates



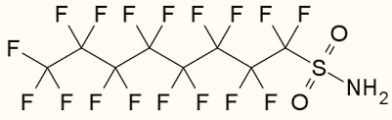
Perfluorooctane Sulfonate (PFOS)

Fluorotelomer sulfonates



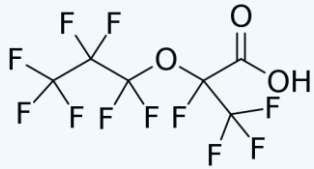
6:2 Fluorotelomer Sulfonate (FTS)

Sulfonamides, sulfonamide acids

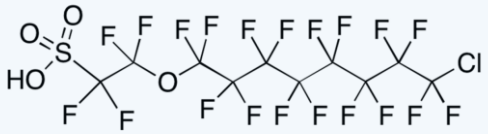


Perfluorooctane Sulfonamide (PFOSA)

Perfluorinated ether carboxylates and sulfonates



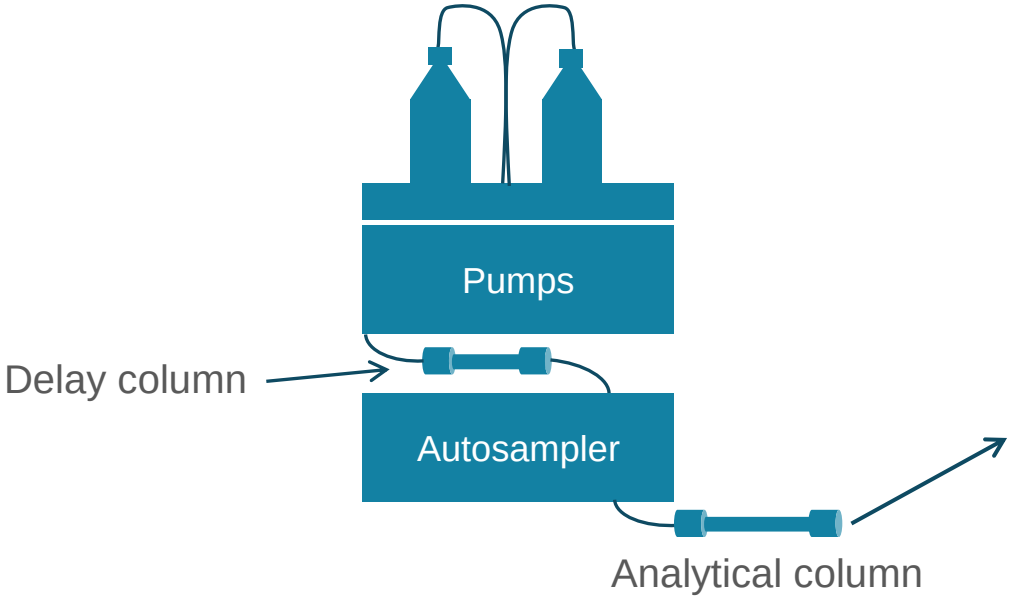
HFPO-DA (GenX)

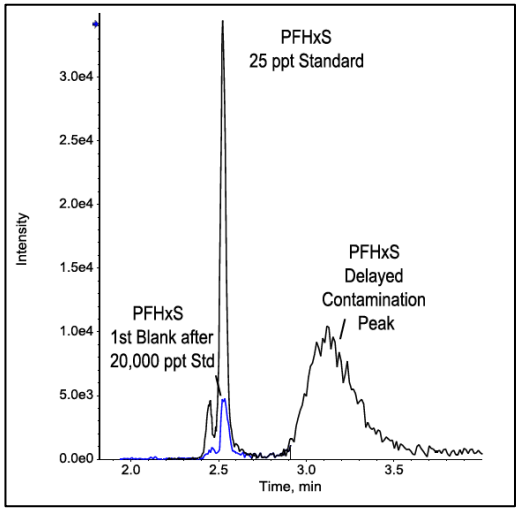


11Cl-PF3OUdS

Controlling contamination: delay columns

- Delay column
  - Also referred to as holdup column, contamination column or cleanup column
  - Retains contaminants from mobile phase and slowly elutes them later in the gradient

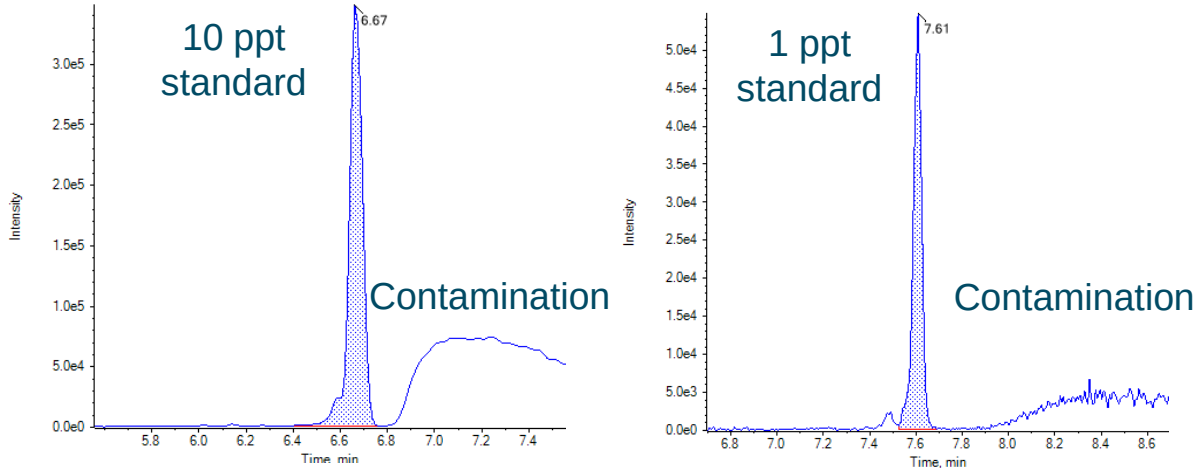




To MS

Controlling contamination: delay columns

HPLC system contaminated with Perfluorobutane sulfonate (PFBS) and Perfluorohexane sulfonate



Delay columns can be the same column chemistry with larger diameter and larger particle size or a more retentive column chemistry

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Find yourself a PFAS free HPLC

ExionLC 2.0 system PFAS kit

Natural PEEK tubing (0.125 in OD x .080 in ID)

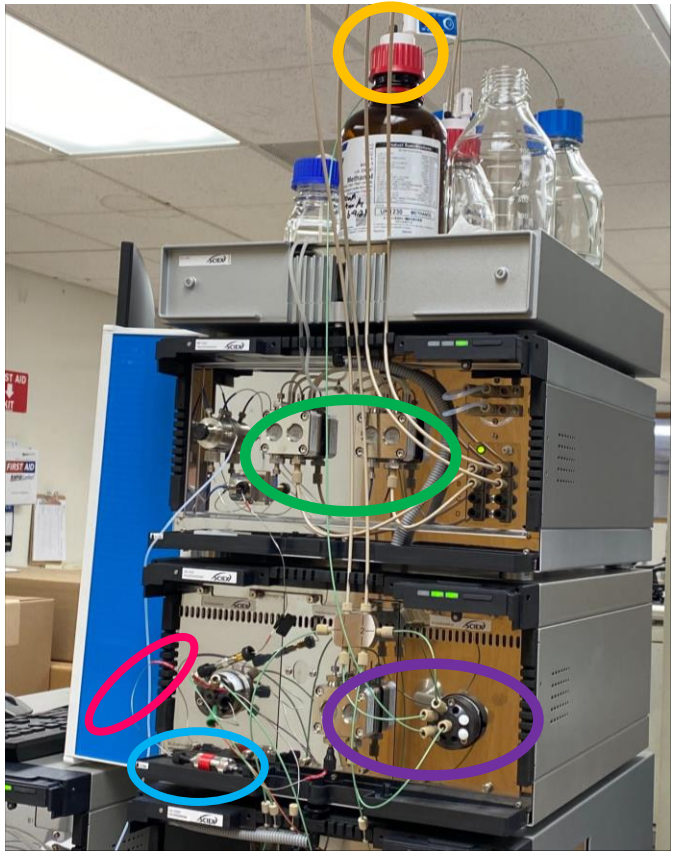
Green PEEK tubing (1/16 in OD x .030 in ID)

SecurityLINK stainless steel connection system

Luna Omega PS C18 (50x3 mm 5 µm)

Luna Omega PS C18 (100x2 mm 3 µm)
(analytical column not shown)

Solvent inlet filter



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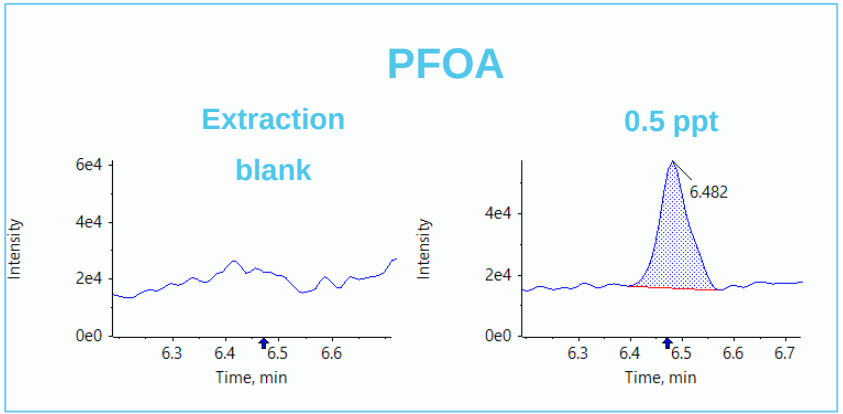
Controlling contamination

