



PFAST Team 1: Sample Acquisition and Analysis Plan for PFAS in Water

TEAM #1: PFAS TESTING: WATER

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Research Questions

What are the concentrations of targeted legacy and emerging PFAS contaminants in North Carolina public drinking water sources?

- Collect and analyze raw water samples during two consecutive quarters of 2018/2019 at all 191 municipal surface water intakes and all 149 municipal drinking water systems treating groundwater in NC for PFAS measurement
- Repeat this sampling for systems with detectable PFAS in the third quarter of 2019

What unanticipated and untargeted PFAS compounds occur in North Carolina public drinking water sources?

- Apply high-resolution mass spectrometry methods to screen samples collected above for presence of > 5,100 known PFAS compounds as well as for unknown fluorinated organic compounds

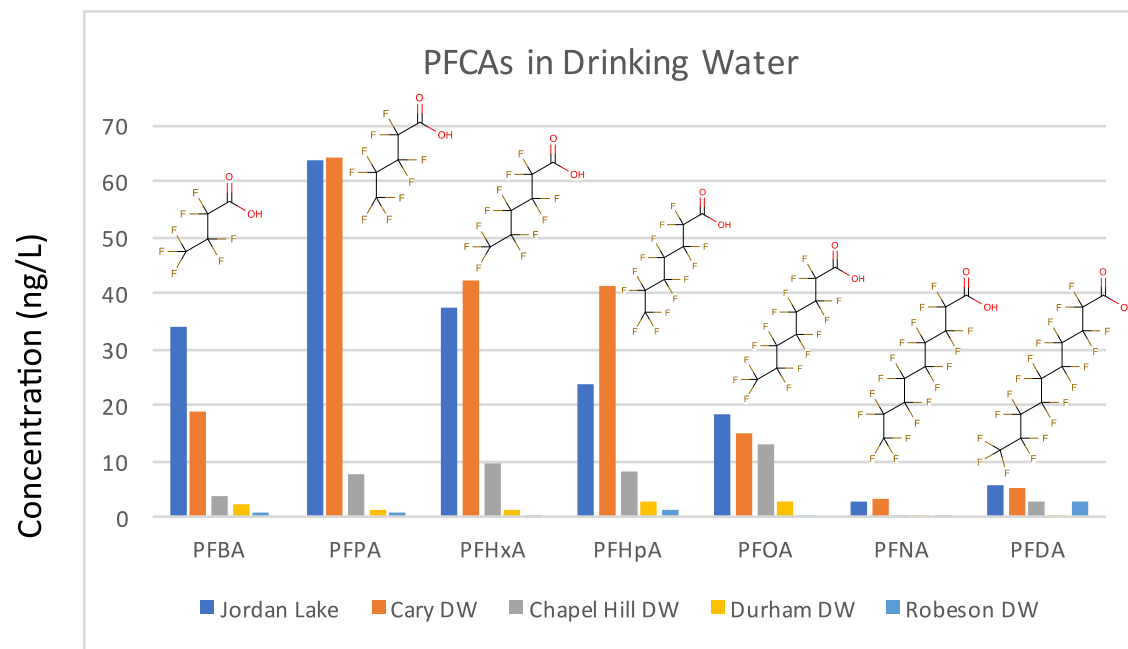
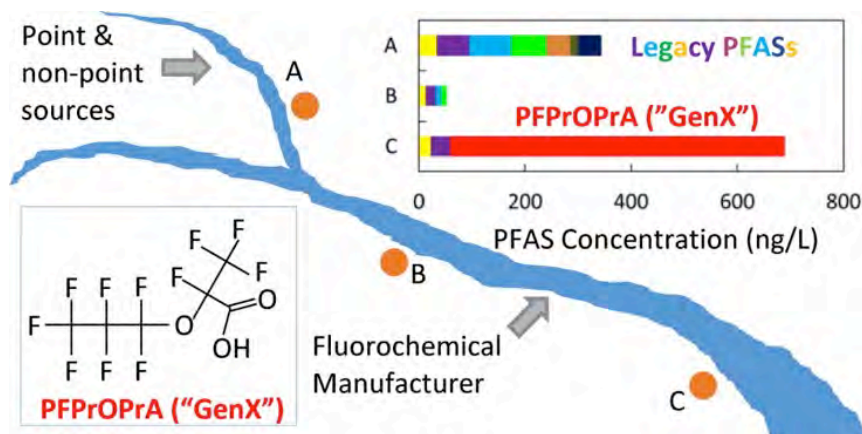
How much of the total organic fluorine in North Carolina public drinking water sources can be accounted for by targeted PFAS quantitation?

