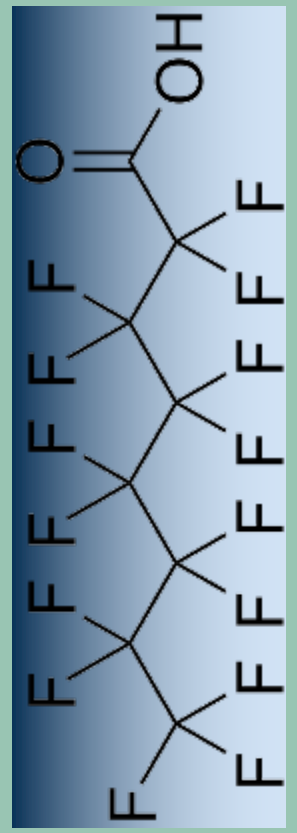
PFAS Monitoring and Assessment: Sampling Considerations and Findings

STAC PFAS Workshop. May 18, 2022



Team

- Amy Williams, Water Program Specialist, PA DEP, Bureau of Clean Water
- Lee Eicholtz, Technician, USGS PAWSC
- Emily Woodward, Chemist, USGS PAWSC
- Jerry Casile, Chemist, USGS WMA
- Ian Lynch, Technician, USGS PAWSC
- Tim Oden, AD, PAWSC
- Stan Skrobialowski, USGS WMA
- Heather Heckathorn, Water Quality Specialist, USGS NJ WSC
- Irene Fisher, Hydrologist, USGS NY WSC
- Andrea Tokranov, Hydroilogist, USGS New England WSC
- So many field scientists in the USGS, SRBC, and at the DEP who helped process blanks, collect samples, prepare paperwork, and coordinate field work.

PFAS Sampling-evaluation Work – USGS PAWSC

- **USGS PAWSC Statewide Surface Water Monitoring (WQN)**
  - 2019/2020 statewide surface-water sampling
    - 50 equipment cleaning blanks on SW equipment
    - 18 field blanks & 18 field replicates
    - 180 environmental samples
    - 16 composite-sample split (splits between 2 labs, with some replication)
    - 18 Polar Organic Chemical Integrative Sampler (POCIS) samples collected by DEP
  - Validating sampling methods across a range of PFAS concentrations currently underway

- **USGS National Water Quality Program Groundwater Sampling**
  - Draft sampling procedures since late 2018
  - Started with public-water supply wells
  - Some specific networks validated monitoring-well procedures
  - Validating across a range of PFAS currently underway

- **USGS Water Mission Area PFAS Sampling Quality Control Study**
  - To inform USGS National Field Manual for the Collection of Water Quality Samples
  - Sampling Completed
    - Data is returning. Spiking was successful, determining max number of methanol rinses that could be needed to properly decontaminate equipment.
  - Questions to answer:
    - Do we lose PFAS to adsorption to our equipment, if so, which substances?
    - When we *know* our equipment is exposed to PFAS, can we clean it?
    - Does it make a difference if the equipment is exposed to lower or higher concentrations?
  - Testing:
    - Both Poly(ethylene and propylene) and Teflon surface water sampling gear
    - 6 different pumps

USGS PAWSC & WMA Development of Procedures for PFAS Sampling for Surface Water and Groundwater

Goal:

Collect a representative sample & robust QC from the WQN network in collaboration with our partners

- No different for PFAS!
- DEP Bureau of Clean Water, SRBC, USGS PAWSC (all field offices), USGS WMA HNB

Approach:

Keep it simple.