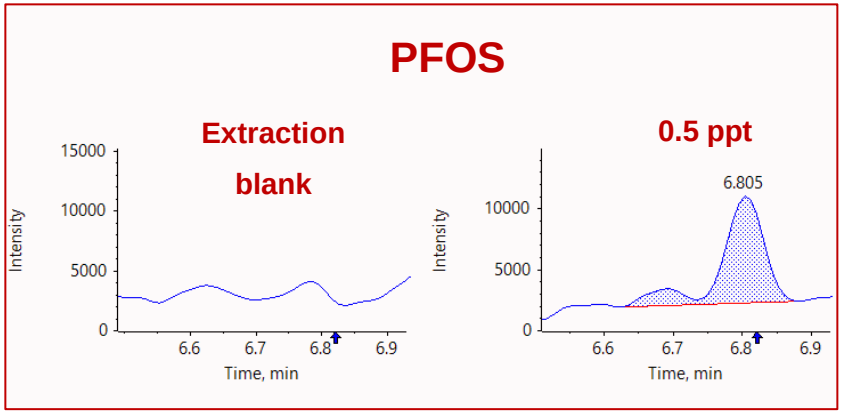
Desired characteristics for PFAS analysis

- Retention of PFBA and PFPeA, which is difficult with a standard C18 column
- Particle size >2 µm for compatibility with HPLC
- No void volume breakthrough in large volume injections (up to 1 mL)

Other considerations

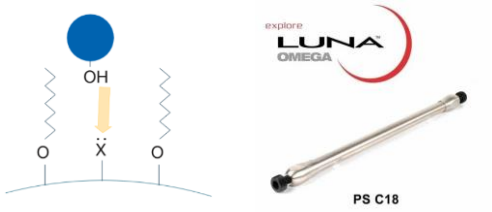
- Delay column must contain the same column chemistry or more retentive chemistry
- pH stable up to pH 10 for compatibility with ammonium hydroxide in sample extracts





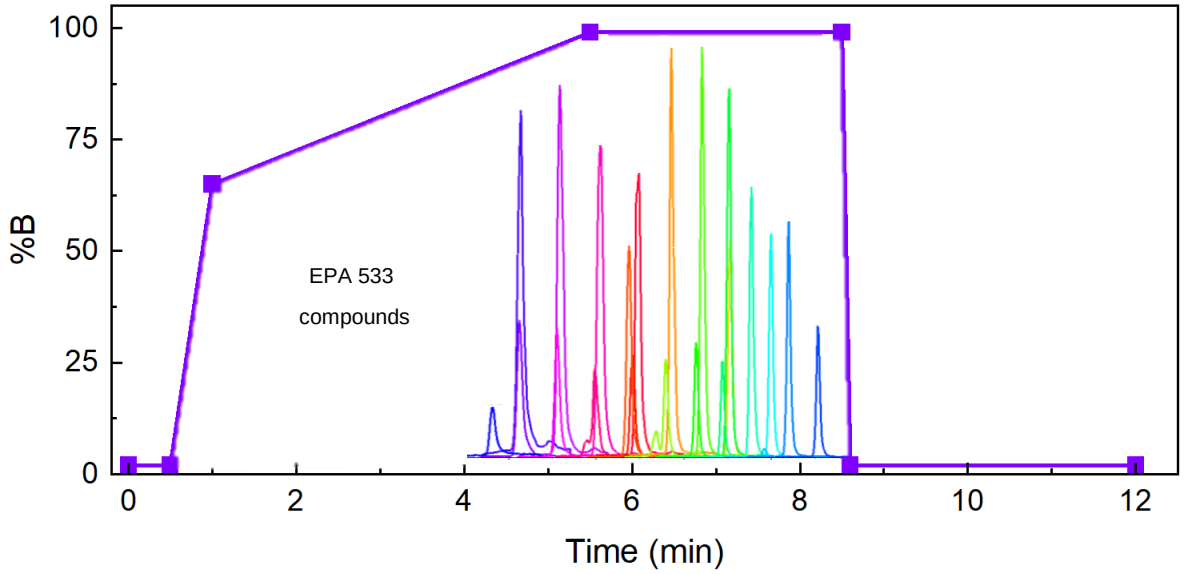
Chromatography: large volume injection

Column: Luna Omega PS C18

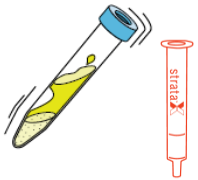


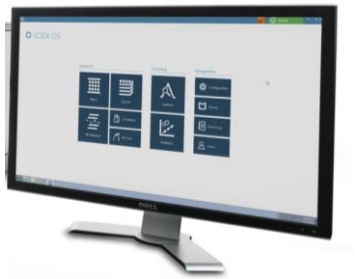
Mobile phase A: H2O + 10 mM ammonium acetate

Mobile phase A: MeOH + 10 mM ammonium acetate



The typical LC-MS/MS PFAS workflow





Sample preparation

HPLC system

Column

Mass spectrometer

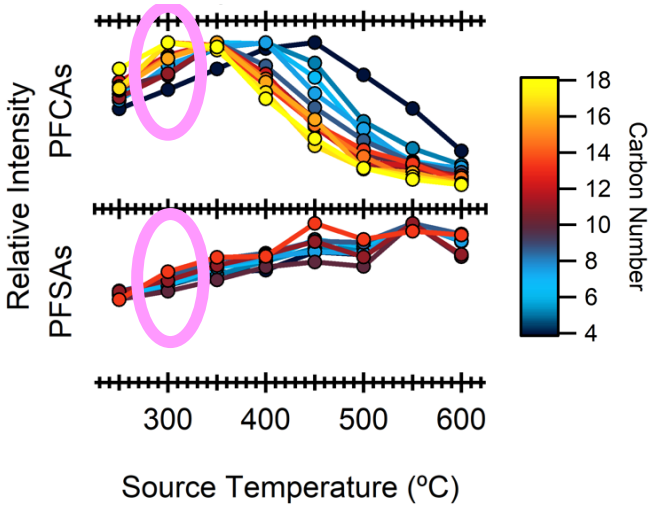
Data processing

RUO-MKT-11-14298-A

Mass spectrometry

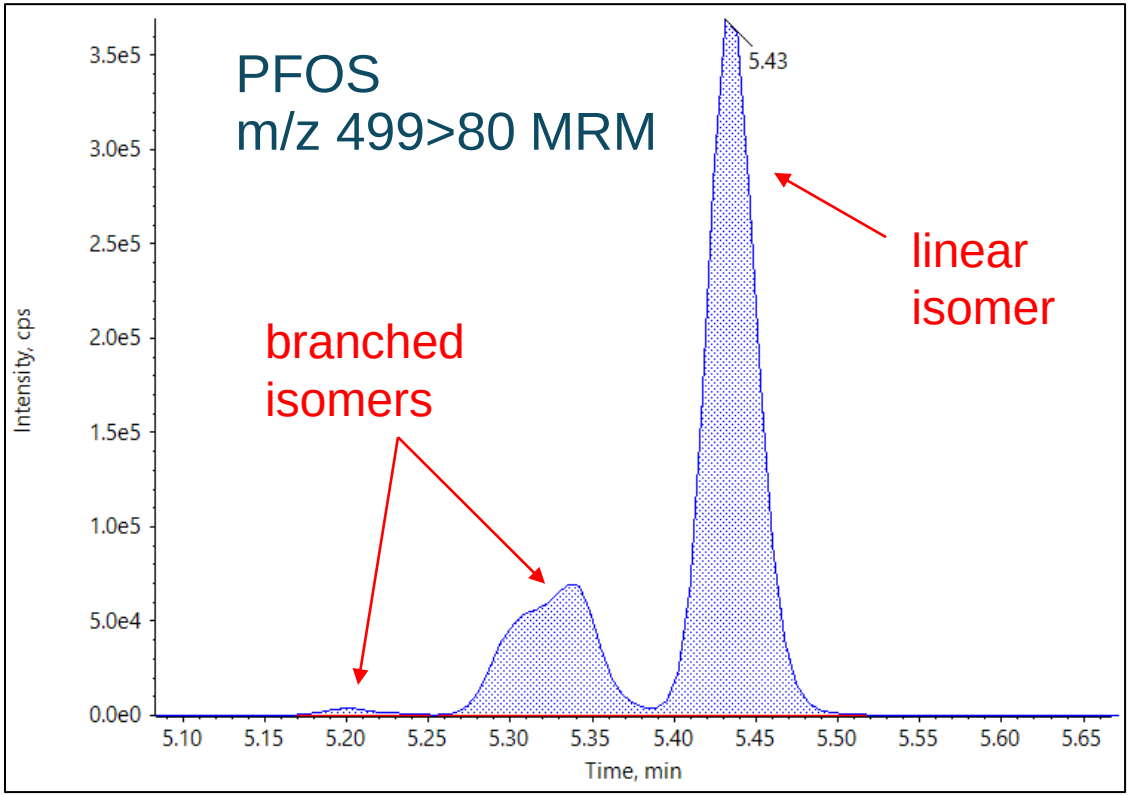
Curtain gas:	45 psi
Ion source gas 1:	35 psi
Ion source gas 2:	70 psi
ISV:	-1250 V
Source temperature (°C):	300°C

