Investigation of PFAS and other Emerging Contaminants in New Jersey Fish Tissue, Sediment, and Surface Water

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Executive Summary

Per- and polyfluoroalkyl substances (PFAS) are a large family of man-made chemicals that have been extensively used in numerous products and industrial procedures since the 1940s. PFAS can enter the environment in numerous ways, including through industrial releases, or from the discharge of Class B Aqueous Film-Forming Foams (AFFF). Releases of PFAS have also been related to aggregate sources such as wastewater discharges and leaching and emissions from landfills that have accepted PFAS in waste. New Jersey's environment has been particularly impacted by PFAS, with it being found in multiple environmental media, related to known or unknown sources. An earlier occurrence study collected single samples at eleven targeted sites in New Jersey (Goodrow et al, 2020). The current study sought to expand on the earlier study and to provide a directed investigation of several known types of sources that have not been fully characterized. The study examined five categories of potential PFAS releases that relate to AFFF, biosolid application, car washes, industrial activities, discharges of wastewater treatment plants (WWTPs). The study was intended to only provide an initial indication of potential impact from a source, as samples were generally limited to grab samples taken at a single sample collection event. Twenty-one locations were selected for investigation, with 58 subsampling locations chosen to characterize the locations. Grab samples of surface water and sediment were collected at each site and fish were collected where available, for a total of 74 surface water, 71 sediments, and 118 fish samples evaluated for this project. Samples were also collected from a sixth category, five isolated ponds in the state with limited impact from overland migration of contaminants.

Data summaries included assessing the percentage of samples above the parameter-specific detection level and concentration averages. Calculations of average concentration or other summary statistics were determined only from samples with detectable levels of the PFAS analyzed, i.e., all parameters recorded as below detection were excluded from calculations, and it was assumed that every result reported to be below the detection limit was not present in any concentration. Evaluating values below the detection limit inevitably introduces bias into an assessment (Helsel, 2012); omitting them from computations of the average or measures of central tendency and percentiles will bias the reported summary concentration high and underrepresents the sample size. Similarly, omitting values below detection when summing for a total concentration of PFAS in a sample will bias the sum low since the omitted parameter may in fact be present in the sample, albeit below detection. However, a consistent handling of the data was preferred given the goal of the evaluation was to screen the environment surrounding various activities that, in the past, have been known to lead to contamination.

Multiple perfluoroalkyl acids (PFAAs) were regularly detected in surface water, with PFOA, PFHpA, and PFHxA occurring most frequently for the perfluoroalkyl carboxylic acids (97.3, 93.2 and 93.2%, respectively) and PFOS, PFHxS, and PFBS occurring most frequently for the carboxylic acids (95.9, 86.5, and 83.8%, respectively). The dominant PFAAs in sediment were found to be PFUnA and PFDoA for the carboxylic acids (80.3 and 71.8%, respectively) and PFOS and PFDS for the sulfonic acids (95.8 and 36.6%, respectively). PFOS, a sulfonic acid, and PFDA, PFDoA, and PFTrDA were detected in a high proportion of fish tissue samples, with 100, 100, 99.2, and 99.2%, respectively.

Concentrations of PFAS at all 21 sites varied and were not generally consistent by site type. The individual grab samples of surface water collected from the streams leaving the Monmouth County Fire Training Facility, the Ashland biosolid application site, and the Ocean County Training Center had the highest total PFAS levels recorded in this study. These sites also showed among the highest levels of total PFAS in the sediment samples. Fish collected near the military base and from streams in industrial areas near wastewater treatment discharges were found to have some of the higher levels of PFAS in this study.

In addition to the evaluation of PFAS in multiple media, several other contaminants of emerging concern were investigated in samples collected for this study or leveraged from a related New Jersey occurrence study investigating levels of intersex in fish. The contaminants quantified for this study include polybrominated diphenyl ethers (PBDEs), chlorinated paraffins (CPs), pharmaceuticals and personal care products (PPCPs) and pesticides. All composite fish tissue samples contained multiple PBDEs, with the fish from the Passaic River at Elmwood Park containing a reported 10,029 ppt of total PBDEs. The data from the chlorinated paraffin analysis were difficult to review, with high detection limits and few detections. A high number of detections of PPCPs, particularly N,N-diethyl-meta-toluamide (DEET) and cocaine, indicated their well distributed presence in the environment.

This study provided a preliminary view of the levels and distribution of PFAS and other CECs in environmental media in New Jersey. Where levels have been shown to be high, additional investigation is recommended. Where a specific analyte or combination of analytes has been detected, a source investigation may be warranted.

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Introduction/Background- PFAS

Per- and polyfluorinated substances (PFAS) are used in the manufacture of useful products that impart stain resistance, water resistance, heat resistance and other desirable properties. PFAS are also used in various Aqueous Film Forming Foams (AFFF) that are used in firefighting. These substances are in wide use today, found at industrial sites that use or manufacture them and at military bases, airports and other areas known for fire-fighting activities. When released to the environment, many PFAS persist indefinitely and can travel distances from their source in surface water, groundwater, or in the atmosphere. PFAS are considered "emerging contaminants" because additional information on their presence and toxicity to ecosystems and humans continues to become available.

Previously, the Division of Science, Research and Environmental Health (DSREH) performed an initial assessment of 13 PFAS at 11 waterways across the state (Goodrow, et al., 2020). Fourteen surface water and sediment samples and 94 fish tissue samples were collected at sites along these waterways. The sites were selected based on their proximity to potential sources of PFAS and their likelihood of being used for recreational and fishing purposes.

The current study discussed herein describes a second phase of a statewide occurrence study that investigates additional potential sources and evaluates multiple areas around those potential sources to further characterize impact.

Objectives- PFAS

This project is a targeted study to determine PFAS occurrence data in environmental media collected near locations of known or suspected release of PFAS. Five general categories of potential releases were included for consideration and include Biosolid Land Application Management Areas (LLAMAs), Aqueous Film-Forming Foams (AFFF) release, industrial areas, car washes, and wastewater treatment facilities. A sixth category, ponds, was to assess potential impact to bodies of water with limited impact from overland migration of contaminants.

The criteria for inclusion in a category of this study were a reasonable assumption that site may have experienced impacts from PFAS releases and that the environmental media to be collected were accessible.

Site Selection- PFAS

The selection of 21 sites grouped into five categories are discussed below. One additional category includes samples collected from remote ponds. The categories, site identification numbers, and names are detailed in Table 1. The following text and the text discussing results will follow the order provided in the table.

Table 1: Study Categories with Site Identification, name and location

1	**Biosolid Land Application Management Areas (LLAMAs)**		**Municipality**	**County**
	Site 1	Pemberton LLAMA	Pemberton Twp.	Burlington
	Site 2	Landis LLAMA	Deerfield/Vineland	Cumberland
	Site 3	Ash Lane LLAMA	Alloway Twp.	Salem
2	**Aqueous Film Forming Foams (AFFF)**		**Municipality**	**County**
	Site 4	The Ocean County Training Center	Ocean Twp.	Ocean
	Site 5	Monmouth County Fire Academy	Howell Twp.	Monmouth
	Site 7	Somerset County Emergency Services Training Academy	Hillsborough Twp.	Somerset
	Site 8	Bergen County Electrical Fire Event	Waldwick Borough	Bergen
	Site 16	Joint Base McGuire-Dix-Lakehurst	Jackson/Manchester	Ocean
	Site 17	Atlantic City International Airport	Egg Harbor/Absecon	Atlantic
3	**Industrial Areas**		**Municipality**	**County**
	Site 9	Allgrind Plastics and Ico Plastics	Bethlehem Twp.	Hunterdon
	Site 17	Atlantic City Industrial Area	Egg Harbor Twp.	Atlantic
	Site 18	Ringwood Mines/Ford Motor Company Disposal Site	Ringwood Borough	Passaic
	Site 19	Forge Pond/Brick MUA Water Intake	Brick Twp.	Ocean
	Site 20	Deal Lake	Loch Arbour/Asbury Park	Monmouth
	Site 21	Manasquan Reservoir	Howell Twp.	Monmouth
4	**Car Washes**		**Municipality**	**County**
	Site 6	New Hanover Car Wash	N. Hanover Twp.	Burlington
	Site 10	Toms River Car Wash	Toms River Twp.	Ocean
5	**Wastewater Treatment Facilities**		**Municipality**	**County**
	Site 11	Wayne Township-Mountain View	Wayne Twp.	Passaic
	Site 12	Two Bridges Sewage Authority	Lincoln Park Boro	Morris
	Site 13	Parsippany Troy Hills Sewage Authority	E. Hanover Twp.	Morris
	Site 14	Caldwell Boro Sewage Treatment Plant	W. Caldwell Twp.	Essex
	Site 15	Wayne-Sheffield Hills	Wayne Twp.	Passaic
6	**Ponds**		**Municipality**	**County**
	Pond 1	Echo Lake	West Milford Twp.	Passaic
	Pond 3	Locustwood Pond	Cherry Hill Twp.	Camden
	Pond 4	Butterworth Pond	Woodland Twp.	Burlington
	Pond 5	Cedar Pond	Buena Vista Twp.	Atlantic

Subsampling Stations and Media Collected

Media Collection

Surface Water and Sediment

One grab sample each of surface water and sediment was collected at each of the subsampling locations within all 21 sites. The surface water was collected from six (6) inches below the surface to eliminate surface debris and to provide a consistent collection approach throughout this study. Sediment samples were collected from representative deposition areas adjacent to water sample collection locations. Each site and the related sample IDs for each subsampling location can be seen in Table 2.

Table 2: Sites and Related Subsampling IDs for Surface Water Sample Collection (See Appendix 1 for more details)

Site	Sample Collection Date	Aqueous Subsample IDs							
1	9/24/2018	LL1A	LL1B	LL1C	LL1D				
2	7/21/2021	LL2A	LL2B	LL2C	LL2D0721211042	LL2E	LL2F	LL2G	LL2H6in
3	7/21/2021	LL3A	LL3B	LL3CPOND	LL3CSW	LL4			
4	7/8/2021	04-FF1B-070821	04-FFIA-070821						
5	8/3/2021	5-FF2A	5FF2B						
6	11/1/2018	CW 1	CW 2	CW 3	CW 3.1	CW 4			
7	10/7/2019	7SFFdwn071019							
8	10/7/2019	FF 5 dwn	FF6up100719						
9	10/7/2019	9AGPdwn071019	9AGPup071019						
10	7/28/2021	10-CW6-070821	10-CW7-070821						
11	10/15/2019	POTW 11 dwn	POTW 11 up						
12	10/7/2019	POTW 8							
13	8/2/2021	13-WHIP							
14	10/15/2019	POTW 14 dwn							
15	10/7/2019	POTW 6	POTW6-15-DS	POTW6-15-UPS					
16	5/22/2019	FSW1	FSW2	FSW3					
16	8/12/2021	FTM20/Horicon 8/3/2021	HORICON 081623						
17	5/31/2023	ATLMST (4)	ATLSBA6	ATLSU3 (4b)					
17	5/31/2023	ATLPC1	ATLPC2						
18	11/2/2022	18RNG2	18RNG4	18RNG6 FR	18RNG9CNT				
19	8/12/2021	FTM022							
20	8/10/2021	FTM028							
21	8/3/2021	TSB080321							

Sediment samples were generally collected along with the surface water samples and often have similar sample IDs. Sediment subsampling station IDs can be seen in Table 3, below.

Table 3: Sites with Related Subsampling IDs for Sediment Sample Collection (See Appendix 1 for more details)

Site	Sample Collection Date	Sediment Subsample IDs							
1	9/24/2018	LL1A	LL1B	LL1C	LL1D				
2	7/21/2021	LL2A	LL2F	LL2E	LL2D	LL2C	LL2B	LL2H	LL2G
3	7/21/2021	LL4	LL3B	LL3C SW	LL3C Pond	LL3A			
4	7/8/2021	04-FF1B-070821	04-FF1A-070821						
5	8/3/2021	5FF2A	5FF2B						
6	11/1/2018	CW 1	CW 2	CW 3	CW 3.1	CW 4			
7	10/7/2019	7SFFdwn071019	7SFFup071019						
8	10/7/2019	FF5dwn100719	FF6up100719						
9	10/7/2019	9AGPdwn071019	9AGPup071019						
10	7/28/2021	10-CW6-070821	10-CW7-070821						
11	10/15/2019	POTW 11 #1 up	POTW 11 #2 dwn						
12	10/7/2019	POTW8100719							
13	8/2/2021	13-WHIP							
14	10/15/2019	POTW 14 #2 dwn							
15	10/7/2019	POTW6100719	POTW6-15-UPS	POTW6-15-DS					
16	5/22/2019	FSW1	FSW2	FSW3					
16	8/16/2023	HORICON 081623		FTM023					
17	5/31/2023	ATLSBA2	ATLSBA	ATLPC2	ATLPC1	ATLMST	ATLSU3		
18	11/2/2022	18RNG9CNT	18RNG4	RNG6FR	18RNG2				
19	8/12/2021	FTM022	FTM028						
21	8/3/2021	TSB080321							
Pond	4/24/2018	ECHO LAKE POND #1							
Pond	4/24/2018	ECHO LAKE POND #2							
Pond	4/24/2018	LOCUSTWOOD POND							

Fish Tissue

Samples of fish tissue were collected from those water bodies with fish present and where there was accessibility. Fish were collected from waterbodies adjacent to eight subsampling locations, as listed in Table 4. Fish were collected at three waterbodies around the JBMDL and one waterbody at each of the other seven subsampling locations, for a total of 10 waterbodies.

Table 4: Sites with Related Subsampling IDs for Fish Tissue Sample Collection with number of specimens collected (See Appendix 1 for more details)

Site Number	**Fish Tissue Sample ID**	**Sample Collection Date**	**Waterbody**	**Largemouth bass**	**Brown bullhead**	**Black Crappie**	**Pumpkinseed**	**Yellow Perch**	**Common carp**	**Bluegill**	**Yellow bullhead**	**Chain pickerel**	**Striped bass**	**White catfish**	**White perch**
2	FTM073	7/29/2021	Union Lake	3	3	3	3	3							
6	CW2	7/21/2021	Oakford Lake	3	3				3	3					
8	FF5	10/14/2021	Whites Lake	3					3	3					
12	POTW8	8/12/2021	Pompton River	3				3	3	3	3				
16	FSW1	10/8/2021	Impoundment above Manpaqua Brook							3		3			
16	FTM145	10/13/2021	Pine Lake		1					3		3			
16	FTM020	6/3/2021	Horicon Lake								3	3			
19	FTM022	10/26/2021	Forge Pond				3	3					3	2	
20	FTM028	10/28/2021	Deal Lake	3	2				3	3				1	2
21	FTM030	6/2/2021	Manasquan Reservoir	3						3					

Note: LMB= Largemouth bass; BBH=Brown bullhead; BC=Black crappie; PS=Pumpkinseed; YP=Yellow perch; CC= Common carp; BG=Bluegill; YBH=Yellow bullhead; CP=Chain pickerel; SB=Striped bass; WCF=White catfish; and WP=White perch

Sample Collection

Fish tissue, surface water, and sediment samples were collected in a manner described in the July 28, 2021, Quality Assurance Project Plan (QAPP), entitled, “Multimedia Investigation of PFAS and other Emerging Contaminants in New Jersey Fish Tissue, Sediment, and Surface Water.” See Appendix 2 for QAPP.

Fish Tissue

Fish tissue was collected through Direct Current (DC) electrofishing; however, other methods were used if necessary (e.g., traps, nets, trot line, or angling). Live fish that were not impacted by collection gear and that did not have skin lacerations or fin deterioration were selected. Initial efforts were intended to collect a minimum of three each of three species. Appropriate alternate species were collected if available, if initial species could not be collected. Targeted species and alternate species are detailed in the QAPP (Appendix 2).

Surface Water

At least one surface water grab sample was collected at each of the subsampling locations as per “NJDEP Field Sampling Procedures Manual", 2005. Water samples were collected from six inches below the surface to eliminate surface debris. Samples were taken in an area where water was flowing or circulating in in a well-mixed area considered representative of the waterbody.

Sample containers for water samples were provided by the contract laboratory and were in strict compliance with operating procedures that avoid cross contamination with all fluoropolymer products. For more details on the sample collection, including holding times and temperatures for surface water, please refer to the QAPP.

Sediment

At least one sediment sample was collected at each of the subsampling locations. Sediment samples were collected at a location of sediment deposition and, where possible, within the area of fish collection. Field equipment used for sediment sampling was decontaminated prior to sample collection at the initial site according to methodologies described in the QAPP and were decontaminated in between sites if needed. Sample containers for sediment samples were provided by the contract laboratory and were in strict compliance with operating procedures that avoid contamination. Holding times and temperatures are described in the QAPP.

Categories and Select Sites and Subsampling Locations

Six categories of potential PFAS contamination were the focus of this project (See Table 1). These categories included biosolid application, aqueous film forming foam (AFFF), industrial areas, car washes, wastewater treatment plants, and isolated natural ponds.

Biosolid Application

The placement of the residuals on agricultural land of those solids or liquids that originate from the treatment of sewage sludge, has been a common practice that allows for its disposal while providing nutrient enrichment to agricultural practices. These residuals, or biosolids, are tested for various contaminants before placement, but this testing does not currently require the quantification of any PFAS analytes. If PFAS is present in these biosolids, their placement onto agricultural lands has the potential to impact not only the immediate soil and vegetation, but the PFAS may migrate to groundwater and become a drinking water or irrigation contaminant, in addition to migrating to surface waters. While evaluating the potential introduction of PFAS into the environment from this type of activity could be more optimally performed with soil and groundwater investigations, this study was limited to easily accessible waterbodies in adjacent areas that could be impacted.

There are three locations in New Jersey that have been provided with a Letter of Land Application Management Approval (LLAMA) to regularly accept biosolids for placement on land. The three Land Application Management sites include Pemberton in Burlington County (Site 1), Landis in Cumberland County (Site 2), and Ash Lane in Salem County (Site 3). Figure 1, below, shows the location of these three sites.

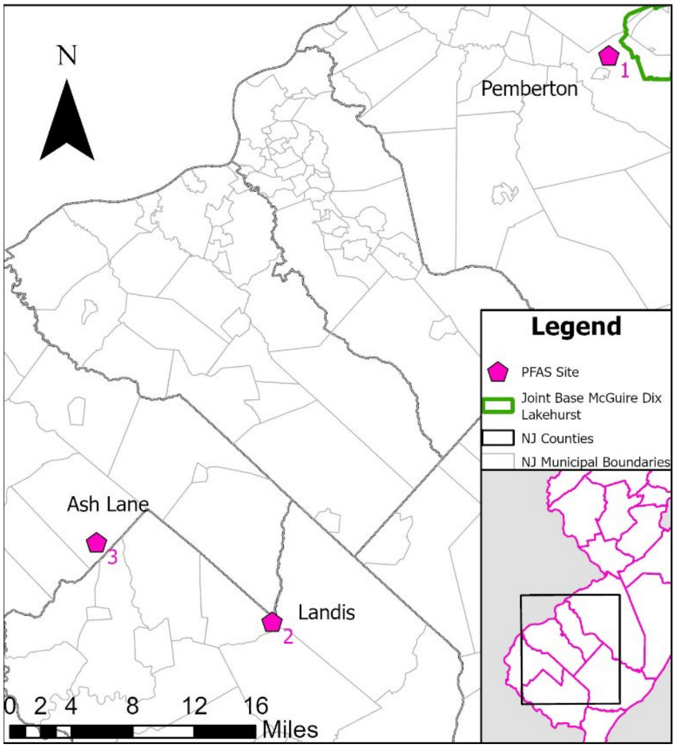

Figure 1: Three NJ LLAMA Sites with Site Numbers and Names

Biosolid Application Sites and Subsampling Locations

Pemberton (Site 1)

The Pemberton Land Application Management (a.k.a. LLAMA) site in Burlington County and the parcels that comprise the site were identified through a New Jersey Environmental Management System (NJEMS) search for land application permits. The local hydrology was evaluated, and four sampling locations were chosen to represent upstream and downstream of each the eastern and western portions of the site. See Figure 2 for the location of the four sub-sampling locations, LL1A, LL1B, LL1C, and LL1D. The site is located less than one mile from the western boundary of the Joint Base McGuire-Dix-Lakehurst. The water originating from the area on the west flows in a south-southwest direction approximately two miles before it joins the North Branch Rancocas Creek. The Budd Run tributary on the eastern portion of the site is designated a PL (Pinelands) waterway.

Refer to Figure 1, above, to see the proximity of the Joint Base to the Pemberton Land Application Management site.