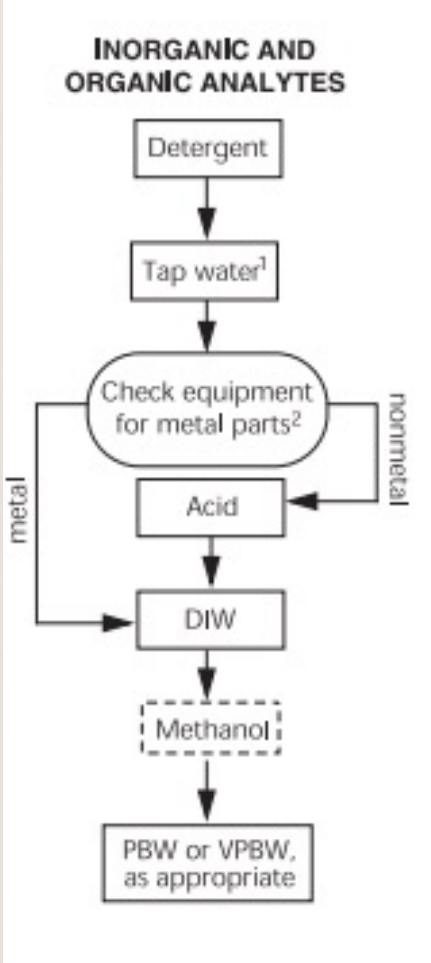
- Use cleaning protocols for trace organics sampling on non-leaching equipment
- Use as many existing protocols from USGS National Field Manual as possible
- Assume concentration of total PFASs is low (<200 ng/L)
- Training session with all sampling agencies
- Provide cleaning supplies, sampling supplies, & blank water

Surface-water Equipment Selection

https://water.usgs.gov/owq/FieldManual/

- HDPE
  - Bottles
  - Nozzle holders
  - Churn splitters
  - Bag samplers
- Delrin
  - Nozzles
- Polypropylene (sorbs)
  - For Methanol rinsing
    - Normally Teflon.
- Certified PFAS free Optima reagent grade blank water

Equipment Cleaning



All equipment used for PFAS sampling will be cleaned with the following process.

Tap water rinse

Liquinox detergent soak (30 min)

Scrub equipment with soft bristle brush

Warm Tap Water Rinse

Acid rinse (this is for our sampling program because we collect metals) (ok for nozzles during this effort)

DI (lab produced) water rinse

Methanol rinse (from a non TFE spray bottle)

