

PFAS Regulatory State of the Union & Wastewater Characterization

June 1, 2022

Taryn McKnight
PFAS Practice Leader
Eurofins Environment Testing America

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Topics



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FEDERAL ACTIONS

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RESEARCH

increase understanding of PFAS exposures and toxicities, human health and ecological effects

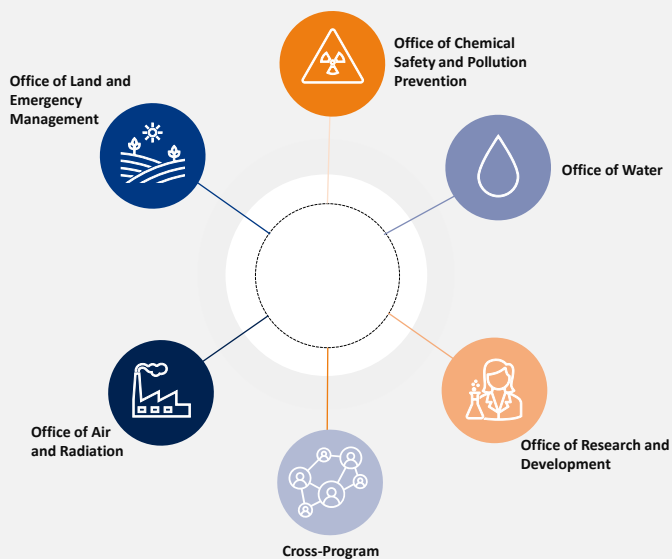
RESTRICT

proactively prevent PFAS from entering air, land, and water at levels that can adversely impact human health and the environment

REMEDIATE

Broaden and accelerate the cleanup of PFAS contamination to protect human health and ecological systems

PFAS STRATEGIC ROADMAP



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TASK FORCE
Identify FFF & Monitor health effects

INCINERATION
Moratorium until guidance is in place

PROCUREMENT
Study items procured containing PFAS

REMEDICATION SCHEDULE
For PFAS release sites

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2022 NATIONAL DEFENSE AUTHORIZATION ACT (NDAA)

The Fiscal Year 2022 NDAA; S. 1605 was signed into law with funding for addressing PFAS with a focus on AFFF and Drinking Water



Temporary Moratorium on Incineration of PFAS Materials
until DoD publishes guidance, or the EPA publishes a final rule regarding destruction and disposal

Creation of a PFAS Task Force
to identify an effective alternative to PFAS AFFF and monitor health aspects of exposure to PFAS

Directs GAO to Audit DoD Procurement
of items containing PFAS, and the feasibility of prohibiting said items

Completion of Remediation Schedule
and cost estimates submitted within 270 days for DoD sites identified as having a release of PFAS



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<https://www.govinfo.gov/app/details/BILLS-117s1605enr>

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HAZARDOUS SUBSTANCES - CERCLA

EPA Takes Steps to Designate PFOA and PFOS as Hazardous Substances Under CERCLA

Issue advance notice of proposed rulemaking on various PFAS under CERCLA

Implications include: cleanup actions, cost recovery, re-open closed sites, ASTM Phase I AAI

<https://www.epa.gov/pfas/pfas-strategic-roadmap-epas-commitments-action-2021-2024>



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Proposed Rule

EPA Submits to OMB, 1/10/22

OMB Review

90 days on average

Comments

Public comment period expected Spring 2022

Final Rule

Summer of 2023

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HAZARDOUS CONSTITUENTS - RCRA

If existing data for the four PFAS constituents support listing any or all as RCRA hazardous constituents, EPA will propose to list the constituents in the Federal Register for public comment

In response to a petition from NM Gov. EPA is evaluating the existing toxicity and health effects data on four PFAS to determine if they should be listed as RCRA Hazardous Constituents

Those chemicals listed are subject to corrective action requirements under RCRA at hazardous waste treatment, storage, and disposal facilities.



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<https://www.epa.gov/newsreleases/epa-responds-new-mexico-governor-and-acts-address-pfas-under-hazardous-waste-law>

<https://www.jdsupra.com/legalnews/epa-announces-plans-for-waste-rules-3519848/>

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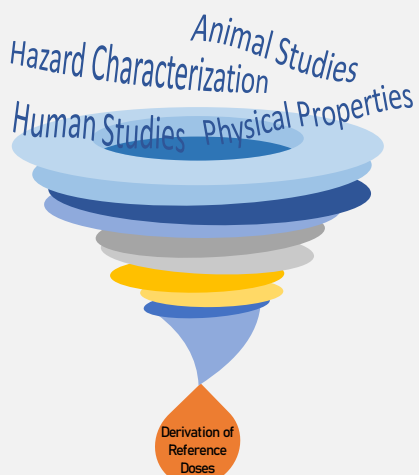
PFOA

PFOS

PFBS

GenX

TOXICITY ASSESSMENTS



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https://cfpub.epa.gov/ncea/iris_drafts/recordisplay.cfm?deid=350051

<https://www.epa.gov/chemical-research/human-health-toxicity-assessments-genx-chemicals>
<https://www.epa.gov/chemical-research/learn-about-human-health-toxicity-assessment-pfbs>

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PFBS

Final, April 2021

GENX

Final, October 2021

PFBA, PFHxA

In Process

PFHxS, PFNA, PFDA

In the queue

TOXICITY ASSESSMENTS



Date	Status	Analyte	RfD (mg/kg/day)
2016	Final	PFOA/PFOS	0.00002
2021	<i>Released for Public Comment</i>	PFOA	0.0000000015
2021	<i>Released for Public Comment</i>	PFOS	0.0000000079
2021	Final	HFPO-DA (GenX)	0.000003
2021	Final	PFBS	0.00003
2021	<i>In Peer Review</i>	PFBA	0.01



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https://iris.epa.gov/AtoZ/?list_type=alpha

https://sab.epa.gov/ords/sab/f?p=100:19:14408101720086::RP_19:P19_ID:963

**PFOA
&
PFOS**
Revisited

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Preliminary Effluent Guidelines Program

Preliminary Plan 15

- Propose limits on PFAS for the chemical, plastics, and synthetic fiber manufacturers by summer 2023.
- Propose limits for the electroplating and metal finishing industries by summer 2024



NPDES News

<https://www.epa.gov/eg/preliminary-effluent-guidelines-program-plan>

March 17, 2021 ANPR
~30,000 comments to date



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Addressing PFAS Discharges in EPA-Issued NPDES Permits

- For federally-issued permits, EPA will include requirements to monitor for PFAS
- Monitoring should include each of the 40 PFAS parameters detectable by draft method 1633
- Draft 1621 can be used in conjunction with draft method 1633, if appropriate.
- This program will enable EPA to obtain comprehensive information on the sources and quantities of PFAS discharges and will use these data to inform the agency's ELG actions
- *EPA plans to issue additional guidance to state permit writers and local pretreatment authorities to address PFAS.*

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<https://www.epa.gov/ep/preliminary-effluent-guidelines-program-plan>

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TSCA SECTION 8(a)(7)

Proposed Rule to Require Reporting on PFAS Manufactured in the United States

EPA proposes to require certain persons that manufacture (including import) or have manufactured these chemical substances in any year since **January 1, 2011**, to electronically report information regarding PFAS uses, production volumes, disposal, exposures, and hazards.