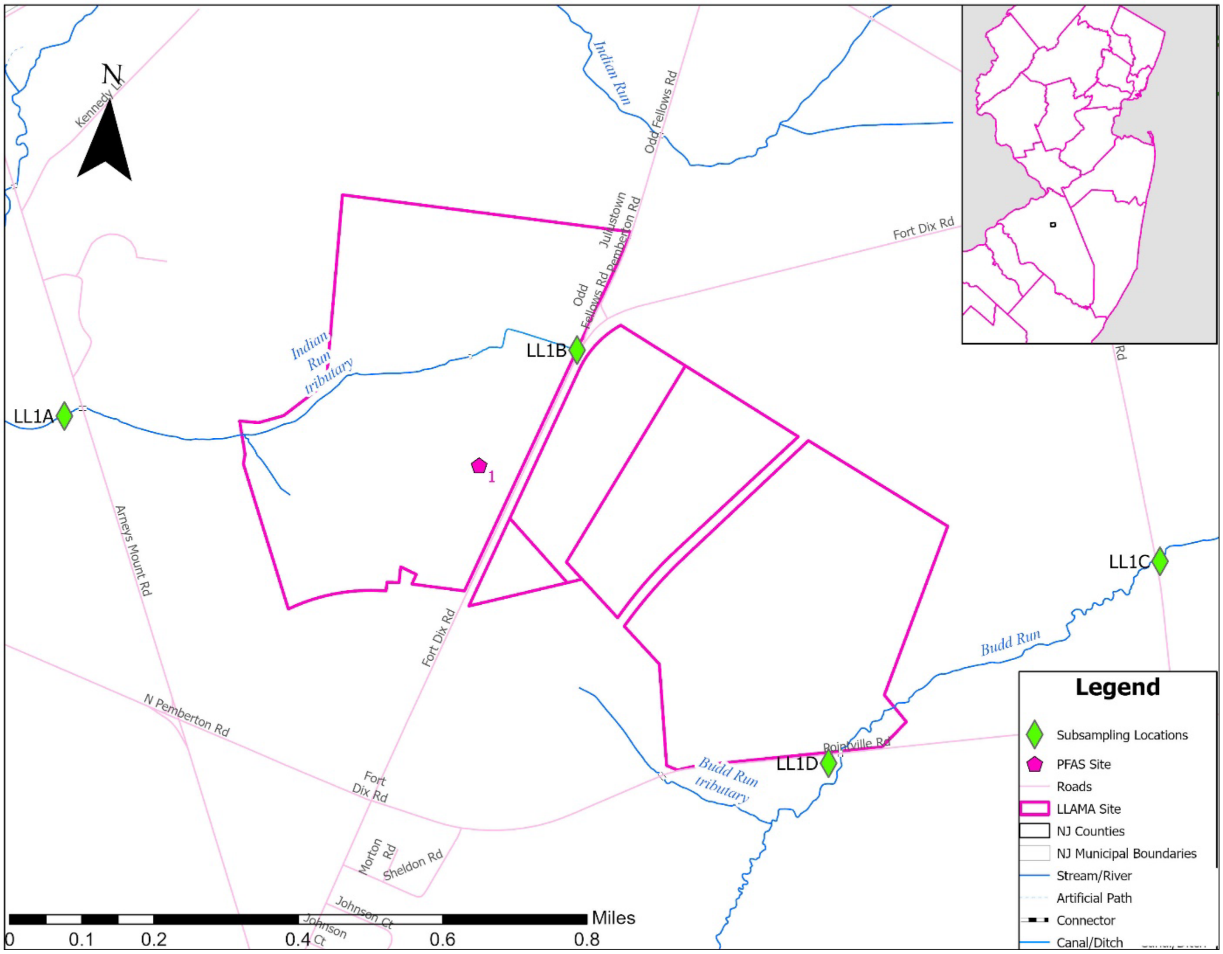

Figure 2: Site 1, Pemberton Land Application Management Site with four sub-sampling locations (LL1A, LL1B, LL1C, and LL1D)

Landis (Site 2)

The site that is used for biosolid land application by the Landis Sewage Authority straddles the Maurice River in the municipalities of Deerfield and Vineland within Cumberland County. The site is located just upstream of Union Lake in Millville (see Figure 3). In addition to the application of biosolids, the Landis Sewerage Authority is permitted for Spray Irrigation and Infiltration Percolation Lagoon- Sanitary and has had these permits modified to include monitoring of this wastewater for PFOA, PFOS, and PFNA at those locations in June 2020.

Nine surface water and sediment subsampling locations were chosen according to proximity to land application, assumed or potential groundwater to surface water interaction, and accessibility (road crossings favored). Five subsampling locations, LL2A, LL2E, LL2D, LL4, and LL2F, were positioned upgradient of any potential groundwater to surface water, or runoff, interaction. These subsampling locations were included to investigate the area that would be considered unimpacted by the application of biosolids. The three subsampling stations, LL2G, LL2C, and LL2B, are the locations expected to represent the most direct potential impact from PFAS input from the application sites. The sample collected at LL2H was to represent the water quality of Union Lake which is located downstream from the Landis application site. Fish were also collected from Union Lake.

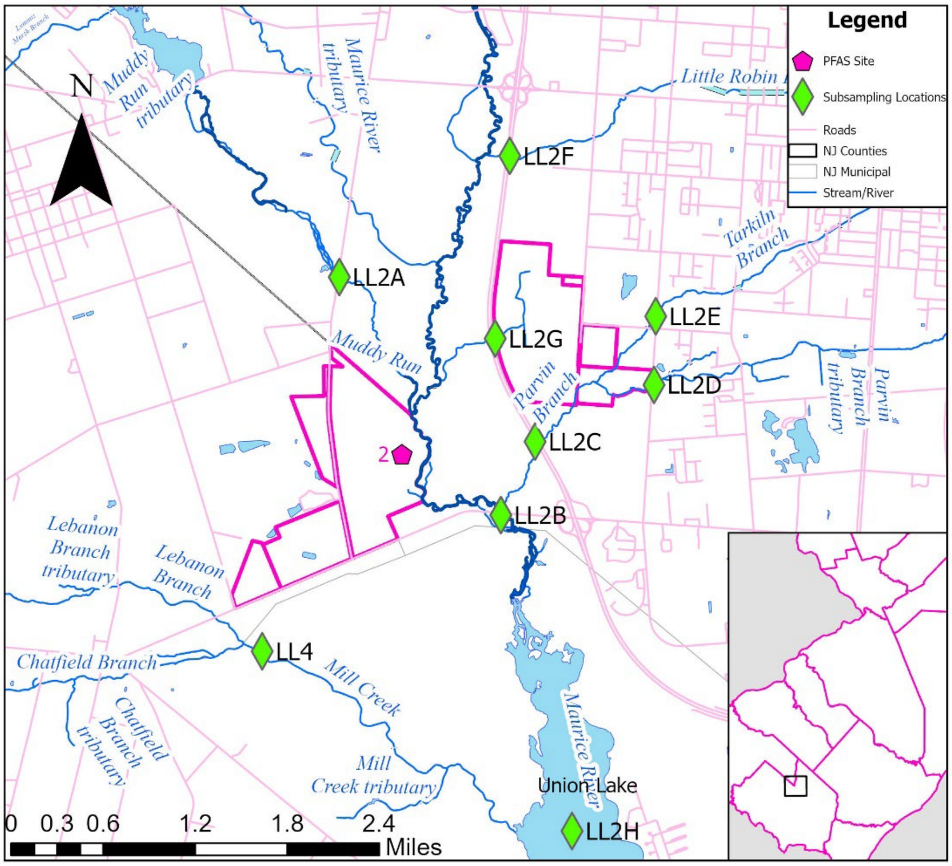

Figure 3: Site 2, Landis Land Application Management Area with nine subsampling stations (LL2A, LL2B, LL2C, LL2D, LL2E, LL2F, LL2G, LL2H, and LL4)

Ash Lane (Site 3)

The Ash Lane Farm is in Alloway Township and has been permitted to accept and land-apply Class B residual sewage sludge generated by the Pennsville, Woodstown, Salem City, and Cumberland County Utilities Authorities (See Figure *4*). The site has three subsampling locations outside of the site boundary where streams originate (headwaters). There are no mapped streams on the site, and therefore the three nearby headwaters were selected as sediment and surface water subsampling locations. These surface water and sediment sampling locations were selected to provide information on the potential groundwater exchange with the nearby surface water and could potentially include contributions from surface water runoff. The three subsampling locations can be seen in the figure below: LL3B located on Deep Run to the northwest, LL3A located on a tributary to Deep Run to the southwest, and LL3CSW located on a tributary to the Cohansey River directly east of the site. LL3CSW was collected at a road crossing downstream from an impoundment that would likely have some interaction with the groundwater flow from the site but also collects runoff from the site and the neighboring farm.

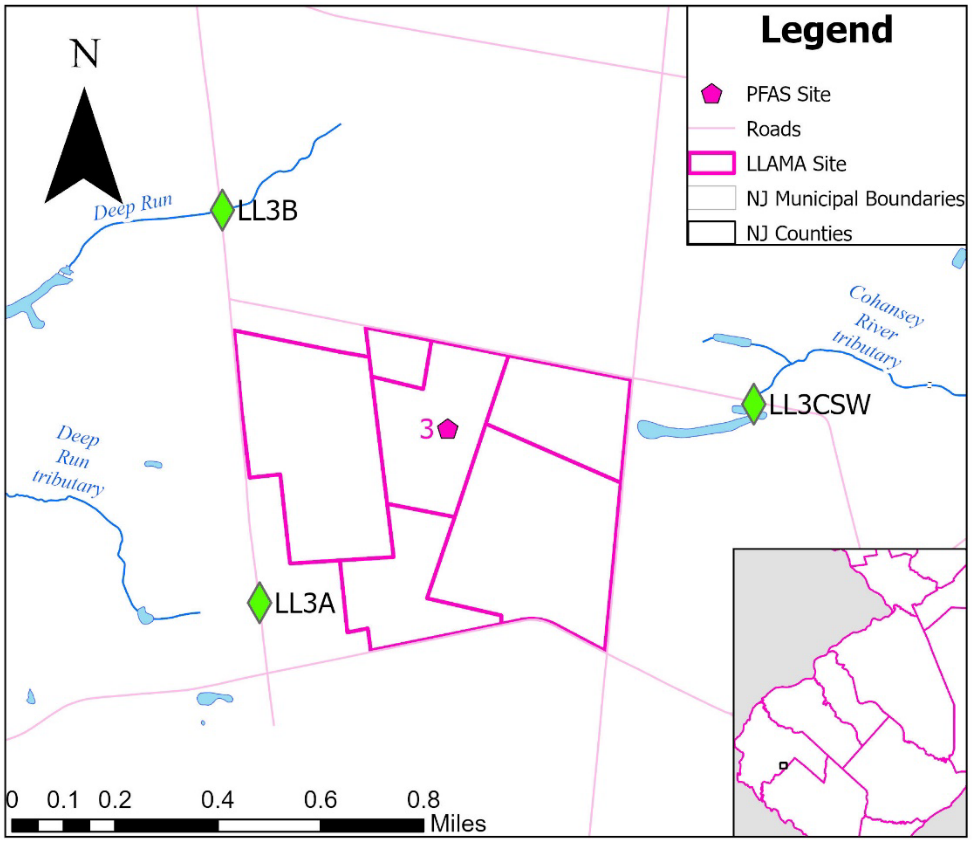

Figure 4: Site 3, Ash Lane Land Application Management Area with subsampling locations LL3A, LL3B, and LL3CSW

Aqueous Film-Forming Foam

Class B Aqueous Film-Forming Foams (AFFF) have been a significant tool to fight large scale hydrocarbon fires and have been widely used by the military, as well as by industrial and public organizations. Much of the valuable properties attributed to these foams are imparted by fluorochemical surfactants that contain mixtures of PFAS, many of which are considered proprietary. Large scale releases of AFFF have occurred over decades through firefighting training activities and uncontrolled spills. PFAS from the AFFF that is released largely impact soils and ultimately migrate to groundwater and surface waters, impacting drinking water and consumable fish tissue.

This study included an initial investigation of the areas surrounding three county fire training academies and addition samples collected from two areas near federal installations.

Three county fire training academies, Ocean County, Monmouth County, and Somerset County Fire Training Academies, were selected out of a list of 19 county firefighting training academies identified statewide. There was no report of PFAS previously being detected in these areas and no discharges reported from these sites. Their location adjacent to surface water bodies increased their prioritization for selection for review.

The first federal installation, the Joint Base McGuire-Dix-Lakehurst, has ongoing investigations by the EPA and the Navy that show the impacts of AFFF releases and was included in the earlier PFAS occurrence study in New Jersey (Goodrow et al., 2020). The second federal installation, the Atlantic

City Federal Aviation Administration's William J. Hughes Technical Center, has had past activities that have led to the discharge of AFFF. The Federal Aviation Administration (FAA) is working with EPA and the Formerly Used Defense Sites (FUDS) Military Munitions Response Program (MMRP) to assess the issues on site.

Aqueous Film-Forming Foam Sites and Subsampling Locations

Ocean County (Site 4)

The Ocean County Training Center is located south of Oyster Creek, slightly over one mile west of Barnegat Bay and upgradient to Fresh Creek and Waretown Creek, both of which discharge to the Barnegat Bay (See Figure 5). Samples of surface water and sediment were collected at the headwaters to the Fresh Creek in the north (FF1A), and on the Waretown Creek by Route 9 (FF1B), south of the center.

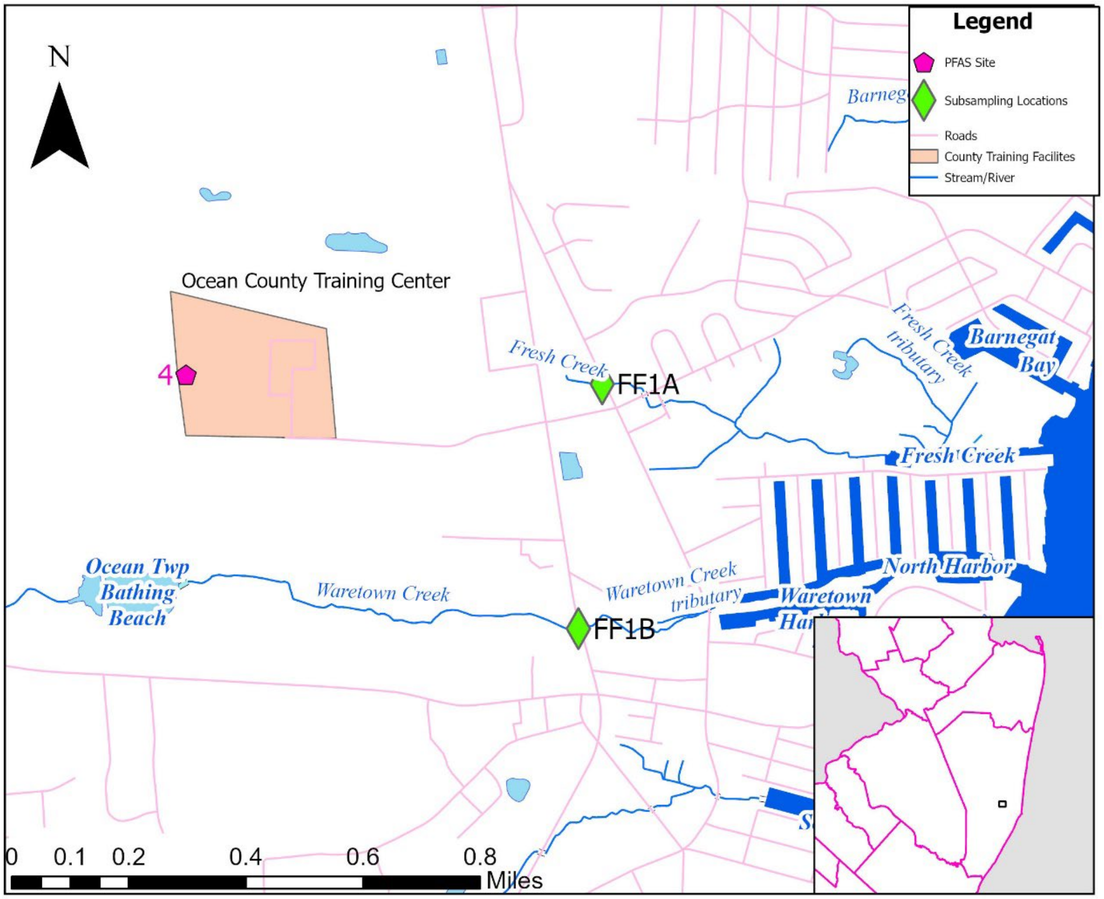

Figure 5: Site 4, Ocean County Training Center

Monmouth County (Site 5)

The Monmouth County Fire Academy is located in central Monmouth County on Route 33 in Howell. Two tributaries, the East Fork Yellow Brook and the West Fork Yellow Brook, run through the site, flowing into the main branch of the Yellow Brook at a small distance downstream (See Figure 6). The Yellow Brook discharges to the Manasquan River nearly two miles downstream.

Water and sediment samples were collected from the West and East Fork tributaries immediately after flowing through the county site. The sample collected from the West Fork is 5FF2A and the sample collected from the East Fork is 5FF2B.

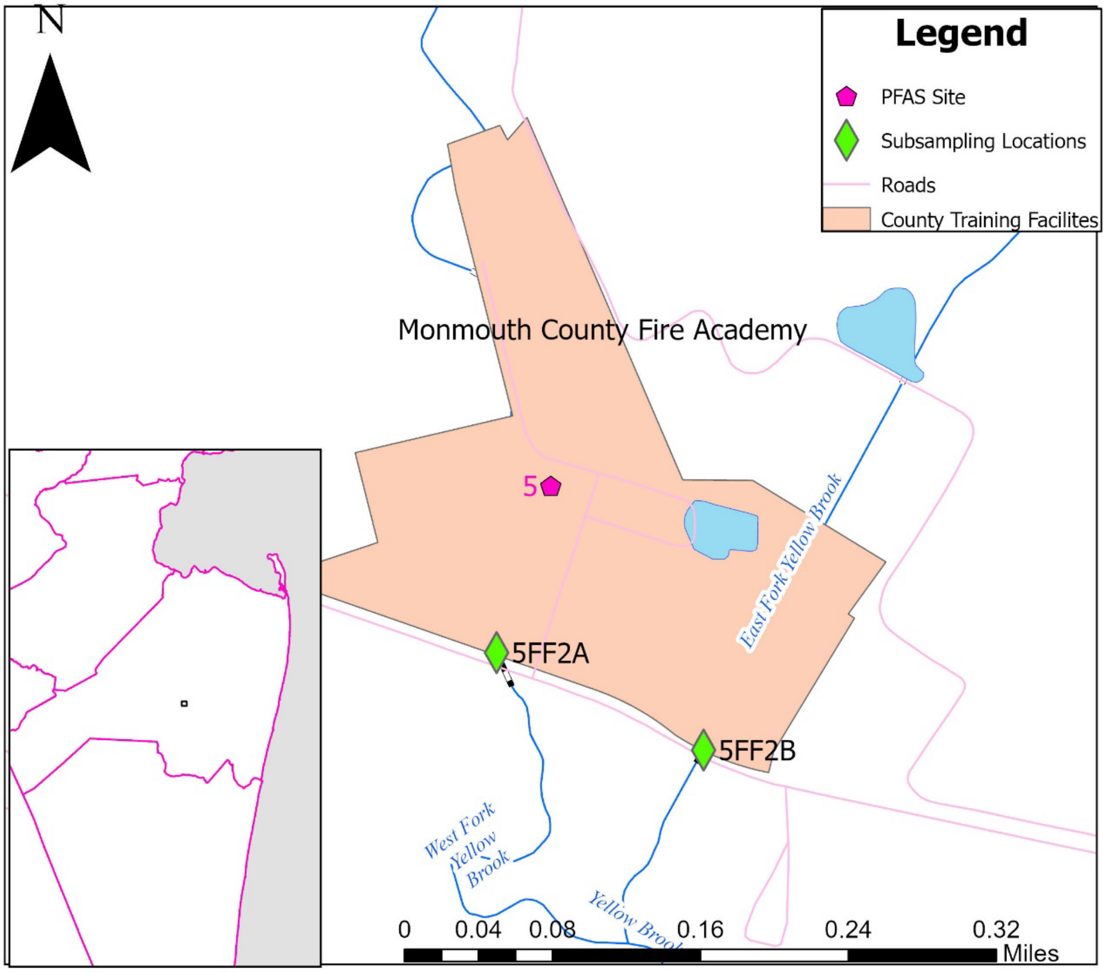

Figure 6: Site 5, Monmouth County Fire Academy with subsampling locations 5FF2A and 5FF2B.

Somerset County (Site 7)

The Somerset County Emergency Services Training Academy is in central Somerset County, just south of the Dukes Brook tributary to the Raritan River that runs through Duke Farms and along Dukes Parkway West (See Figure 7).