OptiFlow Pro ion source



SCIEX 7500 system

Quantifying PFAS isomers



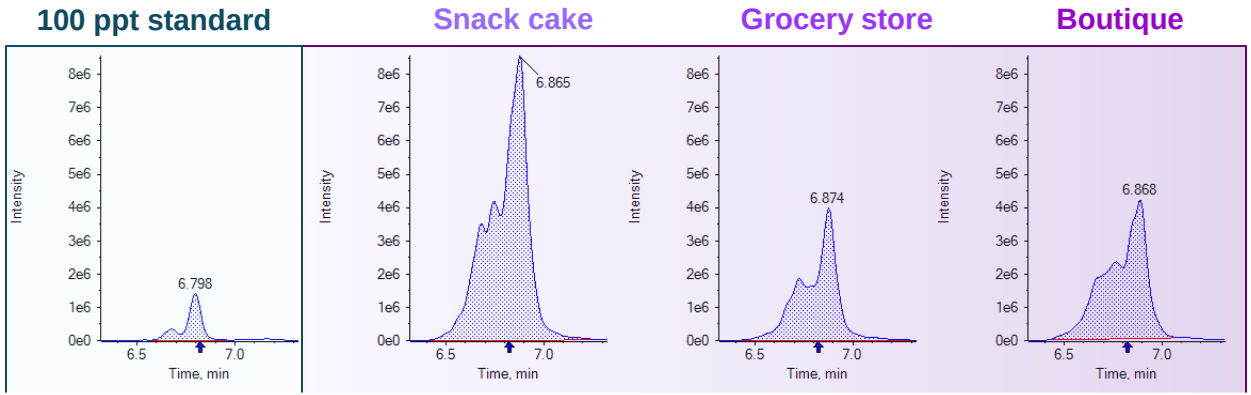
- PFAS can contain branched and linear chain isomers (e.g., PFOS, PFHxS)
- Some regulated methods require that isomers be chromatographically separated
- However, some regulated methods also require data to be reported as “total” integration of branched and linear isomers

Cake

PFOS

Transition 1

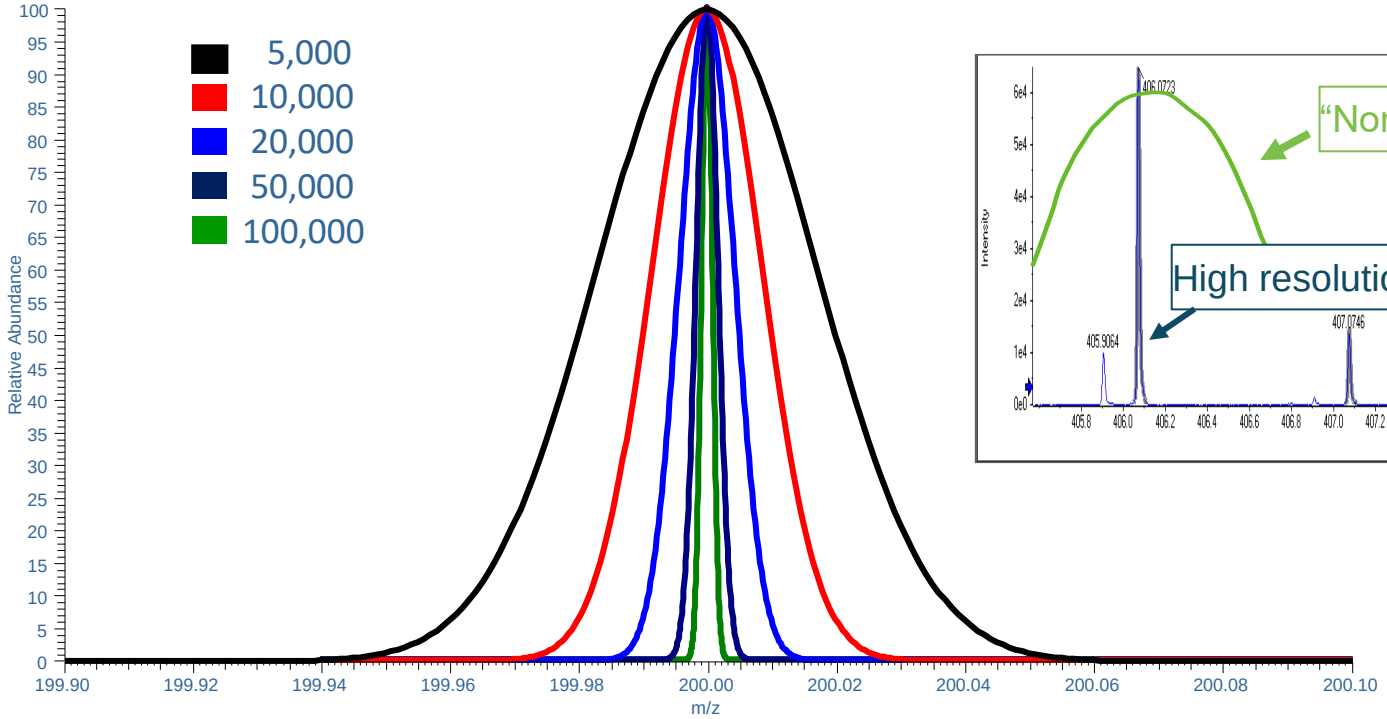
499 ➡ 80

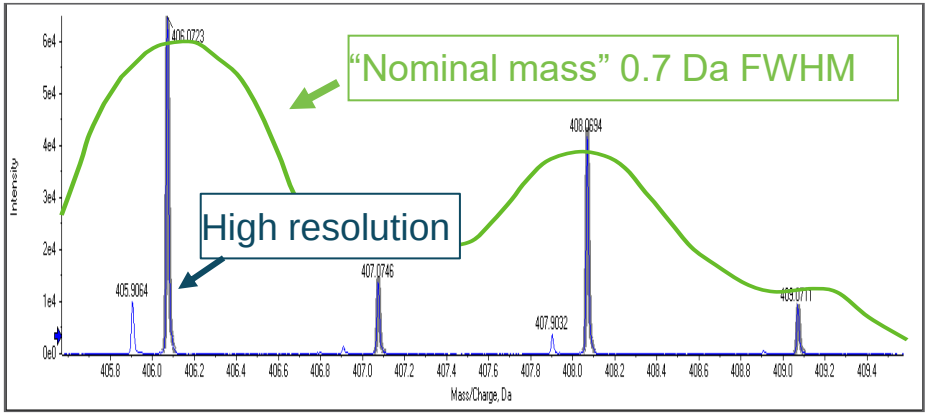


Least expensive ➡ Most expensive

HRMS vs. nominal mass

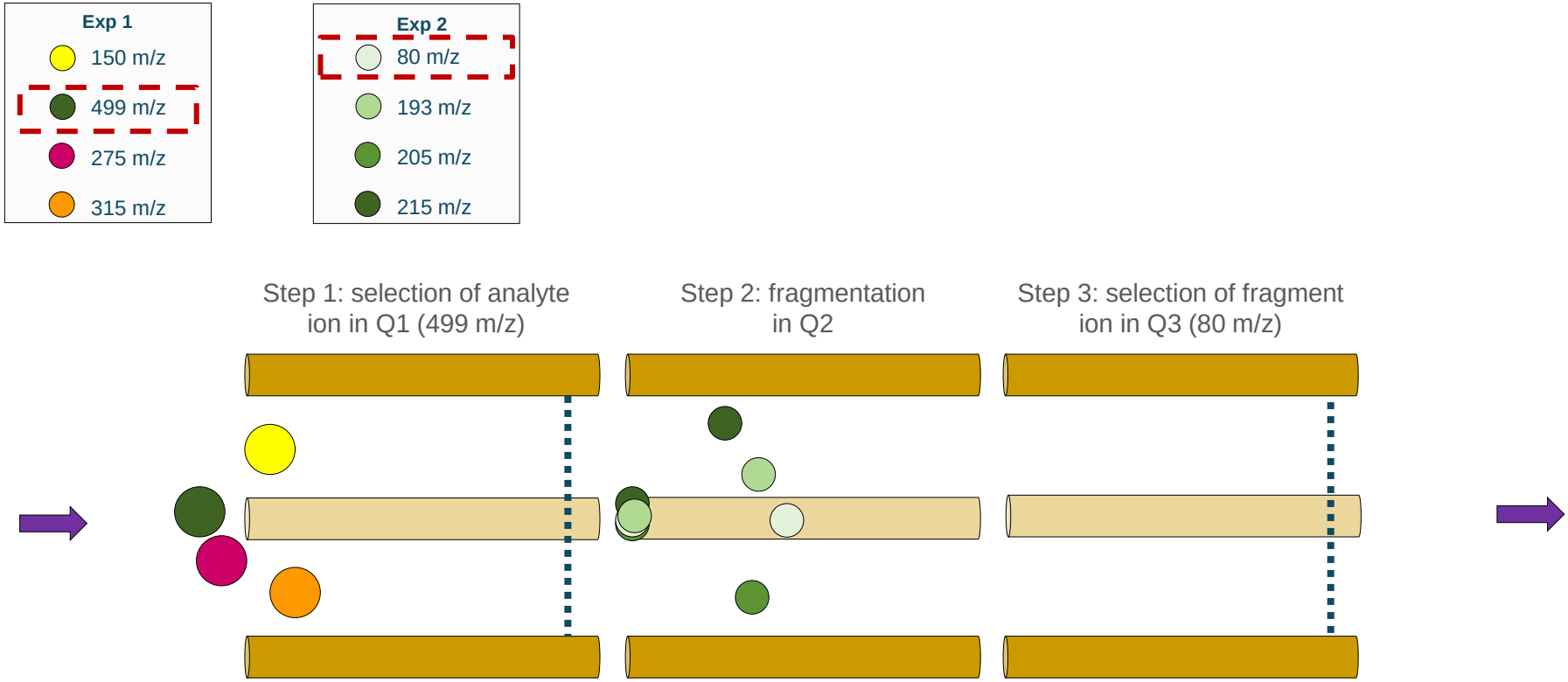
Effects of increased resolution and mass accuracy