<https://www.regulations.gov/document/EPA-HQ-OPPT-2020-0549-0001><https://www.epa.gov/assessing-and-managing-chemicals-under-tsca/tsca-section-8a7-reporting-and-recordkeeping>

Docket Number
EPA-HQ-OPPT-
2020-0549

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179 PFAS

100lbs for each PFAS

REPORTING

Annually on July 1st

EMISSIONS

Air, Water, Land Disposal

deMINIMIS

PFOA is 0.1%

All other PFAS are 1%

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TOXICS RELEASE INVENTORY (TRI)

2020 TRI Factsheet: PFAS Chemicals

Data Source:
2020 National Analysis Dataset
(released October 2021)



2020 1st Reporting Year

39 Facilities, 5 Sectors, 43 PFAS

Industry Sectors Represented

Chemicals, Hazardous Waste,
Food, Petroleum, Non-Metallic
Mineral Product

Total Production-Related Waste Managed

841.4 thousand lbs

Total On & Off-site Disposal or Other Releases:

90% or 60.0 thousand lbs

<https://www.epa.gov/chemicals-under-tsca/epa-releases-updated-2020-tri-data>
<https://www.epa.gov/toxics-release-inventory-tri-program/addition-certain-pfas-tri-national-defense-authorization-act>



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4 NEW PFAS

Added Jan 24, 2022

LAWSUIT FILED

To Remove TRI Exemptions

RULEMAKING

“Chemicals of Special Concern”

MORE PFAS

Possible under NDAA 2020

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TOXICS RELEASE INVENTORY (TRI)

What is to come...

The agency acknowledges the TRI
exemptions “significantly limited the
amount of data that EPA received.”



PFBS & related salt, + 2 PFAS by CAS#

EPA plans to propose a rule removing de minimis exemption and list PFAS as “Chemicals of Special Concern”

Per NDAA 2020 triggers, additional PFAS will likely continue to be added to the TRI

Advocacy group files lawsuit to remove TRI exemptions

<https://www.epa.gov/toxics-release-inventory-tri-program/addition-certain-pfas-tri-national-defense-authorization-act>



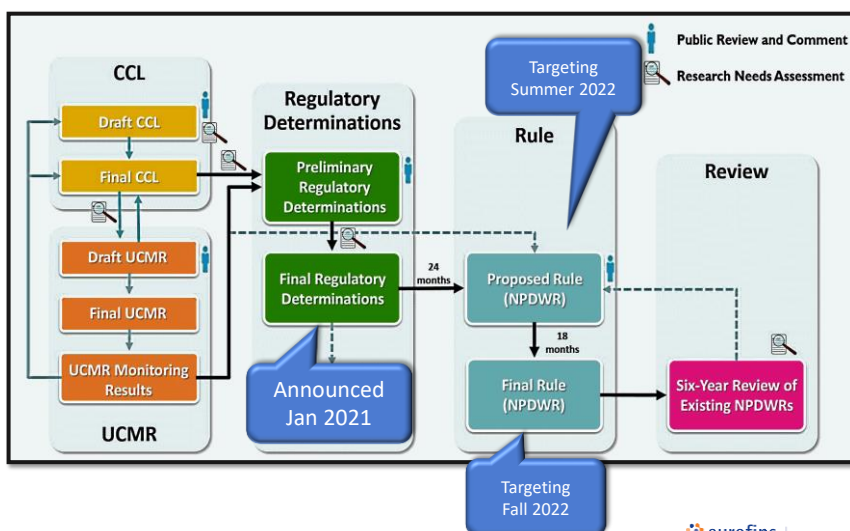
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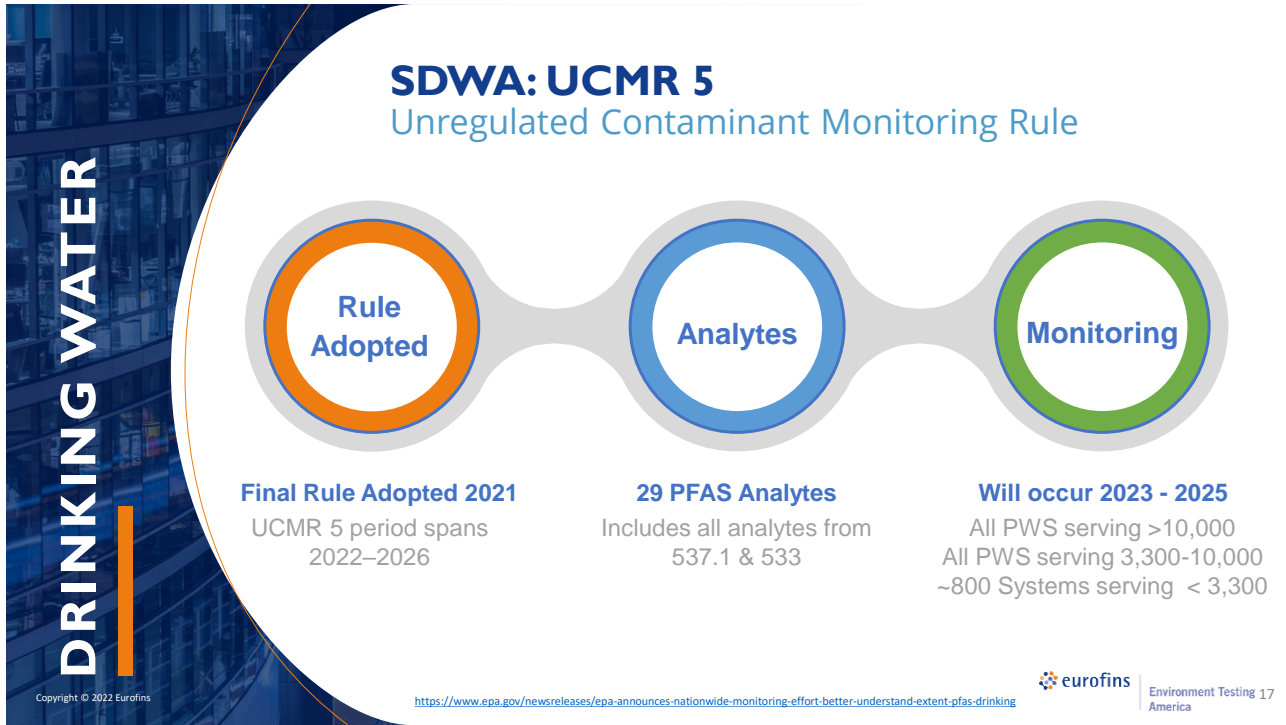
DRINKING WATER