Access was difficult, and only a single surface water and sediment sample (7SFF) was collected from the Dukes Brook tributary where it flows under Roycefield Road at Dukes Parkway West. The direction of flow is from the west to the east-northeast where the Dukes Brook tributary joins the Raritan River less than one mile downstream. Therefore, the subsampling location is upstream of a tributary originating on the county site, but there may be potential groundwater exchange.

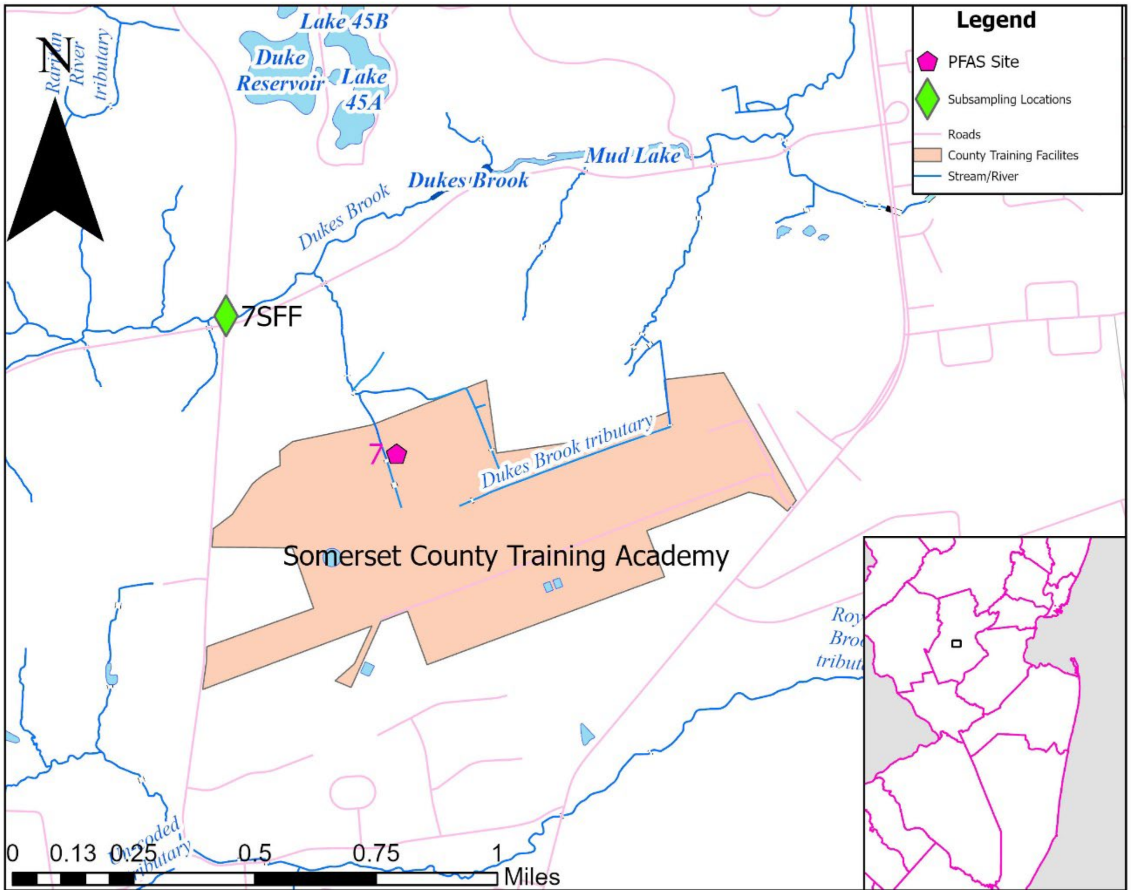

Figure 7: Site 7, Somerset County Emergency Services Training Academy with single subsampling location, 7SFF.

Joint Base McGuire-Dix-Lakehurst (Site 16)

In a previous NJDEP study on PFAS in fish tissue, sediment, and surface water (Goodrow et al., 2020), four waterways were sampled south and east of the Joint Base McGuire-Dix-Lakehurst. These waterways included Little Pine Lake and Mirror Lake to the south, and Horicon Lake and Pine Lake to the east. As a part of the current study, the intention was to capture the character of a waterbody not included in the first phase, which included an impoundment located immediately adjacent to the base and upstream of Manapaqua Brook tributaries, FSW1 (See Figure 8). Samples of surface water, sediment, and fish tissue were collected from this impoundment. Surface water and sediment were also collected at two subsampling locations along the Ridgeway Branch running along the northern boundary of the base to the east (FSW2 and FSW3).

In addition to subsampling locations FSW1, FSW2, and FSW3, as a follow up to the initial NJDEP PFAS statewide occurrence study (Goodrow et al., 2020), and as a complement to the statewide fish tissue monitoring program, fish, surface water, and sediment samples were collected from Horicon Lake (16-X3). Fish tissue samples were also collected at Pine Lake (location 16-X1) as part of the statewide fish tissue monitoring program and discussed herein.

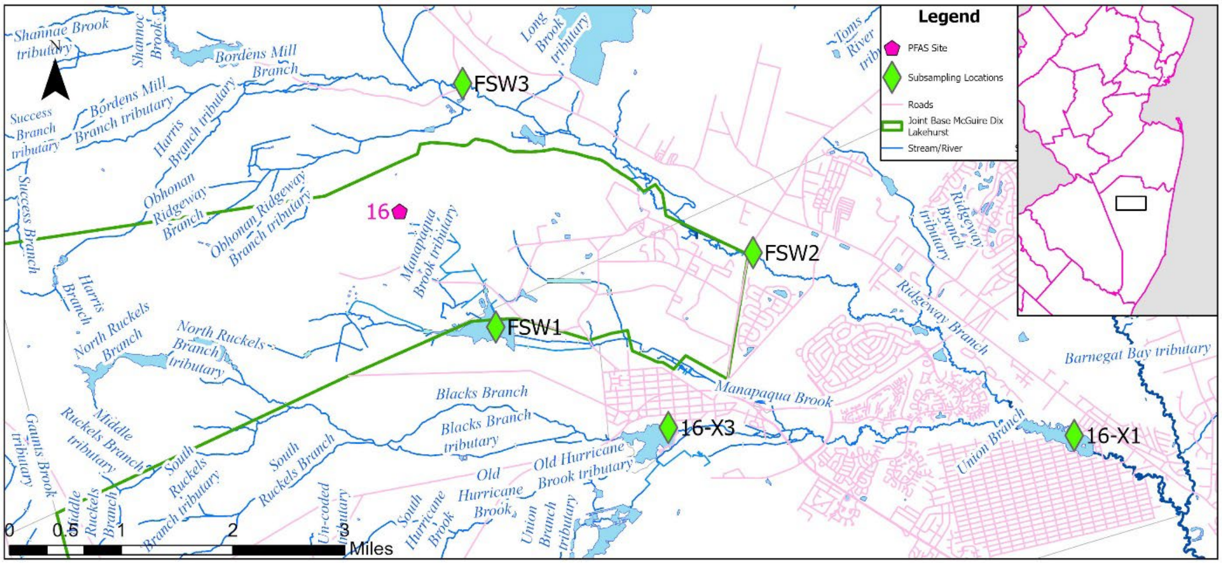

Figure 8: Site 16, Joint Base McGuire-Dix-Lakehurst and five subsampling locations, FSW1, FSW2, FSW3, 16-X3, and 16-X1

Atlantic City International Airport and Industrial Area (Site 17)

The Atlantic City International Airport is a shared civil and military airport located in Egg Harbor Township near the border with Galloway Township. The facility is located upgradient of the Atlantic City Reservoir and the stream to which the reservoir discharges, Absecon Creek. It is located approximately 10 miles from where the Absecon Creek empties into the Absecon Bay, before the barrier island that includes Atlantic City.

Restrictions protecting the reservoir limited sampling options. An upstream sample on the South Branch Absecon Creek near County Road 604 (ATLSU3) was collected for surface water and sediment analysis. A second sample of both surface water and sediment were collected downstream after the reservoir discharges to Absecon Creek by Route 30/Absecon Blvd (ATLMST). One sample of surface water only was collected from the connection between the upper and lower reservoir near the crossing with the Garden State Parkway (ATLSBA6)

Two additional samples were collected in an area of an industrial facility, located slightly over four miles south-southwest of the FAA Atlantic City Airport (ATLPC1 and ATLPC2). These subsampling locations can be seen in Figure 9 and the results are discussed in the Atlantic City (Site 17) section in Site Results.

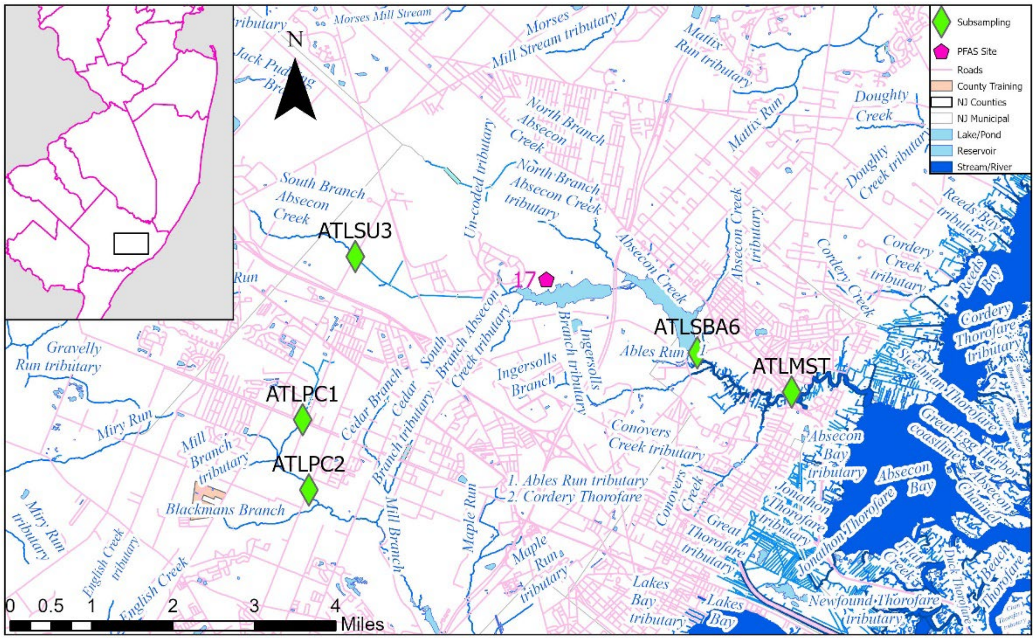

Figure 9: Site 17, Atlantic City International Airport and Industrial Area, with subsampling locations ATLSU3, ATLSBA6, ATLMST, ATLPC1, and ATLPC2.

Bergen County Electrical Station Fire (Site 8)

In 2015, there was a major transformer fire and spill of dielectric fluid (confirmed to be non-PCB containing fluid) where 15,000 gallons of AFFF was used for fire suppression, as reported to the NJDEP (NJDEP Communication Center No. 15-09-13-1237). The runoff AFFF is reported to have entered the Smokis Voll Brook, a tributary of Hohokus Brook in Waldwick, NJ.

The Smokis Voll Brook is an ephemeral stream and did not have notable flow during sample collection. Therefore, samples were collected from Whites Pond, an impoundment upstream of the confluence with the Smokis Voll Brook tributary (FF6), and from a location downstream of the confluence at Wyckoff Avenue (FF5). Fish tissue was collected at Whites Pond and will be discussed here (Figure 10).

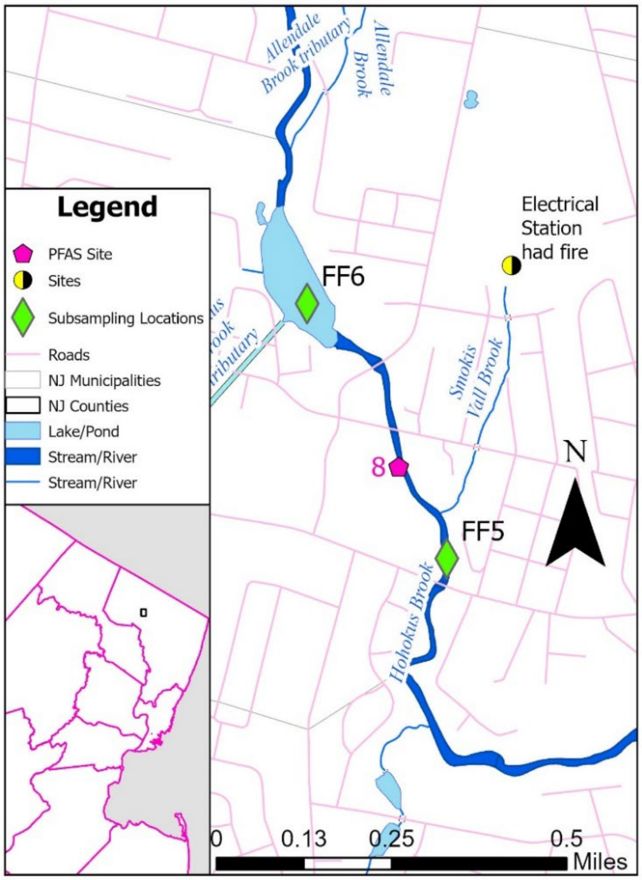

Figure 10: Site 8, Fire Event with discharge of AFFF to Smokis Voll Brook, a tributary of Hohokus Brook

Industrial Areas

Attempts have been made to identify potential PFAS release facilities by SIC or NAICS codes, or by some identifying characteristic. These methods have pros and cons, as they may capture relevant facilities, but may also easily identify a facility with no releases or even with no use or manufacture of a PFAS product. Several industrial sites identified by this method were selected for evaluation here. This selection included sites located near facilities with known or suspected PFAS releases, as well as sites that are in urbanized settings with potential localized or regional sources.

Industrial Areas Sites and Subsampling Locations

Allgrind and Ico Plastics (Site 9)

Included in a broad interpretation of the SIC/NAICS codes, two facilities in Bethlehem Township in Hunterdon County were identified as potential sources. These two companies, Allgrind Plastics and Ico Plastics, are located along a tributary to the Musconetcong River (See Figure 11). No releases of PFAS have been reported from these sites.

Sediment and surface water samples were collected at two subsampling locations- both on the Turkey Hill Brook tributary to the Musconetcong, one upstream of the two facilities, where it passes under Brunswick Pike (AG_up), and the other downstream, after the location of the two facilities (AG_dwn).

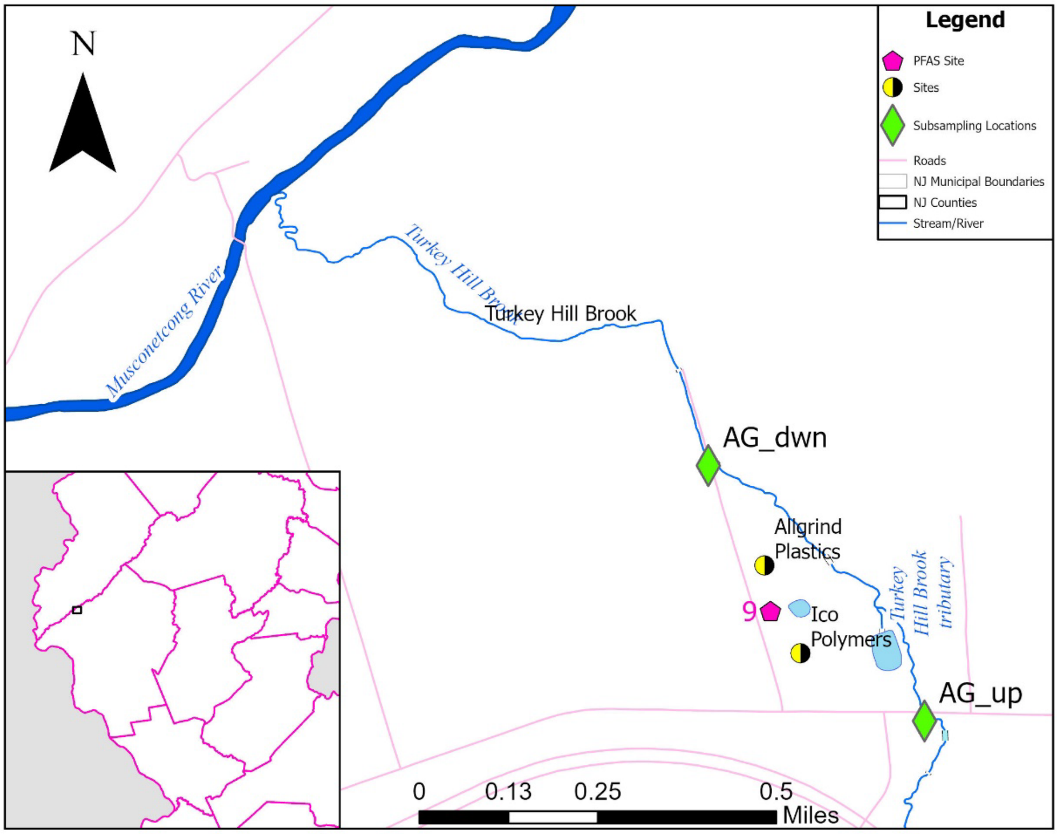

Figure 11: Site 9, Bethlehem Township Industrial Area with subsampling locations AG_up and AG_dwn

Atlantic City Industrial Area (Site 17)

South of the Atlantic City Airport and the Absecon Creek, there is an industrial area with plastic container manufacturing on the Mill Branch tributary to the Patcong Creek. No releases of PFAS have been reported at either of these facilities. Two subsampling stations, ATLPC1 and ATLPC2, were selected to characterize the upper reaches of this stream. A county fire training facility is also located in the area, but subsampling locations were not optimized in this study to understand its potential impact. See Figure 9 for details on this area.

Ringwood Mines/Ford Motor Company Disposal Site (Site 18)

The northernmost area included in this study, Site 18, is in Ringwood Boro in Passaic County. Situated north of the Wanaque Reservoir, this area is an active remediation site, so sampling locations were limited (Figure 12).

Three subsampling locations were selected upstream of the reservoir- Ringwood Creek Tributary at Peter’s Mine Road (RNG2), further downstream on Ringwood Creek Tributary at Boro Parkway (RNG4), and on Ringwood Creek after discharge from Ringwood Mill Pond (RNG6FR, at Farm Road). A final subsampling location, to the east of the lower Wanaque River, RNG9CNT (at Chestnut) was possibly connected to a direct discharge from the reservoir, but exact connections were difficult to ascertain in the field.

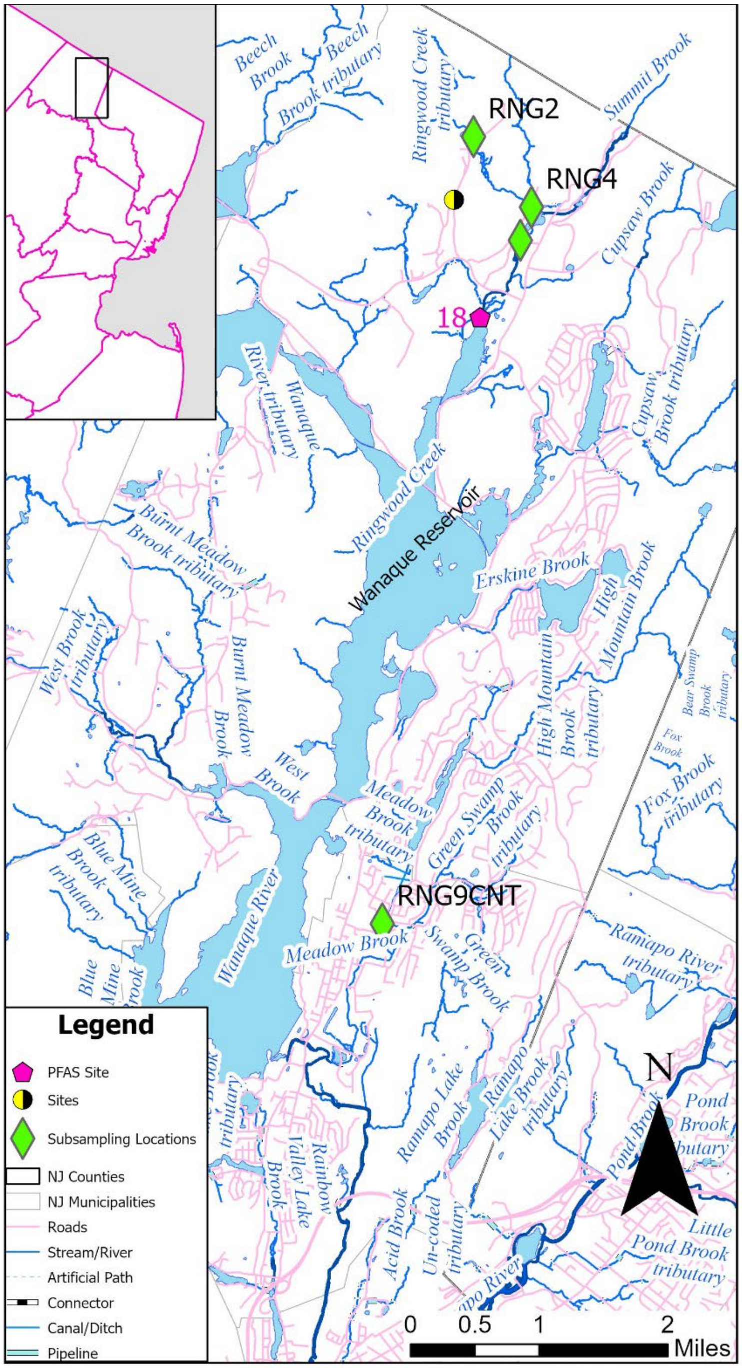

Figure 12: Site 18 Ringwood Mines/Ford Motor Company Disposal Site with subsampling locations RNG2, RNG4, RNG6FR, and RNG9CNT

Forge Pond/Brick MUA Water Intake (Site 19)

Forge Pond is an impoundment in Brick Township (Figure 13), Ocean County, that includes a drinking water intake for the Brick Municipal Utilities Authority (Brick MUA). After finding elevated levels of PFOA in their intake, a track down study was initiated by the Brick MUA in cooperation with NJDEP (Procopio et al. 2017). During an iterative sampling process, the source of the PFAS was found to be a small industrial facility that used products containing PFAS.