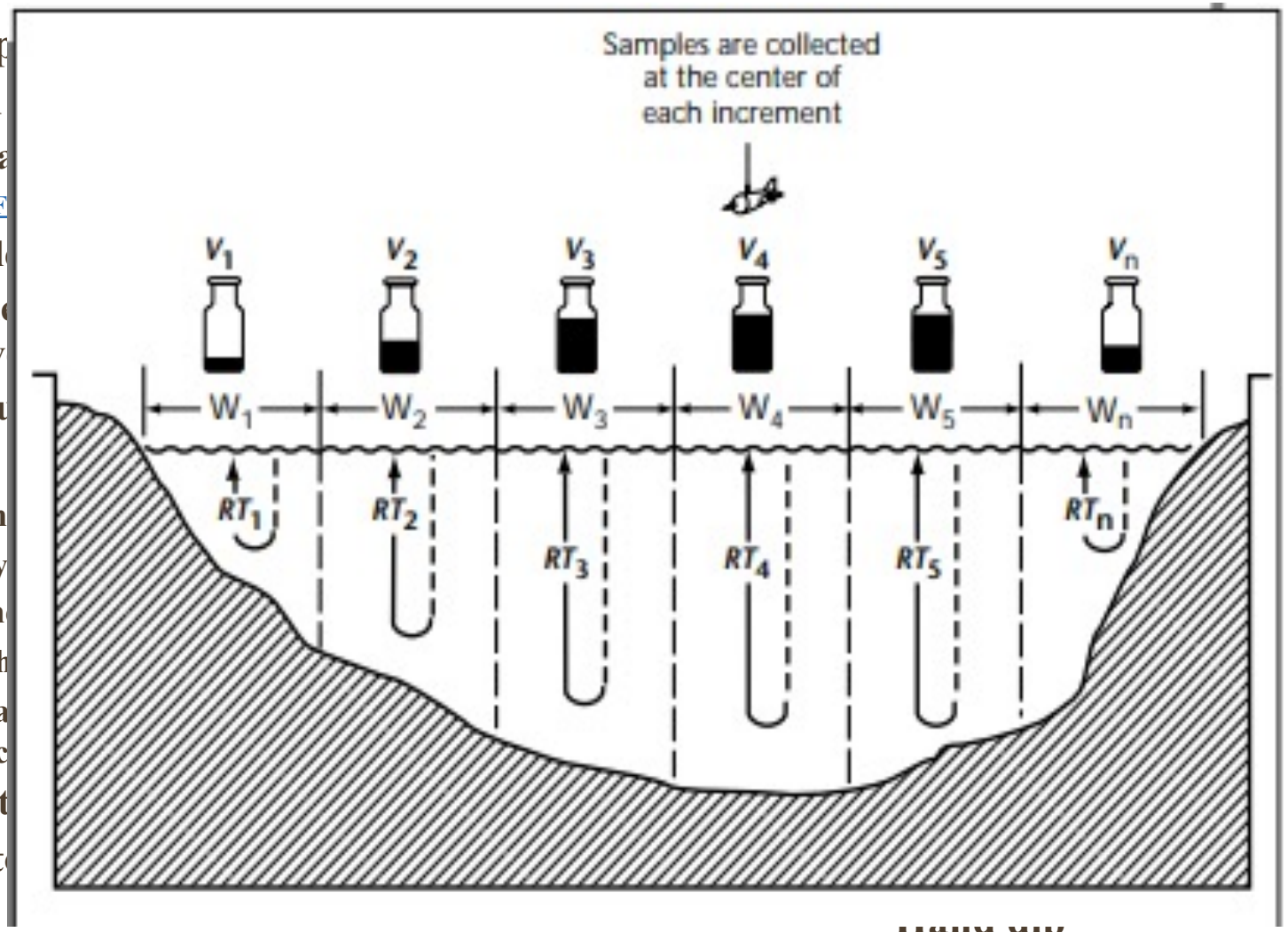
Air dry (or rinse with lab DI if in field)

Stored double bagged in chamber bags

https://water.usgs.gov/owq/FieldManual/

Surface-water Sampling Methods

- Trace (ppt) sampling p… l sampling
  - Composite, integrated …
  - Clean-Hands/Dirty-Ha…
    - https://water.usgs.gov/owq/F…
  - Specially selected & cl… ı, nozzle holder, bags, and Delrin
  - Person opening sample… length poly gloves is only additional elbow length poly glov…
  - USGS, NFM, Techniqu…
  - Normal cautions
    - Only open bottle when…
    - Close bottles promptly…
    - If you drop a bottle an… ıls
    - Avoid any contact with…
    - Avoid spraying or crea… being collected or proc…
  - No special rules for att… er
  - Essential on medium t… estimation



Hand dip

Sample for accurate Concentration
Measure flow for accurate Mass

- Since we're collecting integrated samples and we want to understand sources, fate, and transport
  - Need to convert to mass from concentration
  - Requires accurate streamflow
- 161 of 180 samples have measured or computed streamflow, so all data for statewide assessments and ongoing assessments with the PADEP can be converted into an instantaneous mass.
- Mass can then be divided by drainage area to compute a yield of PFAS from a watershed.
  - Useful in source attribution.