- Utilize adsorbable organic fluorine (AOF) measurements in concert with the quantitative PFAS measurements outlined above to assess fluorine “mass balance” in water samples

Why is this research needed?

Legacy and Emerging Perfluoroalkyl Substances Are Important Drinking Water Contaminants in the Cape Fear River Watershed of North Carolina

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Jordan Lake; n=3
Cary DW; n=4
Chapel Hill; n=1

Durham; n=2
Robeson; n=9

Heather M. Stapleton and P. Lee Ferguson, Unpublished

Sample acquisition strategy

- 191 Municipal surface water sources
- 149 Municipal well water sources

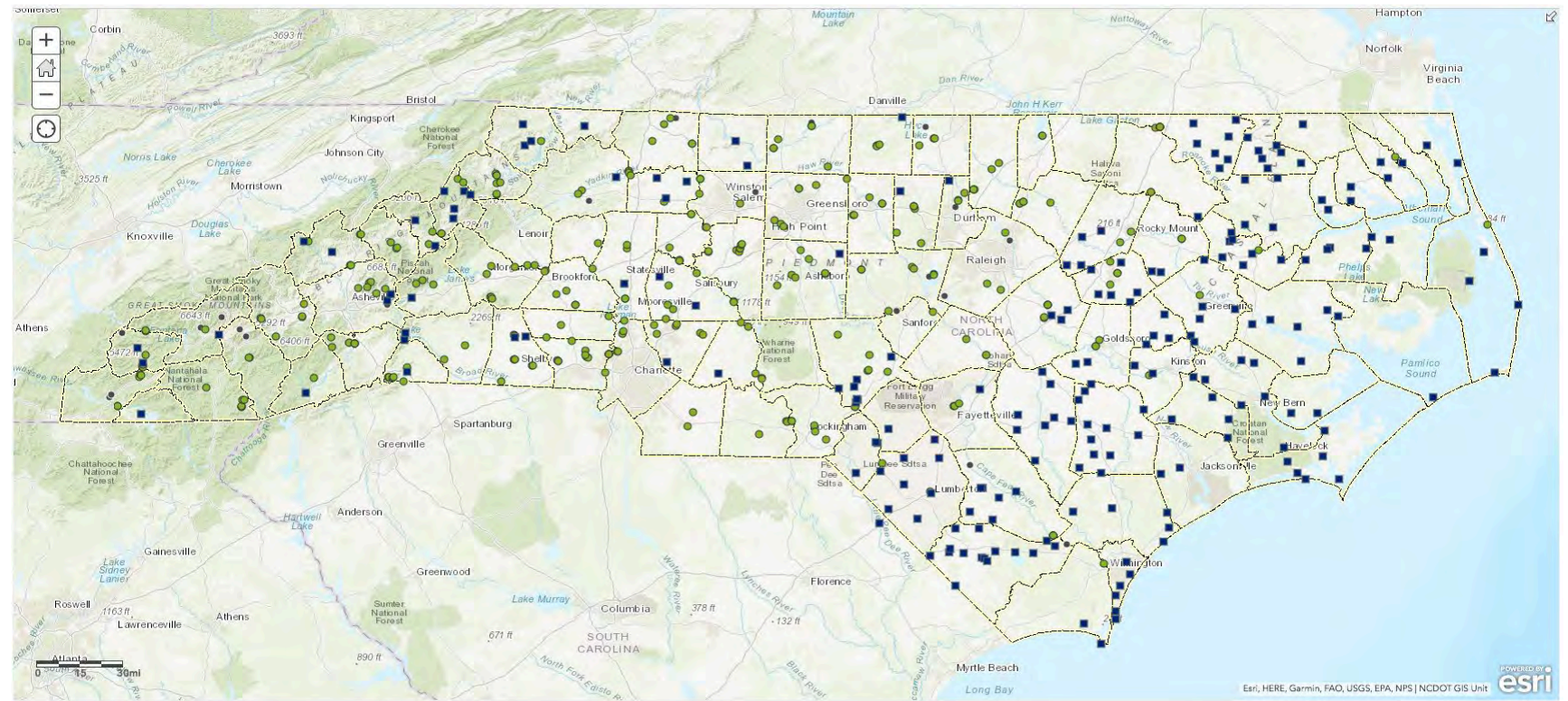


Figure 1. Surface (green circle) and groundwater (blue square) sampling sites for drinking water sources to be analyzed for PFAS compounds.