Fish were collected in Forge Pond as a part of the statewide routine monitoring effort. Surface water and sediment samples were collected as a part of this study to complement routine monitoring and to evaluate the current condition of the area.

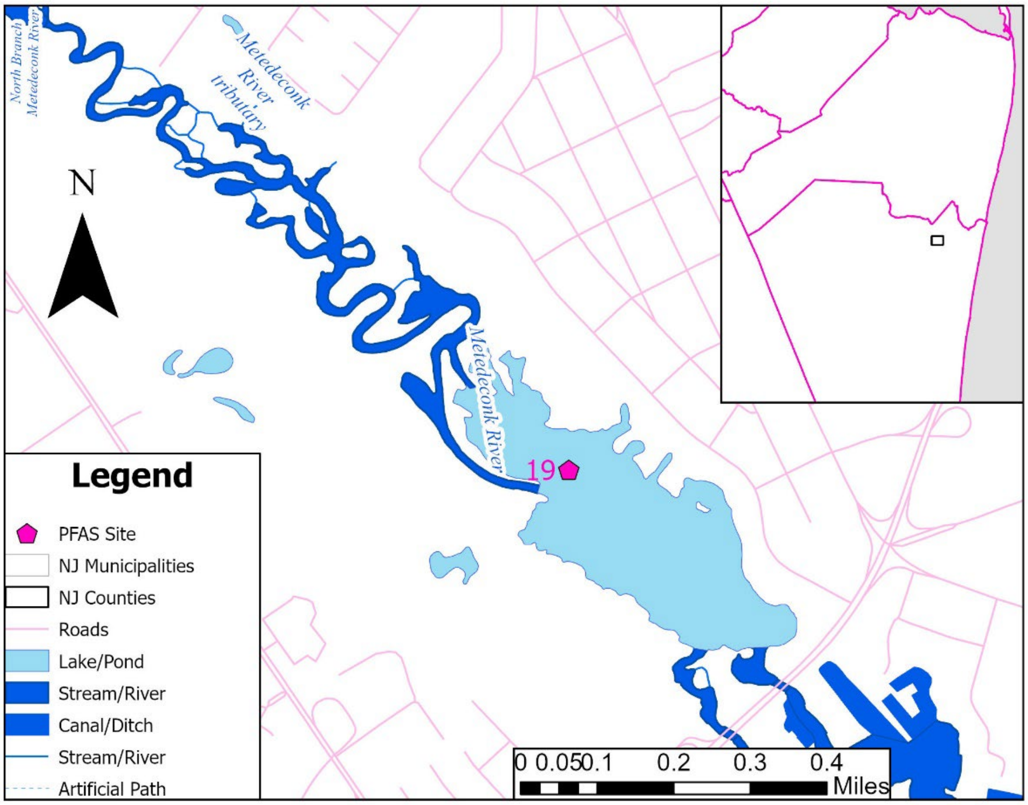

Figure 13: Site 19, Forge Pond

Deal Lake (Site 20)

Deal Lake is a man-made impoundment discharging to the ocean in Ocean County, NJ (Figure 14). Deal Lake has experienced water quality issues related to stormwater runoff and is included in the statewide routine monitoring efforts. Surface water and sediment were collected along with the fish tissue as a complementary effort to characterize PFAS in this system.

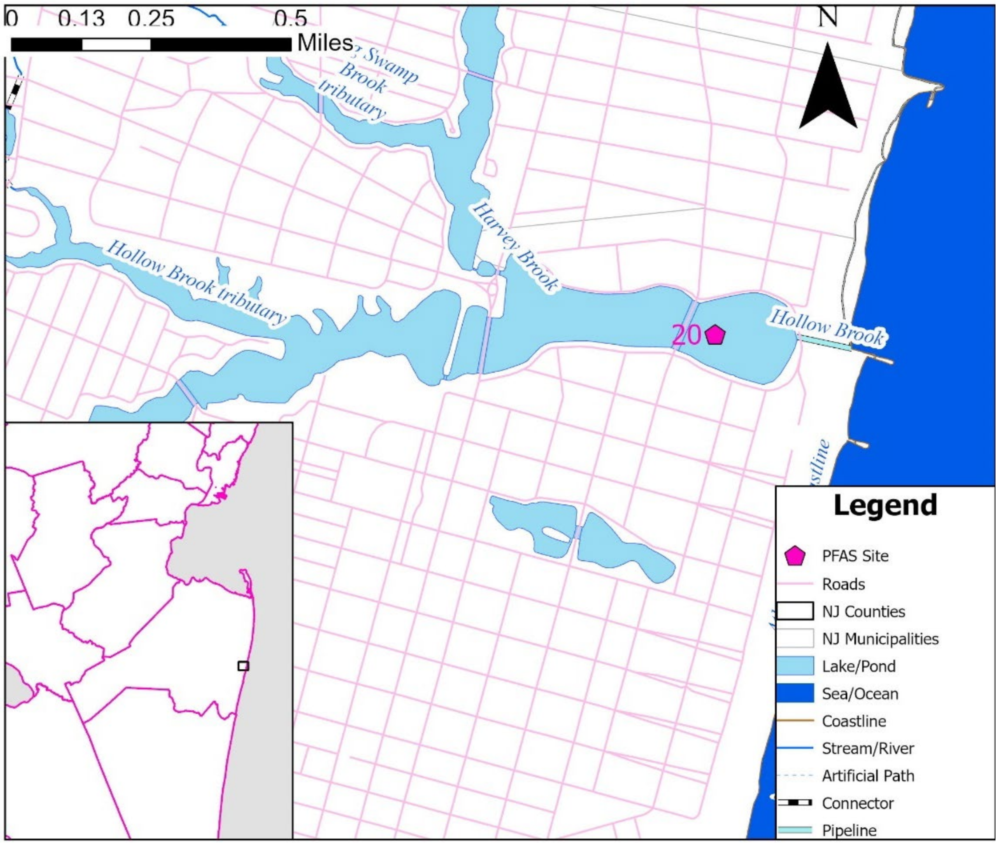

Figure 14: Site 20, Deal Lake in Ocean County, NJ

Manasquan Reservoir (Site 21)

The Manasquan Reservoir is a large drinking water reservoir in Monmouth County, NJ (Figure 15). Included in the statewide routine fish monitoring program, samples of surface water and sediment were collected to compliment the analysis of the fish tissue and to characterize the site's PFAS signature.

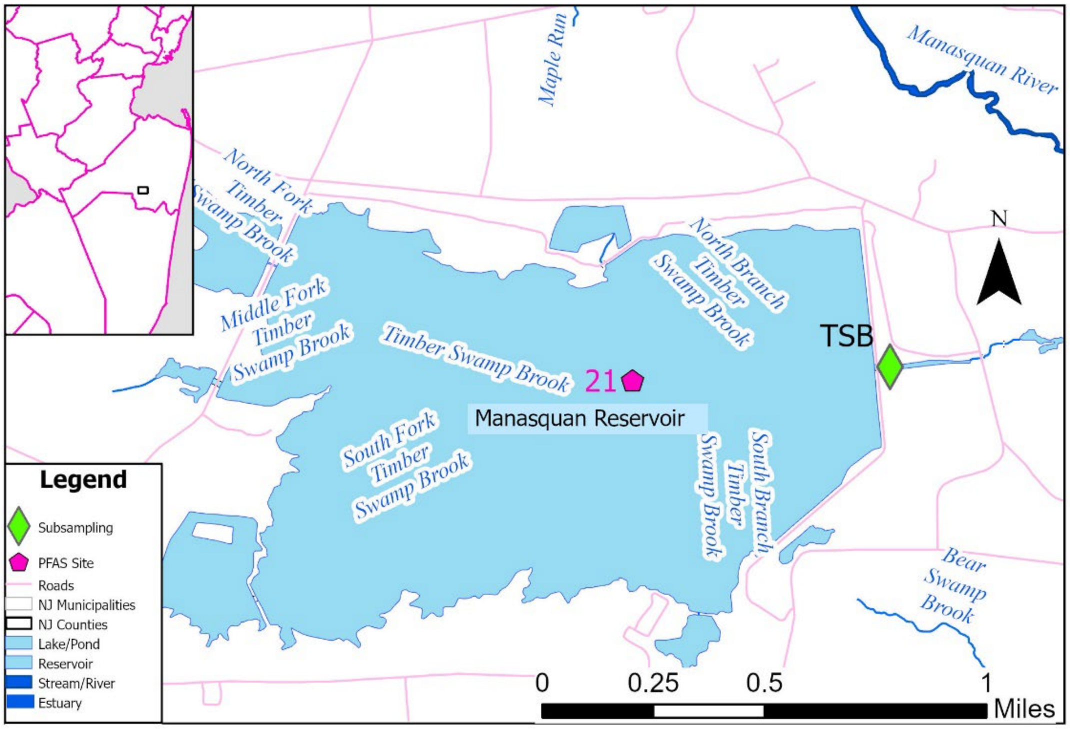

Figure 15: Site 21, Manasquan Reservoir

Car Washes

Two areas in the state were selected to represent potential discharges from commercial car washes. The first site, Site 6, is proximate to a car wash facility formerly known as the Tiny Bubbles Car Wash. The car wash is in New Hanover Township in Burlington County, just north of the Joint Base McGuire-Dix-Lakehurst. Tiny Bubbles was a car wash that had reportedly the only discharge to groundwater permit from the DEP. The second site, which is in Toms River (Site 10), is an area near a commercial car wash known as Surf City Car Wash. Suds were reported where the stream that flows near the car wash ultimately discharges to Barnegat Bay.

Car Wash Sites and Subsampling Locations

New Hanover Car Wash (Site 6)

The commercial car wash located in New Hanover, previously known as “Tiny Bubbles,” is currently operating under a new name. The Tiny Bubbles facility at 245 Cookstown-New Egypt Road, operated with a Discharge to Groundwater Permit for several years. This direct discharge to groundwater had the potential to release surfactants, potentially containing PFAS, to the groundwater. If PFAS release was substantial and had enough time to migrate to the surface water, it was anticipated that PFAS would be evident in samples collected in Oakford Lake and downstream.

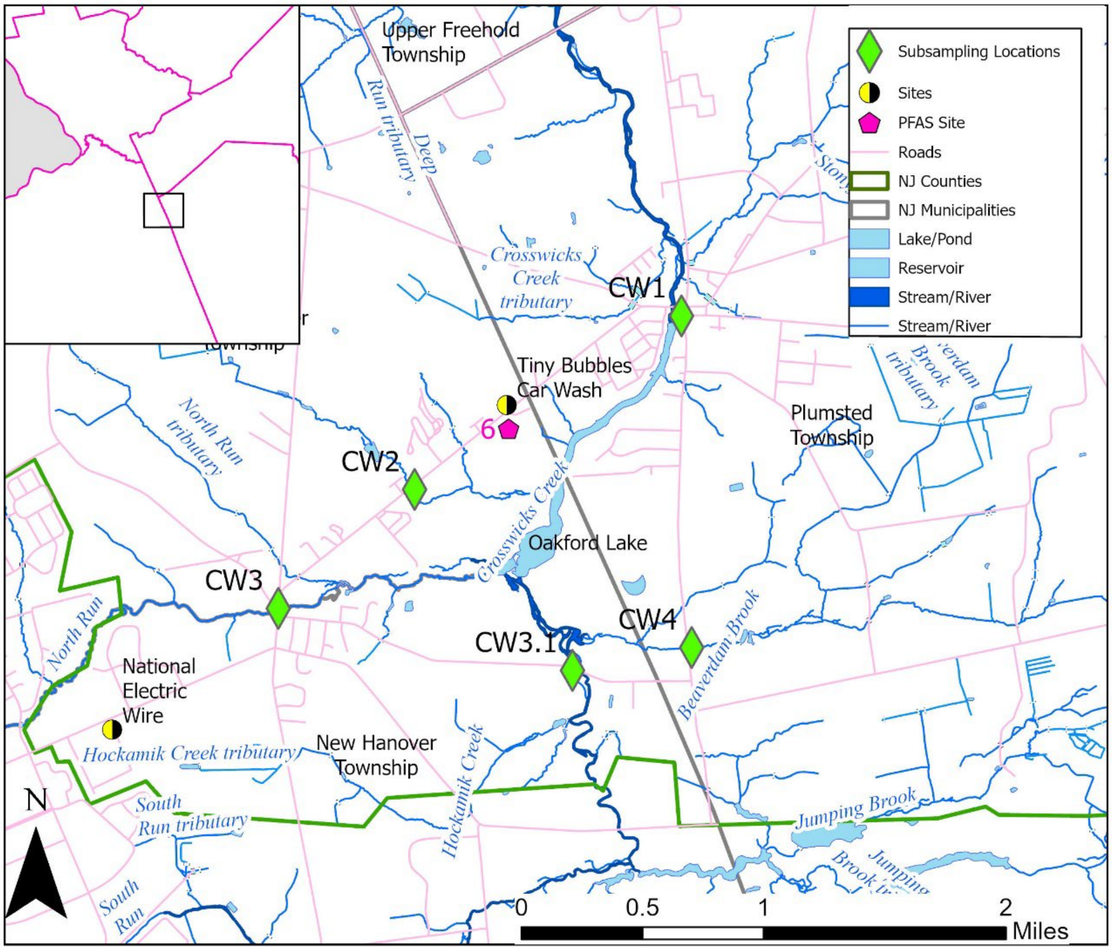

Figure 16: Site 6, New Hanover Car Wash

This area of interest was characterized by the selection of five subsampling locations where samples of surface water and sediment were collected. In addition, fish were collected from an impoundment known as Oakford Lake, where four of the subsampling stations are discharged and one is located at the outlet. The car wash and three of the upstream subsampling locations are located in New Hanover in Burlington County, and one upstream subsampling station and the lone downstream sampling location are in Plumstead/New Egypt Township in Ocean County.

Samples collected at subsampling stations (CW1, CW2, CW3, CW3.1, and CW4) used to represent the potential impact on this area can be seen in Figure 16. The four upstream subsampling stations, CW2, CW3, CW3.1, and CW4, are all tributaries to Oakford Lake. CW1 represents the outlet of Oakford Lake.

The upgradient/upstream subsampling locations were selected to represent the groundwater contributions to the surface water before any potential impact from the car wash. While subsampling locations could have been optimized near and downstream of the car wash location, the stations that were included allowed for evaluation of not only the car wash, but also the potential contribution from PFAS potentially migrating from the Joint Base. Note that subsampling station CW3.1 was collected from the Jumping Brook, a tributary originating on and traversing land area included within the Joint Base McGuire-Dix-Lakehurst. The results from this subsampling location and potential impacts to Oakford Lake at subsampling station CW1 are discussed in the Car Wash section and are also cross referenced in the AFFF section where the JB MDL is discussed.

Toms River Car Wash (Site 10)

The presence of suds in the water in this area was reported to NJDEP during the planning phase of this project, many months before sample collection took place. Given the presence of a car wash upstream from where the suds were observed, this site was included for evaluation. Three subsampling locations were selected to assess the area around Site 10 which includes the Surf City Car Wash in Toms River. The three subsampling locations for Site 10 included CW6, which is located upgradient of the car wash, CW7 located on the stream just downgradient of the car wash, and CW5 located at the outlet of the stream to Barnegat Bay (Figure 17). Surface water and sediment samples were collected for the two upstream subsampling locations, and surface water only was collected from the subsampling station located on the bay due to lack of cohesive sediments. No fish were collected for this site.

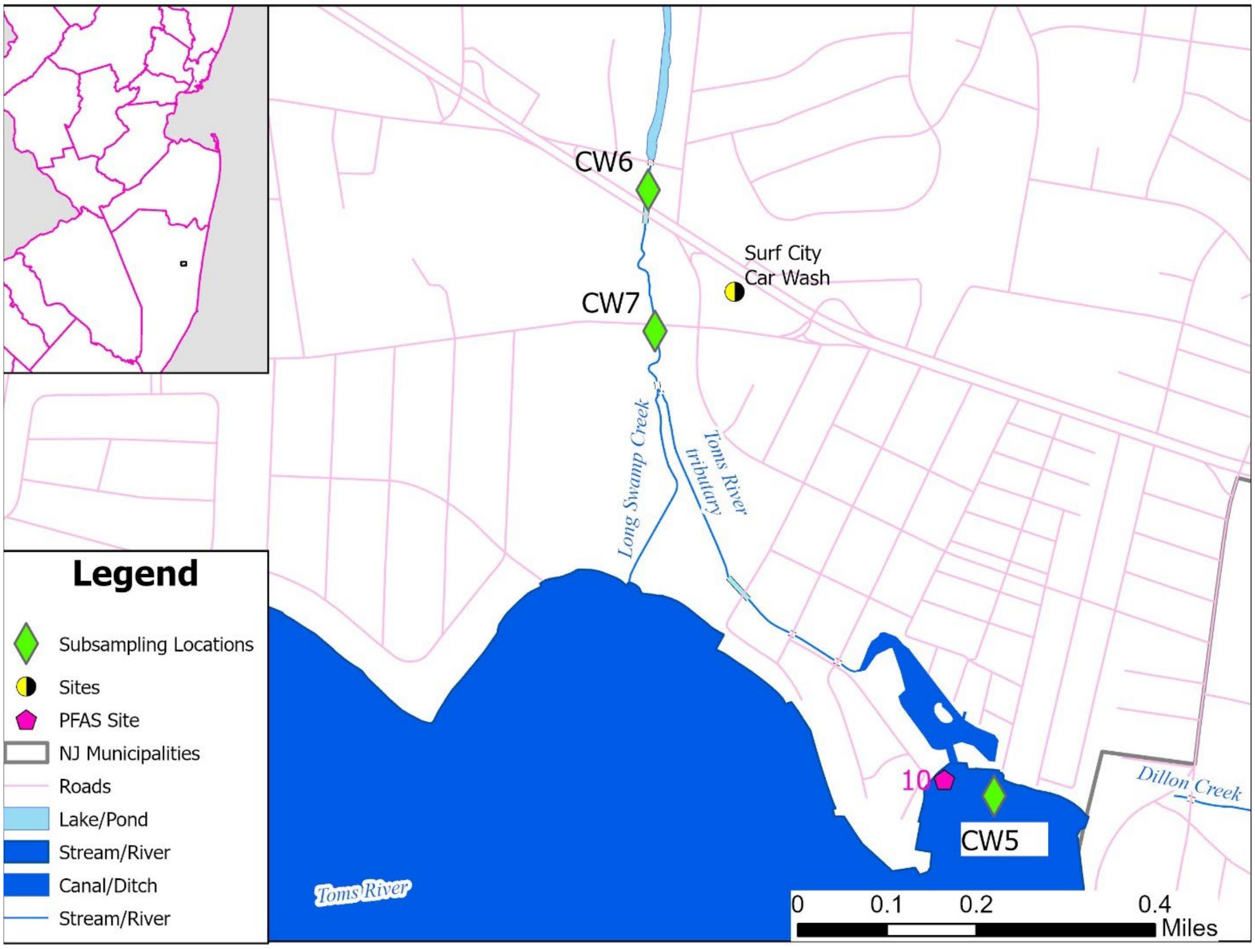

Figure 17: Site 10, Toms River Car Wash

Wastewater Treatment Plants

The discharge of treated domestic and industrial wastewater have been characterized as sources of PFAS to the environment. While not the original source, wastewater treatment facilities could aggregate various sources of PFAS from domestic or industrial sources and discharge PFAS contaminated water when treatment does not adequately remove these chemical compounds.