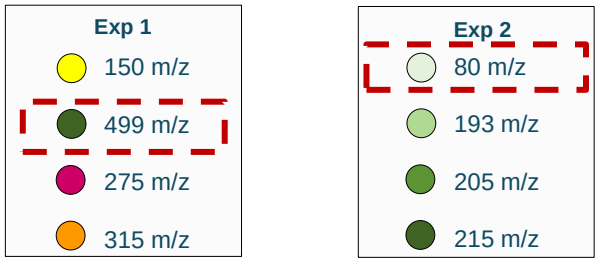


Fernández-Alba 2019

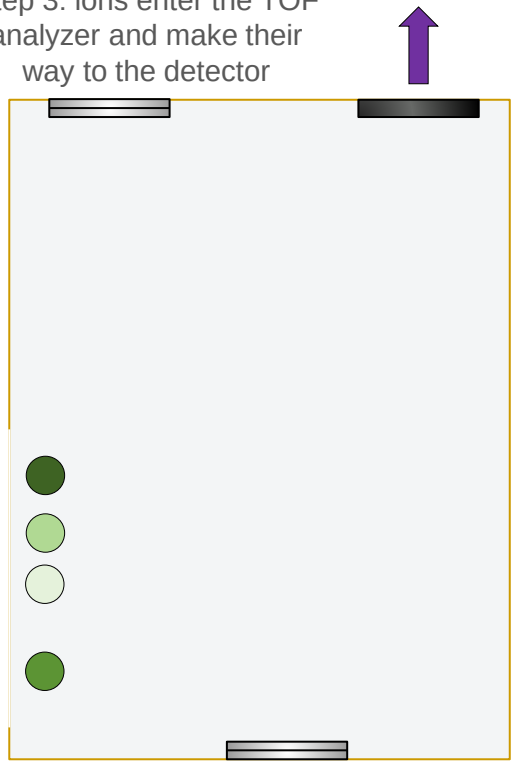
MRM scan for quantification



MS/MS scan using a QTOF system

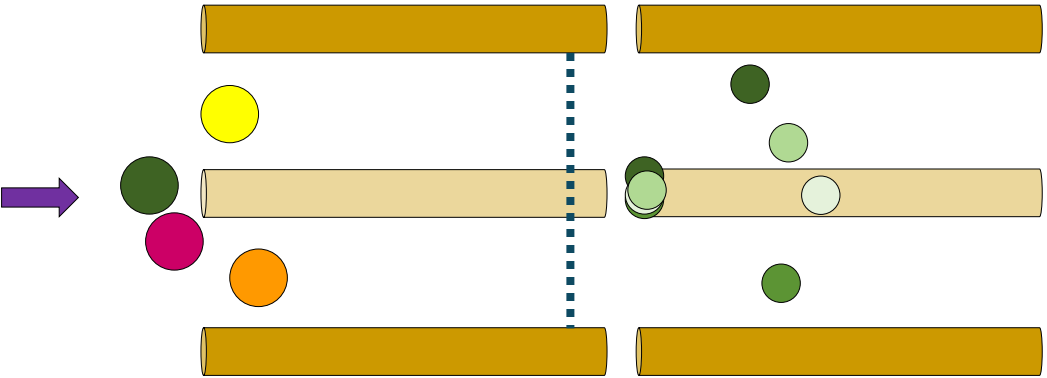


Step 3: ions enter the TOF analyzer and make their way to the detector



Step 1: selection of analyte ion in Q1 (499 m/z)