PFAS analysis strategy

Sample collection:

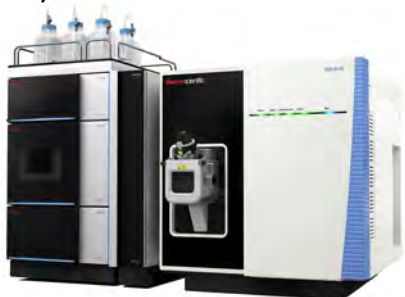
- Coordinated between NCSU and Duke
- ~25-30 samples per week, organized in geographic sectors
- Samples collected in polypropylene bottles and stored on ice during transport

QA/QC and replication:

- Trip blanks included with every sampling
- Trip spikes (50 ng/L analyte addition) included with every sampling
- Duplicate samples for 10% of sites
- NCSU & Duke will split sample analyses evenly, with 20% samples analyzed by both labs

PFAS analysis strategy

Triple Quadrupole MS/MS (target quantitation)



Analyte	Abbreviation	CAS #
Perfluoroalkanoic Acids		
Perfluorobutanoic acid	PFBA	375-22-4
Perfluoropentanoic acid	PFPA	2706-91-3
Perfluorohexanoic acid	PFHxA	307-24-4
Perfluoroheptanoic acid	PFHpA	375-35-9
Perfluorooctanoic acid	PFOA	335-07-1
Perfluorononanoic acid	PFNA	375-05-1
Perfluorodecanoic acid	PFDA	335-26-2
Perfluoroundecanoic acid	PFUdA	2626-94-8
Perfluorododecanoic acid	PFDDA	307-25-1
Perfluorotridecanoic acid	PFTrDA	7403-94-8
Perfluorotetradecanoic acid	PFTeDA	375-05-7
Perfluorohexadecanoic acid	PFDHxA	67005-19-5
Perfluoropolyether acids		
Perfluorooctanesulfonic acid	PFOS	375-75-5
Perfluorodecane sulfonic acid	PFDS	2706-91-4
Perfluorododecane sulfonic acid	PFDDA	369-46-4
Perfluorotetradecane sulfonic acid	PFTEA	375-02-2
Perfluorohexadecane sulfonic acid	PFHSA	1703-23-1
Perfluorooctanesulfonamide	PFOSA	6825-13-1
Perfluorodecane sulfonamide	PFDSA	335-77-3
Perfluorododecane sulfonamide	PFDDSA	67761-39-5
Perfluoropolyether sulfonamides		
N-ethylperfluorooctanesulfonamide	NPEFOA	2091-56-6
N-methylperfluorodecane sulfonamide	NMPFDA	7855-31-9
Perfluorooctane sulfonamide	PFOSA	754-08-6
N-ethylperfluorodecane sulfonamide	NPEFDA	6091-99-2
N-methylperfluorododecane sulfonamide	NMPDDA	7446-09-7
N-ethylperfluorododecane sulfonamide	NPEDDA	6151-50-2
N-methylperfluorotetradecane sulfonamide	NMPTEA	6156-13-8

PFAS mass balance

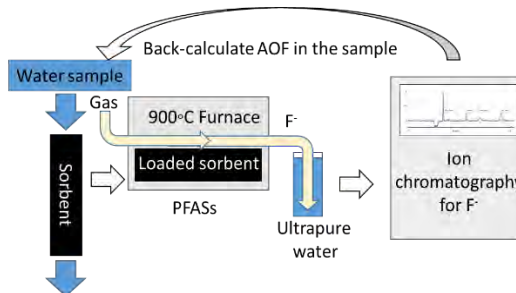
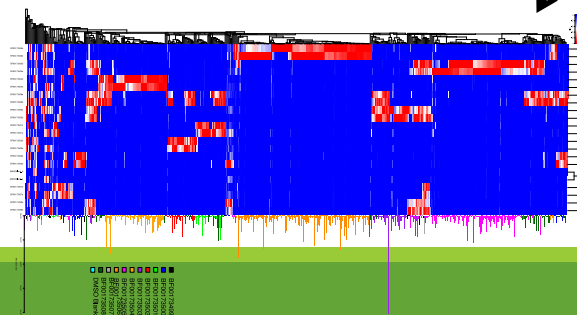
Sample Collection



