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Optimized Method for Analyzing PFAS in Drinking Water to Assess the 2024 Health Canada Objective

Tammy Chartrand, National PFAS Program Lead

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240 Life Sciences Locations

65+
Countries

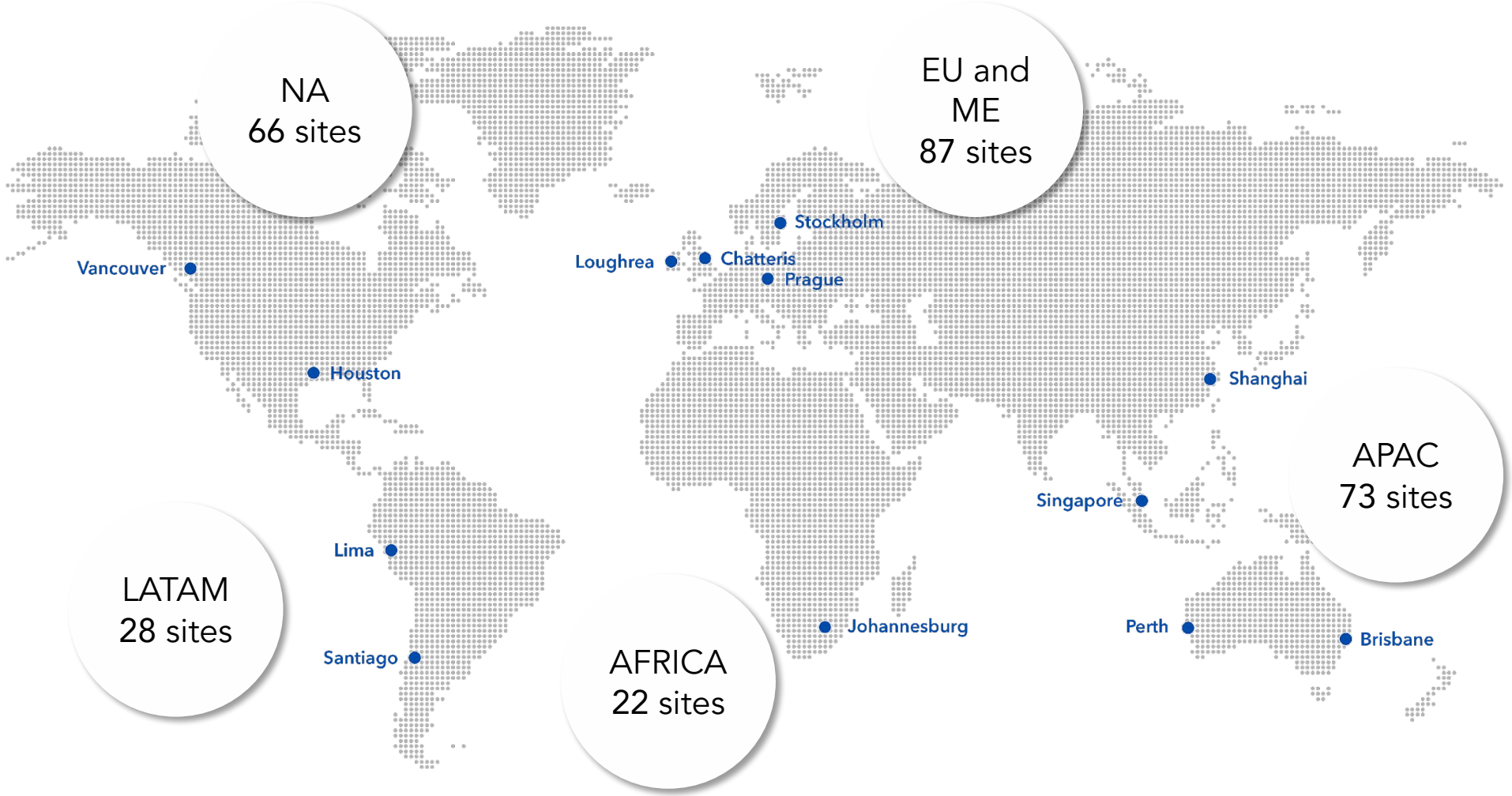
350+
Offices

40+
Years

17,000+
Staff Worldwide

40M+/yr.
Samples Processed

AU$1.8B
Global Revenue



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Our Canadian Presence

10
Laboratories
*including Halifax - 2022

20+
Support Locations

20+
Years

175K+/yr.
Samples Processed @ WT Hub

Complimentary pick-ups within service areas

24/7 secured drop-off and pick-up access

Dedicated, responsive Client Service team

AVERAGE EXPERIENCE AT THE ALS ENVIRONMENTAL LABORATORIES IN CANADA

Managers:	Quality & Technical Services:	Customer Service:	Laboratory:
16.7 years	16 years	9 years	7 years