A selection of five areas where domestic wastewater treatment facilities discharge to surface water was a part of this investigation (Figure 18). The surface water discharge points for these facilities (major discharge points) and other major and minor permitted discharge points[1] are located on streams in Morris, Essex, and Passaic Counties and are shown in Figure 18. While wastewater treatment plants are located throughout the state, these closely located plants were selected as an initial evaluation to represent typical wastewater discharges and to maintain sampling efficiency. The five main sites include Wayne Township-Mountain View (Site 11), Two Bridges Sewage Authority (Site 12), Parsippany Troy Hills Sewage Authority (Site 13), Caldwell Boro Sewage Treatment Plant (Site 14), and Wayne-Sheffield Hills (Site 15). These locations can be seen in Figure 18. Access to streams adjacent to discharge locations was difficult and limited optimal sampling strategies.

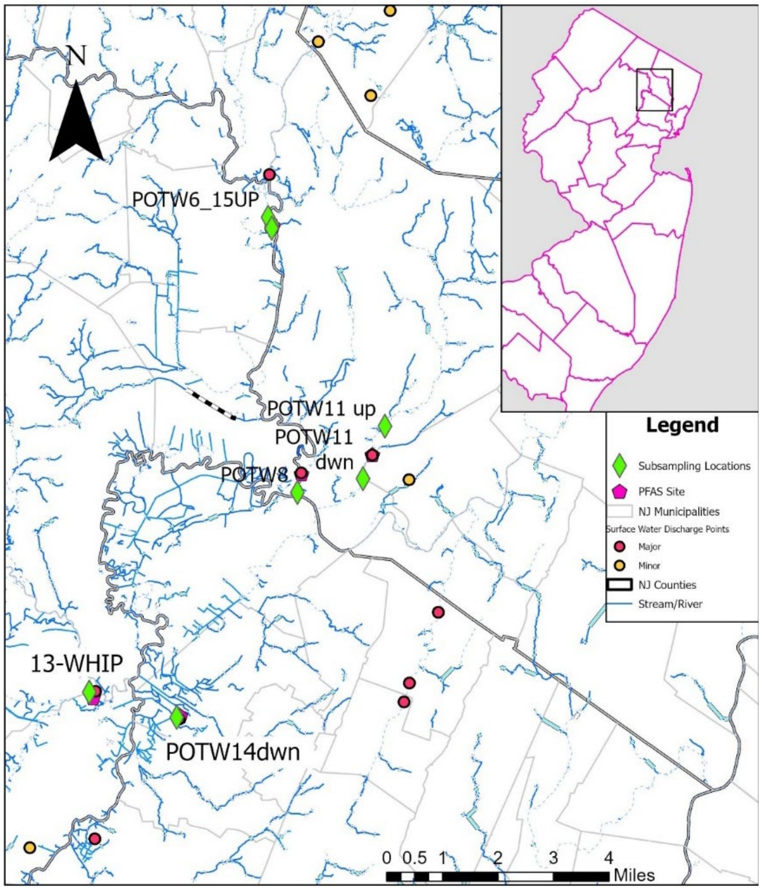

Figure 18: Wastewater Treatment Plant Locations

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[1] Major and minor surface water discharges are regulated under the New Jersey Pollutant Discharge Elimination System (NJPDES) program. Major discharges are generally larger and often require an individual permit, while minor discharges are typically smaller and often covered under a general permit.

Wayne Township-Mountain View (Site 11)

Wayne Township – Mountain View STP has a major discharge point to Preakness Brook east of West Belt Parkway in Wayne. Sediment and surface water samples were collected at road crossings upstream (POTW11 up) and downstream (POTW11 dwn) of discharge point (Figure 19).

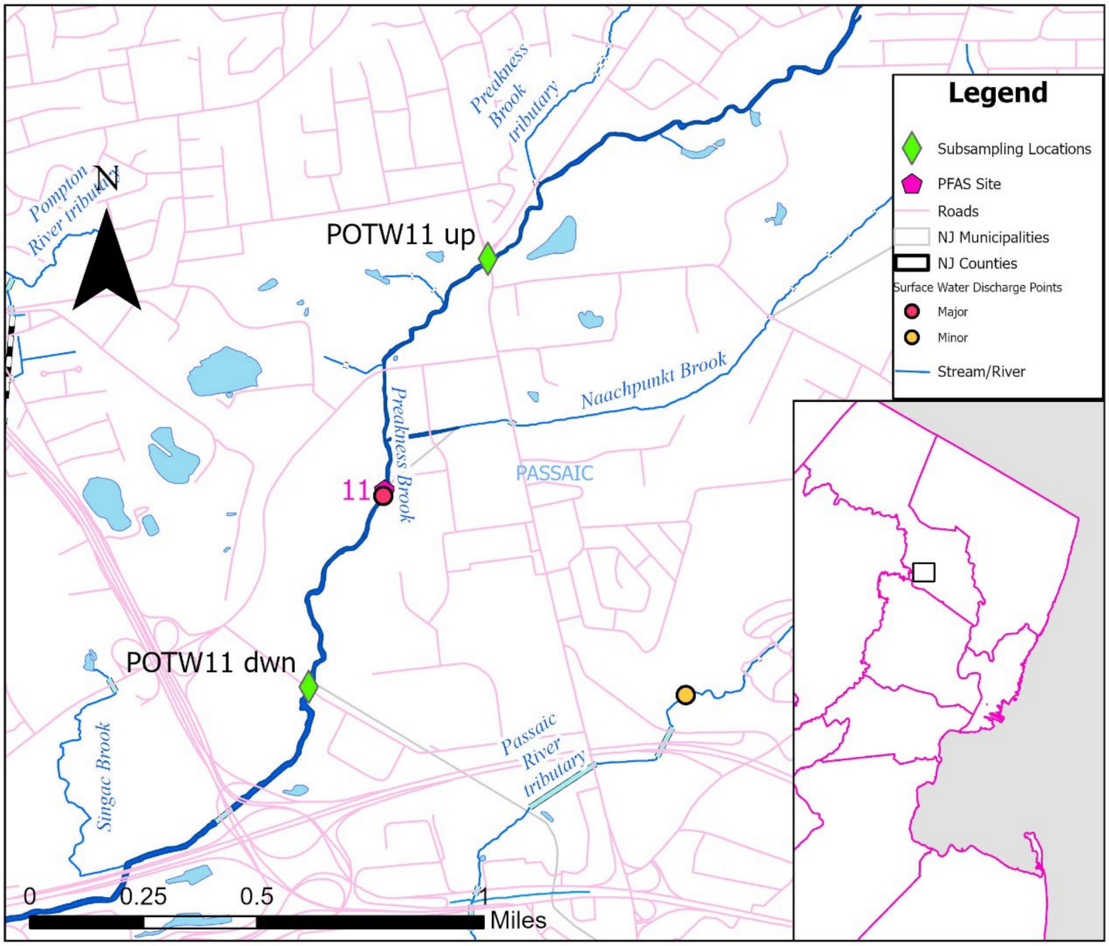

Figure 19: Site 11 Wayne Mountain View Wastewater Treatment Facility

Two Bridges Sewage Authority (Site 12)

Two Bridge Sewage Authority has a major discharge permit for a location on the Pompton River (i.e., Pequannock River), approximately five miles downstream of the Wayne-Sheffield discharge location. The discharge location is less than one half mile before the Pompton River empties into the Passaic River.

A single sample collection station on the Pompton River at Two Bridge Road was accessible and sediment and surface water were collected. Fish were also collected in this area (Figure 20).

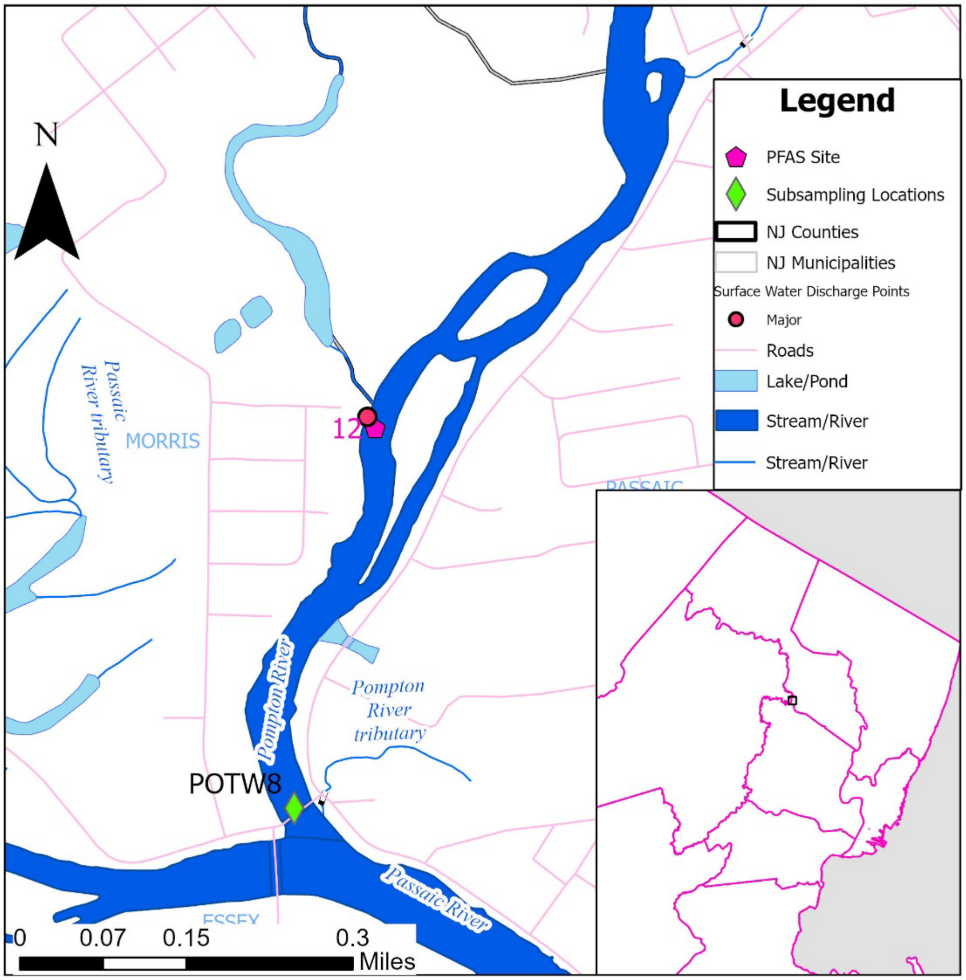

Figure 20: Site 12 Two Bridges Sewage Authority

Parsippany Troy Hills Sewage Authority (Site 13)

The Parsippany-Troy Hills discharges to a point after the Whippany River joins the Rockaway River on the border of Montville Township and East Hanover Township (Figure 21). Access to any location past the discharge point was not possible, as it was deemed unsafe due to natural impediments (difficult brush, wetlands, and water features). This lack of access was not apparent upon sample selection and initial reconnaissance. A single sample of surface water and sediment was collected on the Whippany River upstream from the discharge (13_WHIP).

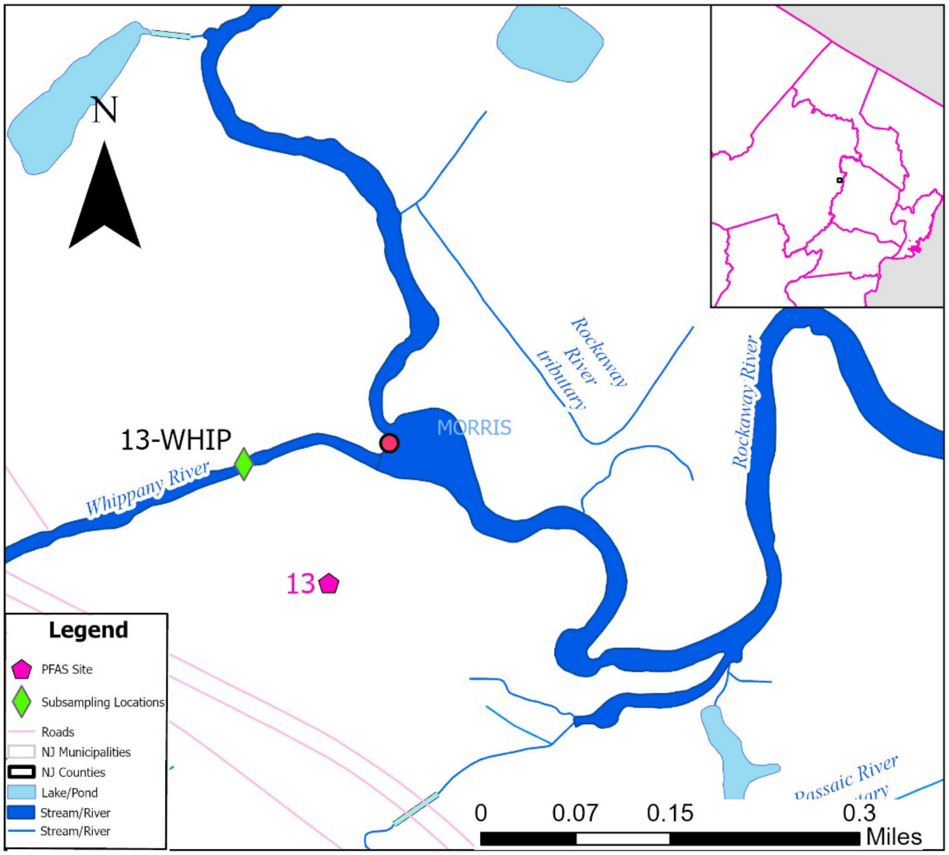

Figure 21: Site 13 Parsippany Troy Hills Sewage Authority

Caldwell Boro Sewage Treatment Plant (Site 14)

The Caldwell Boro Sewage Treatment Plant discharges to a small tributary in West Caldwell that joins the Passaic River. The location nearest to the discharge point had limited accessibility. One sample of sediment and surface water was collected after the discharge point (POTW14dwn, Figure 22).

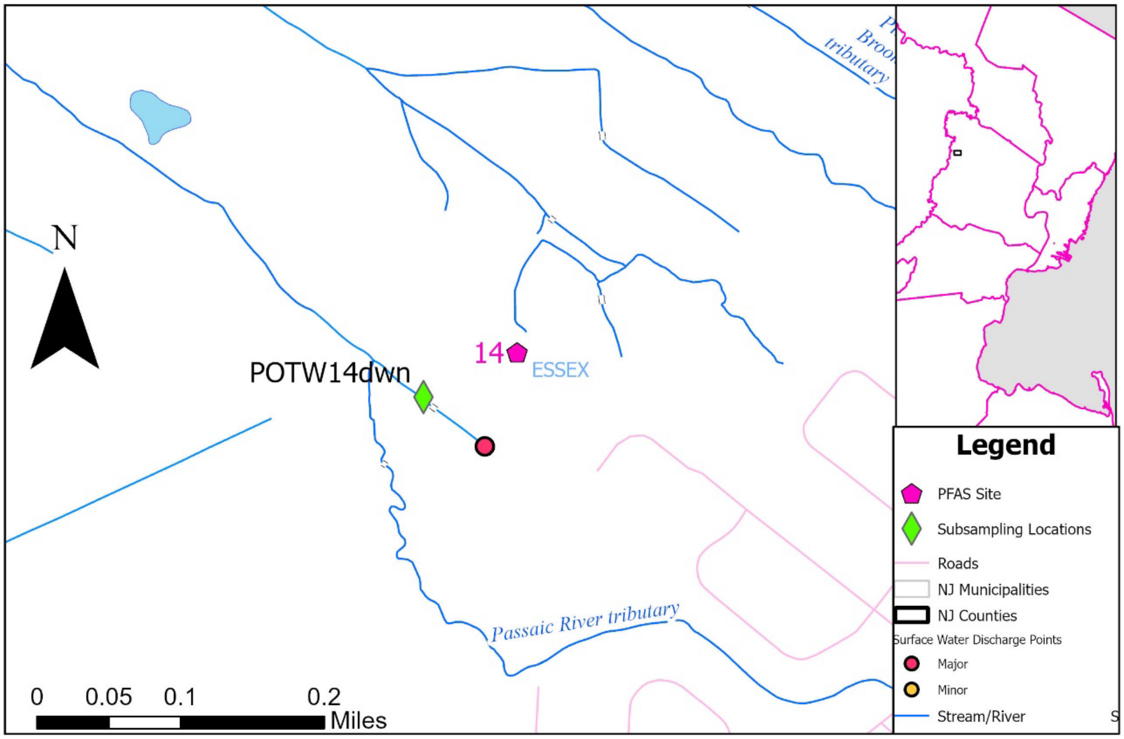

Figure 22: Caldwell Boro Sewage Treatment Plant

Wayne-Sheffield Hills (Site 15)

Wayne Township-Sheffield Hills Sewer Treatment Plant (STP) has a major discharge point on the Pompton River *(*i.e., Pequannock River*)* behind the Plains Plaza Shopping Center, and the Shopping Center has a minor discharge point.

This location was sampled during two different sampling events- 10/7/2019 where both the upstream location (POTW6_15up) and downstream location (POTW6_15ds) were sampled, and on 8/2/2021 where a single sample was collected downstream of the discharge point (POTW6) (See Figure 23).

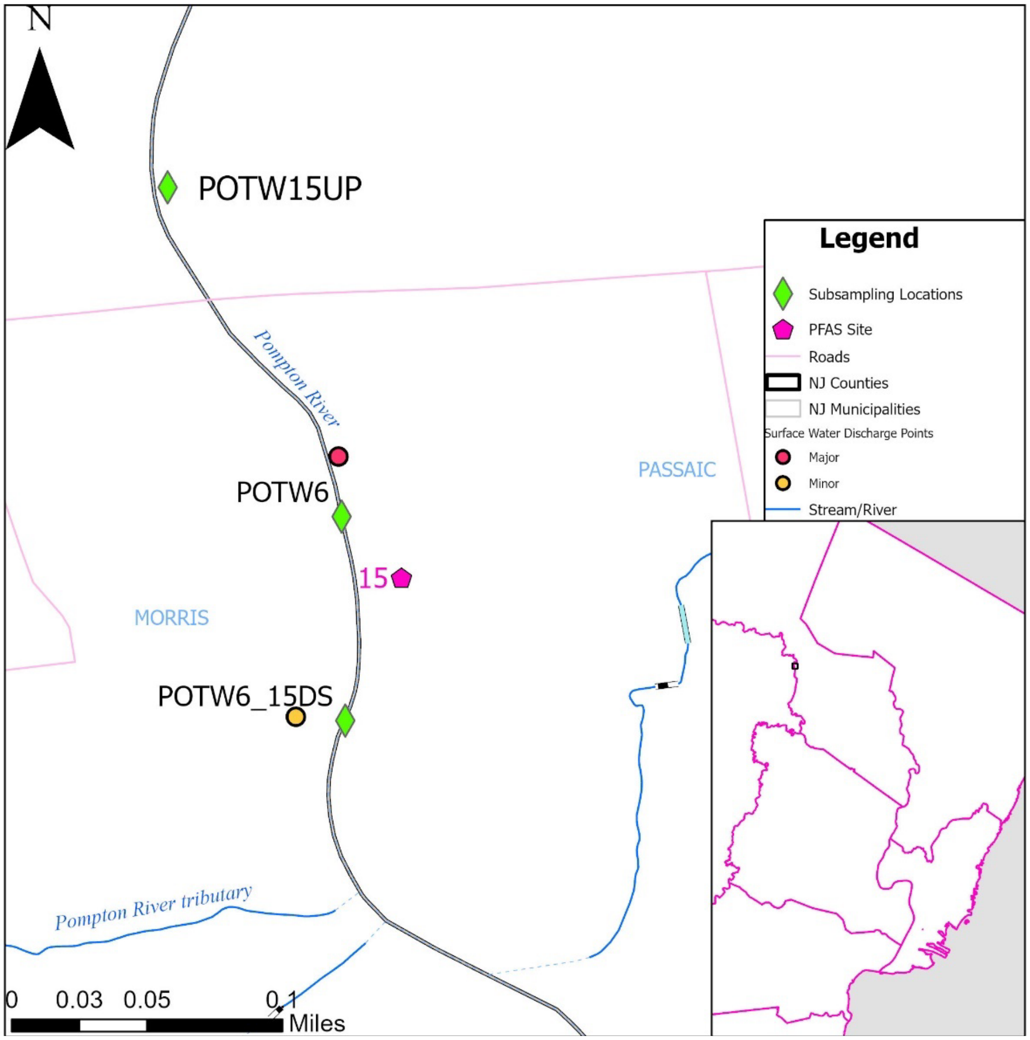

Figure 23: Wayne Sheffield Hills Wastewater Treatment Plant

Ponds

Natural isolated ponds without any surface water connections were included to assess potential occurrence where upgradient surface water inputs are absent. Three of the subsampling locations are in areas with low surrounding disturbance, and one is in a suburban community. One of the larger lakes sampled for this effort, Echo Lake in West Milford Township, Passaic County, was also sampled as a part of the first occurrence study (Goodrow et al., 2020). At the time of the first occurrence study, PFAS levels in the fish tissue collected from Echo Lake indicated the need for advisories recommending consumption not more than once per week or not more than once per month, depending on the fish species, while the PFAS levels in the surface water were largely below detection limits. For this study, grab samples were collected at two sampling locations that varied in size and could be described as ponds or groundwater seeps with little overland connection to the lake (Echo_1 and Echo_2). Butterworth Pond, in Woodland Township in Burlington County, and Cedar Pond, in Buena Vista Township in Atlantic County, were the other isolated ponds with minimal local impacts or surface water inputs. Locustwood Pond in Cherry Hill, Camden County was selected to include a similar environment in a more developed area.