Step 2: fragmentation in Q2 with optimized CE

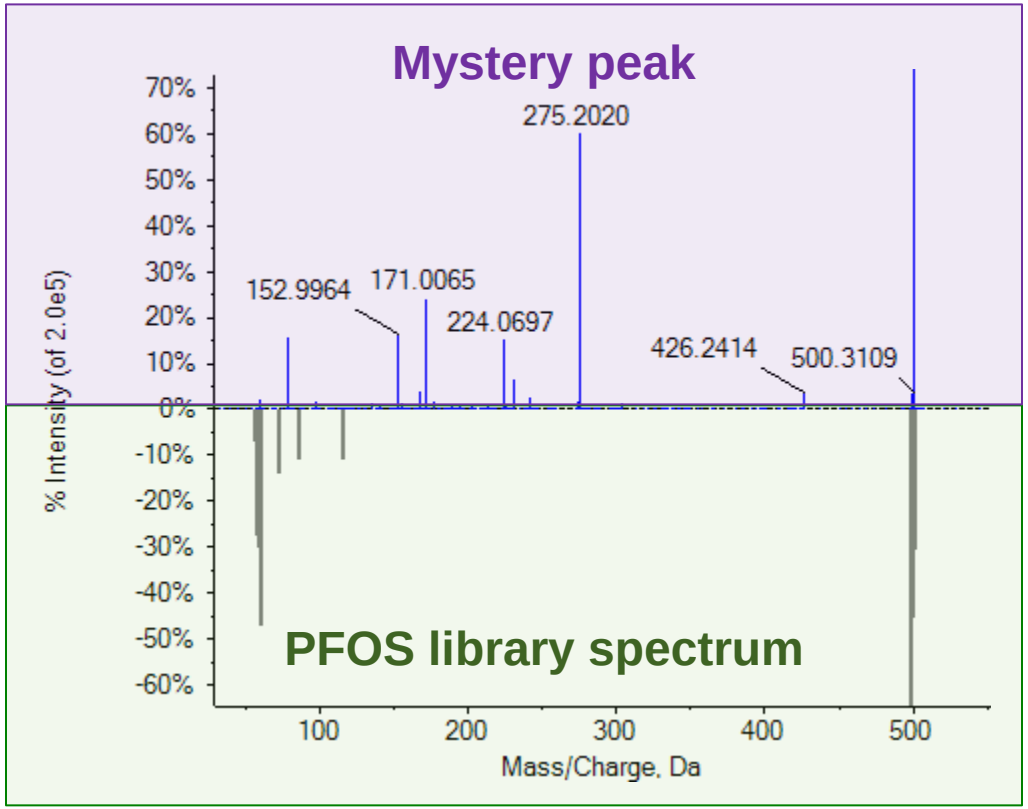


Adding confidence with MS/MS data

Mass error > 1,000 ppm

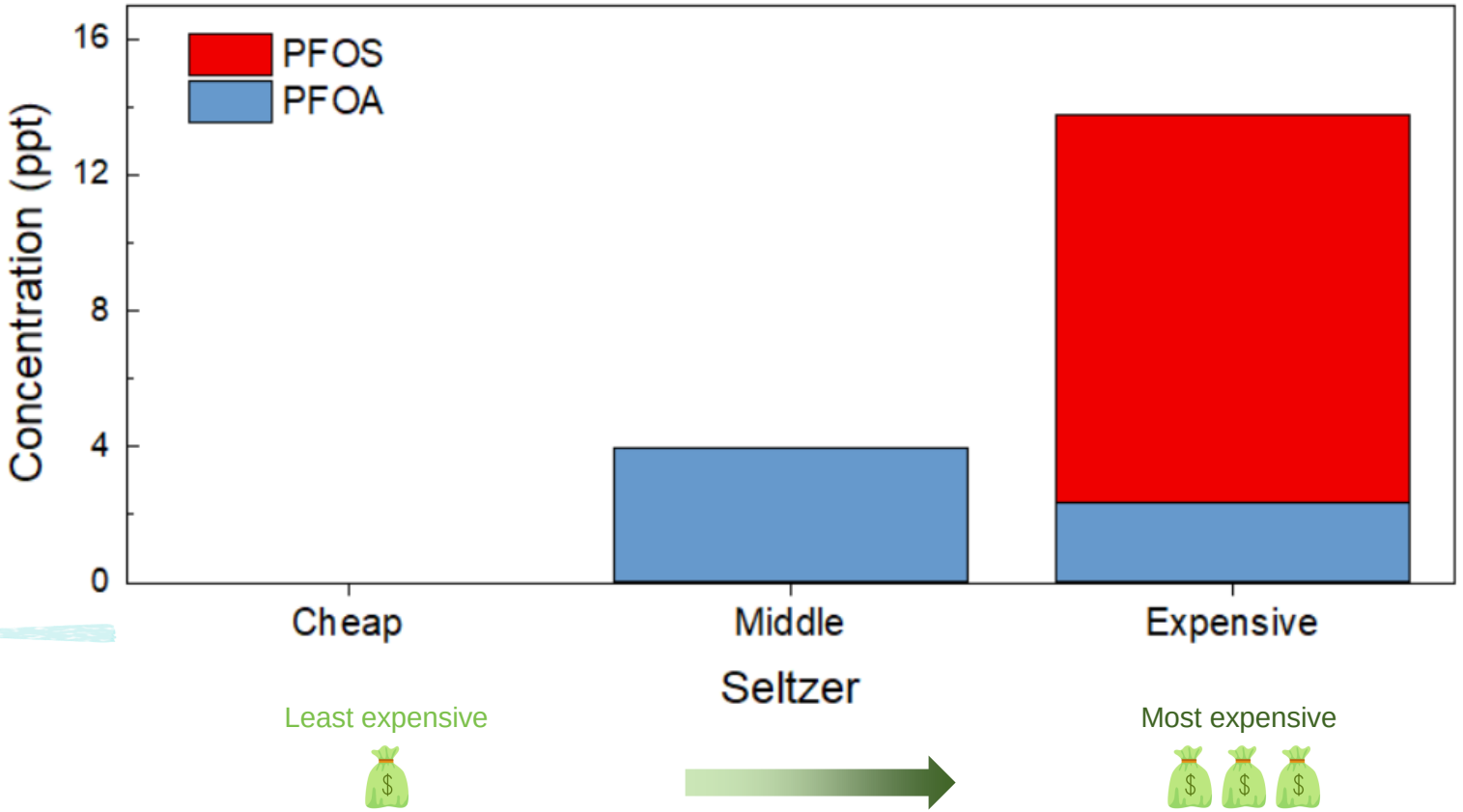
Missing diagnostic ions

Mystery peak ≠ PFOS

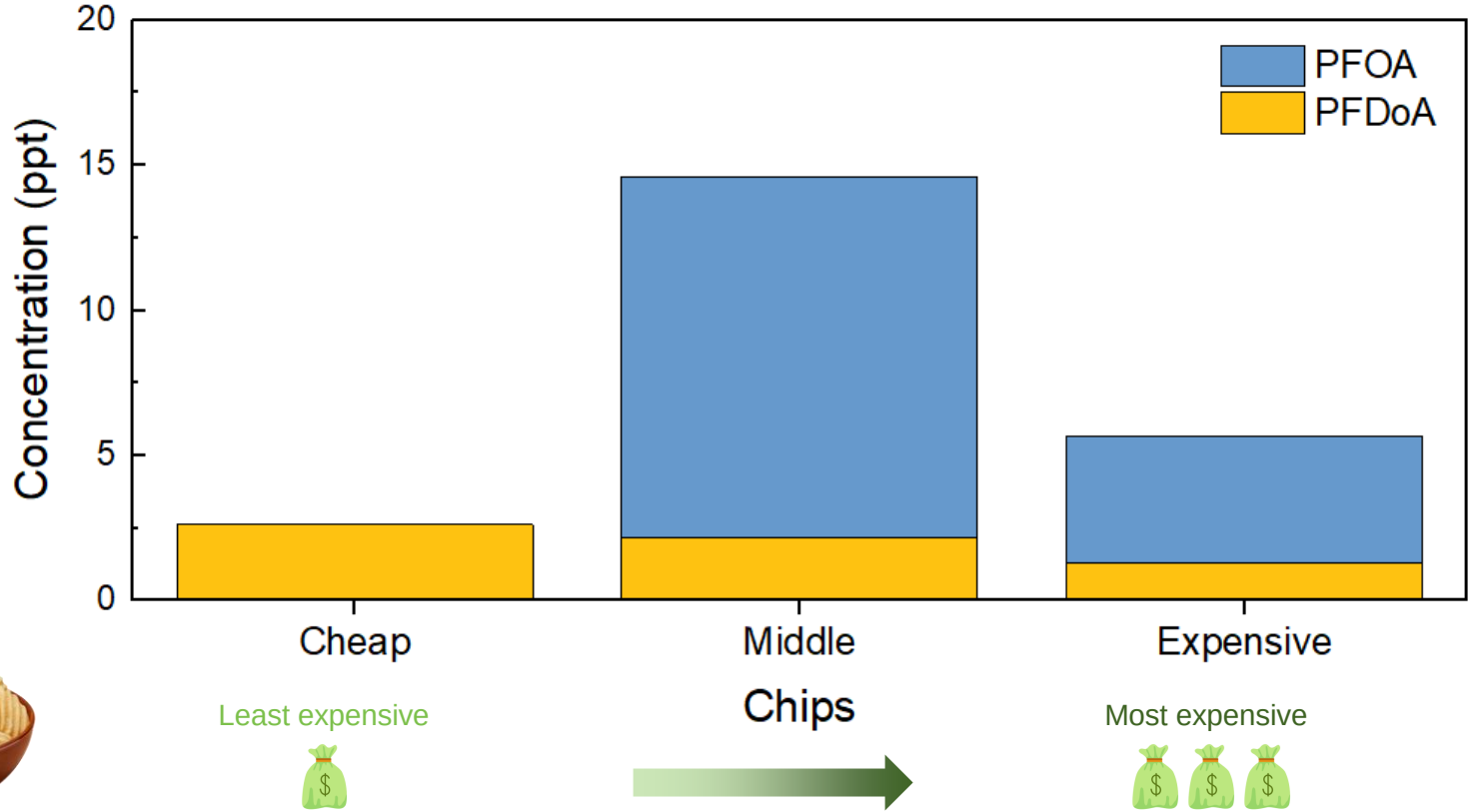


Food samples

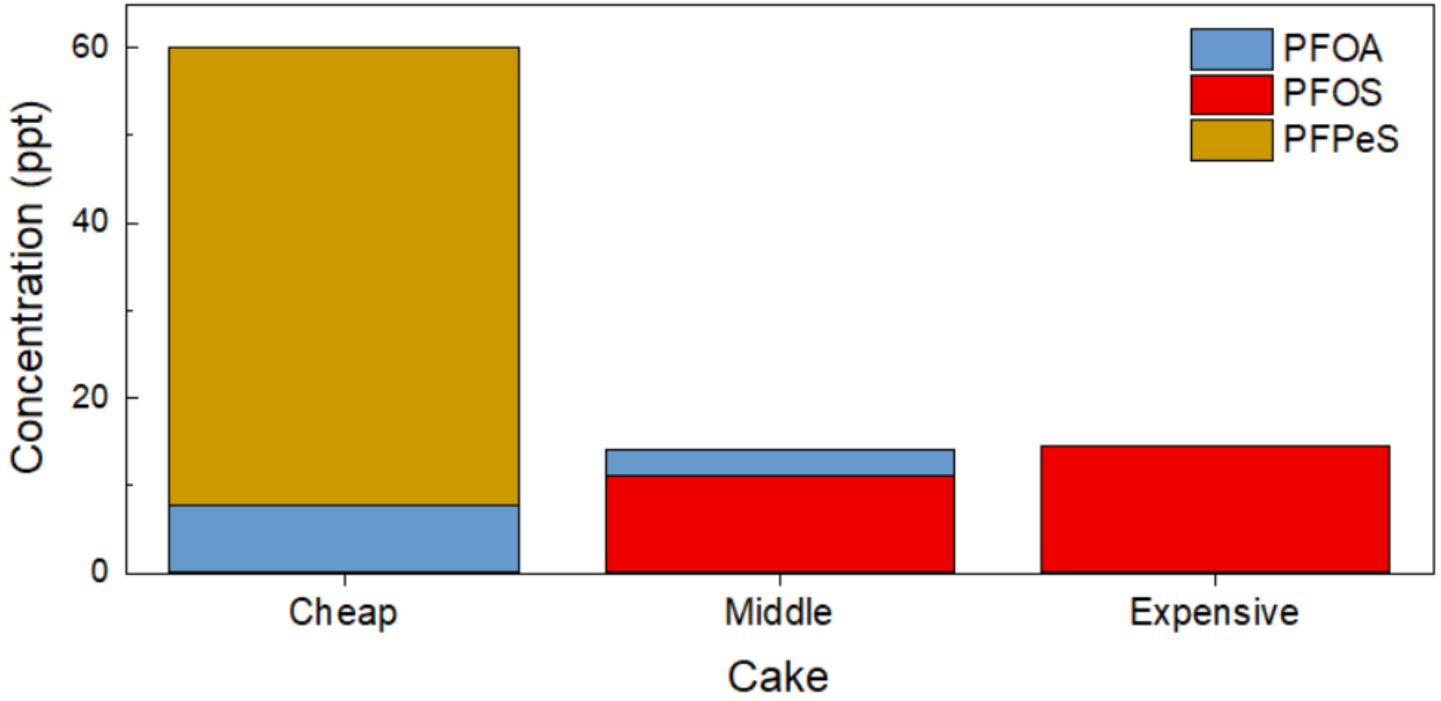
Seltzer



Chips



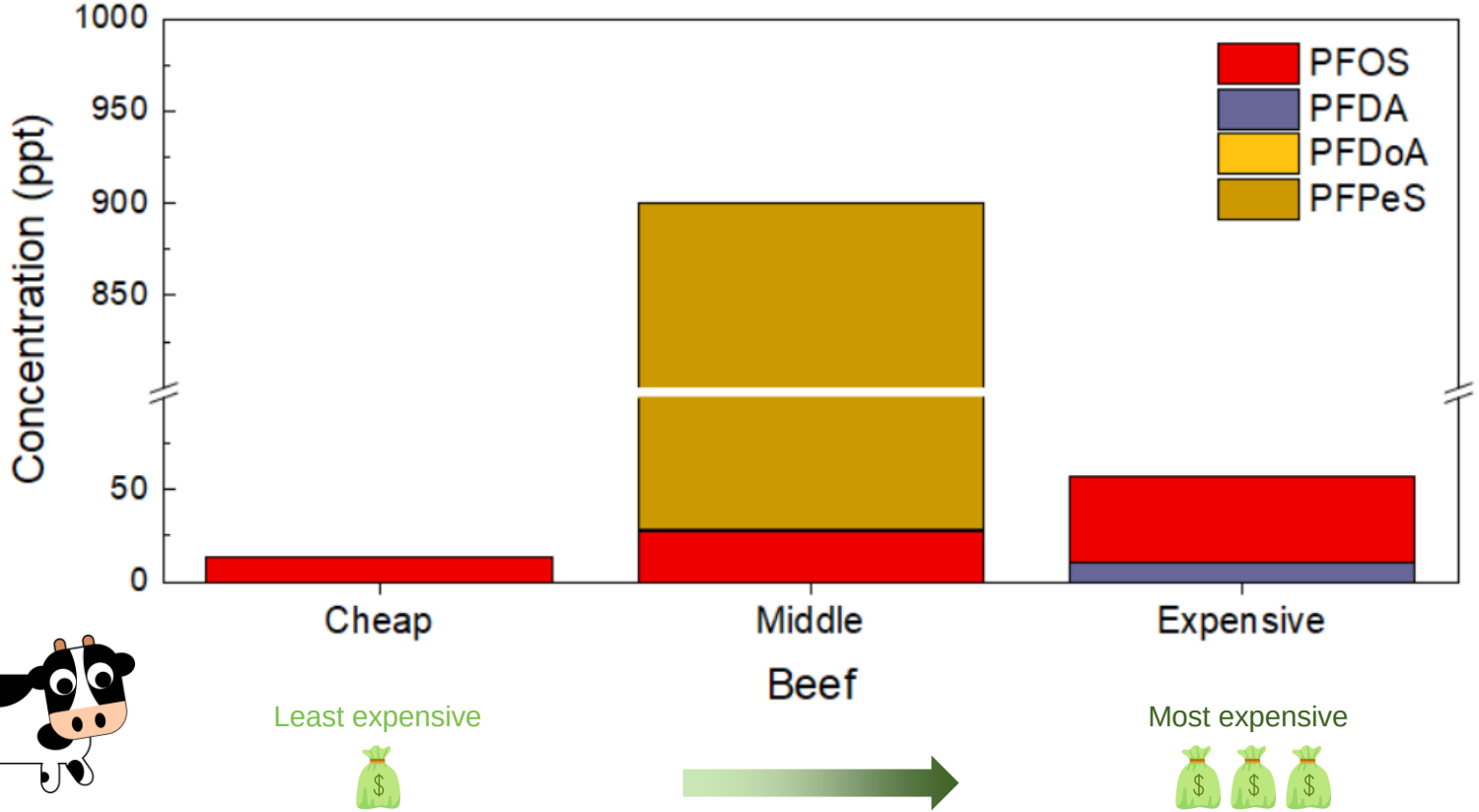
Cakes