Equipment Blanks: 9 sets of equipment, 7 teams of 2 people each

USGS_equipment_cleaning_blank_sample_description	PFUnDA	PFHxA	N-MeFOSAA	4:2FTS	6:2FTS	8:2FTS	GenX	N-EtFOSAA	FOSA	PFTeDA	PFTrDA	FBSA	FHx-SA	N-MeFOSA-M	N-EtFOSA-M	11Cl-PF3OUds	PFHxS Linear	PFHxS Total	PFOS Linear	PFOA	PFBA	PFPeA	PFHpA	PFNA	PFDA	PFDoDA	PFBS	PFDS	9Cl-PF3ONS	NaDONA	PFHpS	PFOS Total	PFPeS	PFNS
Source Solution Blank, Optima	<25	<5	<10	<5	<5	<10	<5	<10	<10	<100	<50	<25	<25	<100	<250	<10	<5	<5	<10	<2.5	<1	<2.5	<5	<2.5	<5	<25	<1	<10	<5	<1	<10	<10	<1	<10
USGS NCO HDPE DH-81 Bottle Optima Rinse Treatment	<25	<5	<10	<5	<5	<10	<5	<10	<10	<100	<50	<25	<25	<100	<250	<10	<5	<5	<10	<2.5	<1	<2.5	<5	<2.5	<5	<25	<1	11.8	<5	<1	<10	<10	<1	<10
USGS NCO HDPE DH-81 Bottle Optima Rinse Treatment_replicate	<25	<5	<10	<5	<5	<10	<5	<10	<10	<100	<50	<25	<25	<100	<250	<10	<5	<5	<10	<2.5	<1	<2.5	<5	<2.5	<5	<25	<1	<10	<5	<1	<10	<10	<1	<10
USGS NCO Delrin DH-81 Nozzle Optima Rinse Treatment	<25	<5	<10	<5	<5	<10	<5	<10	<10	<100	<50	<25	<25	<100	<250	<10	<5	<5	<10	<2.5	<1	<2.5	<5	<2.5	<5	<25	<1	<10	<5	<1	<10	<10	<1	<10
USGS NCO HDPE DH-81 Nozzle Holder Optima Rinse Treatment	<25	<5	<10	<5	<5	<10	<5	<10	<10	<100	<50	<25	<25	<100	<250	<10	<5	<5	<10	<2.5	<1	<2.5	<5	<2.5	<5	<25	<1	<10	<5	<1	<10	<10	<1	<10
USGS NCO HDPE DH-2 Bag Optima Rinse Treatment	<25	<5	<10	<5	<5	<10	<5	<10	<10	<100	<50	<25	<25	<100	<250	<10	<5	<5	<10	<2.5	<1	<2.5	<5	<2.5	<5	<25	<1	<10	<5	<1	<10	<10	<1	<10
USGS NCO HDPE 14L Churn Optima Rinse Treatment	<25	<5	<10	<5	<5	<10	<5	<10	<10	<100	<50	<25	<25	<100	<250	<10	<5	<5	<10	<2.5	<1	<2.5	<5	<2.5	<5	<25	<1	<10	<5	<1	<10	<10	<1	<10
USGS NCO HDPE DH-81 Bottle	<25	<5	<10	<5	<5	<10	<5	<10	<10	<100	<50	<25	<25	<100	<250	<10	<5	<5	<10	<2.5	<1	<2.5	<5	<2.5	<5	<25	<1	<10	<5	<1	<10	<10	<1	<10
USGS NCO Delrin DH-81 Nozzle	<25	<5	<10	<5	<5	<10	<5	<10	<10	<100	<50	<25	<25	<100	<250	<10	<5	<5	<10	<2.5	<1	<2.5	<5	<2.5	<5	<25	<1	<10	<5	<1	<10	<10	<1	<10
USGS NCO HDPE DH-81 Nozzle Holder	<25	<5	<10	<5	<5	<10	<5	<10	<10	<100	<50	<25	<25	<100	<250	<10	<5	<5	<10	<2.5	<1	<2.5	<5	<2.5	<5	<25	<1	<10	<5	<1	<10	<10	<1	<10
USGS NCO PE DH-2 Bag	<25	<5	<10	<5	<5	<10	<5	<10	<10	<100	<50	<25	<25	<100	<250	<10	<5	<5	<10	<2.5	<1	<2.5	<5	<2.5	<5	<25	<1	<10	<5	<1	<10	<10	<1	<10
USGS NCO HDPE 14L Churn	<25	<5	<10	<5	<5	<10	<5	<10	<10	<100	<50	<25	<25	<100	<250	<10	<5	<5	<10	<2.5	<1	<2.5	<5	<2.5	<5	<25	<1	<10	<5	<1	<10	<10	<1	<10
USGS NCO Teflon DH-81 Bottle	<25	<5	<10	<5	<5	<10	<5	<10	<10	<100	<50	<25	<25	<100	<250	<10	<5	<5	<10	<2.5	<1	<2.5	<5	<2.5	<5	<25	<1	<10	<5	<1	<10	<10	<1	<10