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ALS Global PFAS Network

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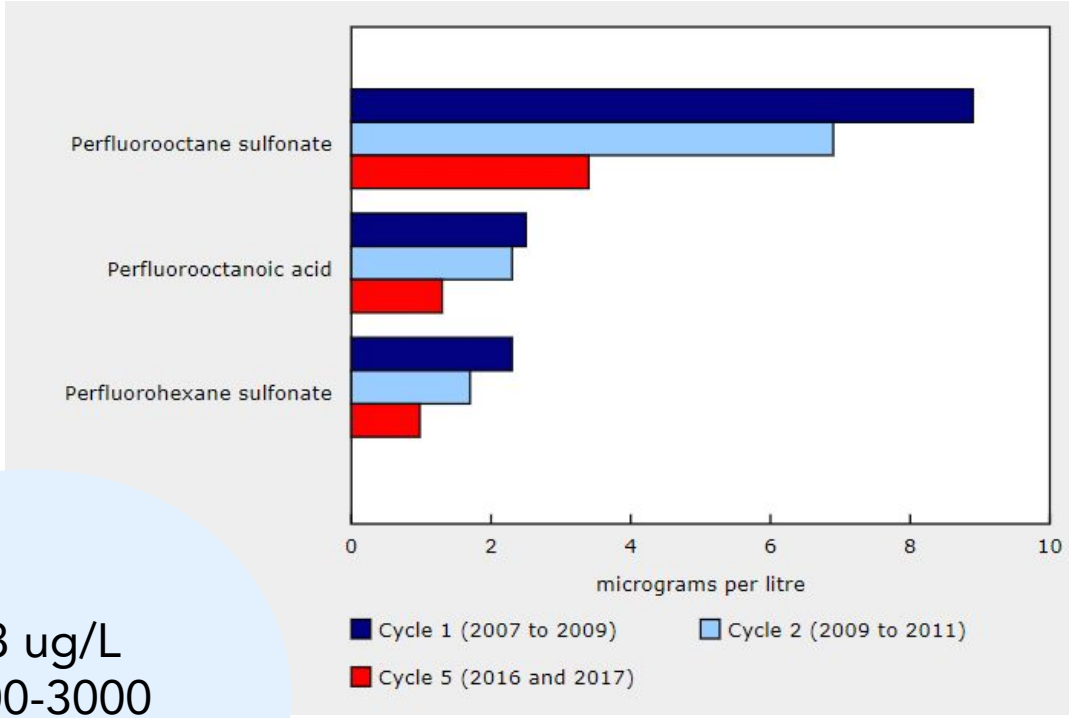
Outline

- Regulatory Framework
- General principles of analysis
- Drinking water method development
- Sample handling and collection
- Other matrices to consider

~98% of Canadians have PFAS in their blood!



1-3 ug/L (1000-3000 ng/L) (2016/17)

The Daily — Canadian Health Measures Survey: Environmental laboratory data, 2016 and 2017 (statcan.gc.ca)

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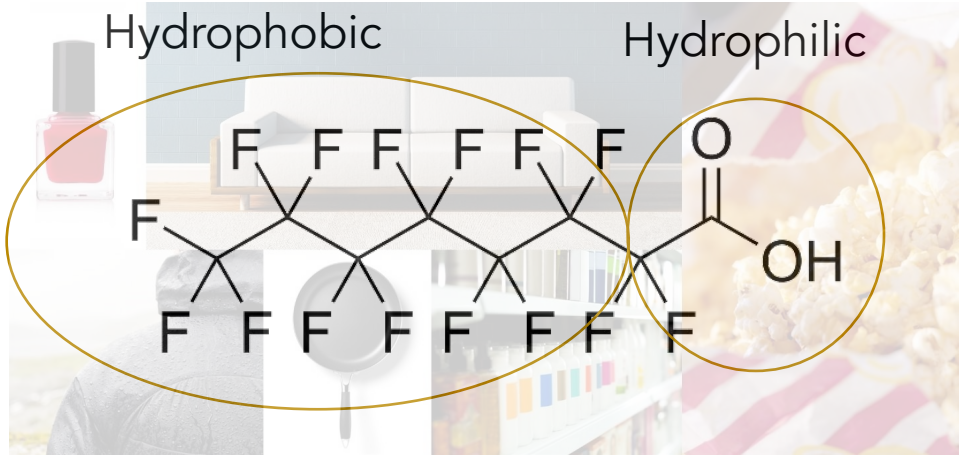
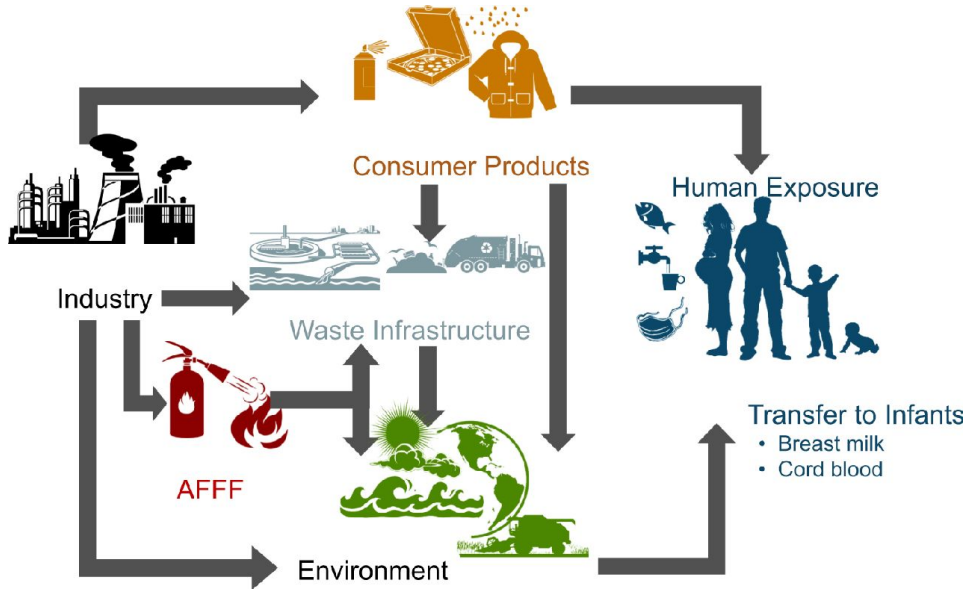
PFAS 101