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PFAS Maximum Contaminant Levels (MCLs)



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DRINKING WATER

SDWA: UCMR 5

PFAS Analyte List

533 Analytes						537.1 Analytes
PFBA	PFOA	PFDaA	PFHpS	8:2 FTS	9Cl-PF3ONS	PFTA
PFPeA	PFNA	PFBS	PFOS	DONA	PFMPA	PFTTrDA
PFHxA	PFDA	PFPeS	4:2 FTS	HFPO-DA	PFMBA	NMeFOSAA
PFHpA	PFUnA	PFHxS	6:2 FTS	11Cl-PF3OUdS	NFDHA	NEtFOSAA
PFEESA						

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<https://www.epa.gov/dwucmr/fifth-unregulated-contaminant-monitoring-rule>

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STATE ACTIONS



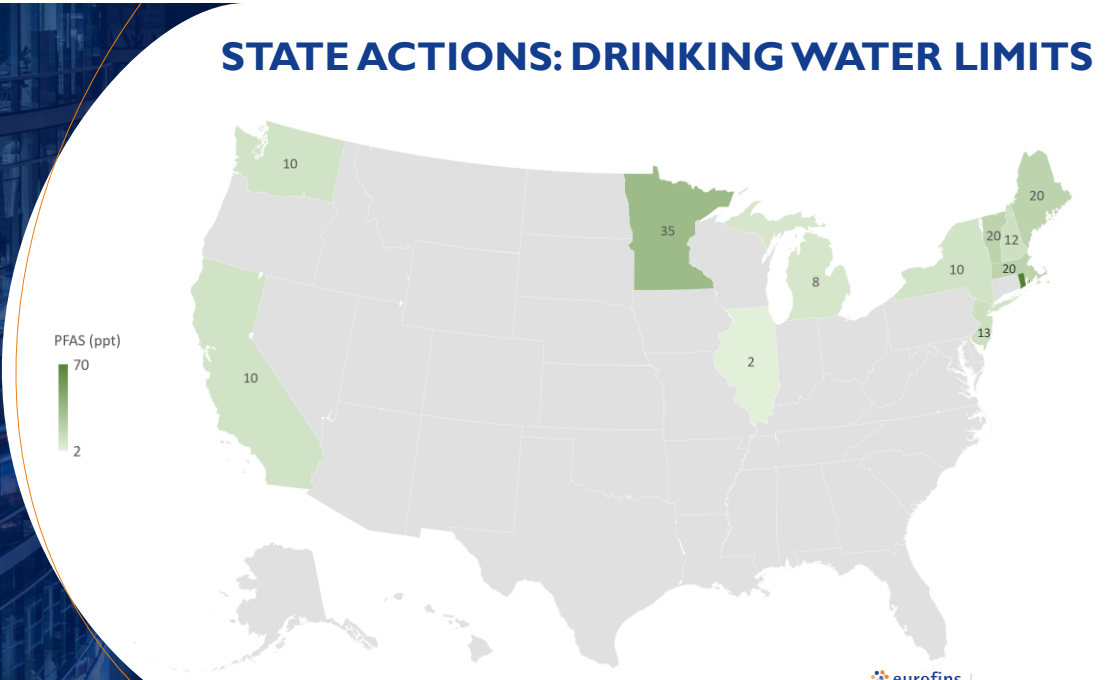
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DRINKING WATER

STATE ACTIONS: DRINKING WATER LIMITS



State	PFAS Limit (ppt)
California	10
Washington	10
Minnesota	35
Illinois	2
Michigan	8
Ohio	10
Connecticut	10
Massachusetts	12
New Hampshire	20
Vermont	20
New Jersey	13

PFAS (ppt)

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STATE ACTIONS: GROUNDWATER LIMITS

	FL	HI	MI	IL	PA
PFOA ppt	70	40	8	2	70
PFOS ppt	70	40	16	14	70
Ratified Y/N	N	N	Y	N	N
Comments	Sum of PFOA & PFOS	+ 16 other PFAS	+ PFHxS/ GenX/PFBs/ PFHxA/PFNA	+ PFBS/ PFHxS/PFHxA	+ PFBS

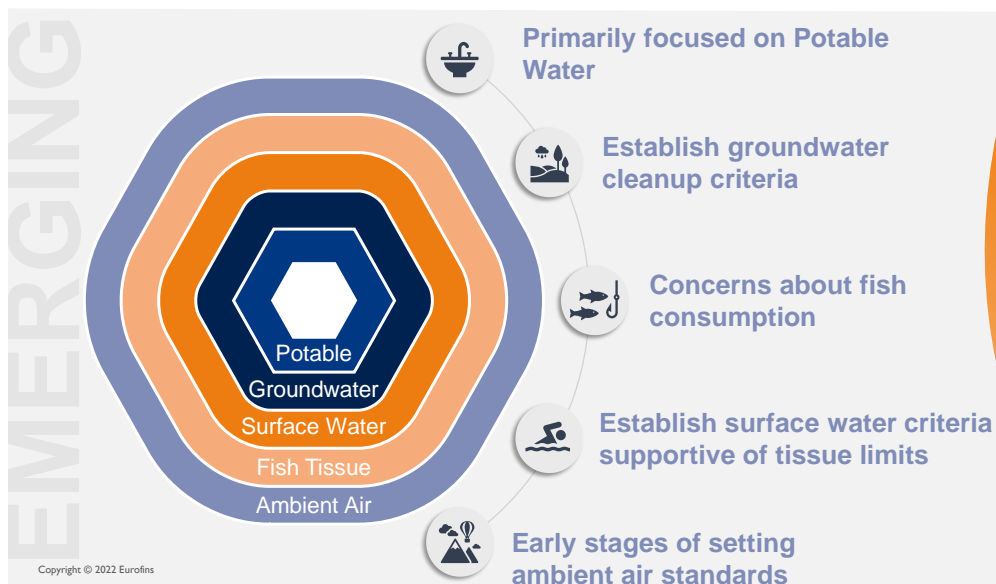
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CLEANUP