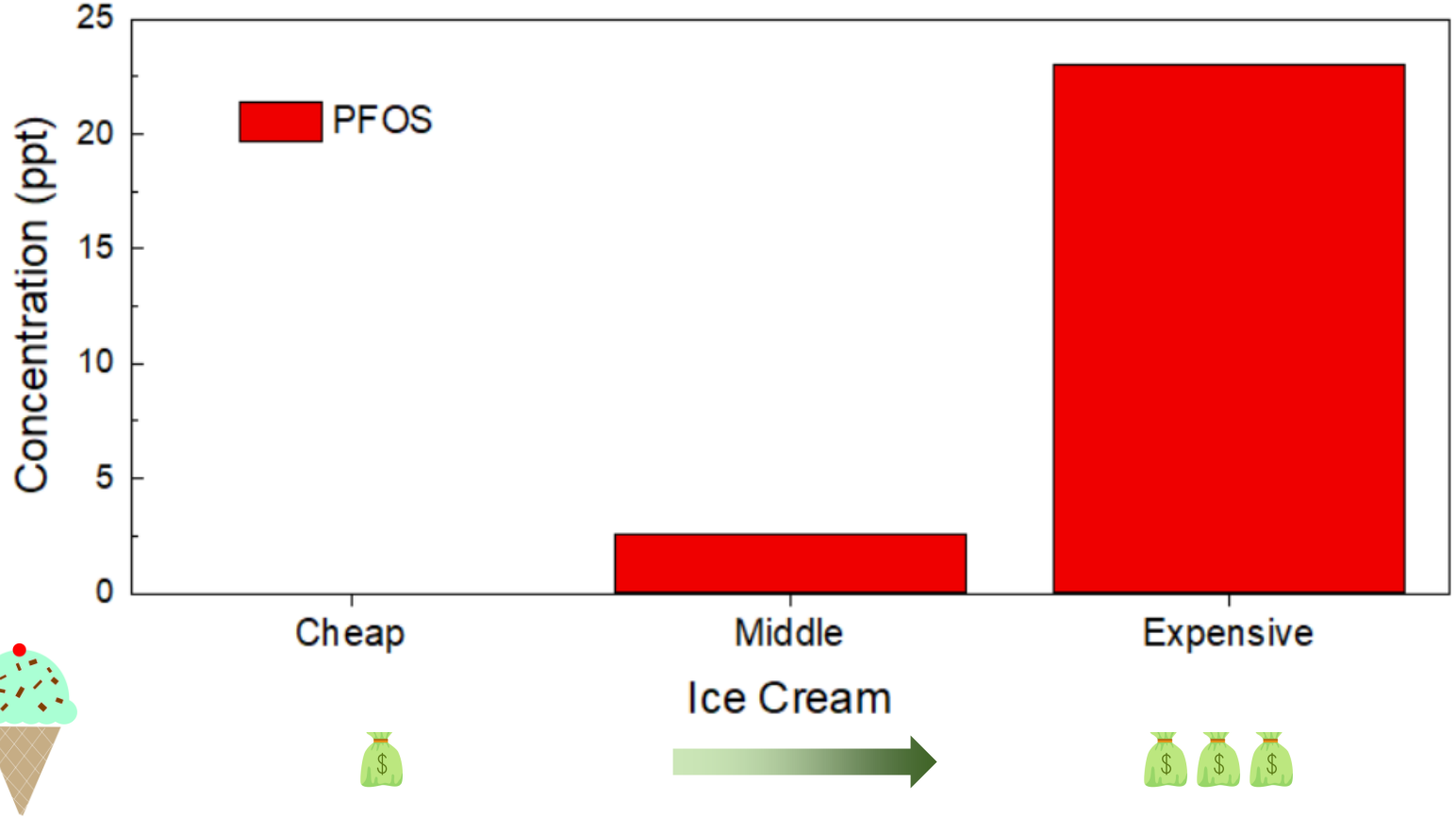
Least expensive

Most expensive

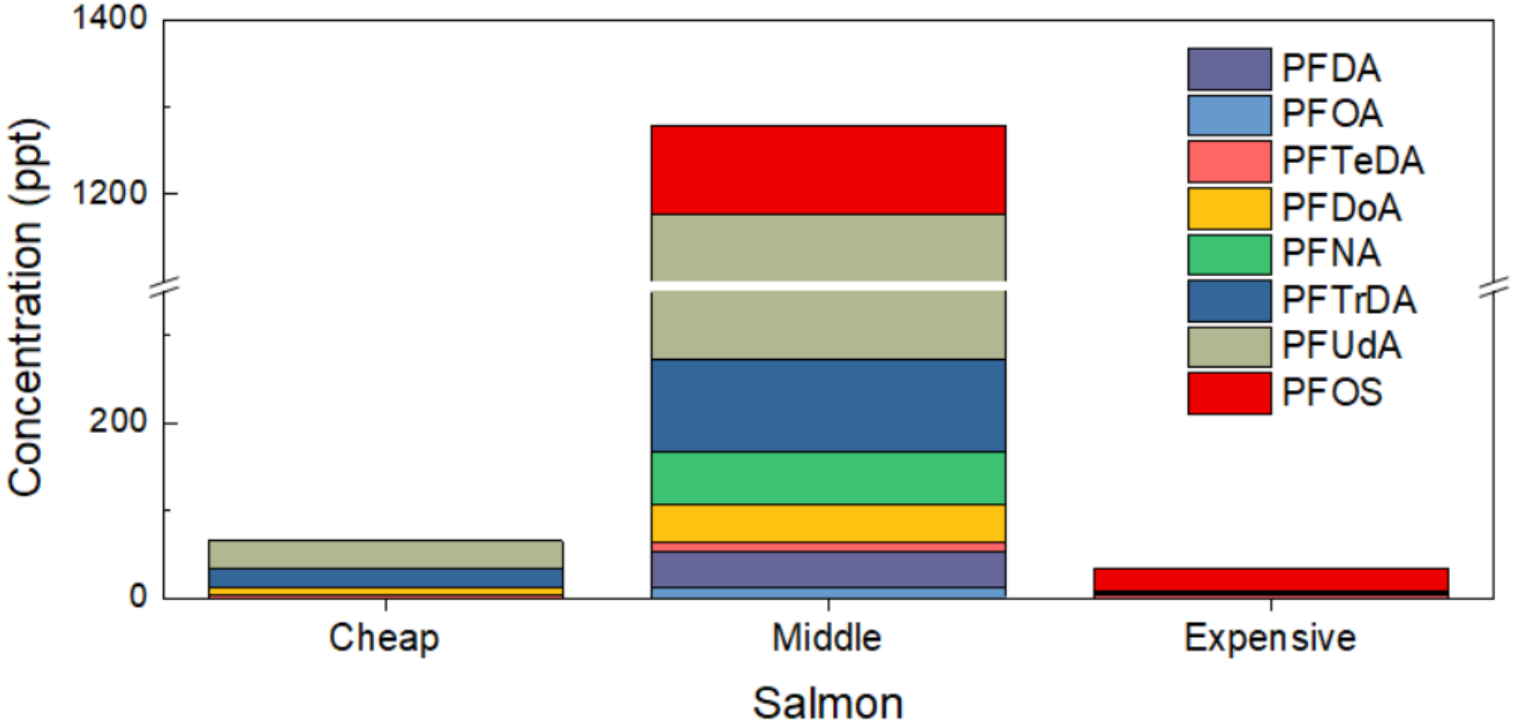
Beef

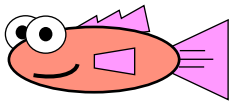


Ice cream



Salmon

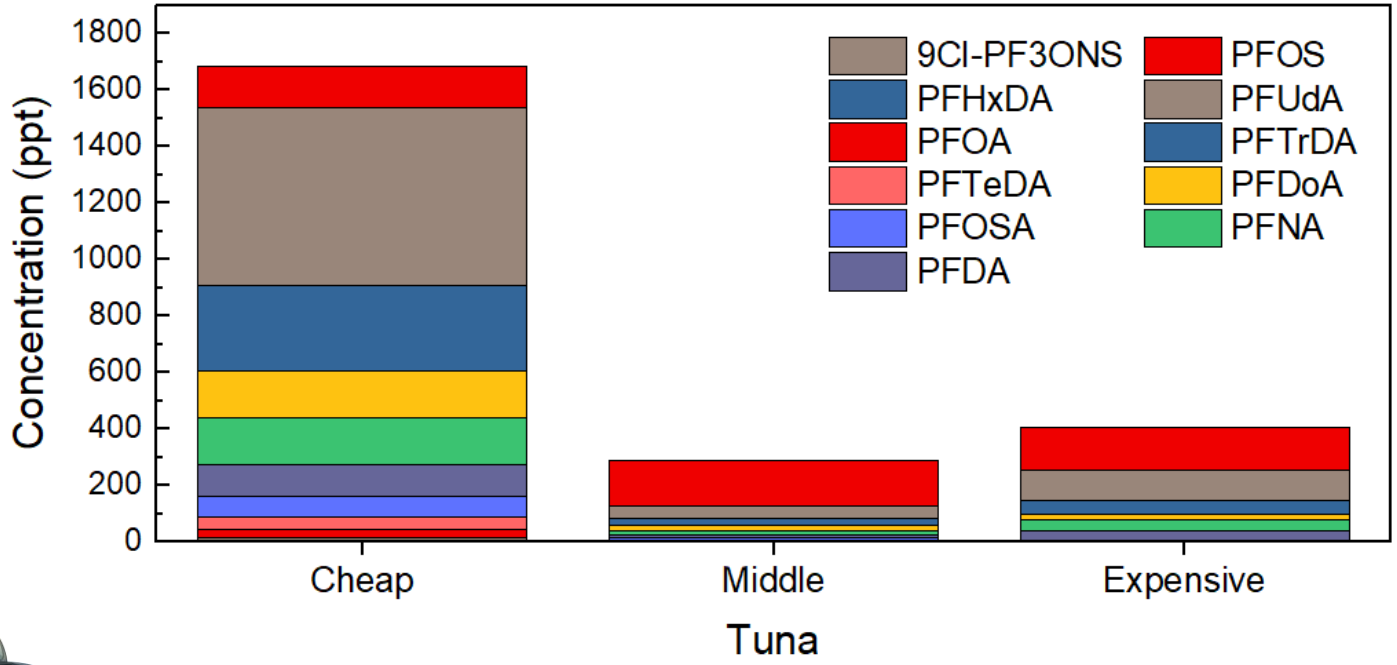




Least expensive

Most expensive

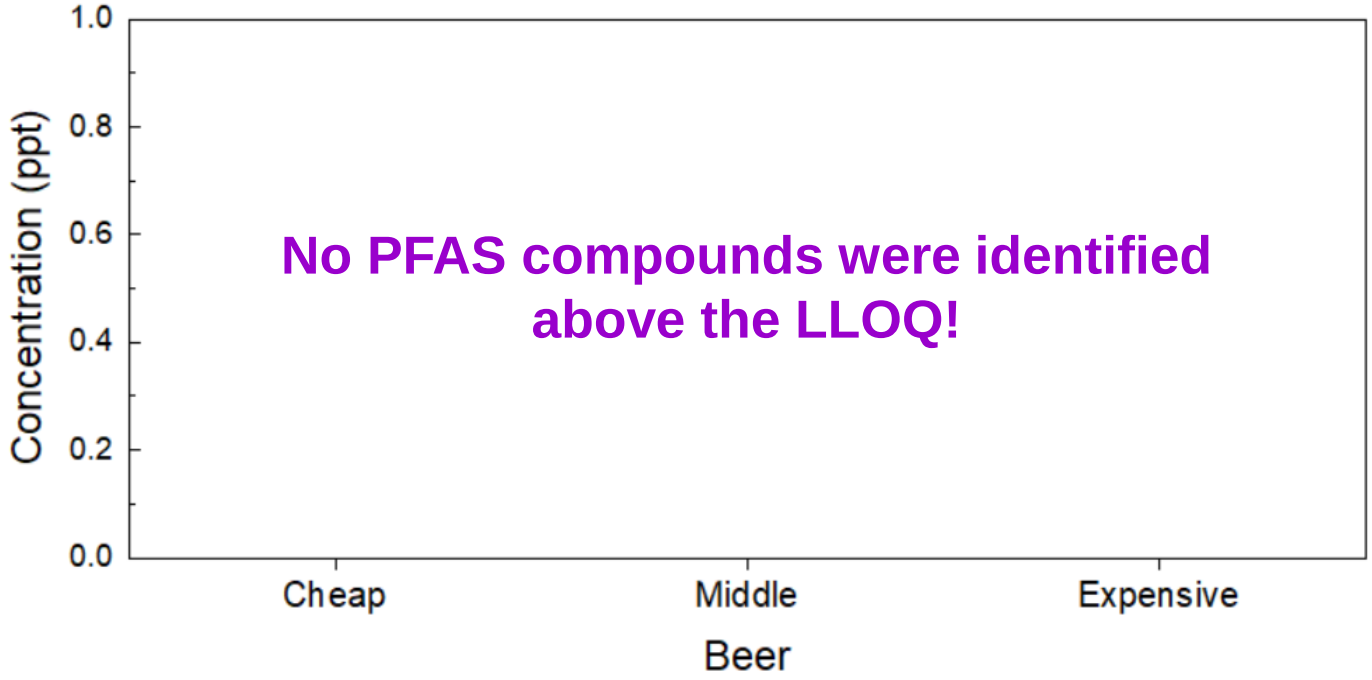
Tuna



Least expensive

Most expensive

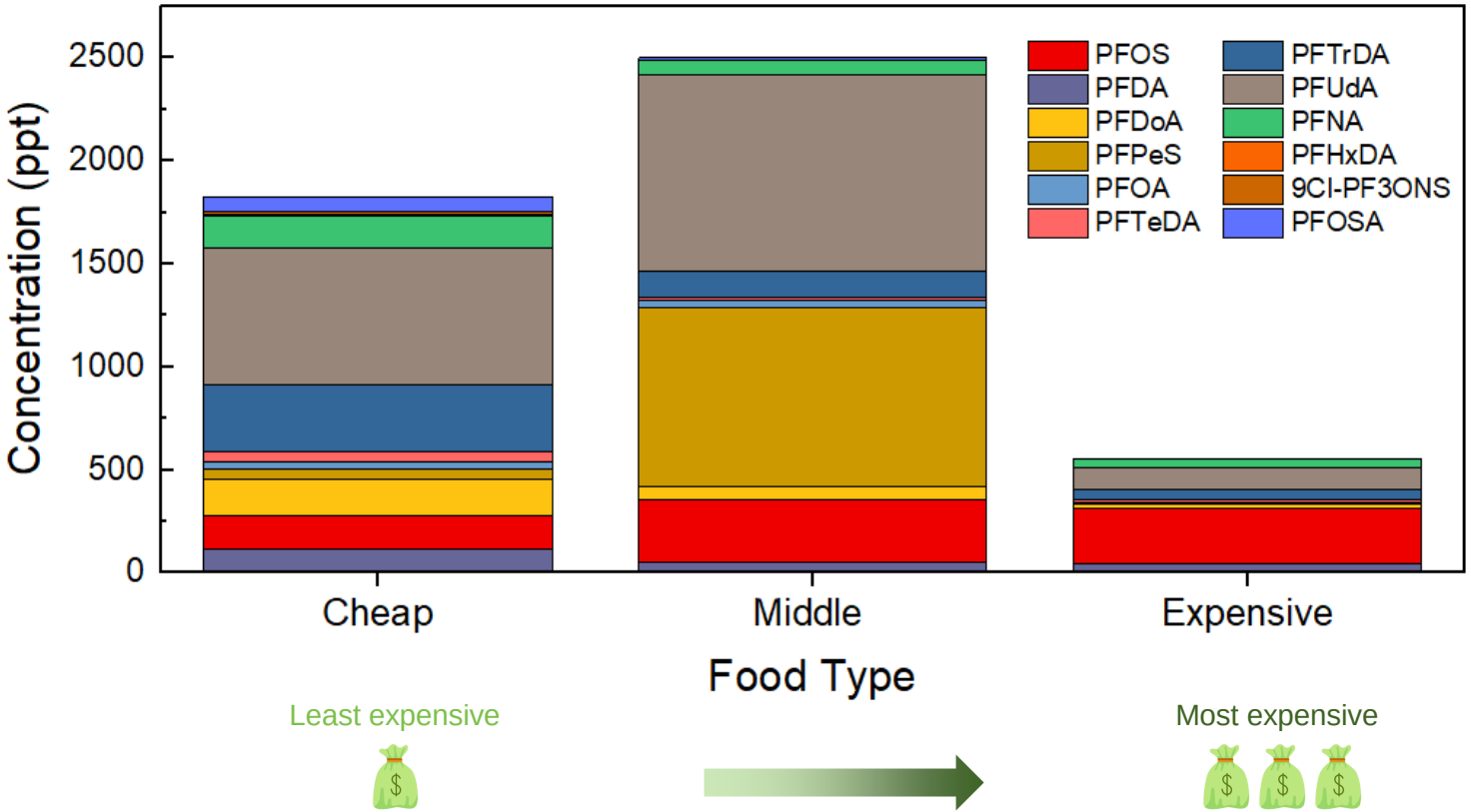
Beer