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USGS PFO PE DH-2 Bag	<25	<5	<10	<5	<5	<10	<5	<10	<10	<100	<50	<25	<25	<100	<250	<10	<5	<5	<10	<2.5	<1	<2.5	<5	<2.5	<5	<25	<1	<10	<5	<1	<10	<10	<1	<10
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USGS WFO HDPE DH-81 Bottle	<25	<5	<10	<5	<5	<10	<5	<10	<10	<100	<50	<25	<25	<100	<250	<10	<5	<5	<10	<2.5	<1	<2.5	<5	<2.5	<5	<25	<1	<10	<5	<1	<10	<10	<1	<10
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PADEP PE DH-2 Bag	<25	<5	<10	<5	<5	<10	<5	<10	<10	<100	<50	<25	<25	<100	<250	<10	<5	<5	<10	<2.5	<1	<2.5	<5	<2.5	<5	<25	<1	<10	<5	<1	<10	<10	<1	<10
PADEP PE DH-2 Bag replicate	<25	<5	<10	<5	<5	<10	<5	<10	<10	<100	<50	<25	<25	<100	<250	<10	<5	<5	<10	<2.5	<1	<2.5	<5	<2.5	<5	<25	<1	<10	<5	<1	<10	<10	<1	<10
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Polypropylene Spray DH-81 Bottle	<25	<5	<10	<5	<5	<10	<5	<10	<10	<100	<50	<25	<25	<100	<250	<10	<5	<5	<10	<2.5	<1	<2.5	<5	<2.5	<5	<25	<1	<10	<5	<1	<10	<10	<1	<10
PADEP Kemmerer Sampler	<25	<5	<10	<5	<5	<10	<5	<10	<10	<100	<50	<25	<25	<100	<250	<10	<5	<5	<10	<2.5	<1	<2.5	<5	<2.5	<5	<25	<1	<10	<5	<1	<10	<10	<1	<10
PADEP Van Dorn Sampler	<25	<5	<10	<5	<5	<10	<5	<10	<10	<100	<50	<25	<25	<100	<250	<10	<5	<5	<10	<2.5	<1	<2.5	<5	<2.5	<5	<25	<1	<10	<5	<1	<10	<10	<1	<10
PADEP Passive Sampler Canister	<25	<5	<10	<5	<5	<10	<5	<10	<10	<100	<50	<25	<25	<100	<250	<10	<5	<5	<10	<2.5	<1	<2.5	<5	<2.5	<5	<25	<1	<10	<5	<1	<10	<10	<1	<10
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Laboratory Preparation Blank, 1	<25	<5	<10	<5	<5	<10	<5	<10	<10	<100	<50	<25	<25	<100	<250	<10	<5	<5	<10	<2.5	<1	<2.5	<5	<2.5	<5	<25	<1	<10	<5	<1	<10	<10	<1	<10
Laboratory Preparation Blank, 2	<25	<5	<10	<5	<5	<10	<5	<10	<10	<100	<50	<25	<25	<100	<250	<10	<5	<5	<10	<2.5	<1	<2.5	<5	<2.5	<5	<25	<1	<10	<5	<1	<10	<10	<1	<10
USGS NCO DH-2 Bag Teflon_matrixspike	72.1	91.4	88.9	86.0	93.3	78.4	83.3	74.5	31.3	<100	<50	92.9	96.7	<100	<250	47.8	62.3	78.8	69.2	117.5	93.6	91.6	89.7	80.3	70.8	40.8	78.1	81.0	86.9	92.6	74.9	89.4	71.9	60.8
USGS NCO HDPE DH-81 Bottle_matrixspike	68.4	70.5	58.1	56.6	<5	72.8	51.3	49.2	33.3	<100	<50	86.8	88.0	<100	<250	24.4	48.2	60.1	45.9	55.5	69.0	65.2	63.0	74.4	64.8	<25	47.6	30.3	71.5	71.0	83.5	65.8	69.7	67.7
PADEP Passive Sampler Stake_matrixspike	47.9	75.2	55.2	76.2	60.6	72.6	63.9	45.3	35.1	<100	<50	76.5	87.4	<100	<250	39.5	56.1	69.0	51.5	76.1	72.2	76.2	77.0	70.8	58.1	46.7	58.1	80.4	71.5	61.4	67.9	68.1	63.1	49.5