- Class of synthetic compounds
- C-F backbone = good stability under heat and chemical stress
- Dual hydrophobic, hydrophilic structure = Oil, grease, water and stain repellent
- Estimates now > 15,000 different chemicals



- Common Applications
  - Fire fighting foams (AFF)
  - Cookware
  - Clothing, paper, leather, textiles
  - Cosmetics
  - Food Packaging
  - Cleaners and paints
  - Wire insulation
  - Surfactants and lubricants



Source: Sunderland et al. 2019 A review of the pathways of human exposure to poly- and perfluoroalkyl substances (PFASs) and present understanding of health effects

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Regulatory Framework for PFAS in Drinking Water

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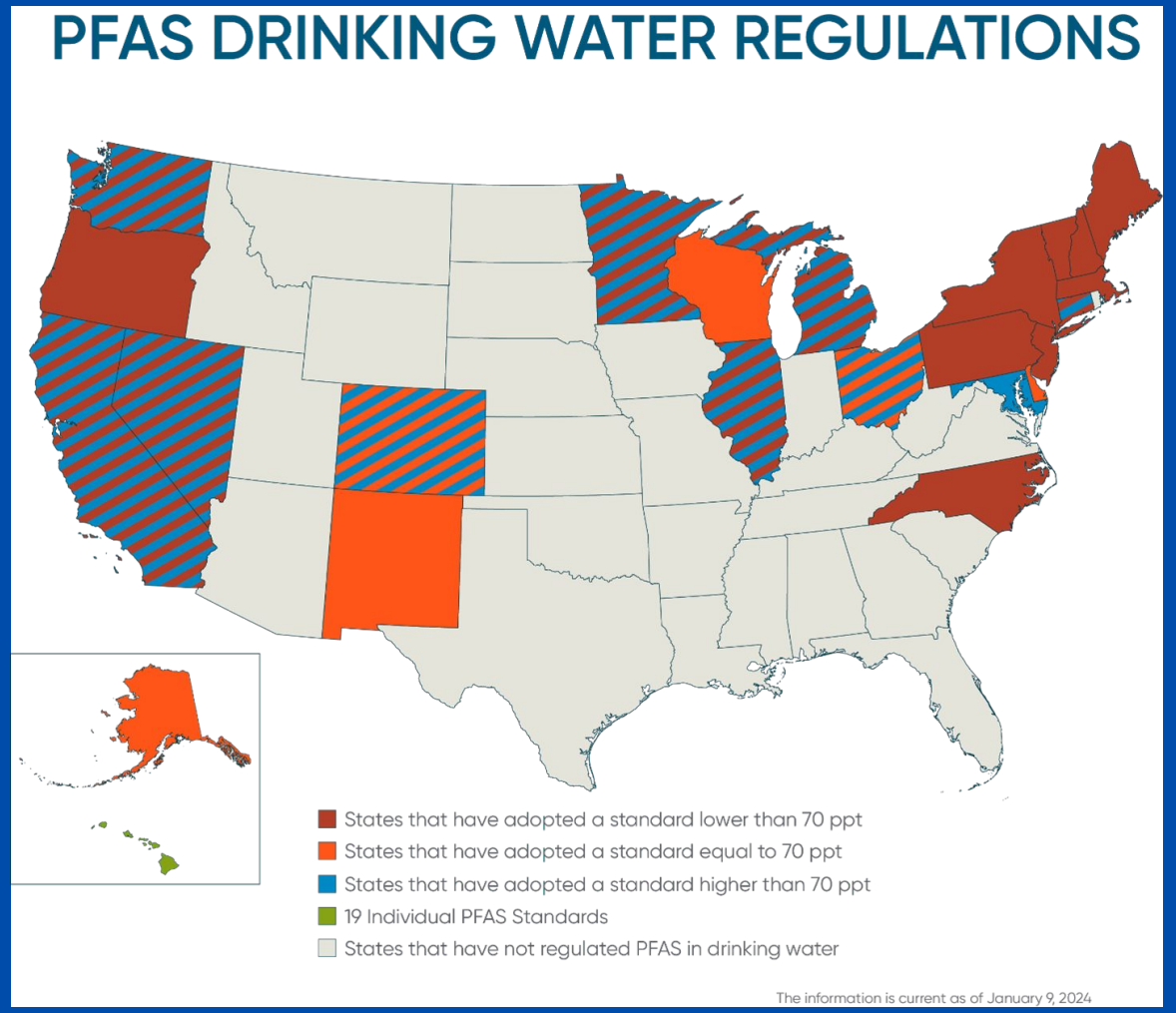
Drinking water Criteria and Guidelines

	Drinking Water	Non-Potable Water	Soil / Solids
Health Canada	Drinking Water Objective 30 ng/L (sum of 25)		Screening values for 11 PFAS *update coming soon*
CCME		PFOS (GW) (PFOA under development)	PFOS (PFOA under development)
ECCC		PFOS (SW)	
CFIA			PFOS 50 ppb (Biosolids - interim)
Nova Scotia	9 PFAS (potable GW)	9 PFAS (GW) 1 PFAS (SW)	9 PFAS
Alberta	PFOS and PFOA	PFOS and PFOA (GW/SW)	PFOS
British Columbia and Yukon (2027)	PFOS, PFOA, PFBS	PFOS	PFOS and PFBS
Ontario	Interim guidance value – 70 ng/L (sum of 11)		
Quebec		PFOS and PFOA (SW)	PFOS, PFOA, sum of 11 additional PFAS

US National Primary Drinking Water Regulation (NPDWR)