Solid Phase Extraction



High-resolution MS (suspect screening)



Adsorbable organic fluorine analysis

PFAS target compounds (quantitative measurements)

Analyte	Abbreviation	CAS #
<i>Perfluorocarboxylic Acids</i>		
Perfluorobutanoic acid	PFBA	375-22-4
Perfluoropentanoic acid	PFPeA	2706-90-3
Perfluorohexanoic acid	PFHxA	307-24-4
Perfluoroheptanoic acid	PFHpA	375-85-9
Perfluorooctanoic acid	PFOA	335-67-1
Perfluorononanoic acid	PFNA	375-95-1
Perfluorodecanoic acid	PFDA	335-76-2
Perfluoroundecanoic acid	PFUnA	2058-94-8
Perfluorododecanoic acid	PFDoDA	307-55-1
Perfluorotridecanoic acid	PFTriDA	72629-94-8
Perfluorotetradecanoic acid	PFTeDA	376-06-7
Perfluorohexadecanoic acid	PFHxDA	67905-19-5
<i>Perfluoroalkylsulfonic acids</i>		
Perfluorobutanesulfonic acid	PFBS	375-73-5
Perfluoropentanesulfonic acid	PFPeS	2706-91-4
Perfluorohexanesulfonic acid	PFHxS	355-46-4
Perfluoroheptanesulfonic acid	PFHpS	375-92-8
Perfluorooctanesulfonic acid	PFOS	1763-23-1
Perfluorononanesulfonic acid	PFNS	68259-12-1
Perfluorodecanesulfonic acid	PFDS	335-77-3
Perfluorododecanesulfonic acid	PFDoS	79780-39-5
<i>Perfluoroalkylsulfonamides</i>		
N-ethyl perfluorooctanesulfonamidoacetic acid	NEtFOSAA	2991-50-6
N-methyl perfluorooctanesulfonamidoacetic acid	NMeFOSAA	2355-31-9
Perfluorooctane sulfonamide	PFOSA	754-91-6
N-ethylperfluorooctane sulfamidoethanol	NEtFOSE	1691-99-2
N-methylperfluorooctane sulfamidoethanol	NMeFOSE	24448-09-7
N-ethylperfluorooctane sulfamide	NEtFOSA	4151-50-2
N-methylperfluorooctane sulfamide	NMeFOSA	31506-32-8

Analyte	Abbreviation	CAS #
<i>Fluorotelomer sulfonic acids</i>		
4:2 Fluorotelomer sulfonic acid	4:2 PFS	757124-22-4
6:2 Fluorotelomer sulfonic acid	6:2 PFS	27619-97-2
8:2 Fluorotelomer sulfonic acid	8:2 PFS	39108-34-4
10:2 Fluorotelomer sulfonic acid	10:2 PFS	120226-60-0
<i>Perfluoroalkyl ether carboxylic and sulfonic acids</i>		
Perfluoro-2-propoxypropanoic acid	GenX	13252-13-6
Dodecafluoro-3H-4,8-dioxanonanoic acid	ADONA	958445-44-8
9-chlorohexadecafluoro-3-oxanonane-1-sulfonate	F-53B (Major)	73606-19-6
11-chloroeicosafluoro-3-oxanonane-1-sulfonate	F-53B (Minor)	83329-89-9
Perfluoro-2-methoxyacetic acid	PFMOAA	674-13-5
Perfluoro-2-methoxypropanoic acid	PMPA	13140-29-9
Perfluoro-2-ethoxypropanoic acid	PEPA	N/A
Perfluoro(3,5-dioxahexanoic) acid	PFO2HxA	39492-88-1
Perfluoro(3,5,7-trioxaoctanoic) acid	PFO3OA	39492-89-2
Perfluoro(3,5,7,9-tetraoxadecanoic) acid	PFO4DA	39492-90-5
Perfluoro(3,5,7,9,11-pentaoxadodecanoic) acid	PFO5DoDA	39492-91-6
Ethanesulfonic acid, 2-[1-[difluoro[(1,2,2-trifluoroethenyl)oxy]methyl]-1,2,2,2-tetrafluoroethoxy]-1,1,2,2-tetrafluoro-	Nafion by-product 1	29311-67-9
Ethanesulfonic acid, 2-[1-[difluoro(1,2,2,2-tetrafluoroethoxy)methyl]-1,2,2,2-tetrafluoroethoxy]-1,1,2,2-tetrafluoro-	Nafion by-product 2	749836-20-2
2,2,3,3,4,5,5,5-4-(1,1,2,2-tetrafluoro-2-sulfoethoxy)pentanoic acid	Nafion by-product 4	N/A
Propanoic acid, 3-[1-[difluoro(1,2,2,2-tetrafluoroethoxy)methyl]-1,2,2,2-tetrafluoroethoxy]-2,2,3,3-tetrafluoro-	Hydro-EVE acid	773804-62-9
1,1,2,2-tetrafluoro-2-(1,2,2,2-tetrafluoroethoxy)ethane sulfonic acid	NVHOS	N/A