Only 2 detections in all equipment blanks. 0.11% of all results had a detection in a blank. Compounds detected in equipment blanks were PFDS and PFNS.

Summary of Equipment and Field Blank Data

- **Detections in <u>Blanks:</u>**
  - **Equipment Blanks** (cleaned equipment tested in lab)
    - Analyzed by USGS NWQL as part of initial QC study. Some RLs are higher than those at SGS AXYS.
    - Clean all equipment using standard procedures, touch each type of equipment listed for 30 seconds then collect a full sample.
      - PFDS 11.8 ng/L (LRL 10 ng/L)- 1L HDPE DH95 sample bottle
      - PFNS 1.9 ng/L (LRL 10 ng/L) – 14L HDPE churn splitter
    - Different samples
    - Different cleaning crews
  - **Field Blanks**
    - Touch all sampling equipment with PFAS-free blank water), then collect a full sample.
      - All analyzed at SGS AXYS, with the same methods as the samples, same RLs
        - PFOS- 1.2 ng/L (LRL 0.856)-Downingtown FO
        - 6:2 FTS- 13 ng/L (LRL 5.460)-New Cumberland FO
          14 ng/L (LRL 5.610)-Pittsburgh FO
      - 0.3% detection of PFAS in field blanks
      - Different samples
      - Different field offices
      - Different equipment
      - Different dates
- ***No systematic contamination detected in equipment blanks or field blanks***

Groundwater-sampling Equipment Selection (to date)

- For public supplies and homes with in-place pumps, purging and direct fill is recommended
  - If not possible, use of standard cleaning and disposable HDPE tubing, and 3 volume purge of well
- For monitoring wells (no in-place pump):
  - Fultz, Grundfos Rediflo-2/3, Bennett, other cheaper pumps have been blank tested
  - Use HDPE tubing alone for PFAS or split the flow at the pump
    - Run HDPE and Teflon™ tubing side by side from the pump to the surface

Public Supply

Fultz

Grundfos

Bennett

Data summary, conclusions from groundwater sampling and quality control

- Using this approach for two years nationally
  - No detects in blanks
  - Reasonable replicate and split data
  - Good matrix spike recoveries