Compound	Maximum Contaminant Level Goal (MCLG)	**Maximum Contaminant Level (MCL)** Enforceable
PFOS	0	**4 ng/L**
PFOA	0	**4 ng/L**
PFHxS	~~10 ng/L~~	~~10 ng/L*~~
PFNA	~~10 ng/L~~	~~10 ng/L*~~
HFPO-DA (GenX)	~~10 ng/L~~	~~10 ng/L*~~
Mixtures containing 2 or more : PFNA, PFBS, GenX, PFHxS	~~Hazard index of 1~~	~~Hazard index of 1*~~

US EPA has rolled back on all but PFOS and PFOA



30 States with various types of levels for 25 PFAS

Credit: BCLPlaw.com

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Other Regions

Region	# Compounds	Limit
UK	48	100 ng/L (sum)
EU	20	100 ng/L (sum)
Denmark	22	100 ng/L (sum)
Italy	24	100 ng/L (sum)
Australia	4	**PFOS - 8 ng/L** PFOA – 200 ng/L PFHxS – 30 ng/L PFBS – 1000 ng/L

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General Principles of Analysis

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PFAS : Trace - Level Analysis

- Usually looking at contaminants in ug/L, for PFAS we are down to ng/L
- With such low levels, more susceptible to background interference and contamination
- Guidelines and regulations are often limited by the sensitivity on the available methods
- US Maximum Contaminant Level Goal for drinking water = 0 for PFOS and PFOA
  - MCL Limit = 4 ng/L

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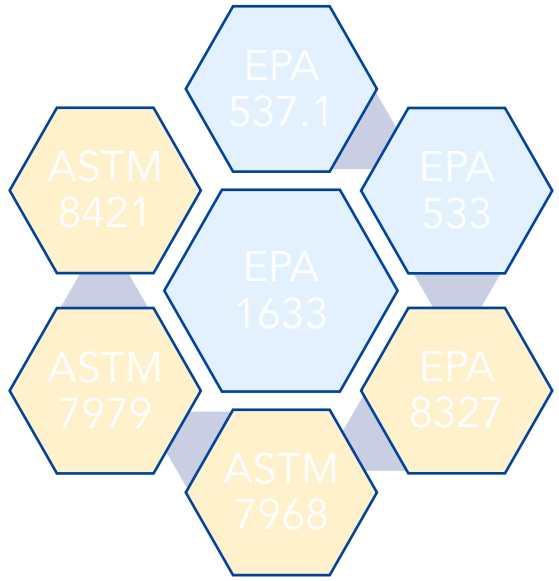
Key Components of PFAS Analytical Methods

LC/MS-MS	• Selectivity and sensitivity
Isotope Dilution	• Accuracy and precision
Solid Phase Extraction	• For Trace-level Analysis



- Not all methods incorporate isotope dilution or SPE
- Co-solvation methods do not rely on SPE for non-trace level objectives

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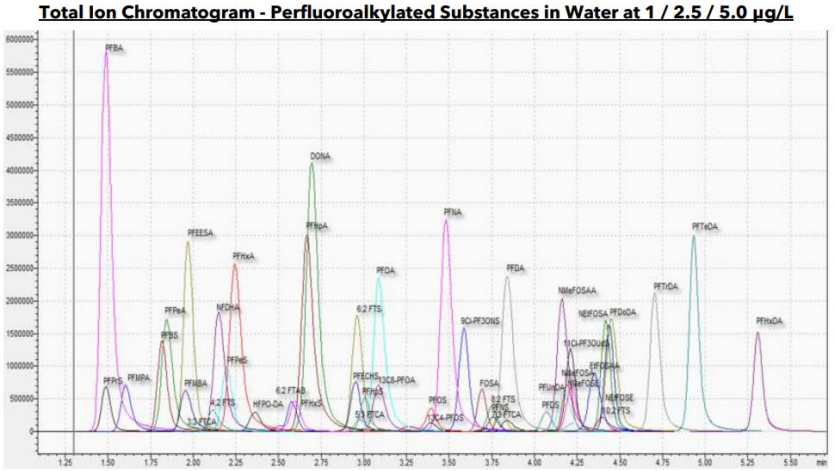
Principles of analysis: LC-MS/MS

- Selective and sensitive
- Fast and reliable
- Versatile
- Low reporting limits
  - ng/L
  - µg/kg