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SURFACE WATER & TISSUE



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CLEANUP

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AMBIENT AIR States Establish Limits

State	PFOA	PFOS	APFO	6:2FTS
NH	N/A	N/A	0.024ug/m ³ (annual)	N/A
TX	0.005ug/m ³ (annual)	0.01ug/m ³ (annual)	0.01ug/m ³ (annual)	N/A
MI	0.07ug/m ³ (24hr)	0.07ug/m ³ (24hr)	N/A	1.0ug/m ³ (annual)
NY	0.0053ug/m ³ (annual)	N/A	N/A	N/A
MN	0.07ug/m ³ (24hr->8yr)	0.07ug/m ³ (24hr->8yr)	N/A	N/A

<http://www2.des.state.nh.us/OneStopPub/Air/330110016520060807TypeAOC.pdf>

https://www.michigan.gov/documents/deq/deq-aqd-toxics-ITSLALPH_244167_7.pdf

https://www.dec.ny.gov/docs/air_pdf/darIproposed.pdf

<https://www.health.state.mn.us/communities/environment/risk/guidance/air/table.html#hbvsraas>



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RESEARCH & SCIENTIFIC DEVELOPMENTS

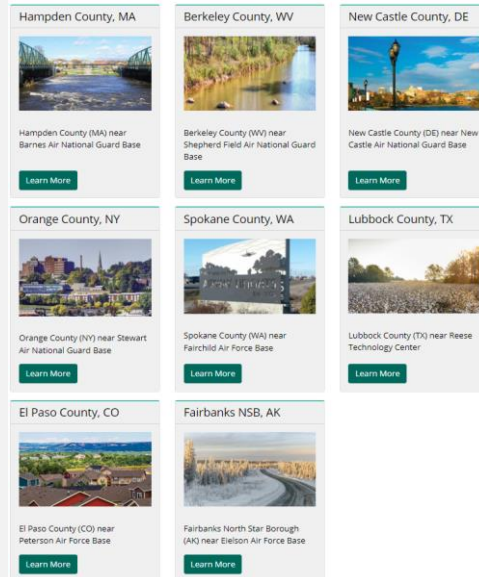


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EXPOSURE STUDIES



- Drinking Water
- NHANES Human Serum
- Indoor Air / Dust

https://www.cdc.gov/exposurereport/pfas_early_release.html
<https://www.atsdr.cdc.gov/pfas/activities/studies/multi-site.html>



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ENVIRONMENTAL STUDIES

- Vapor Intrusion Pathway
- Source Air, Products of Incomplete Combustion (PICs)

<https://www.epa.gov/chemical-research/status-epa-research-and-development-pfas>
<https://www.serdp-estcp.org/serdp-estcp/Program-Areas/Environmental-Restoration/ER19-1408/ER19-1408>
https://www.epa.gov/sites/default/files/2019-09/documents/technical_brief_pfas_incineration_ioaa_approved_final_july_2019.pdf



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EPA RESEARCH

BIOSOLIDS

- The EPA expects to complete a risk assessment of PFOA and PFOS in biosolids by the winter of 2024 to determine if regulations are needed

<https://www.epa.gov/pfas/pfas-strategic-roadmap-epas-commitments-action-2021-2024>

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EPA Draft Methods

EPA Draft 1633

- Targeted Analysis of 40 PFAS
- Non-Potable Water, Soil & Tissue
- LCMSMS, SPE, Isotope Dilution
- Multi-Lab Validation Underway

EPA Draft 1621

- Adsorbable Organic Fluorine (AOF)
- Proxy analysis for 'Total PFAS'
- Single lab validation complete; multi-lab validation has not begun



<https://www.epa.gov/cwa-methods/cwa-analytical-methods-and-polyfluorinated-alkyl-substances-pfas>

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EPA METHODS

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Compounds Included in EPA Draft 1633 (RLs = 2-5ng/L)

Perfluorobutanoic acid (PFBA)	NEtFOSA
Perfluoropentanoic acid (PFPeA)	NMeFOSA
Perfluorohexanoic acid (PFHxA)	NMeFOSAA
Perfluoroheptanoic acid (PFHpA)	NEtFOSAA
Perfluorooctanoic acid (PFOA)	NMeFOSE
Perfluorononanoic acid (PFNA)	NEtFOSE
Perfluorodecanoic acid (PFDA)	4:2 FTS
Perfluoroundecanoic acid (PFUnA)	6:2 FTS
Perfluorododecanoic acid (PFDoA)	8:2 FTS
Perfluorotridecanoic acid (PFTrIA)	9CI-PF3ONS
Perfluorotetradecanoic acid (PFTeA)	11CI-PF3OUdS
Perfluorobutanesulfonic acid (PFBS)	DONA
Perfluoropentanesulfonic acid (PFPeS)	HFPO-DA (GenX)
Perfluorohexanesulfonic acid (PFHxS)	3:3 FTCA
Perfluoroheptanesulfonic Acid (PFHpS)	5:3 FTCA
Perfluorooctanesulfonic acid (PFOS)	7:3 FTCA
Perfluorononanesulfonic acid (PFNS)	NFDHA
Perfluorodecanesulfonic acid (PFDS)	PFMBA
Perfluorododecanesulfonic acid (PFDoS)	PFMPA
Perfluorooctanesulfonamide (FOSA)	PFEESA

Target Compounds Not Part of EPA Draft 1633 (RLs = 2-5ng/L)

10:2 FTS	EVE Acid
6:2 FTCA	PFO5DA
8:2 FTCA	PMPA
10:2 FTCA	PEPA
6:2 FTUCA	MTP
8:2 FTUCA	PS Acid
10:2 FTUCA	Hydro-PS Acid
PFECHS	R-PSDA
PFPrS	Hydrolyzed PSDA
PFPrA	R-PSDCA
PFMOAA	6:2 diPAP
PFECAG	8:2 diPAP
PFO4DA	6:2/8:2 diPAP
PFO3OA	10:2 diPAP
PFO2HxA	10:2 FTOH (RL=1ug/L)
R-EVE	8:2 FTOH (RL=1ug/L)
NVHOS	7:2 FTOH (RL=1ug/L)
Hydro-EVE Acid	6:2 FTOH (RL=1ug/L)
Perfluoro-n-octadecanoic acid (PFODA)	4:2 FTOH (RL=1ug/L)
Perfluoro-n-hexadecanoic acid (PFHxDA)	

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In-depth Characterization of PFAS in Wastewater, a More Comprehensive Analysis



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Where to Begin?

