



# PFAS, PFOS, PFOA, PTFE, and GenX Data:

---

REQUIREMENTS AND PITFALLS FOR  
SAMPLE COLLECTION AND YOUR LIMS



***Edward F. Askew PhD***

## Askew Scientific Consulting



### Provides Guidance and Direction to Municipal Clients for:

- Compliance with EPA and IDNR regulatory pretreatment requirements.
- Laboratory accreditation and certification
- Onsite training and remote training
- Updating city codes and ordinances

### Provides Industrial Clients

- Approval of new methods by EPA
- Approval of Alternate Test Procedures (ATP) by EPA
- Approval of instruments by EPA

The quality someone has  
when they are able to  
make sensible decisions  
about what to do.

---

**(GOOD SENSE)**

# Sample Collection and Your LIMS

---

# EPA Method Being Reviewed

---

<b>EPA Method</b>	<b>Matrix</b>	<b>EPA Method</b>	<b>Matrix</b>
<b>Method 533</b>	<b>Drinking Water</b>	<b>Draft 1633, Revision 3</b>	<b>Wastewater Water, Industrial Pretreatment, BioSolids ???, Landfill Leachate</b>
<b>Method 537 Ver 1.1</b>	<b>Drinking Water</b>		
<b>Method 537.1 Ver 2</b>	<b>Drinking Water</b>	<b>8327, Rev 0 2021</b>	<b>SW 846 40 CFR § 503 ???</b>

EPA Method	Matrix	EPA Method	Matrix
Method 533	<p>This method targets "short chain" PFAS (none greater than C12), including perfluorinated acids, sulfonates, fluorotelomers, and poly/perfluorinated ether carboxylic acids. <u>Method 533 measures a total of 25 PFAS.</u></p>	Draft 1633, Revision 3	<p><u>Draft, single-laboratory validated</u>, direct injection EPA method for <u>40 PFAS in wastewater, surface water, groundwater, soil, biosolids, sediment, landfill leachate, and fish tissue.</u></p>
Method 537 Ver 1.1	<p>EPA method for the determination <u>of 14 PFAS in drinking water.</u></p>		
Method 537.1 Ver 2	<p>EPA method for the determination <u>of 18 PFAS in drinking water,</u> including HFPO-DA (one component of the GenX processing aid technology).</p>	8327, Rev 0 2021	<p>Direct injection method for non-drinking water aqueous (groundwater, surface water, and wastewater) samples. <u>Validated for 24 analytes.</u></p>

# Lets Look at Your Chain of Custody and Sample Collection

---

No Matter How Good  
The Laboratory Is, A  
Bad Sample Negates All  
Of Your Work



# Sampler & Sample Collection Requirements

---

DEVIL IN THE DETAIL

# How Low Are We Going to Analyze For??

---

**EXAMPLES (OH BOY !!!!!)**

# Drinking Water & Wastewater

---

	Gallons	Pounds	Kilograms	Grams
Wastewater	5,000,000	41,700,000	18,914,802	18,914,801,829

3 PPT PFAS in Grams	3 PPT PFAS in Milligrams
0.057	57

# Biosolids

---

Sludge	Metric Tons	Percent Solids	Dry Solids (Kilograms)	Dry Solids (Grams)
	100	20.00%	20,000	20,000,000

3 PPT PFAS in Grams	3 PPT PFAS in Milligrams
0.00006	0.06



# QAPP: Let's Collect a Sample

---

[HTTPS://PFAS-1.ITRCWEB.ORG/11-SAMPLING-AND-ANALYTICAL-METHODS/#11\\_1](https://PFAS-1.ITRCWEB.ORG/11-SAMPLING-AND-ANALYTICAL-METHODS/#11_1)

# Field Clothing

---

- Powderless nitrile gloves.
- Polyvinyl chloride (PVC) or wax-coated fabrics.
- Neoprene.
- Any boots made of polyurethane and/or PVC. If a specific type of boot such as (steel-toed) and PFAS-free cannot be purchased, PFAS- free over-boots may be worn. The overboots must be put on, and hands washed after putting the overboots on before the beginning of sampling activities.
- Overboots may only be removed in the staging area and after the sampling activities have been completed.
- Synthetic and natural fibers (preferably cotton) that are well laundered (more than six times with no fabric softener) clothes and cotton overalls