The five sampling locations included in this study can be seen in Figure 24, below.

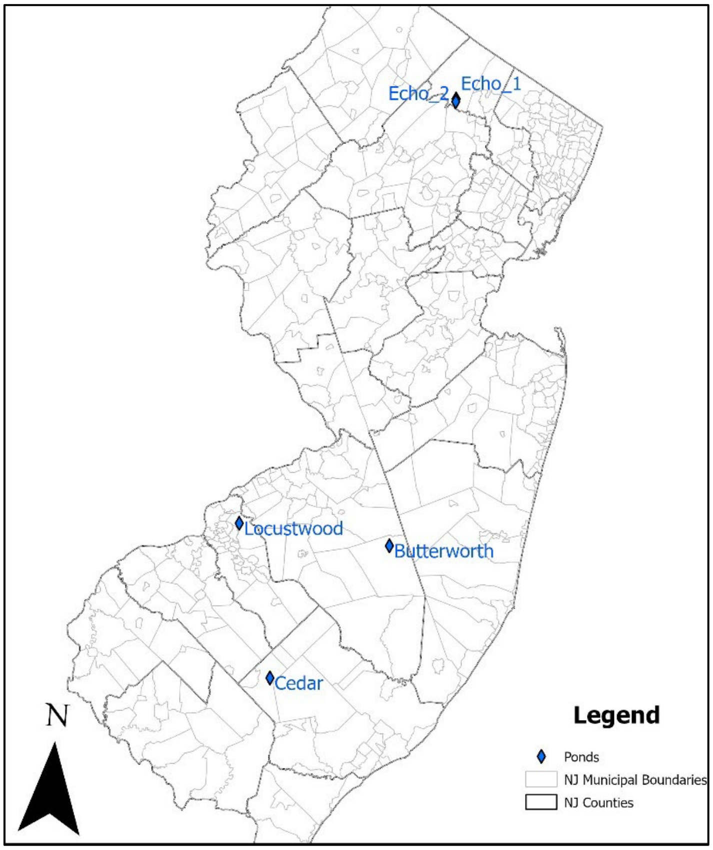

Figure 24: Sample collection sites for ponds

Analytical Methods

Analysis of the samples collected as part of this PFAS study began in 2018 and was completed in 2024. An analytical method that was appropriate for aqueous, solid, and tissue samples, SGS Axys Method MLA-110, was the primary method used in this study. This method provides the total of linear and branched isomers and uses isotopically labeled surrogate standards and cleanup by Solid Phase Extraction and liquid chromatograph/mass spectrometry (LC-MS/MS).

The first version of this analytical method, SGS Axys Method MLA-110 Rev. 01, was used for only the earliest 14 samples collected (Sites 2, 6, and the ponds) and included a quantification of 30 PFAS. SGS Axys Method MLA-110 Rev. 02 was used on samples analyzed starting in 2019 and included 33 analytes in 2019, and 40 analytes for all remaining analysis. The full names, chemical groups, and abbreviations of all analytes quantified under PFAS methods mentioned here can be found in Appendix 3.

Parameters

PFAS Quantified

The initial statewide study to quantify PFAS in surface water, sediments, and fish tissue (Goodrow et al., 2020) used the research analytical method from SGS Axys known as MLA-060. This method included the quantification of 13 compounds. These 13 PFAS will be known as “the original 13” for discussion herein and include the following analytes (Table 5):

Table 5: Original Thirteen Analytes MLA-060

PFBA	PFBS	PFDA	PFDoA	PFHpA	PFHxA	PFHxS	PFNA	PFOA	PFOS	PFOSA	PFPeA	PFUnA

Method SGS Axys MLA-110 Rev 01 and 02 added 17 and 20 additional PFAS, respectively, to the original 13. The 17 additional PFAS that were quantified for Sites 1 and 6 and the ponds, shown in Table 6, included the following analytes (for a total of 30 analytes):

Table 6: Step 1-Additional Seventeen Analytes with Rev 01 Ver 2019

4:2 FTS	6:2 FTS	8:2 FTS	EtFOSAA	HFPO-DA	MeFOSAA	N-EtFOSA	N-EtFOSE	N-MeFOSA
N-MeFOSE	PFDoS	PFDS	PFHpS	PFNS	PFPeS	PFTeDA	PFTrDA	

The three additional analytes added to MLA-110 for analysis in 2019 and 2020, shown in Table 7, included the following (for a total of 33 analytes):

Table 7: Step 2-Additional Three Analytes with Rev 01 Ver 2021

11Cl-PF3OUdS	9Cl-PF3ONS	ADONA

For all samples analyzed in 2022 and beyond, SGS Axys MLA-110 Rev 02 Ver. 12 was used. This method included an additional seven analytes for a total of 40 PFAS. The seven additional analytes, shown in Table 8, included:

Table 8: Step 3-Additional Seven Analytes with MLA-110 Rev. 02 Ver. 12

3:3 FTCA	5:3 FTCA	7:3 FTCA	NFDHA	PFEESA	PFMBA	PFMPA

The 40 PFAS that are included in Tables 5 to 8 above belong to sub-categories of PFAS, such as perfluoroalkyl sulfonates (e.g., PFOS and PFHxS) and fluorotelomer carboxylates (e.g., 5:3 FTCA and 7:3 FTCA). The full names and analyte groups can be found in Appendix 3. For a detailed discussion on the hierarchy of classes, subclasses, groups, and subgroups in the PFAS family, refer to the ITRC PFAS Technical and Regulatory Guidance Document, Section 2 (ITRC, 2023).

Detection Limits

Analytical detection limits have consistently improved over time with the laboratories improving methods to detect lower and lower levels of each individual PFAS in an environmental sample. These lower detection limits have provided the opportunity to recognize low levels of PFAS that may be present in a system to understand potential sources and exposure.

For a list of detection limits in the analyses performed during this study, with some comparison to those detection limits in the analyses in the earlier study, please refer to Appendix 4.

Data Summarization

This evaluation focused on the occurrence, magnitude, and distribution of the analytes. Therefore, data summaries included assessing the percentage of samples above the parameter-specific detection level, measure of central tendency, and total measured PFAS. Calculations of average concentration or other summary statistics, including total concentration at a subsampling location, were determined only from samples with detectable levels of the PFAS analyzed, i.e., all parameters recorded as below detection were excluded from calculations, and it was assumed that every result reported to be below the detection limit was not present in any concentration. Evaluating values below the detection limit inevitably introduces bias into an assessment (Helsel, 2012); omitting them from computations of the average or measures of central tendency and percentiles will bias the reported summary concentration high and underrepresents the sample size. Similarly, omitting values below detection when summing for a total concentration of PFAS in a sample will bias the sum low since the omitted parameter may in fact be present in the sample, albeit below detection. However, a consistent handling of the data was preferred given the goal of the evaluation was to screen the environment surrounding various activities that have been shown to lead to contamination. This approach was also used in the first NJ PFAS occurrence study (Goodrow et al., 2020) and is intended to provide a basis to understand the occurrence of each analyte.

Results

Analytes

The original 13 PFAS that were included in the analytical method used in the initial statewide study of PFAS in surface water, sediments, and fish tissue (Goodrow et al., 2020) were detected in these environmental media and were therefore prioritized for quantification. Additional analytes were included in revisions to the method to represent PFAS that might have been released to the environment or had been known to be part of a product thought to be released. The method now includes a wide range of analytes that could inform many environmental evaluations.

Several PFAS were not detected in any sample collected during this study. These negative results help to characterize the PFAS releases in NJ - what types of products are a concern and what types of risk could be present.

Media

Surface Water

Seventy-four surface water samples were collected from all sites included in this study. Given the expansion of the PFAS analyte list over the course of sample collection, the number of PFAS that were quantified for each sample ranged between 30 and 40. PFAS data for the surface water samples collected in this study are found in Appendix 5.

Of the analytes that were quantified in all surface water samples, there were eight PFAS that were not detected in any of the 74 water samples. These analytes include: 4:2 FTS, 8:2 FTS, N-EtFOSA, N-EtFOSE, NFDHA, N-Me-FOSA, PFDoS, and PFNS.

The 10 analytes added to the analysis of samples collected later in the study were quantified in at least 46 of the 74 samples, as shown in Table 9, below. Seven of the 10 had zero detections, while the remaining three were detected in only one sample.

The three compounds that had only a single detection are 3:3 FTCA, PFMBA, and PFMPA. Concentrations of these three were near detection level which would limit conclusions based on these results alone.

Table 9: Ten PFAS Added to Analysis of *Later* Surface Water Samples

Analyte in Surface Water	11Cl-PF3OUdS	3:3 FTCA	5:3 FTCA	7:3 FTCA	9Cl-PF3ONS	ADONA	N-MeFOSE	PFEESA	PFMBA	PFMPA
Number of samples	59	46	47	47	60	60	50	47	47	47
Number of detections	0	1	0	0	0	0	0	0	1	1
% detections	0.0%	2.2%	0.0%	0.0%	0.0%	0.0%	0.0%	0.0%	2.1%	2.1%
Max concentration (ng/L)	0	5.33	0	0	0	0	0	0	1.45	0.555

Perfluoroalkyl Acids (PFAAs)

Carboxylic Acid PFAAs in Surface Water

The eleven carboxylic acids (carbon chain lengths ranging from 4 to 14) were quantified in all 74 surface water samples that were collected. The most frequently observed PFAS in surface water was PFOA, the eight carbon carboxylate, being detected in just over 97% of samples collected. PFPeA, PFHxA, PFHpA, and PFNA were all observed in over 85% of the samples collected (See Table 10).

Table 10: Descriptive Statistics for Carboxylic Acid PFAS in Surface Water

Carbon Chain Length	4	5	6	7	8	9	10	11	12	13	14
Caboxylates- AQUEOUS (ng/L)	PFBA	PFPeA	PFHxA	PFHpA	PFOA	PFNA	PFDA	PFUnA	PFDoA	PFTrDA	PFTeDA
n	74	74	74	74	74	74	74	74	74	74	74
detections	53	64	69	69	72	66	37	1	3	1	2
% detections	71.6%	86.5%	93.2%	93.2%	97.3%	89.2%	50.0%	14.9%	4.1%	1.4%	2.7%
min	1.5	0.8	0.5	0.5	0.5	0.4	0.4	0.4	0.8	0.4	0.4
max	56.1	99.4	143.0	73.5	136.0	21.4	38.9	93.7	5.0	0.4	2.2
average	7.9	11.0	11.5	6.9	14.5	3.1	2.5	9.6	2.2	0.4	1.3
median	4.2	3.9	3.7	2.8	6.7	1.6	0.7	0.7	0.9	0.4	1.3
geomean	4.7	4.8	4.2	2.9	6.8	1.8	1.0	1.3	1.5	0.4	1.0

Note: Min, median, and averages based only on detectable values

Sulfonic Acid PFAAs in Surface Water

Eight sulfonic acid (chain lengths ranging from 4 to 12) were evaluated in all 74 samples collected for this study, and PFNS (chain length of 9) was evaluated in 68 samples.

Table 11: Descriptive Statistics for Sulfonic Acid PFAS in Surface Water

Carbon Chain Length	**4**	**5**	**6**	**7**	**8**	**8**	**9**	**10**	**12**
Sulfonates- AQUEOUS (ng/L)	PFBS	PFPeS	PFHxS	PFHpS	PFOS	PFOSA	PFNS	PFDS	PFDoS
n	74	74	74	74	74	74	68	74	74
detections	62	30	64	12	71	16	0	1	0
% detections	83.8%	40.5%	86.5%	16.2%	95.9%	21.6%	0.0%	1.4%	0.0%
min	0.5	0.4	0.4	0.5	0.6	0.4	0.0	0.6	0.0
max	115.0	14.2	215.0	14.9	537.0	4.5	0.0	0.6	0.0
average	8.2	1.7	9.2	2.3	20.9	0.9		0.6	
median	2.3	0.8	2.8	1.2	6.3	0.6		0.6	
geomean	2.4	1.0	3.2	1.3	6.9	0.7		0.6	

Note: Min, median, and averages based only on detectable values

PFOS, PFBS and PFHxS are all water soluble and commonly associated with historic PFAS use. These compounds are often widely found in surface water, and were found in 96%, 84%, and 87% of the samples in this study, respectively (Table 11).

Sediments

Seventy-one sediment samples were collected from sites included in this study. Given the expansion of the PFAS analyte list over the course of sample collection, the number of PFAS that were quantified for each sample ranged between 30 and 40 PFAS data for sediment samples collected in this study are found in Appendix 6.

Of the analytes that were quantified in all 71 samples, there were only four PFAS that were not detected in any sample. These four analytes include: 4:2 FTS, N-EtFOSA, N-EtFOSE, and NFDHA. Note that 8:2 FTS, N-Me-FOSA, PFDoS, and PFNS, which were not found in surface water, were detected in sediments, but only infrequently and at levels near the detection limit.

The 10 analytes added to the analysis of samples collected later in the study were quantified in at least 43 of the 71 samples, as shown in Table 12. Eight of the 10 had zero detections, while the remaining two were detected in only one sample.

The two compounds that had only a single detection are 5:3 FTCA and 7:3 FTCA. The detection of 22 ng/L of 5:3 FTCA and 5.37 ng/L of 7:3 FTCA in the East Fork of the Yellowbrook, a stream offsite from the Monmouth County Fire Training facility, along with the other PFAS found here, indicate that re-sampling efforts are warranted (see AFFF section).

Table 12: Ten Sediment Analytes Added Last

SOLID (ng/g (dry weight basis))	11Cl-PF3OUdS	3:3 FTCA	5:3 FTCA	7:3 FTCA	9Cl-PF3ONS	ADONA	NFDHA	PFEESA	PFMBA	PFMPA
n	57	43	43	43	57	57	43	43	43	43
detections	0	0	1	1	0	0	0	0	0	0
% detections	0.0%	0.0%	2.3%	2.3%	0.0%	0.0%	0.0%	0.0%	0.0%	0.0%
min	0	0	22	5.37	0	0	0	0	0	0
max	0	0	22	5.37	0	0	0	0	0	0

Note: Min and max based only on detectable values

Carboxylic Acids in Sediment

The longer chain carboxylic acids are known to preferentially partition to sediments when present in a surface water system. As can be seen in Table 13, below, the majority of the sediment samples contained detectable levels of the carboxylates with lengths of eight through 14-carbons. PFOA, a commonly detected PFAS, was found in just over 50% of the samples, while PFUnA, a less soluble compound that would preferentially partition to sediments, was found in just over 80% of the sediment samples.

Table 13: Descriptive Statistics for Carboxylic Acid PFAS in Sediment

Sediments- dry weight ng/g. Carbon chain length:	**4**	**5**	**6**	**7**	**8**	**9**	**10**	**11**	**12**	**13**	**14**
Carboxylates- SOLID (ng/g (dry weight basis))	**PFBA**	**PFPeA**	**PFHxA**	**PFHpA**	**PFOA**	**PFNA**	**PFDA**	**PFUnA**	**PFDoA**	**PFTrDA**	**PFTeDA**
n	71	71	71	71	71	71	71	71	71	71	71
detections	4	20	19	15	40	39	37	57	51	49	37
% detections	5.6%	28.2%	26.8%	21.1%	56.3%	54.9%	52.1%	80.3%	71.8%	69.0%	52.1%
min	0.3	0.1	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0
max	3.7	0.9	1.6	0.8	3.4	5.0	10.6	30.6	8.0	12.0	2.1
average	1.3	0.3	0.2	0.2	0.3	0.3	0.5	0.9	0.4	0.5	0.3
median	0.5	0.2	0.1	0.1	0.2	0.1	0.2	0.2	0.2	0.2	0.2
geomean	0.7	0.2	0.1	0.1	0.2	0.1	0.2	0.2	0.2	0.2	0.2

Note: Min, median, and averages based only on detectable values

Sulfonic Acids in Sediment

Shorter chain sulfonic acids tend to be more soluble than those with longer chain lengths and would therefore partition to the surface water. For the sulfonic acids quantified here, only PFOS was detected in the majority of sediment samples. PFOS is a common PFAS that has been widely released to the environment. It can be considered quite soluble but also possesses a strong ability to partition to sediments. In this study, PFOS was found in 96% of all sediment samples collected, with a maximum detection of 283 ng/g. Another long chain sulfonic acid PFAS, PFDS, was detected in 37% of samples collected. See Table 14 for more details

Table 14: Descriptive Statistics for Sulfonic Acid PFAS in Sediment

	4	**12**	**10**	**7**	**6**	**9**	**8**	**5**
Sulfonates- SOLID (ng/g (dry weight basis))	**PFBS**	**PFDoS**	**PFDS**	**PFHpS**	**PFHxS**	**PFNS**	**PFOS**	**PFPeS**
n	71	71	71	71	71	71	71	71
detections	5	1	26	1	13	2	68	2
% detections	7.0%	1.4%	36.6%	1.4%	18.3%	2.8%	95.8%	2.8%
min	0.1	1.0	0.0	1.6	0.0	0.0	0.0	0.2
max	0.2	1.0	3.5	1.6	9.9	2.7	283.0	0.3
average	0.1	1.0	0.3	1.6	0.9	1.4	4.9	0.3
median	0.1	1.0	0.1	1.6	0.1	1.4	0.5	0.3
geomean	0.1	1.0	0.2	1.6	0.2	0.4	0.5	0.3

Note: Min, median, and averages based only on detectable values

Fish Tissue

All fish tissue samples were analyzed for all 40 PFAS, including the 10 “newer” PFAS (i.e., those that were included in analysis of the surface water and sediment samples collected later in the study). PFAS data for fish tissue samples collected in this study are found in Appendix 7.

Of the analytes that were quantified in all surface water, sediment, and fish tissue samples in this study, there were seven PFAS that were not detected in any of the 118 fish tissue samples. These analytes include: 4:2 FTS, 8:2 FTS, HFPO-DA, N-EtFOSA, N-MeFOSA, PFBA, and PFPeA. N-EtFOSE was detected in 35 samples, ranging from 1.03 to 11 ng/g. HFPO-DA, PFBA, and PFPeA would generally be considered more soluble, and less likely to bioaccumulate in fish tissue, than PFAS with longer chain lengths. However, whether the reason for non-detection is due to solubility or lack of presence in the environment can be evaluated on a site-specific basis in concert with data on concentrations found in the other media.

Seven of the 10 “newer” PFAs, were not detected in any fish tissue sample. There was one detection of PFMPA, while 5:3 FTCA and 7:3 FTCA were found in 16 and 32% of the fish tissue samples, respectively. Detectable levels of 5:3 FTCA ranged from 2.48 ng/L to 44.3 ng/g in the fish tissue, while 7:3 FTCA ranged from 2.42 to 90 ng/g. These PFAS are commonly associated with AFFF and are precursors to the perfluoroalkyl carboxylic acid with the same fluorinated carbon chain length. Additionally, they have their own inherent potential for toxicity and bioaccumulation. These PFAS will be further discussed in the site evaluations where their concentrations play a role in the overall assessment (AFFF section, see Little Pine Lake).

FTCAs can be formed as transformation products of fluorotelomer alcohols (FTOHs) in surface water and the atmosphere. FTOHs are released from wastewater treatment plants (Chen, 2020). They are also the building blocks of fluorotelomer-based side chain fluorinated polymers (SCFPs) and can be released from degradation of these polymers in landfills, as well as from biosolids and consumer products (Titaley, 2024).

Carboxylates in fish tissue

Perfluoroalkyl carboxylates with a carbon chain length of nine or longer (i.e., eight or more fluorinated carbons and an additional carbon in the carboxyl group) are highly bioaccumulative in fish (Conder, 2008). However, PFOA, with a carbon chain length of eight (i.e., seven fluorinated carbons), does not possess a high degree of bioaccumulation in fish[2], and it was only detected in just over 8% of all tissue samples collected in this study. The longer chain carboxylates, from 10 to 14 carbons were detected between 97 (PFUnA) and 100% (PFDA) of the time. Details can be found in Table 15. . There is limited or no toxicology information for some of these longer chain carboxylates that are frequently detected in fish tissue, and obtaining information on health effects of these compounds should be a consideration for further research.