Confirmation of approach and publication

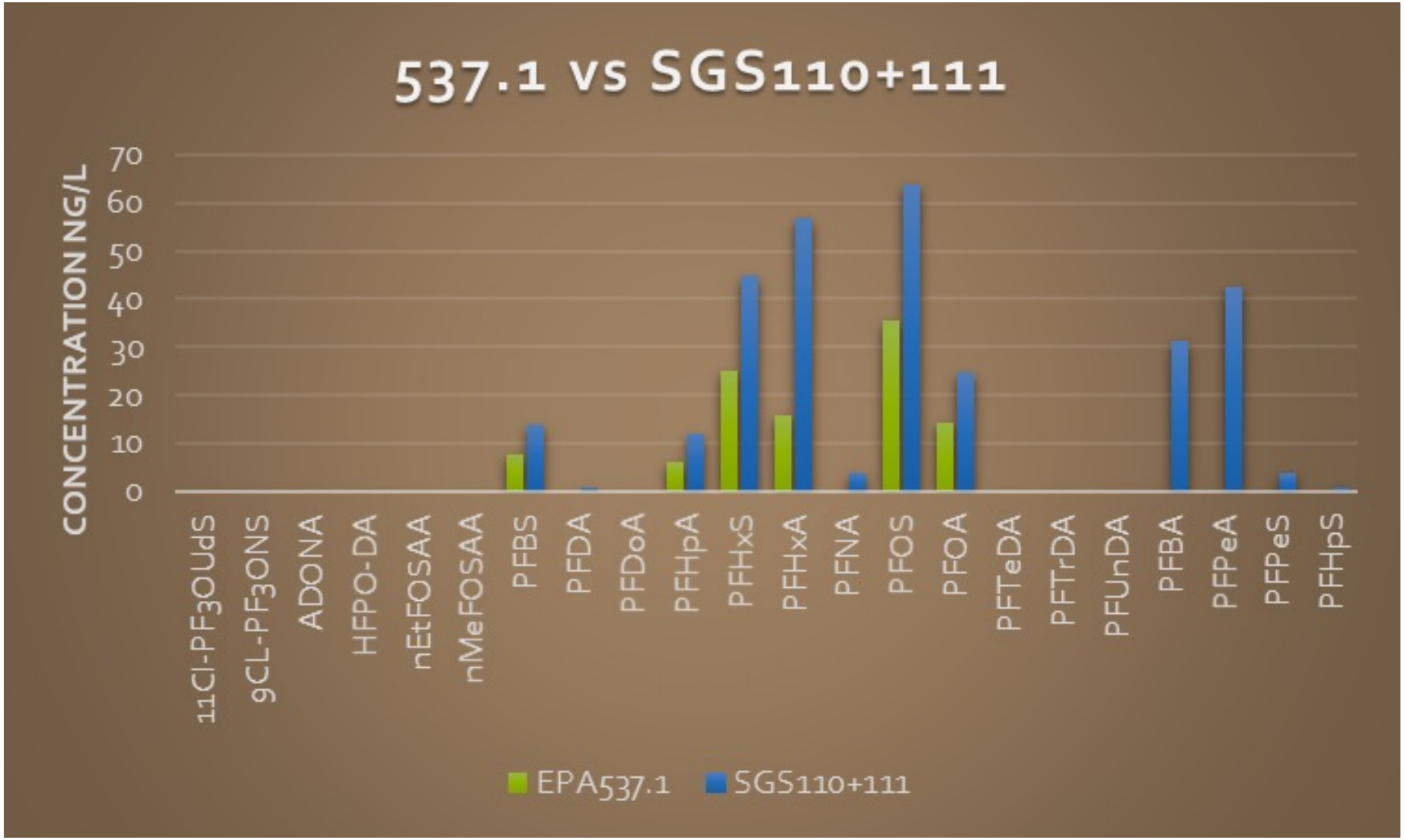
- Surface-water and groundwater sampling equipment
- Spike solutions at two levels (low and high), cleaning protocols, blanks, pre- and post-solution samples, equipment pour/store (SW) and pump throughs (GW), replicates/splits to two labs, 300+ samples submitted
- Analysis for cleaning, reuse, sorption, contamination
- Journal article and Integration to The USGS National Field Manual-NFM