# Sun Screen and Insect Repellant

---

### ■ Allowable Sunscreens

- Banana Boat for Men Triple Defense Continuous Spray Sunscreen SPF 30
- Banana Boat Sport Performance Coolzone Broad Spectrum SPF 30
- Banana Boat Sport Performance Sunscreen Lotion Broad Spectrum SPF 30
- Banana Boat Sport Performance Sunscreen Stick SPF 50
- Coppertone Sunscreen Lotion Ultra Guard Broad Spectrum SPF 50
- Coppertone Sport High-Performance AccuSpray Sunscreen SPF 30
- Coppertone Sunscreen Stick Kids SPF 55
- L'Oréal Silky Sheer Face Lotion 50+
- Meijer Clear Zinc Sunscreen Lotion Broad Spectrum SPF 15, 30 and 50
- Meijer Wet Skin Kids Sunscreen Continuous Spray Broad Spectrum SPF 70
- Neutrogena Beach Defense Water + Sun Barrier Lotion SPF 70
- Neutrogena Beach Defense Water + Sun Barrier Spray Broad Spectrum SPF 30
- Neutrogena Pure & Free Baby Sunscreen Broad Spectrum SPF 60+

### ▲ Materials That Require Screening

**Sunscreens:** Alba Organics Natural Sunscreen, Yes To Cucumbers, Aubrey Organics, Jason Natural Sun Block, Kiss My Face, and baby sunscreens that are "free" or "natural."

**Insect Repellents:** Jason Natural Quit Bugging Me, Repel Lemon Eucalyptus Insect repellent, Herbal Armor, California Baby Natural Bug Spray, Baby Ganics.

**Sunscreen and Insect Repellent:** Avon Skin So Soft Bug Guard Plus – SPF 30 Lotion.



# Personnel Hygiene and Personal Care Products (PCPs)

---

***BOY, DO YOU STINK !!!!***

A number of sampling guidance documents recommend that personal hygiene and personal care products (PCPs) (e.g., cosmetics, shampoo, sunscreens, dental floss, etc.) not be used prior to and on the day(s) of sampling because the presence of PFAS in these products has been documented.

- Do not handle or apply PCPs in the sampling area.
- Do not handle or apply PCPs while wearing PPE that will be present during sampling.
- Move to the staging area and remove PPE if applying personal care products becomes necessary.
- Wash hands thoroughly after the handling or application of PCPs and, when finished, put on a fresh pair of powderless nitrile gloves.

# Snack and Lunch Time

---

**OH BOY, I'M ON A DIET !!!!**

**Do not handle, consume, or otherwise interact with pre-wrapped food or snacks, carryout food, fast food, or other food items while on-site during sampling !!!!!**

# Sample Bottles

---

**PFAS FREE**

EPA Method	Sample Bottle	EPA Method	Sample Bottle
Method 533	<u>Polypropylene bottles fitted with polypropylene screw-caps, or polyethylene bottles with polypropylene screw caps.</u>	Draft 1633, Revision 3	<b>HDPE</b>
Method 537 Ver 1.1	<u>Polypropylene bottle fitted with a polypropylene screw-cap.</u>		
Method 537.1 Ver 2	<u>Polypropylene bottle fitted with a polypropylene screw-cap.</u>	8327, Rev 0 2021	<u>Polypropylene containers.</u> Other types of container materials, such as high-density polyethylene (HDPE), may be used if performance is acceptable for the project.

# PFAS FREE ????

EPA Method	PFAS Free Requirement	EPA Method	PFAS Free Requirement
Method 533	Extract an LRB prepared with reagent water using the same lot of sample bottles destined for shipment to the sampling site and ensure that analyte concentrations are less than one-third the MRL,	Draft 1633, Revision 3	All containers must be demonstrated to be PFAS-free at the laboratory's MDLs for the target analytes by testing one or more representative containers from each lot.
Method 537 Ver 1.1	All items such as these must be routinely demonstrated to be free from interferences (less than 1/3 the MRL for each method analyte) under the conditions of the analysis by analyzing laboratory reagent blanks		
Method 537.1 Ver 2		8327, Rev 0 2021	Blanks are generally considered to be acceptable if target analyte concentrations are less than one half the LLOQ or are less than project-specific requirements.