LC-MS/MS – Liquid chromatography coupled with tandem mass spectrometry



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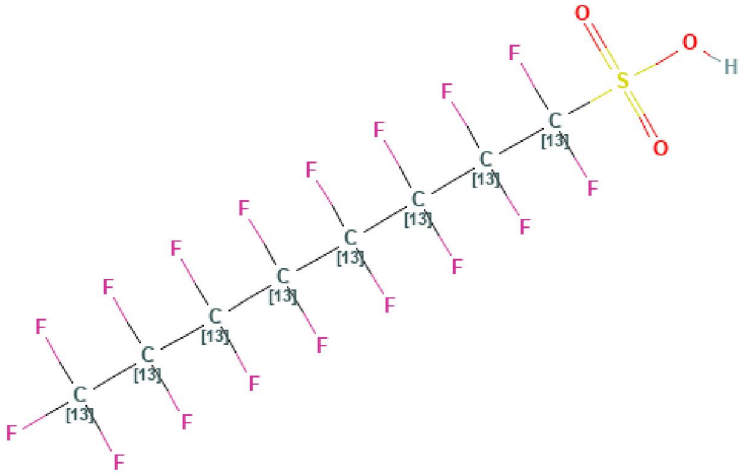
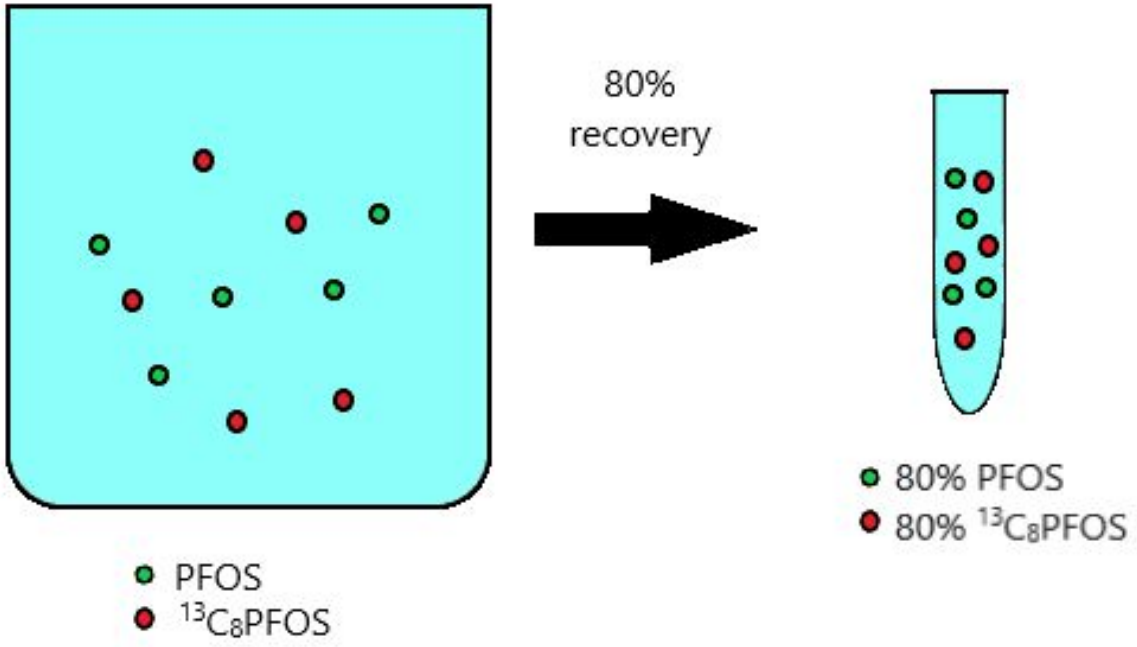
Principles of Analysis: Isotope Dilution

- Isotopically labeled standards
- Same physical and chemical properties as native compounds, but with different mass
- Used to track and correct for losses and matrix interference
- Great accuracy and precision





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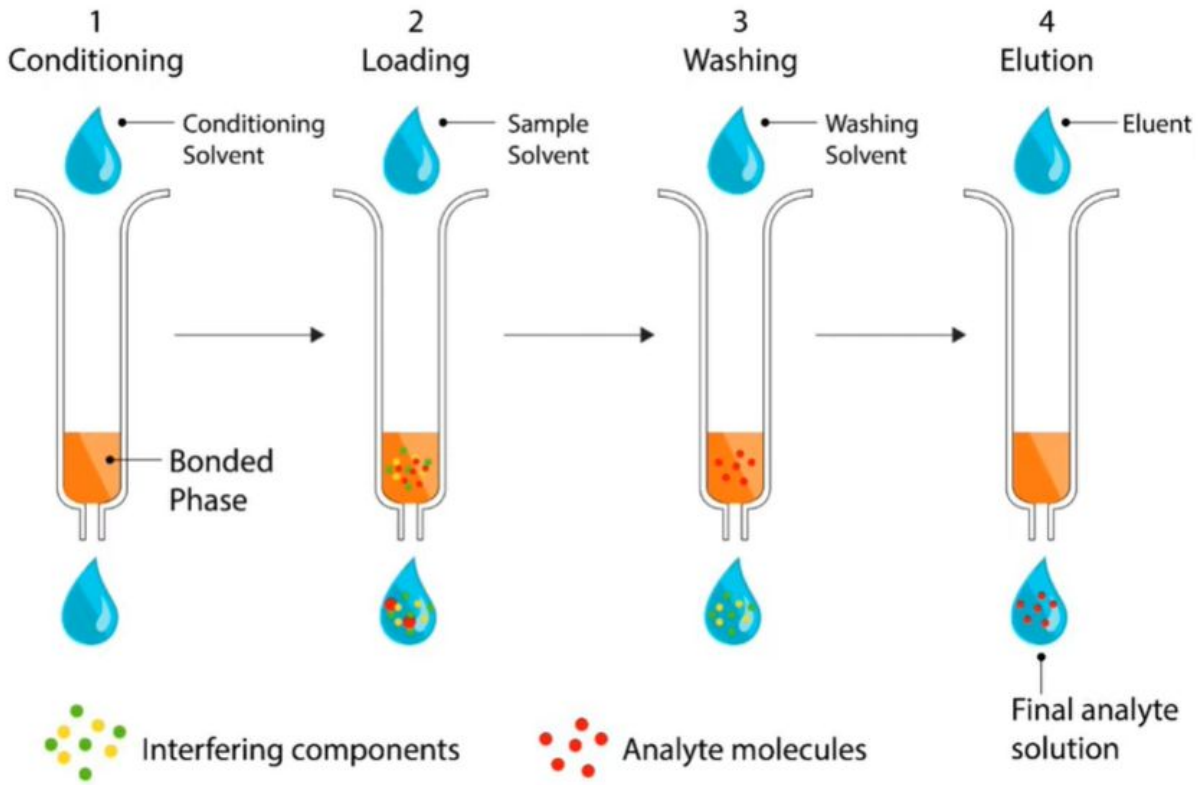
Sample preparation : Solid Phase Extraction (SPE)

- Sample preparation done prior to analysis using an SPE cartridge
- Remove interfering components
- Cleanup and concentrate sample
- Complex and difficult matrices (including soil)
- Needed to achieve low level RLs (2 ng/L) for drinking water



Can take up to 7.5 hrs!