Analytical Methods EPA Methods

- Regulatory Methods:
- **Drinking water**
  - EPA method 537 Revision 1
    - 14 compounds by SPE, LC/MS/MS
    - Published in 2008 **superseded** by 537.1
  - EPA method 537.1 Revision 2
    - 18 compounds by SPE LC/MS/MS, includes "replacement compounds like GenX, ADONA, F-53B
    - Revision 2 published in 2020
  - EPA method 533
    - UCMR 5 public drinking water sampling effort
    - 25 PFAS all "short chain" i.e. less than C12
    - Isotope Dilution
    - Published in 2019
- **Non-potable water, soil, biosolids, sediment, leachate, fish tissue**
  - EPA Draft Method 1633 (single lab validated, multi-lab validation underway)
  - 40 compounds by SPE LC/MS/MS with Isotope Dilution
  - Published in 2021, Errata 2022
- **Many other options**
  - Air methods
  - Different isotope dilution methods
  - Total organic fluorine (all the organic fluorine in a sample, also called total extractable fluorine)
  - Total oxidizable precursors (converts unknown precursors in a sample into known PFAS)
  - Non-target analysis

Analytical method comparison on split samples: USEPA 537.1 conducted by PADEP, and draft USEPA 1633 (MLA 110 + TOP 111) conducted by SGS AXYS Analytical



Differences due to:

- Larger array of compounds included in USEPA 1633.
- Reporting limits are lower in USEPA 1633
- Inclusion of mass converted to detectable compounds by TOP using SGS AXYS MLA 111.



pennsylvania
DEPARTMENT OF ENVIRONMENTAL PROTECTION

BUREAU OF CLEAN WATER

Pennsylvania Passive Water Sampler PFAS Monitoring 2019

Amy Williams, Water Program Specialist, Water Quality Division

Joe Duris, Water Quality Specialist, USGS Pennsylvania Water Science Center - Presenting

Tom Wolf, Governor

Patrick McDonnell, Secretary

USGS science for a changing world

Use of POCIS Time-weighted Average Sampling for PFAS

- Sum PFAS concentrations were calculated by summing the detected concentrations of PFBS, PFOS, PFOA, PFHxA, PFNA, PFDA, and PFDoA in WQN discrete samples and in POCIS samples.
  - These seven compounds had reliable solubility constants at the time of data release. Several more are available now.
- The highest PFOS + PFOA POCIS concentration was 242 ng/L at Neshaminy Creek near Langhorne, PA (compare to 62.5 from the discrete sample at the same site).
- POCIS TWA concentrations for PFOS + PFOA were higher than the PFOS + PFOA concentrations in discrete water samples at all sites where both types of samples were collected
- Detectable POCIS concentrations of PFOS + PFOA were observed in some POCIS samples from sites where PFOS + PFOA concentrations were not detectable in the discrete samples.
- Deployment period for POCIS overlapped discrete sample, but discrete sample was only on a single day during a single time period at each site.

	Sum of PFAS		Number of Substances	
USGS Station Name	POCIS	Discrete	POCIS	Discrete
Delaware River at Trenton, NJ	41.4	8.8	18	4
Schuylkill River at Pottstown, PA	71.5	24.2	17	5
Neshaminy Creek near Langhorne, PA	242.5	62.5	17	5
Lehigh River at Glendon, PA	51.3	15.4	18	5
Jordan Creek at mouth at Allentown, PA	44.4	20	17	5
Brodhead Creek at Minisink Hills, PA	37.9	9.9	15	4
Delaware River near Richmond, PA	12.1	2.1	12	2
Yellow Breeches Creek at New Cumberland, PA	16.0	5.3	12	4
Lackawanna River at Old Forge, PA	39.6	38.2	17	4
Kettle Creek at Cross Fork, PA	0.9	nd	4	0
Hyner Run near Hyner, PA	0.7	nd	4	0
Monongahela River at Elizabeth, PA	20.5	9	16	4
Tunungwant Creek at Bradford, PA.	43.1	9.1	16	3
Ohio River at Sewickley, PA	24.7	13.7	17	4
WEST BRANCH MAHANTANGO CREEK AT MAHANTANGO, PA	25.1	na	11	na
Susquehanna River at Marysville, PA (left bank)	14.0	na	12	na
Susquehanna River at Marysville, PA (center channel)	11.8	na	12	na
Susquehanna River at Marysville, PA (right bank)	13.4	na	13	na

PFAS Concentration comparison POCIS vs Discrete EWI Sample

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Questions?

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