Table 15: Descriptive Statistics for Carboxylic Acid PFAS in Fish Tissue

Carbon chain length	**5**	**6**	**7**	**8**	**9**	**10**	**11**	**12**	**13**	**14**
Carboxylates- Fish Tissue (ng/g)	**PFPeA**	**PFHxA**	**PFHpA**	**PFOA**	**PFNA**	**PFDA**	**PFUnA**	**PFDoA**	**PFTrDA**	**PFTeDA**
n	118	118	118	118	118	118	118	118	118	118
detections	0	3	3	10	77	118	115	117	117	115
% detections	0.0%	2.5%	2.5%	8.5%	65.3%	100%	97.5%	99.2%	99.2%	97.5%
min	0.0	0.1	0.1	0.1	0.1	0.1	0.4	0.1	0.2	0.1
max	0.0	0.1	0.2	0.7	1.2	3.4	6.0	12.5	4.5	6.2
average		0.1	0.2	0.4	0.3	0.9	1.8	1.5	1.4	1.1
median		0.1	0.1	0.4	0.2	0.7	1.4	1.1	1.2	0.8
geomean		0.1	0.2	0.3	0.2	0.6	1.4	1.0	1.1	0.8

Note: Min, median, and averages based only on detectable values

Sulfonates in Fish Tissue

Similar to the carboxylate PFAS, sulfonates with a carbon chain length of eight or longer (i.e., PFOS and longer-chain sulfonates) are highly bioaccumulative in fish and they generally possess a higher rate of bioaccumulation than the carboxylate with the same carbon chain length. In this study, PFOS was found in every fish tissue sample and PFDS was found in just over 75% of samples but at a much lower average concentration (Table 16).

PFOS is commonly found in environmental media, including water, sediment, and fish tissue, often at notable levels. It should also be mentioned that PFNS, although not detected at all in surface water and only twice in sediments, was detected in 18 fish tissue samples, or 15% of all samples collected in this study.

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[2] It should be noted that PFOA is highly bioaccumulative in humans and other mammals, in contrast to its relatively low bioaccumulation in fish.

Table 16: Descriptive Statistics for Sulfonic Acid PFAS in Fish Tissue

Carbon Chain	**4**	**5**	**6**	**7**	**8**	**8**	**9**	**10**	**12**
Sulfonates- Fish Tissue (ng/g)	**PFBS**	**PFPeS**	**PFHxS**	**PFHpS**	**PFOS**	**PFOSA**	**PFNS**	**PFDS**	**PFDoS**
n	118	118	118	118	118	118	118	118	118
detections	1	3	41	22	118	74	18	89	3
% detections	0.8%	2.5%	34.7%	18.6%	100%	62.7%	15.3%	75.4%	2.5%
min	0.3	0.1	0.1	0.1	0.5	0.1	0.1	0.1	0.1
max	0.3	0.3	9.1	1.1	249.0	24.9	0.5	2.7	0.2
average	0.3	0.2	1.1	0.3	26.6	1.4	0.2	0.5	0.2
median	0.3	0.2	0.4	0.2	12.7	0.2	0.1	0.3	0.1
geomean	0.3	0.2	0.5	0.2	11.1	0.4	0.2	0.4	0.1

Note: Min, median, and averages based only on detectable values

Consumption Advisories for PFAS in Fish Tissue

It is important to note that data from any samples of fish tissue that are collected by NJDEP can be used as the basis for issuing a consumption advisory if levels of a contaminant exceed trigger values for daily or less frequent consumption. NJDEP has issued consumption advisories for several contaminants, including PFOS and PFNA, and has developed trigger values for PFOA, PFOS, PFNA, and PFUnA. The methods for development of trigger values and fish consumption advisories can be found in the *Methods for the Development of Fish Consumption Advisories in the State of New Jersey* (NJDEP, 2024). All fish tissue data collected as part of this study were considered in the recently released New Jersey Fish Consumption Advisories which can be found at https://dep.nj.gov/dsr/fish-advisories-studies/. While fish tissue data for the four PFAS for which triggers are available have been evaluated to determine the need for site-specific advisories, data for other parameters like mercury or PCBs from the same site may indicate the need for a more stringent advisory than would be based only on PFAS data. For this reason, the PFAS-based advisories indicated by the fish tissue data from this study are not presented in this report.

Site Results

To understand the contribution of the various analytes to the overall PFAS levels detected at each subsampling location, the data have been organized into various groups that describe the distribution of PFAS detected at that site. At some sites, descriptions begin with the original 13 analytes (evaluated in Phase I, shown in Table 5), and proceed to include the subsequent addition of analytes according to the analytical method applied for those sites.

Biosolid application sites

Results of the PFAS analysis in the samples collected from the subsampling locations around the three biosolid application sites are discussed here. The review of these sample results represents a preliminary assessment of the potential impact of the application of wastewater residuals on the surrounding environment. These samples were collected in easily accessible locations near the application site and were intended to capture a potential groundwater to surface water interaction.

These results should not be considered conclusive but could be suggestive of a potential contribution of PFAS to the natural systems surrounding the sites that can only be confirmed with onsite soil and groundwater sampling.

Pemberton (Site 1)

Samples collected from the Pemberton LLAMA site were analyzed to provide quantification of 33 PFAS. Those seven PFAS found in Table 8 were not quantified.

As described in the site selection section on Pemberton (Site 1), the Pemberton site included four subsampling locations, as shown in Figure 2. Surface water detections for the original 13 PFAS can be found in Table 17 and Figure 25 and sediment PFAS results can be found in Table 18 and Figure 26.

On the eastern border: Given the close location of the Joint Base McGuire-Dix-Lakehurst (JB MDL; to the northeast of the Pemberton LLAMA site), it was possible that PFAS contamination could be migrating off the base and could impact the surface waters nearby. Therefore, surface water and sediment samples were collected at two subsampling locations - one upstream of the LLAMA site (LL1C) which would be expected to characterize potential impact from the JB MDL, and a downstream subsampling location after water travels along the edge of LLAMA site (LL1D), which could indicate any impact of land use of the LLAMA site. The upstream total (13) PFAS result was 7.4 ng/L where the downstream site had a result of 33.3 ng/L total (13) PFAS. See Table 17 for all detectable (13) PFAS surface water results from the Pemberton LLAMA subsampling locations.

The sediment samples collected from the eastern border, LL1C and LL1D, showed relatively low levels of total (13) PFAS in both locations, with 0.152 and 0.184 ng/g detected, in the upstream and downstream subsampling locations, respectively (Table 18).

On the western border: The western half of the LLAMA site includes a stream that flows in a westerly direction, where the upstream sampling subsampling location is the headwaters of the stream (LL1B), and the downstream subsampling location was located at a road crossing soon after the stream left the boundary of the LLAMA site (LL1A).

The PFAS concentrations in the surface water and the sediment seem to describe different modes of impact. The surface water collected at the upstream subsampling location contained a total PFAS of 37.5 ng/L. The PFAS levels at the downstream subsampling location were notably elevated to 277.9 ng/L total PFAS, with PFBS, the four-carbon chain perfluorosulfonic acid (PFSA), and PFOA, the 8-carbon chain perfluorocarboxylic acid (PFCA) contributing the majority of the total PFAS (see Table 17). The impacts to the water quality of the downstream subsampling location could be attributed to its passage through the site, although the potential exists for an unknown source to be located between the edge of the site and the subsampling station on the stream. In contrast to the surface water results, the sediment collected at the upstream, headwaters, location showed elevated PFAS when compared with the downstream site. One potential reason for these elevated levels could be that this location is the recipient of runoff (stormwater from roads and farmland or irrigation runoff) leaving areas north of the subsampling location. Given this is an upstream subsampling location, the PFAS in the sediment could be indicative the adsorption of PFAS in the overland stormwater runoff that originated on site or was carried by the upgradient road runoff. The

PFAS in the sediment here could be representative of this contribution over the long term. The downstream subsampling location does not show this impact in the sediments (Table 18).

Table 17: Pemberton LLAMA Surface Water (13) PFAS Results

AQUEOUS (ng/L)	PFBA	PFBS	PFDA	PFDoA	PFHpA	PFHxA	PFHxS	PFNA	PFOA	PFOS	PFOSA	PFPeA	PFUnA	Sum of 13
LL1A	7.96	81.4	<	<	25.4	32.3	18.6	1.87	79.3	9.45	<	21.7	<	**277.98**
LL1B	3.8	1.26	0.658	<	2.75	2.58	1.78	2.4	8.36	9.91	0.743	2.72	0.505	**37.466**
LL1C	<	0.811	<	<	0.556	0.893	0.967	0.655	1.34	1.79	0.407	<	<	**7.419**
LL1D	<	5.86	<	<	2.55	2.41	2.57	1.09	12.3	4.41	0.493	1.58	<	**33.263**

Note: Sum of 13 include only the detectable levels reported. "<" in table are non-detects. Detection limits can be seen in Appendix 4.

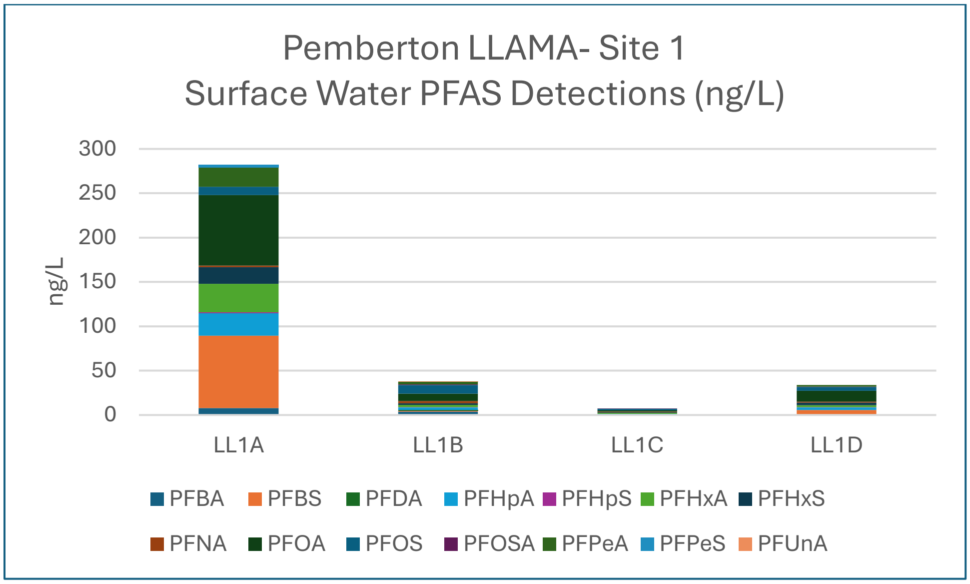

Figure 25: Pemberton LLAMA- Site 1 Surface Water PFAS Detections (ng/L)

Table 18: Pemberton LLAMA Sediment (13) PFAS Results

SOLID (ng/g)	PFBA	PFBS	PFDA	PFDoA	PFHpA	PFHxA	PFHxS	PFNA	PFOA	PFOS	PFOSA	PFPeA	PFUnA	Sum of 13
LL1A	<	<	<	<	<	<	<	<	<	0.052	<	<	<	**0.052**
LL1B	<	<	0.438	0.952	<	<	<	0.17	0.094	2.58	<	<	1.75	**5.984**
LL1C	<	<	<	<	<	<	<	<	<	0.089	<	<	0.063	**0.152**
LL1D	<	<	<	<	<	<	<	<	<	0.103	<	<	0.081	**0.184**

Note: Sum of 13 include only the detectable levels reported. "<" in table are non-detects. Detection limits can be seen in Appendix 4.

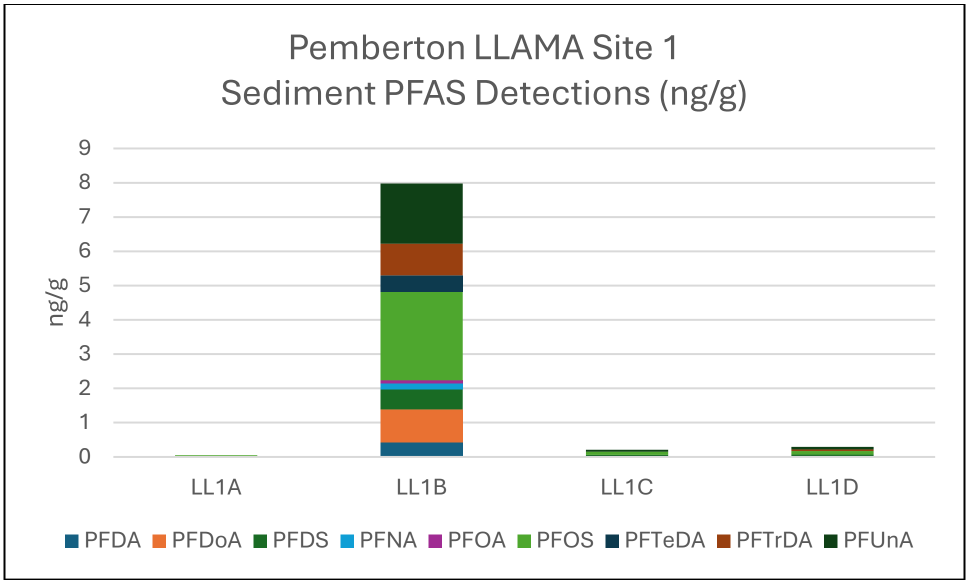

Figure 26: Pemberton LLAMA Site 1 Sediment PFAS Detections (ng/g)

Beyond the original 13 PFAS, there was a small contribution of other analytes to the total of the 33 quantified analytes. However, including these analytes in the total does not alter the overall view of these subsampling stations, that show the downstream subsampling location on the west side with high levels of total PFAS in the surface water coming off of the site, and the sediment collected at the roadway crossing also on the west side having higher levels of PFAS detected.

Most or all (89.6 to 100%) of the total PFAS in all surface water samples was from the original 13 analytes, with PFHpS and PFPeS, which were not included in the original 13, making small contributions (Table 19). Total PFAS in sediment samples, in addition to the original 13, included PFDS, PFTeDA, and PFTrDA in small percentages (Table 20).

Table 19: Pemberton Sum of 13 and Percentage of Total (33) PFAS in Surface Water

AQUEOUS (ng/L)	**Sum of 13**	**Total 33**	**Percent of Total**	**PFHpS**	**PFPeS**
LL1A	277.98	281.8	**98.6%**	0.991	2.91
LL1B	37.466	37.8	**98.9%**	0.421	<
LL1C	7.419	7.4	**100.0%**	<	<
LL1D	33.263	33.7	**98.6%**	0.458	<

"<" in table are non-detects. Detection limits can be seen in Appendix 4.

Table 20: Pemberton Sum of 13 and Percentage of Total (33) PFAS in Sediment

SOLID (ng/g (dry weight basis))	**Sum of 13**	**Total 33**	**Percent of Total**	**PFDS**	**PFTeDA**	**PFTrDA**
LL1A	0.052	0.052	**100.0%**	<	<	<
LL1B	5.984	7.9	**75.0%**	0.586	0.474	0.93
LL1C	0.152	0.2	**71.0%**	0.062	<	<
LL1D	0.184	0.3	**61.7%**	0.074	<	0.04

"<" in table are non-detects. Detection limits can be seen in Appendix 4.

Summary and Recommendations for Site 1

The impact of the land use on the surface water quality on the Pemberton LLAMA site can be seen in the higher levels of PFAS at the downstream sample collection location, most notably on the western side at subsampling location LL1B. The total PFAS concentration of 281.88 ng/L is among the highest total PFAS concentrations in surface water this study. While it is possible there could be a contributing source between where the western stream leaves the LLAMA site and where the sample was collected, the notably high total PFAS detected in surface water at this site at a minimum indicates the need for additional sampling, which may include an evaluation of the groundwater that is contributing to stream base flow. The follow-up sampling could also include a re-evaluation of the other three subsampling locations, to understand potential impacts.

Landis LLAMA (Site 2)

Samples collected from the Landis LLAMA site were analyzed to provide quantification of 40 PFAS.

As described in the site selection section Landis (Site 2), the Landis LLAMA site included nine subsampling stations, as shown in Figure 3.

Surface Water

Total PFAS (40) at the upgradient sites ranged from 21.9 ng/L in the surface water at LL4, a tributary that appears not to be hydraulically connected to the farm area, up to 44.0 ng/L at sampling site LL2A. This elevated value found at LL2A suggests that additional investigation in this area is warranted.

The LL2G sample collection station was located on an eastern tributary to the Maurice River located immediately upon discharge from the site. LL2G had the highest total PFAS (40) concentration of 115.0 ng/L, with 15 different analytes detected. The water sample collected from the downgradient subsampling locationLL2C was positioned to represent potential impact from two tributaries draining the southern portion of the eastern side of the farm and had a total PFAS concentration of 51.7 ng/L with 12 PFAS analytes detected. PFAS concentrations for all locations can be seen in Figure 27, below.

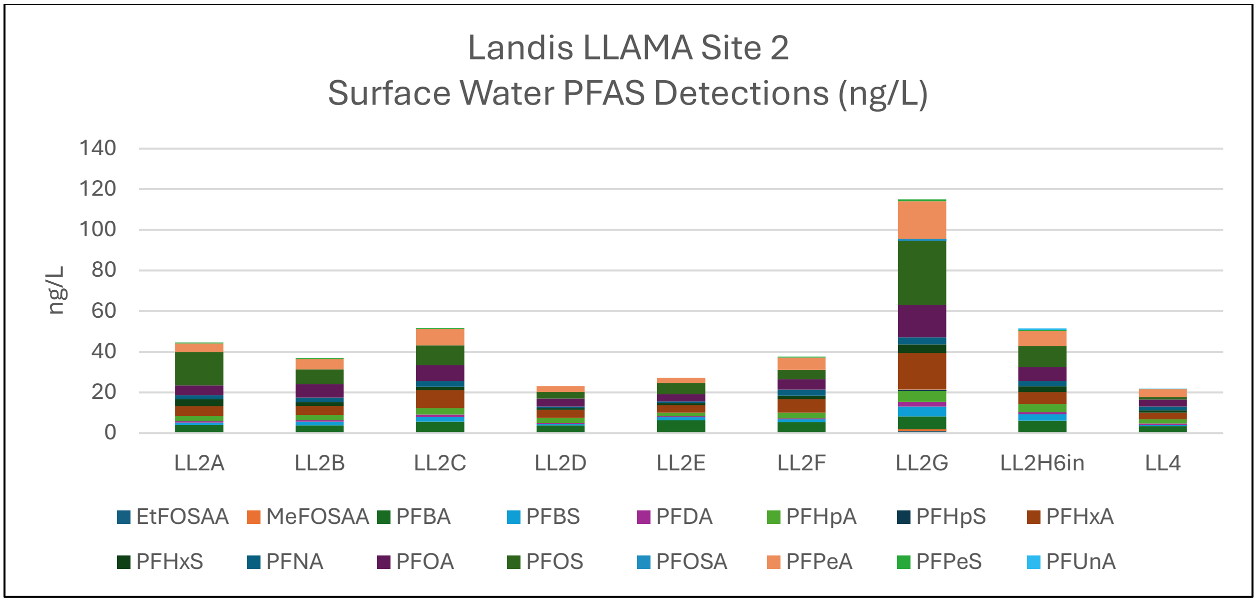

Figure 27: Landis LLAMA Site 2 Surface Water PFAS Detections (ng/L)

LL2B and LL2H sampling stations were in areas of flow that capture and characterize a larger land contribution. LL2B was located on the Maurice River just downstream of the confluence with the Parvin Branch and upstream of Union Lake and would be expected to represent all land inputs along with a higher flow. LL2B showed a total PFAS of 36.8 ng/L, with 11 analytes contributing. LL2C was located on Parvin Branch downstream of the application site.

Total PFAS in Union Lake surface water was found to be 49.5 ng/L, with detection of 12 analytes and PFOS having the highest concentration of 9.9 ng/L.

Sediment

The results from the sediment analysis shows partitioning of the longer chain PFAS, particularly PFUnA, in the sediments compared with levels detected in surface water. Concentrations of PFTeDa and PFTrDA were also present in six and seven of sediment samples collected at the nine total subsampling locations, respectively. PFOS, PFPeA, and PFUnA were large contributors to the total PFAS found at many of the subsampling locations, but the fingerprint of all contributing PFAS in this area included 16 PFAS (See Figure 28). PFPeA often partitions to groundwater given the higher solubility of shorter chain PFAS, and seeing these higher levels may indicate a unique fingerprint in the source material.