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Sample flow

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ALS Drinking Water Method

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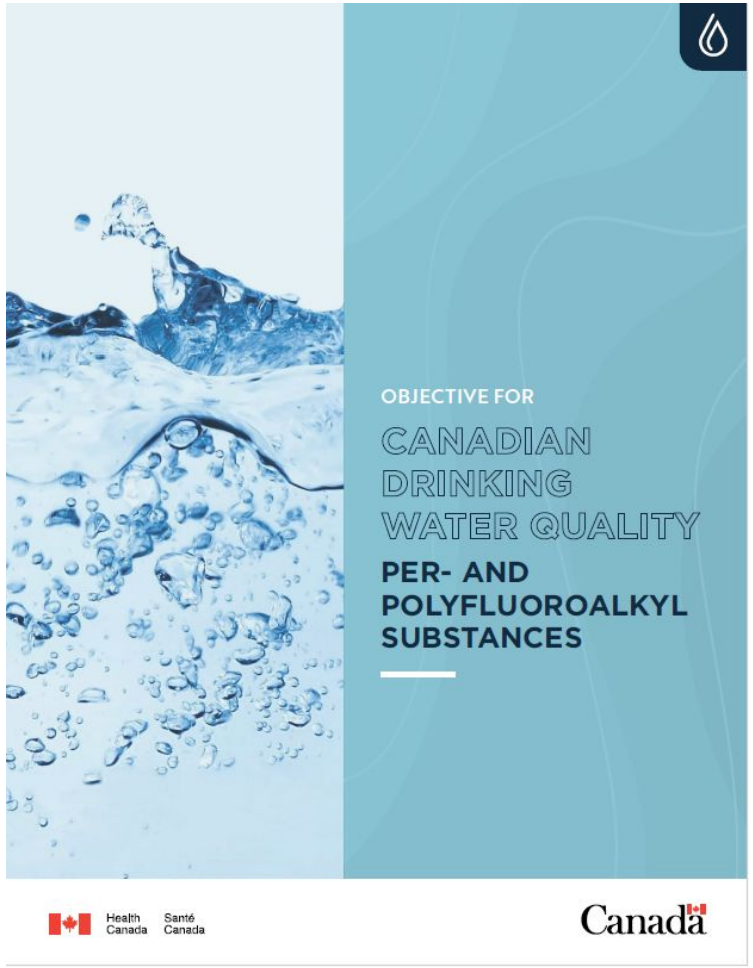
Health Canada Drinking Water Objective (DWO)

- 30 ng/L for the sum of 25 PFAS (EPA 533)
- Not enforceable limit
- EPA 533 is recommended over 537.1 because it incorporates isotope dilution, and provides good coverage of compounds, noting other methods could be approved for use by jurisdiction.
- Recommends utilities analyze for additional compounds where possible to better understand what PFAS may be present which could impact treatment and exposure
- Samples should be collected minimum once a year
- Formal drinking water guidelines are currently being developed



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Drinking Water Methods

	EPA 537.1	EPA 533	EPA 1633	MECP E3457
Drinking water method?	Yes	Yes	No	Yes
Number of analytes	18	25	40	16
Isotope dilution	No	Yes	Yes	yes
SPE	Yes	Yes	Yes	yes
Preservative	Trizma	Ammonium Acetate	None (not a DW method)	Sodium Thiosulfate

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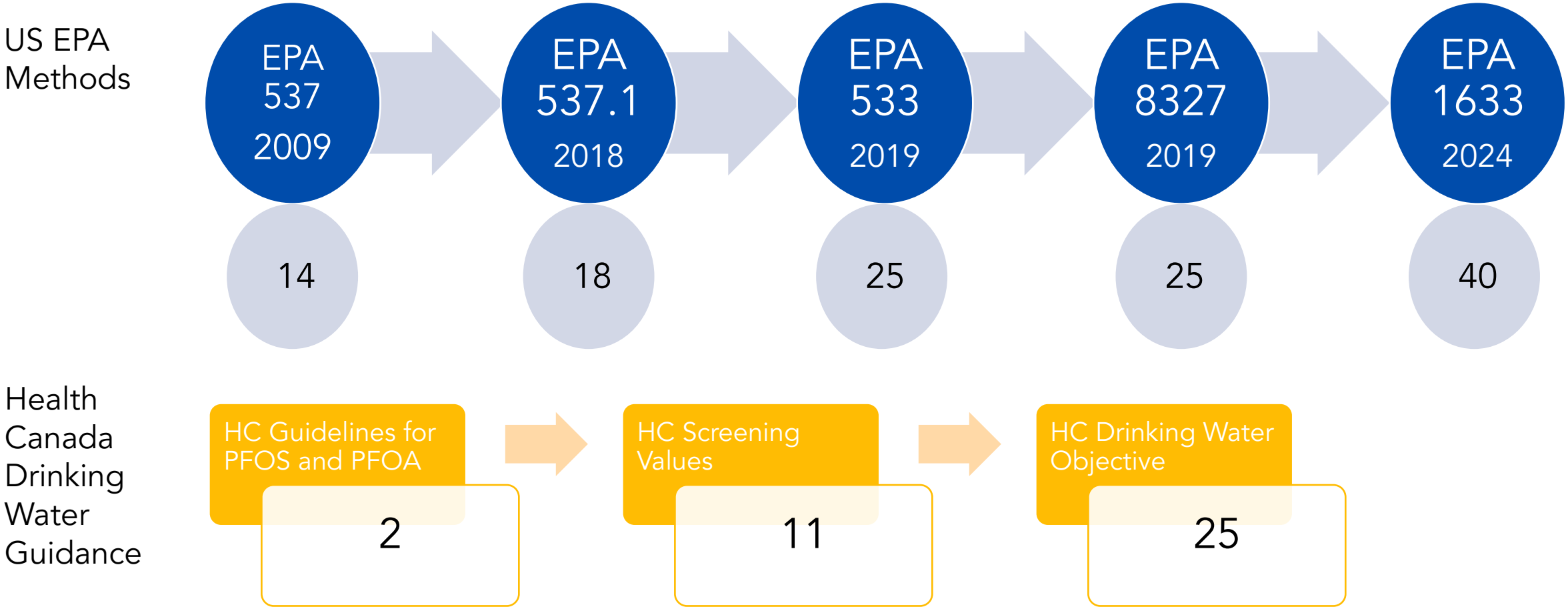
Number of Compounds