*A mountain of questions
when addressing the
mostly unknown*

WHAT WILL THE DATA BE USED FOR

- Developing a Conceptual Site Model
- Determining the extent of contamination
- Investigating sources of contamination
- Assessing human health impacts
- Implementing a remediation plan

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CONVENTIONAL TOOLS

TARGETED ANALYSIS

The analysis of specific target analytes with known CAS numbers and analytical reference standards

- EPA Standard Methods
- User-Defined Methods
 - Regulatory derived target analyte lists
 - Laboratory derived target analyte lists

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EPA Methods

EPA 537.I
EPA 533
EPA 8327
EPA Draft 1633

User-defined Methods

“537 Modified”
ASTM D7979 Mod
Laboratory SOP

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EMERGING TECHNOLOGIES

NON-TARGETED ANALYSES

The analysis of analytes without known CAS numbers or analytical reference standards, or the analysis of a proxy analyte(s)

- Draft or Non-Standard Methods
- User-Defined Methods
 - Program specific targets
 - Screening applications
 - Litigation derived targets

TOP Assay

Total Oxidizable Precursors

AOF/EOF

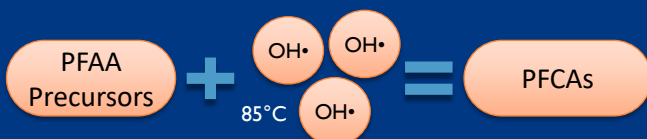
Adsorbable Organic Fluorine
Extractable Organic Fluorine

NTA

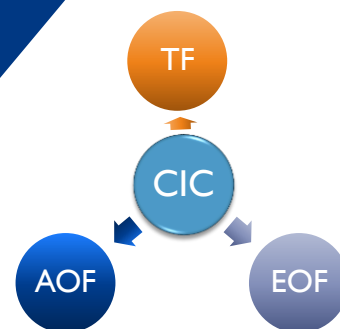
Non-Target Analysis

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Total Oxidizable Precursors TOP Assay



	Pre-TOP	Post-TOP
PFOA	13	14 ng/L
PFHxA	11	42 ng/L
PFPA	15	40 ng/L
PFHpA	ND	6.3 ng/L
PFBA	ND	46 ng/L
Total PFCA	39	148.3 ng/L
Total PFCA Difference		109.3 ng/L



CIC: Combustion Ion Chromatography

Total Organofluorine Analysis

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Non-Target Analysis



LC-QToF-MS

Liquid Chromatography
Quadrupole Time of Flight
Mass Spectrometry



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STUDY OBJECTIVES



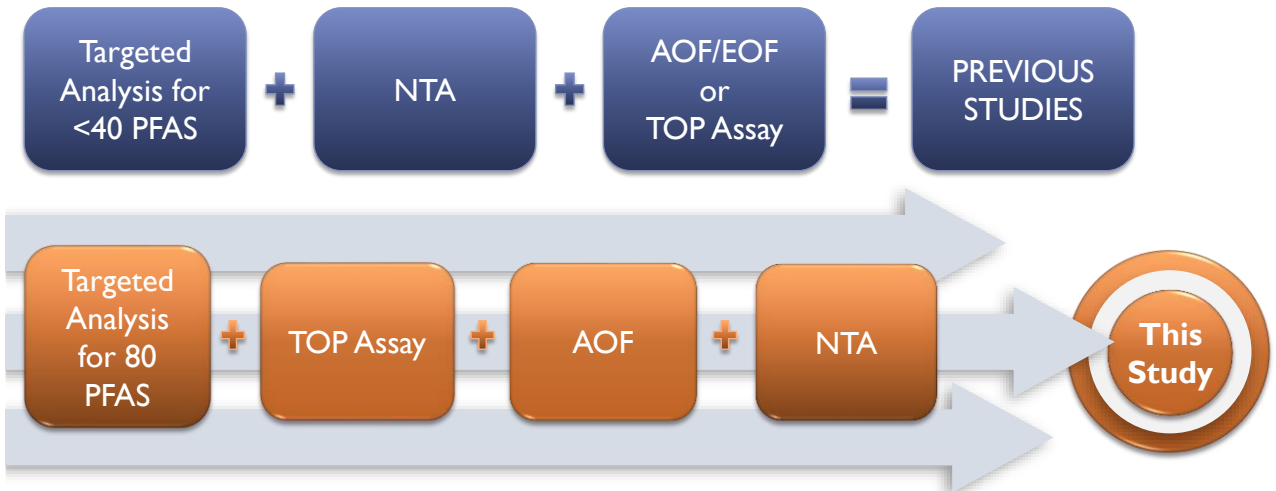
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Study Goals



Fluorine Mass Balance in Marine Mammals from the Northern Hemisphere - A combination of targeted, total (organo)fluorine, and non-targeted analysis K. Spaan et al
 Detection and Transformations of Poly- and Perfluoroalkyl Acids Downstream from Fire Training Areas in Groundwater-fed Coastal Watersheds B. Ruyle et al
 Characterization of Per- and Polyfluoroalkyl Substances (PFAS) in Firefighting Training Areas across Eastern Canada M. Liu et al

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SAMPLES COLLECTED

POTW Influent & Effluent
 Industrial Discharge Effluent

The slide features a background image of a waterfall and a circular inset showing two researchers in waders collecting samples from a stream. One researcher is using a tablet while the other is reaching into the water.