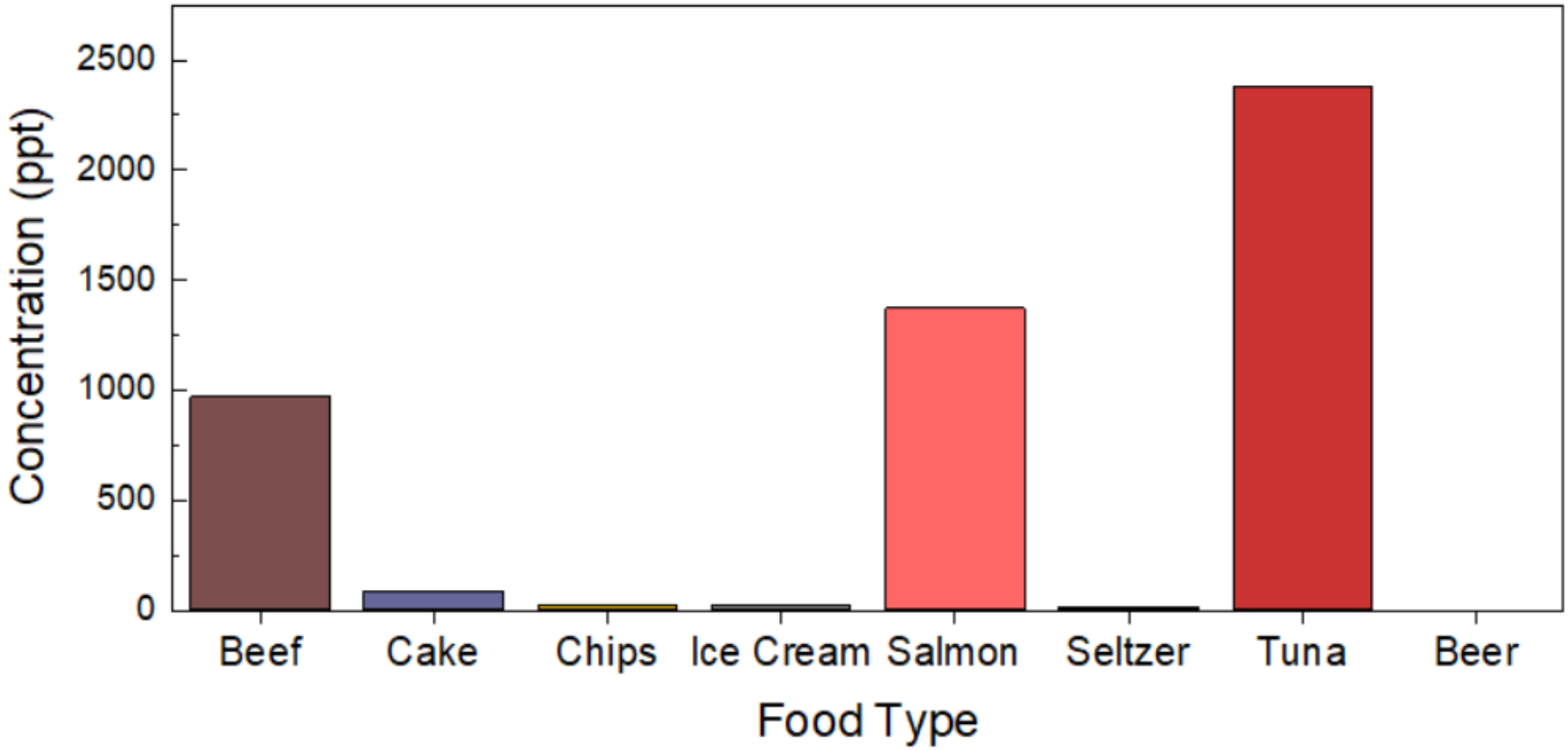
Least expensive

Most expensive

Summary by cost



Summary by food type

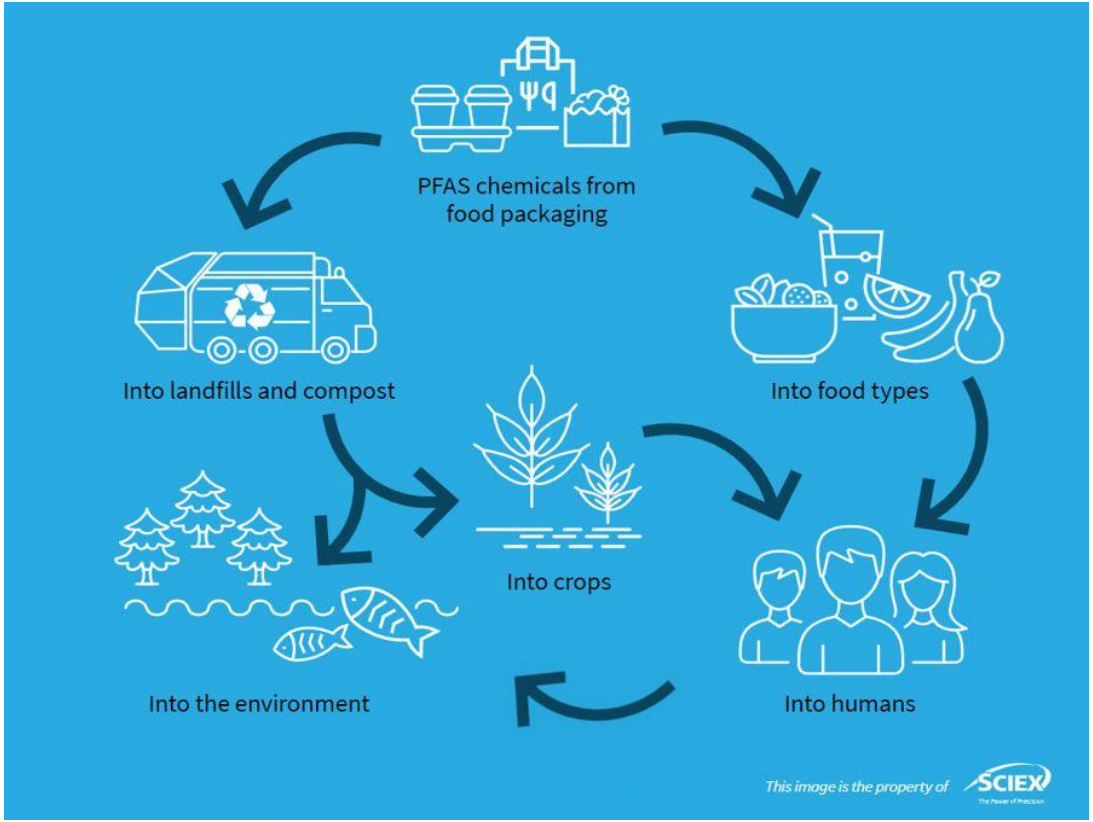


Conclusions

What compounds are we going to focus on?

Are targeted methods enough?

What does PFAS in food mean for human health?



Compounds included in this study

Component name			
PFDS	PFNA	PFPeS	10:2 FTS
PFPrS	PFOSA	6:2 FTUCA	11Cl-PF3OUdS
PFPeA	PFOS	PFHpA	PFTrDA
PFECA A	PFDA	PFO4DA	PFDoS
HFPO-DA	8:2 FTS	DONA	PFTeDA
PFECA B	9Cl-PF3ONS	PFECA_G	PFHxDA
5:3 FTCA	PFNS	PFHxS	PFODA
PFBS	10:2 FTUCA	PFOA	PFDoA
PFO3OA	PFUdA	6:2 FTS	PFHpS
PFHxA	N-MeFOSAA	7:3 FTCA	8:2 FTUCA
PES	N-EtFOSAA	PFECHS	4:2 FTS

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The Power of Precision

Questions and answers

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