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Compound Selection Considerations

Compound	EPA 1633	Other Regions	Other Rationale
PFTrDA	Y	EU, DK, IT, UK	
PFTeDA	Y	UK	
PFNS	Y	EU, DK, IT, UK	
PFDS	Y	Ontario, EU, DK, IT, UK	
PFDoS	Y	UK	
PFOSA	Y	Ontario, DK	
NMeFOSA	Y	UK	
NEtFOSA	Y	UK	
NMeFOSAA	Y	UK	
NEtFOSAA	Y	UK	
NMeFOSE	Y	UK	
NEtFOSE	Y	UK	
3:3 FTCA	Y	UK	degradation product
5:3 FTCA	Y	UK	degradation product
7:3 FTCA	Y	UK	degradation product

Compound	EPA 1633	Other Regions	Other Rationale
PFECHS		UK	
PFHxDA		UK	
PFODA		UK	
PFTrDS		EU, DK, IT	
PFUdS		EU, DK, IT, UK	

20 additional compounds beyond HC DWO
45 TOTAL

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Preservative Selection

Preservative	Method	Hold Time	# Analytes
Trizma	EPA 537.1	14 days	18
Ammonium Acetate	EPA 533	28 days	25
Sodium Thiosulfate	MECP E3457	56 days	16

- Stability study of dechlorinating agents Trizma vs Sodium Thiosulfate
  - Support sodium thiosulfate PFAS stability for 14 days (most conservative)
  - Samples should be extracted within 7 days if NMeFOSE, NEtFOSE, NMeFOSAA or NEtFOSAA are analytes of concern (EPA 1633A)

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ALS PFAS Methods

Matrix	Solids		Drinking Water	Water	
Sale Item	**Routine level**	**Low Level**	**Low Level**	**Routine level**	**Low Level**
Reference Method	ASTM 8535 (mod)	EPA1633	EPA 533 / 1633 (mod)	ASTM 8421 (mod)	EPA 1633
Preparation	Co-solvation with methanol	Solvent Extraction, Solid phase extraction and carbon cleanup Clean up	Solid phase Extraction	Co-solvation with methanol	Solid phase extraction and Carbon Cleanup
LOR Range		0.0001-0.0005 mg/kg (soil) 0.001- 0.01 mg/kg (biosolids)	0.4 - 4 ng/L	20-100 ng/L	0.4-5 ng/L
Bottle Requirement (MEOL #)	120 mL HDPE	120 mL HDPE	3x 250 mL HDPE	2 x 5 mL Vials	2x 250 mL HDPE 1x 60 mL HDPE
Preservative	None	None	Sodium Thiosulfate	None	None
TAT (business days)	Regular - 5 Rush- 3 (100% surcharge)	Regular - 15 Rush - 10 (100% surcharge) *please contact lab in advance*	Regular - 10 Rush- 5 (100% surcharge)	Regular - 5 Rush- 3 (100% surcharge)	Regular - 15 Rush - 10 (100% surcharge) *please contact lab in advance*
Hold time		28 Days	14 days	28 Days	28 Days

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Why a separate Method?

Method based and modified from EPA 533 (drinking water method) and EPA1633 (non-DW water method)

- Method type: Drinking water, and non-drinking water
- Includes all elements of 1633 with exception of Carbon cleanup
- QA/QC: segregation and quality control
- Some 533 QC requirements are more stringent

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Sample Collection and Handling

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Consulting with the Lab

- What type of sample is it? Are samples finished potable water or raw source water?
- Are there criteria or regulations that apply?
- List of target PFAS?

Engage with your lab early!

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Sample collection and handling

- Avoid materials where PFAS can adsorb to surface (glass)
- Avoid any potential sources of contamination including all sampling material
- Use of QC samples (blanks)
- Good standard field protocols and practices with close consideration for anything coming into direct contact with the sample
- Take a representative sample

- For drinking water, open cold water tap and allow system to flush for 3 mins prior to collection – filling to shoulder
- Once filled, invert 5 times to dissolve preservative

Avoid	Acceptable alternative
Teflon™ pump or tubing	HDPE or Silicone tubing
Decon 90	Alconox®, Liquinox®, Citrinox®
LDPE or glass sample containers	HDPE or polypropylene containers ** ensure no Teflon™ liner
Chemical Blue Ice packs	Free ice
Waterproof field book	Metal clipboard / loose paper
Markers	Ball point pen or pencil
Water resistant or treated gloves / clothing	Powderless nitrile gloves / cotton clothing
Cosmetics, creams, sunscreen and related products	--
pre-packaged food, aluminum foil, fast food wrappers or containers	--
Plastic bags / packaging – screen before use	Polyethylene bags (Ziplock®)

Other resources: ITRC, Michigan Sate sampling guidance, Transport Canada Sampling guidance

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Field Blanks / QC samples

Trip Blanks. Analyte-free water prepared in a sealed sample container at the laboratory, transported from lab to sampling site and back to lab without exposure to sampling procedures.