# Preservative & Preservation

---



EPA Method	Preservative	EPA Method	Preservative
Method 533	<p><b>Based on sample volume, add <u>ammonium acetate</u> to each sample bottle as a solid (prior to shipment to the field or immediately prior to sample collection) to achieve a 1g/L concentration of ammonium acetate. Ammonium acetate will sequester free chlorine to form chloramine.</b></p>	Draft 1633, Revision 3	None !!!
Method 537 Ver 1.1	<p><b>TRIZMA<sup>®</sup> PRESET CRYSTALS, pH 7.0 (Sigma cat# T-7193 or equivalent) – Reagent grade. A premixed blend of Tris [Tris(hydroxymethyl)aminomethane] and Tris HCL [Tris(hydroxymethyl)aminomethane hydrochloride].</b></p>		
Method 537.1 Ver 2	<p><b>TRIZMA<sup>®</sup> PRESET CRYSTALS, pH 7.0 (Sigma cat# T-7193 or equivalent) – Reagent grade. A premixed blend of Tris [Tris(hydroxymethyl)aminomethane] and Tris HCL [Tris(hydroxymethyl)aminomethane hydrochloride].</b></p>	8327, Rev 0 2021	None!!!

EPA Method	Preservation Temperature	EPA Method	Preservation Temperature
Method 533	<p><u>stored at or below 6 °C until extraction. <b>Samples must not be frozen</b></u></p>	Draft 1633, Revision 3	<p><u>Once received by the laboratory, the samples may be stored at 0 - 6 °C <b>or at ≤ -20 °C</b>, until sample preparation.</u></p>
Method 537 Ver 1.1	<p><u>Samples stored in the lab must be held at or below 6 °C until extraction, <b>but should not be frozen.</b></u></p>		
Method 537 Ver 2	<p><u>Samples stored in the lab must be held at or below 6 °C until extraction <b>but must not be frozen.</b></u></p>	8327, Rev 0 2021	<p><u>In the laboratory, samples and <b>sample extracts should be stored in the refrigerator at ≤6 °C while not being analyzed.</b></u></p>

# HOLDING TIME

---



EPA Method	Holding Time	EPA Method	Holding Time
Method 533	28 Days <i>(Extract 28 Days at Room Temperature)</i>	Draft 1633 Revision 3	<b><u>NEXT SLIDE</u></b>
Method 537 Ver 1.1	14 Days <i>(Extract 28 Days at Room Temperature)</i>		
Method 537.1 Ver 2	14 Days <i>(Extract 28 Days at Room Temperature)</i>	8327 Rev 0 2021	<p><b><u>A 14-day limit</u></b> from sample collection to preparation and a <b><u>30-day limit from preparation to analysis</u></b> may be used as a guide until a more formal study is completed.</p>

EPA Method	Holding Time
<p><b>Draft 1633 Revision 3</b></p>	<ul style="list-style-type: none"> <li>• Aqueous samples (including leachates) should be analyzed as soon as possible; however, samples may be held in the laboratory for up to <b><u>28 days</u></b> from collection, when stored at 0 - 6 °C and protected from the light,</li> <li>• When stored at <math>\leq -20</math> °C and protected from the light, aqueous samples may be held for up to <b><u>90 days</u></b>.</li> <li>• Soil and sediment samples may be held for up to <b><u>90 days</u></b>, if stored by the laboratory in the dark at either 0 - 6 °C or <math>\leq -20</math> °C,</li> <li>• Tissue samples may be held for up to <b><u>90 days</u></b>, if stored by the laboratory in the dark at <math>\leq -20</math> °C,</li> <li>• Biosolids samples may be held for up to <b><u>90 days</u></b>, if stored by the laboratory in the dark at 0 - 6 °C, but preferably at <math>\leq -20</math> °C</li> <li>• <b><i>Store sample extracts in the dark at less than 0 - 6 °C until analyzed. If stored in the dark at <math>\leq 0</math> °C, sample extracts may be stored for up to 90 days,</i></b></li> </ul>

# 1633 Question ?

---

- If a water sample is collected in the Field at Day 1 and then is received in the Lab on Day 2:
- If the sample is kept in the refrigerator, how many days does the laboratory have left to analyze the sample?
  - 26 days to Extract, 90 days to analyze extract
- If the sample is frozen, how many days does the laboratory have left to analyze the sample?
  - 90 days?
  - 90 days-2 days?
  - 90 days-2 days~1 to 2 days to melt sample?

So, What Should You  
Have On The Chain of  
Custody and In The  
Sample Receiving LIMS

---

**GOOD SENSE !!!**

# Chain of Custody & Sample Receiving LIMS Requirements

---

1. Sample Collection Date and Time **Start the Clock & Total Time**
2. Sample QAPP Requirements **Sample Collector**
3. Sample Bottle **PE, HDPE, PP, Lot #**
4. Sample Cooler **Wet Ice or Dry Ice, Lot #**
5. List of PFAS to Analyze **Method Dependent , Caveats Included**
6. Blanks **Trip, Field, Instrument ?**



# Questions

---