PFAS suspect list (HRMS screening)

The screenshot shows the EPA's PFAS Master List of PFAS Substances webpage. The browser address bar shows the URL https://comptox.epa.gov/dashboard/chemical_lists/pfasmaster. The page title is "PFAS Master List of PFAS Substances".

Search PFASMASTER Chemicals [Search icon]

Substring search

List Details

Description: Per- and polyfluorinated alkyl substances (PFAS) represent a growing, increasingly diverse inventory of chemicals of interest to the general public, scientific researchers, and regulatory agencies world-wide. Accompanying data-gathering, testing, and environmental monitoring exercises, in turn, have led to the publication and sharing of various lists of PFAS chemicals, some exceeding several thousand substances. A major effort was undertaken by EPA researchers within the National Center for Computational Toxicology to curate and structure-annotate several public lists in DSSTox. The below list of registered PFAS lists, from within and outside EPA, encompass PFAS of potential interest based on environmental occurrence (through literature reports and analytical detection) and manufacturing process data, as well as lists of PFAS chemicals procured for testing within EPA research programs. The consolidated list contains over 5000 PFAS CAS-name substances, with almost 4000 represented with a defined chemical structure. There is no precisely clear definition of what constitutes a PFAS substance given the inclusion of partially fluorinated substances, polymers, and ill-defined reaction products on these various lists. Hence, PFASMASTER serves as a consolidated list of substances spanning and bounded by the below lists, defining a practical boundary of PFAS chemical space (within DSSTox) of current interest to researchers and regulators worldwide. This PFAS Master List will continue to expand as component lists grow.

https://comptox.epa.gov/dashboard/chemical_lists/EPAPFASRL is an EPA research list of PFAS compiled from various internal, literature and public sources.

https://comptox.epa.gov/dashboard/chemical_lists/EPAPFASINV is a complete list of DMSO-solubilized PFAS in EPA's ToxCast inventory.

https://comptox.epa.gov/dashboard/chemical_lists/EPAPFAS75S1 list is a prioritized subset of this larger chemical inventory.

https://comptox.epa.gov/dashboard/chemical_lists/EPAPFASINSOL is a list of chemicals procured, but found to be insoluble in DMSO above 5mM.

https://comptox.epa.gov/dashboard/chemical_lists/PFASOECD is a list of PFAS chemicals in the OECD New Comprehensive Global Database.

https://comptox.epa.gov/dashboard/chemical_lists/PFASKEMI is a list of PFAS chemicals from a KEMI Swedish Chemicals Agency Report (provided by Stellan Fischer).

https://comptox.epa.gov/dashboard/chemical_lists/PFASTRIER is a list of PFAS compiled by a community effort in 2015.

https://comptox.epa.gov/dashboard/chemical_lists/EPAPFASCAT is a list of structure-based Markush PFAS categories (capabilities under development).

Number of Chemicals: 5177

5177 chemicals

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Show info: DTXSID CASRN TOXCAST [Dropdowns] [Select all]

Sort by: DTXSID [Dropdown] [Sort icon]

Filter by: Name or CASRN [Input] Hide [Dropdown]

Data analysis and dissemination

Data validation/curation

- Assessment of compound ID confidence (custom non-targeted analysis routines)
- Cross-validation of suspect screening and quantitative measurements
- Assessment of PFAS mass balance in samples

Data analysis and communication

- PFAST Team 6: Communications – will assess compound identifications and concentrations against known or suspected toxicants for risk assessment based on predicted exposure and hazard
- PFAST Team 3: Removal Performance Testing – will be provided compound ID lists for testing
- Data summaries included in reports to GA, stakeholders
- Curated and georeferenced data will be uploaded regularly to PFAST communications website.