Field Blanks. Analyte-free water poured into a sample container in the field, preserved and shipped with field samples.

Equipment Blanks. Analyte-free water poured over or passed through sampling equipment

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Quality Control In the Lab

- Extensive sample processing and laboratory cleaning procedures to ensure no background PFAS contamination
- Sample preparation equipment dedicated to PFAS
- Laboratory supplies and equipment selection to avoid potential PFAS contamination
- Extensive QA/QC protocols
- Preparation and analysis of Laboratory Blanks

QC Parameter
Initial Calibration Curve
Instrument Sensitivity Check
Interference Check Standard
Calibration Verification Standard (CVS)
Continuing Calibration Verification (CCV)
Extracted Internal Standard (EIS)
Non-extracted Internal Standard (NIS)
Ion Abundance Ratio
Method Blank (MB)
Laboratory Control Sample (LCS)
Laboratory Control Sample (LLLCS), Low-Level
Matrix Spike (MS)
Sample Duplicate

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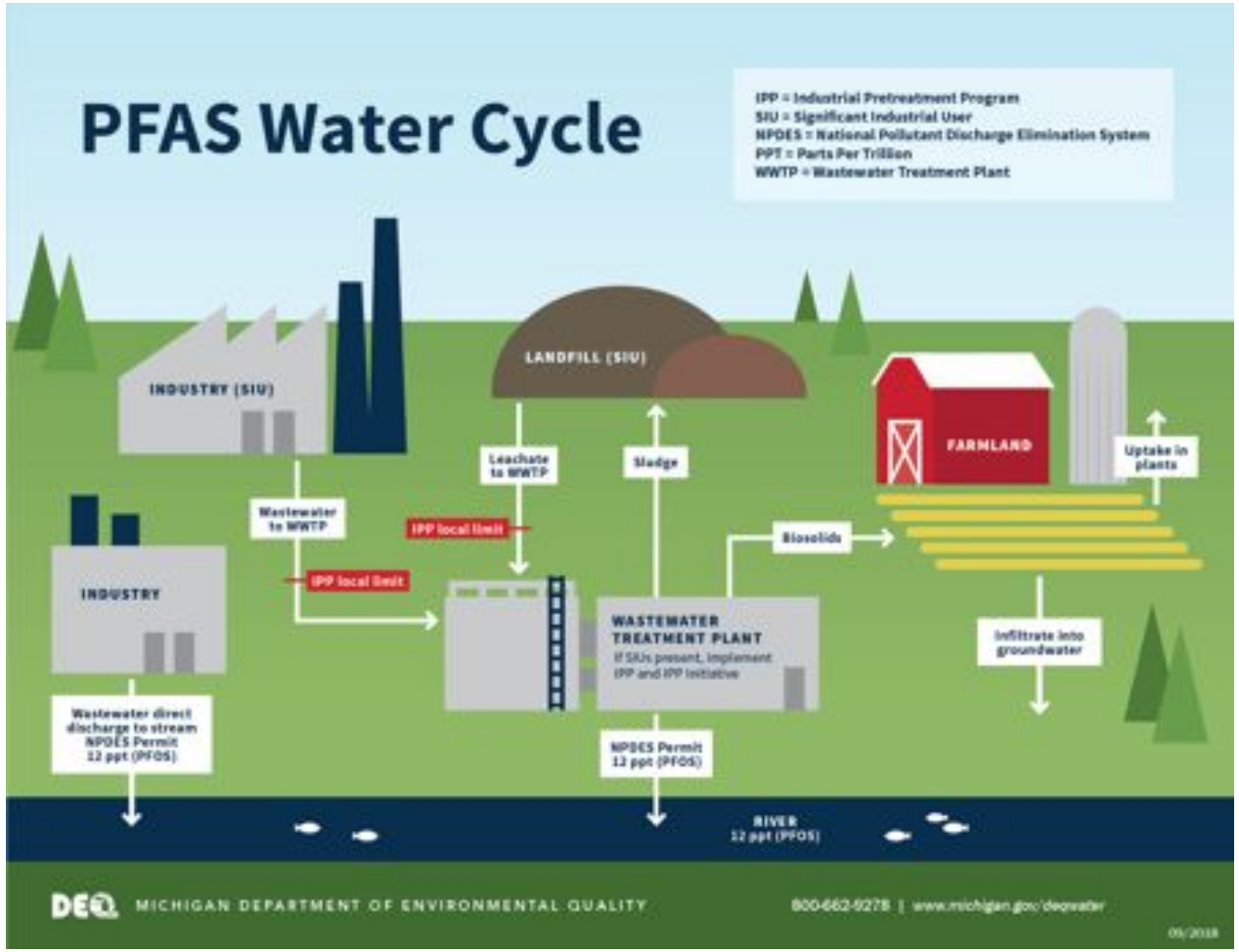
What else?

- Wastewater treatment plants
  - Generally, PFAS is not being removed/treated
  - Effluent and biosolids (secondary source)
- Landfills
  - Disposal of various consumer products
  - Leachate
- Industrial / Manufacturing Effluent
  - PFAS used in process, or released as by product



Source: Michigan Department of Environmental Quality

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Questions?

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Environmental Canada