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Wastewater Samples



Private WWTP

Site 1

1. Influent
2. Influent Duplicate
3. Effluent
4. Effluent Duplicate

POTW

Site 2

1. Influent
2. Influent Duplicate
3. Effluent
4. Effluent Duplicate

POTW

Site 3

1. Influent
2. Influent Duplicate
3. Effluent
4. Effluent Duplicate

INDUSTRIAL

Site 4

1. Eff (Aerospace)
2. Eff (Aerospace) Dup
3. Eff (Circuit Board)
4. Eff (Circuit Board) Dup
5. Eff (Landfill)
6. Eff (Landfill) Dup
7. Eff (Dairy)
8. Eff (Dairy) Dup
9. Eff (Com Laundry)
10. Eff (Com Laundry) Dup
11. Eff (Car Wash)
12. Eff (Car Wash) Dup

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METHODS EMPLOYED

Targeted PFAS by LCMSMS & GCMSMS
 FTOHs by GCMSMS
 TOP Assay by LCMSMS
 AOF by CIC
 NTA by LC-QTOF-MS



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User-Defined LCMSMS	User-Defined GCMSMS	TOP Assay	Adsorbable Organic Fluorine (AOF)	Non-Target Analysis (NTA)
75 PFAS Compounds	5 Fluorotelomer Alcohols (FTOHs)	Total Oxidizable Precursors	Total Organic Fluorine	Non-targeted compounds
RLs: 2ppt-5ppt	RLs: 1ppb	RLs: 2ppt-5ppt	RLs: 1ppb	>1ppb
LCMSMS with confirmation ion	GCMSMS	LCMSMS with confirmation ion	Combustion Ion Chromatography (CIC)	LC-QToF-MS: Data independent acquisition
Isotope Dilution	Internal Standard with FTOH labeled standard	Isotope Dilution	External Standard	SWATH: HRMS/MS data are collected on parent and daughter ions independent of intensity threshold
SPE WAX	Solvent Extraction	SPE WAX	Carbon Sorbent	Direct Injection

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LCMSMS/GCMSMS Analysis	Compounds Included in EPA Draft 1633 (RLs = 2-5ng/L)		Compounds Not Part of Draft 1633 (RLs = 2-5ng/L)	
	Perfluorobutanoic acid (PFBA)	NEtFOSA	10:2 FTS	PFPeA
	Perfluoropentanoic acid (PFPeA)	NMeFOSA	6:2 FTCA	EVE Acid
	Perfluorohexanoic acid (PFHxA)	NMeFOSAA	8:2 FTCA	PFOSDA
	Perfluoroheptanoic acid (PFHpA)	NEtFOSAA	10:2 FTCA	PMPA
	Perfluorooctanoic acid (PFOA)	NMeFOSE	6:2 FTUCA	PEPA
	Perfluorononanoic acid (PFNA)	NEtFOSE	8:2 FTUCA	MTP
	Perfluorodecanoic acid (PFDA)	4:2 FTS	10:2 FTUCA	PS Acid
	Perfluoroundecanoic acid (PFUnA)	6:2 FTS	PFECHS	Hydro-PS Acid
	Perfluorododecanoic acid (PFDoA)	8:2 FTS	PFPrS	R-PSDA
	Perfluorotridecanoic acid (PFTriA)	9Cl-PF3ONS	PFPrA	Hydrolyzed PSDA
	Perfluorotetradecanoic acid (PFTeA)	11Cl-PF3OUdS	PFMOAA	R-PSDCA
	Perfluorobutanesulfonic acid (PFBS)	DONA	PFECAG	6:2 diPAP
	Perfluoropentanesulfonic acid (PFPeS)	HFPO-DA (GenX)	PFO4DA	8:2 diPAP
	Perfluorohexanesulfonic acid (PFHxS)	3:3 FTCA	PFO3OA	6:2/8:2 diPAP
	Perfluoroheptanesulfonic Acid (PFHpS)	5:3 FTCA	PFO2HxA	10:2 diPAP
	Perfluorooctanesulfonic acid (PFOS)	7:3 FTCA	R-EVE	10:2 FTOH (RL=1ug/L)
	Perfluorononanesulfonic acid (PFNS)	NFDHA	NVHOS	8:2 FTOH (RL=1ug/L)
	Perfluorodecanesulfonic acid (PFDS)	PFMBA	Hydro-EVE Acid	7:2 FTOH (RL=1ug/L)
	Perfluorododecanesulfonic acid (PFDoS)	PFMPA	Perfluoro-n-octadecanoic acid (PFODA)	6:2 FTOH (RL=1ug/L)
	Perfluorooctanesulfonamide (FOSA)	PFEESA	Perfluoro-n-hexadecanoic acid (PFHxDA)	4:2 FTOH (RL=1ug/L)

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RESULTS

Summary of Results Comparison Across Methods



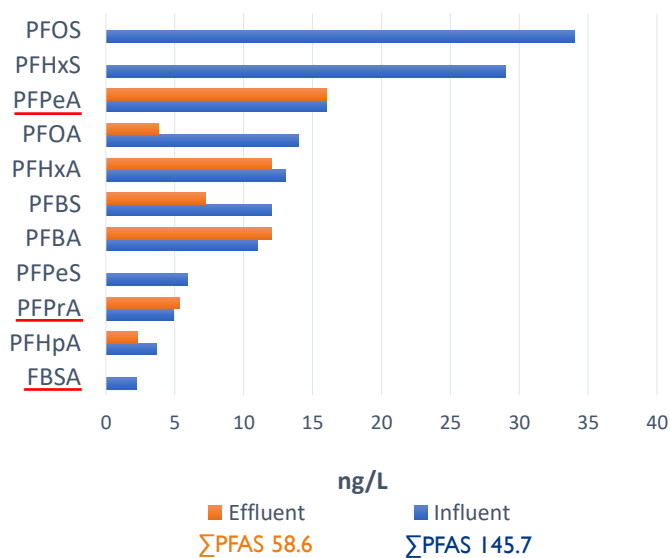
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Site 1 Private WWTP

Sample	AOF (ng/L)	TOP Assay – PFCA Difference (ng/L)
Influent	1,300	110
Influent Dup	1,300	120
Effluent	1,500	220
Effluent Dup	1,100	230

FTOHs are all non-detect @ 1,000 ng/L

Site 1, Private WWTP Influent & Effluent



AOE Equivalent for these samples is 63%

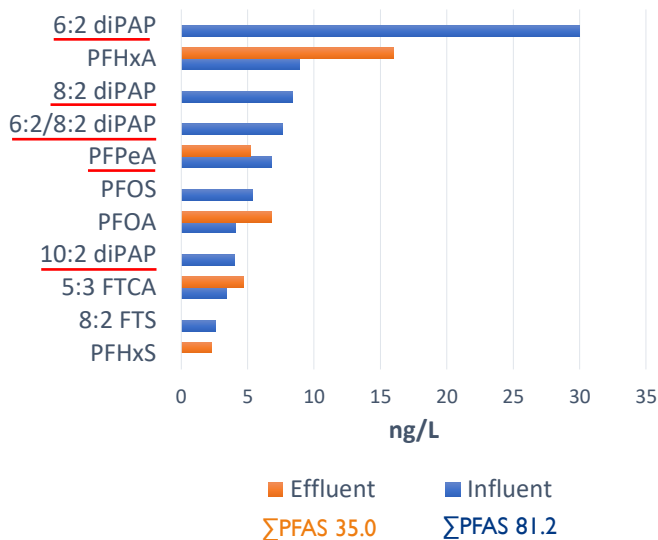
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Site 2 POTW

Sample	AOF (ng/L)	TOP Assay – PFCA Difference (ng/L)
Influent	ND @ 10,000	Not available
Influent Dup	ND @ 10,000	Not available
Effluent	2,400	Not available
Effluent Dup	2,800	Not available

FTOHs are all non-detect @ 1,000 ng/L

Site 2, POTW Influent & Effluent



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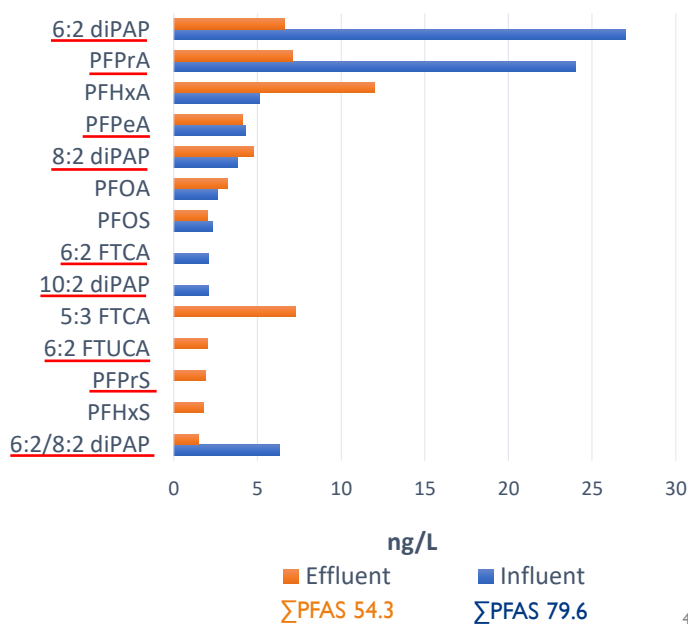
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Site 3 POTW

Sample	AOF (ng/L)	TOP Assay – PFCA Difference (ng/L)
Influent	5,200	170
Influent Dup	4,600	170
Effluent	3,100	94
Effluent Dup	1,800	85

FTOHs are all non-detect @ 1,000 ng/L

Site 3, POTW Influent & Effluent



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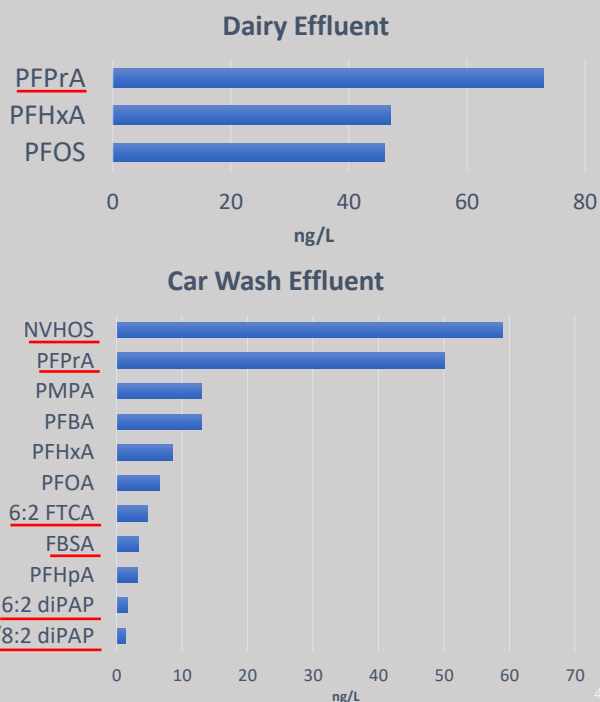
Sample	AOF (ng/L)	TOP Assay – PFCA Difference (ng/L)
Effluent (Circuit Board 1)	2,300	7.1
Effluent (Circuit Board 2)	3,300	140
Effluent (Aerospace)	3,700	43
Effluent (Landfill)	1,400	100

Sample	AOF (ng/L)	TOP Assay – PFCA Difference (ng/L)
Effluent (Circuit Board 1)	2,300	7.1
Effluent (Circuit Board 2)	3,300	140
Effluent (Aerospace)	3,700	43
Effluent (Landfill)	1,400	100

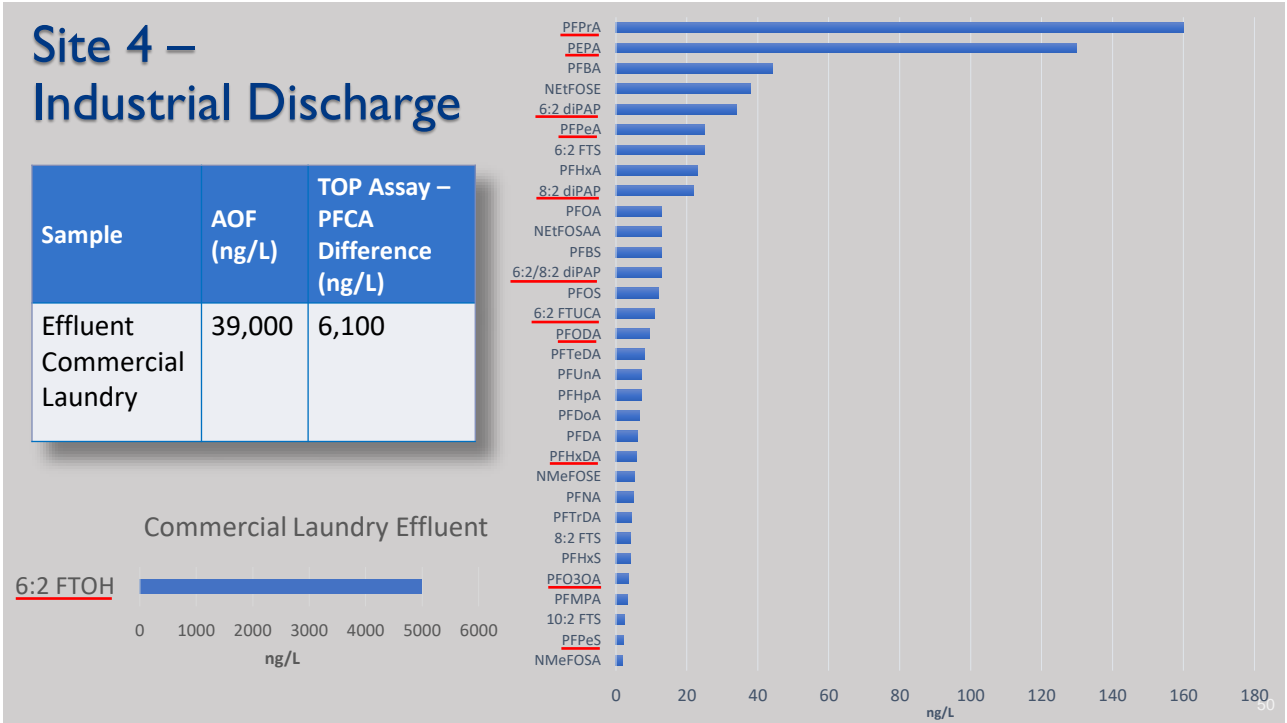
48

Site 4 – Industrial Discharge

Sample	AOF (ng/L)	TOP Assay – PFCA Difference (ng/L)
Effluent Dairy	33,000	360
Effluent Car Wash	ND @ 1,000	84



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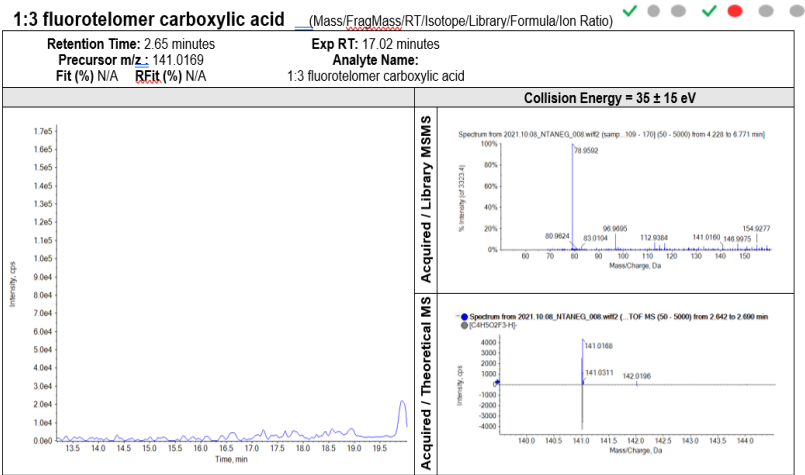


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What did the NTA tell us?

Sites	Detections
Sites 1, 2, & 3	1:3 fluorotelomer carboxylic acid – Moderate to High Confidence
Site 1	N-dimethyl ammonio propyl perfluorohexane sulfonamido propanoic acid - Low Confidence / Possible identification of a group

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CONCLUSIONS

Not all tools are necessary for every job

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Consistent across samples:

Low level detections (i.e. <70ppt)
~10-15 PFAS compounds detected
~Half the compounds captured by
Draft 1633, the other half not
captured by Draft 1633
TOP Assay conversion >100ng/L
AOF at 1000-5000ng/L range

Results



Not so good correlation:

Significant AOF & TOP Assay
detections with minimal detections
in the target analysis

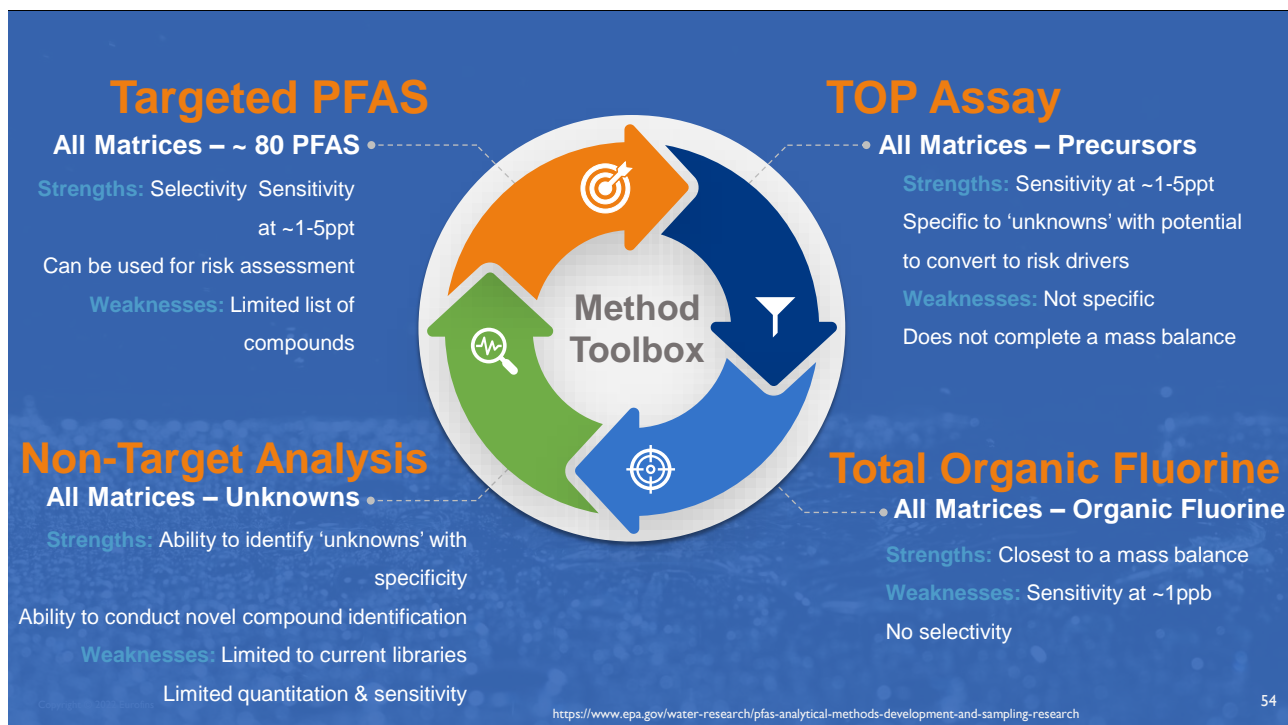
AOF indicates presence of PFAS
where TOP Assay does not

Infrequent correlation or occurrence:

Minimal detections in target
analysis with equally minimal
detections in AOF and TOF

NTA illuminates a novel compound
from PFAS 'dark matter' in several
samples

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