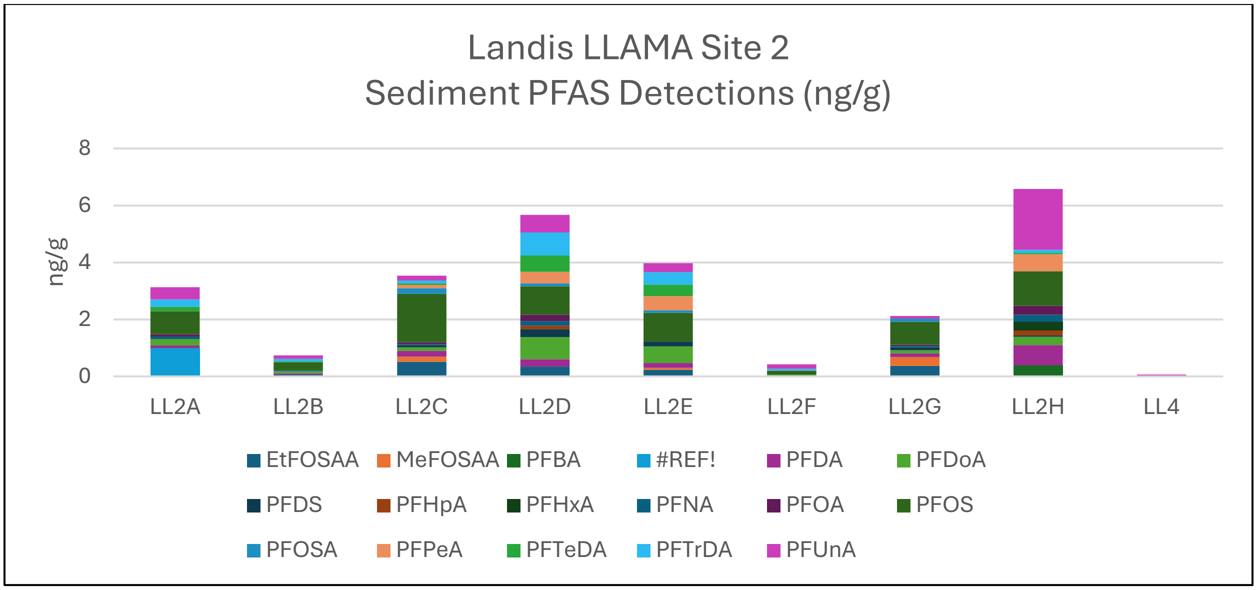

Figure 28: Landis LLAMA Site 2 Sediment PFAS Detections (ng/g)

Fish Tissue- Union Lake

Union Lake has a large catchment area and receives flow from the Maurice River and many tributaries. There is no final determination regarding all sources of PFAS to the system, and residence time and turnover can affect water column concentrations, which would impact the availability of PFAS to the fish.

Three individual Brown bullhead, Black crappie, Pumpkinseed, Yellow Perch, and Largemouth bass were collected from Union Lake, the impoundment located downgradient to the nine subsampling stations around the Landis LLAMA, Site 2. Bioaccumulation dynamics can be seen over the species distribution, with the filet tissue samples collected from the Largemouth bass and Black Crappie containing the highest levels of total PFAS, and the Brown bullhead containing the lowest.

The 15 fish had reported levels of up to 10 PFAS, with PFDA, PFDoA, PFOS, PFTeDA, PFTrDA, and PFUnA being detected in 100% of the samples (see Figure 29). Two of the Largemouth bass contained among the higher levels of PFDA in this study, with 2.57 and 2.09 ng/g, as well as the highest level of PFUnA, 6.03 ng/g. PFOS made up a large proportion of the total PFAS in all fish, ranging from 33-48% of total PFAS in three fish and over 50% of total PFAS in 12 of the 15 fish.

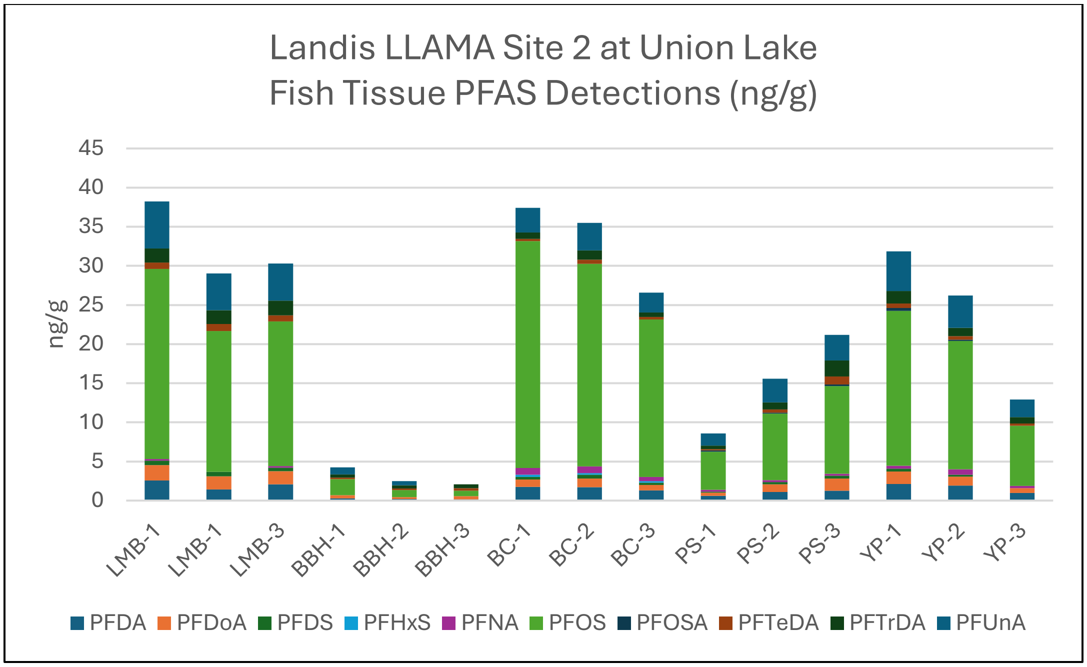

Figure 29: Landis LLAMA Site 2 Fish Tissue PFAS Detections (ng/g).
Note: LMB= Largemouth bass; BBH=Brown bullhead; BC=Black crappie; PS=Pumpkinseed; YP=Yellow perch

Summary and Recommendations for Landis LLAMA Site 2

The drainage areas contributing to the subsampling locations around the Landis LLAMA site are large and could contain other relevant sources of PFAS. However, the surface water collected from a road crossing immediately after waterway leaves the site, LL2G, contained a high level of total PFAS (115 ng/L) along with a wide range of various PFAS analytes not detected in the other samples. In addition, the elevated PFAS found in the sediments of Union Lake (LL2H) along with the elevated PFAS found in the fish tissue, may require a more in-depth investigation of the area.

Ash Lane (Site 3)

The Ash Lane LLAMA Site and the three subsampling collection locations can be seen in Figure *4*. The three subsampling locations were located outside of the boundaries of the LLAMA. The two subsampling locations to the west were located at or near the headwaters of the stream (LL3B and LL3A). The sampling location to the east was at a road crossing immediately after a farm pond discharge point (LL3CSW).

Surface Water

Several of the 40 measured PFAS were found in one or more of the surface water samples collected around Ash Lane Farms (Figure 30). PFAS found in the samples collected at the southwest subsampling location, LL3A, and the outlet of the pond, LL3CSW, were found at elevated levels. Levels of PFAS in the sample collected from the subsampling location downstream of the pond, LL3CSW, had detectable levels of 17 analytes, with total PFAS of 676.7 ng/L. The major components of the total PFAS from this water sample came from PFHxA (143.0 ng/L), PFOA (135.0 ng/L), and PFBS (109.0 ng/L). The sample collected at subsampling location LL3A had 12 analytes detected with a total PFAS of 281.0 ng/L, including PFOA of 74.3 ng/L and PFHxA of 57.8 ng/L. Surface water PFAS concentrations determined from the samples collected from LL3B, the northwest subsampling station, were relatively unremarkable, having PFBA and PFOA of 1.6 ng/L and a total of 10.2 ng/L for nine detected PFAS.

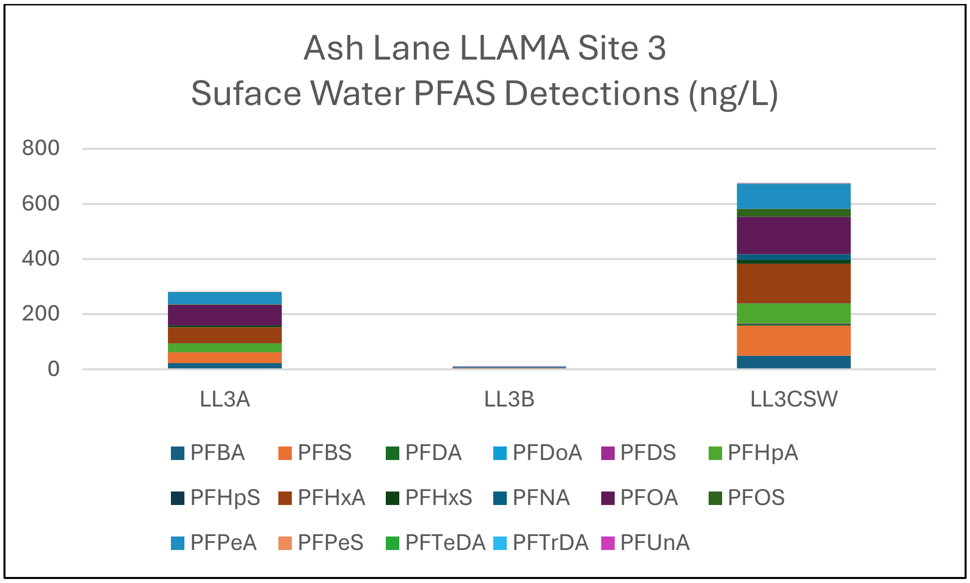

Figure 30: Ash Lane LLAMA Site 3 Surface Water PFAS Detections (ng/L)

<u>Sediment</u>

PFAS detected in the sediments collected at the LL3A and LL3C sampling locations showed elevated levels of PFAS indicating a local contributing source (Figure 31). Neither location showed any single PFAS dominating the contribution to the total PFAS, while the two subsampling locations included detections of multiple analytes. The fingerprints of the two subsampling locations were somewhat similar in the levels and types of PFAS contributing to total PFAS, which included 12 PFAS for the sample location after the pond (LL3CSW) and 14 at LL3A.

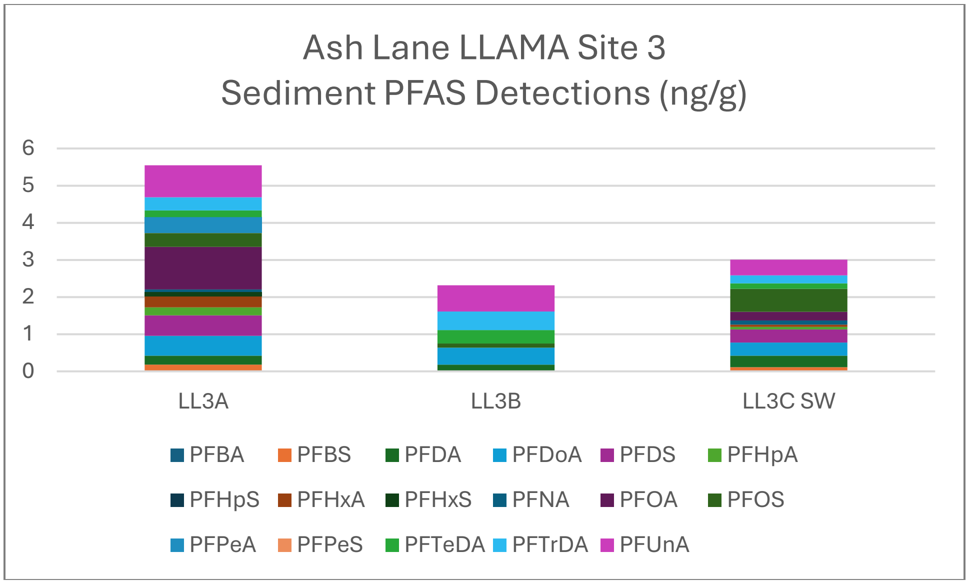

Figure 31: Ash Lane LLAMA Site 3 Sediment PFAS Detections (ng/g)

<u>Summary and Recommendations for Site 3 Ash Lane LLAMA</u>

The limited grab sampling performed at this site indicates a local source that is impacting surface water and sediment, most notably to the southwest at LL3A, and after the farm pond discharge to the east at LL3CSW. The elevated levels of total PFAS, >281 ng/L in the surface water of the LL3A and >676 ng/L in the surface water of the LL3CSW samples clearly indicate that follow up sampling to fully characterize the site is warranted.

AFFF use/Military Base

There are two general categories of firefighting foams - Class A and Class B. Class A foams are intended to be used on fires fueled by items such as paper, wood, or brush, and do not generally contain PFAS. Class B foams are intended to be used on fires caused by flammable or combustible liquids and often include PFAS for their surfactant properties. These foams produce a foam blanket to cut off oxygen to the fire and prevent reignition. The variety of foams that fall into Class B range widely in their PFAS composition but will generally contain some mixture of PFAS that can be dominated by PFOS, or by fluorotelomers that have the potential to transform to recalcitrant perfluoroalkyl acids.

Ocean County Training Center (Site 4)

Surface water and sediment samples were collected in two streams west of the Ocean County Training Center (Figure 5), as the groundwater flow moves toward Barnegat Bay. Samples from the site FF1A were collected from a headwater stream located directly to the west of the training center. FF1B is to the south and is along a stream that originates southwest of the training center.

Samples collected from subsampling station FF1A are expected to represent a hydraulic connection to the groundwater originating near the training facility. Samples collected at FF1B would be expected to represent sources south and upgradient to the site and would likely include only a small contribution from area near the training facility. Analysis for both samples included the quantification of 40 PFAS in both the surface water and the sediments.

Surface Water

The results for the original 13 PFAS quantified in the surface water samples can be seen in Table 21, below.

Table 21: Site 4, Ocean County FF Original 13 PFAS Surface Water Results (ng/L)

Site	Surface Water	PFBA	PFBS	PFDA	PFDoA	PFHpA	PFHxA	PFHxS	PFNA	PFOA	PFOS	PFOSA	PFPeA	PFUnA	Total of 13 PFAS	% original 13 PFAS contribute to total PFAS (40)
4	04-FF1A	32.1	2.9	2.6	<	50.5	78.4	7.0	15.4	22.7	18.1	<	99.4	<	329.1	96.4%
4	04-FF1B	2.2	1.5	<	<	1.4	1.0	0.6	0.8	2.3	1.6	<	2.3	<	13.7	100.0%

Note: "<" represent results below the detection limits. DLs can be found in Appendix 4.

Ninety-six percent (96%) of the total PFAS detections found in the FF1A sample can be attributed to the original 13 PFAS, whereas 100% of the detections in the FF1B sample can be attributed to these PFAS. In the sample FF1A, low levels of PFDA (2.55 ng/L) and PFPeS (0.852 ng/L) contribute to the total PFAS, potentially indicating a unique fingerprint of contributing source material.

The results for the single sample collected for FF1A include high levels of PFHxA (78.4 ng/L), PFPeA (99.4 ng/L), and PFHpA (50.5 ng/L). These PFAS have been related to the release of newer foams known as "C6 foams," which contain short-chain fluorotelomers that do not break down to PFOA or PFOS in the environment (ITRC, 2023). The FF1A sampling location was selected to represent potential groundwater contribution from the area of the fire training center to the headwater stream where the sample was collected and could indicate a source that impacts the groundwater in this area. The FF1A sample, containing more than 20 times the total PFAS of the sample from the site unconnected to the training center groundwater (FF1B), indicates additional sampling would be required to fully characterize the area.

Sediment

The results for the original 13 PFAS quantified in the sediment samples can be seen in Table 22, below. The table also provides the results for five of the remaining PFAS that were detected in one of the samples (lower table).

Table 22: Site 4, Ocean County FF Sediment Original 13 PFAS Results (ng/g dry weight basis)

Site	Sediment	PFBA	PFBS	PFDA	PFDoA	PFHpA	PFHxA	PFHxS	PFNA	PFOA	PFOS	PFOSA	PFPeA	PFUnA	Total of 13 PFAS	% 13 of total (40)
4	04-FF1A	<	<	0.5	0.3	0.3	0.2	<	0.3	0.2	2.0	0.1	0.9	0.3	5.1	78.16%
4	04-FF1B	<	<	<	0.2	<	<	<	0.3	<	0.8	<	<	0.3	1.6	70.42%

Note: "<" represent results below the detection limits. DLs can be found in Appendix 4.

Seventy-eight percent (78%) of all the PFAS detections found in the FF1A sediment sample can be attributed to the original 13 PFAS, whereas 70% of the detections in the FF2A sample can be attributed to these 13 PFAS. Other PFAS found in both samples include-PFTrDA and PFTeDA, the 13- and 14- carbon perfluoroalkyl carboxylates. Other PFAS contributing a smaller input into the total PFAS for the FF1A sample include two perfluoroalkane sulfonamides, EtFOSAA and MeFOSAA, which may serve as precursors to other PFAS in environmental conditions.

The PFOS concentration result of 2.0 ng/g detected in the FF1A sediment sample was notably elevated above the median PFOS concentrations of 0.485 ng/g detected in 61 of the 64 samples collected for this PFAS targeted study. PFOS is known to be a common PFAS found in legacy firefighting foams manufactured by the ECF process.

Summary and Recommendations for Site 4 Ocean County Training Center

The levels and types of PFAS found in the surface water and sediment samples of FF1A, the subsampling station most hydraulically connected to the training center, suggest potential impact from release of AFFF. Additional sample collection of environmental media, including groundwater wells, is recommended to properly characterize the area for PFAS impact from the fire training site or other local source(s).

Monmouth County Fire Academy (Site 5)

Surface water and sediment samples were collected in two streams located downgradient of the Monmouth Couty Fire Academy on Highway 33 in Howell. The samples were collected on Route 33 where each stream crossed the road. The two crossings, located less than 600 feet apart, are where the West Fork Yellow Brook and the East Fork Yellow Brook cross over Route 33 before they join to form the Yellow Brook approximately 600 feet downgradient. These streams represent groundwater discharge or runoff from the fire academy site. The site, streams and sampling locations can be seen in Figure 6.

Both subsampling stations would be hydraulically connected to surface water and groundwater on the academy site. Analysis for both samples included the quantification of 40 PFAS in both the surface water and the sediments. Samples of foam found in the West Fork stream were also collected and analyzed.

Surface Water

The results for the original 13 PFAS quantified in the surface water samples can be seen in Table 23.

Table 23: Site 5, Monmouth County Fire Academy Surface Water Original 13 PFAS Results (ng/L)

Site	Surface Water	PFBA	PFBS	PFDA	PFDoA	PFHpA	PFHxA	PFHxS	PFNA	PFOA	PFOS	PFOSA	PFPeA	PFUnA	Total of 13 PFAS	% 13 of total (40)
5	5-FF2A	56.1	31.9	6.7	<	27.4	39.3	26.4	11.6	66.7	72.9	<	41.7	<	380.7	98.9%
5	5FF2B	19.3	6.9	6.3	<	28.3	72.4	215.0	18.0	45.5	537.0	<	40.0	3.9	992.7	97.2%

Note: "<" represent results below the detection limits. DLs can be found in Appendix 4.

Nearly ninety-nine percent (98.9%) of all the PFAS detected in the West Fork sample (5-FF2A) was made up of PFAS included in the original 13 PFAS, with elevated levels of PFBA, PFOA, and PFOS. Ninety-seven percent of the PFAS quantified in the East Fork sample (5FF2B) was due to PFAS in the original 13, including notably high levels of PFHxS (215.0 ng/L) and PFOS (537 ng/L). The total (13) PFAS in the West Fork was 380.7 ng/L and the East Fork was 992.7 ng/L.

The levels of individual and total PFAS recorded at the two subsampling stations at this site were among the highest in this targeted study. The PFAS that have been found to be elevated are consistent with releases of legacy and more modern AFFFs.

<u>Sediment</u>

The results for the original 13 PFAS quantified in the sediment samples collected from the Yellow Brook can be seen in Table 24, below.

Table 24: Site 5, Monmouth Fire Academy Sediment Original 13 PFAS Results (ng/g dry weight basis)

Site	Sediment	PFBA	PFBS	PFDA	PFDoA	PFHpA	PFHxA	PFHxS	PFNA	PFOA	PFOS	PFOSA	PFPeA	PFUnA	Total of 13 PFAS	% 13 of total (40)
5	5FF2A	<	<	0.1	0.2	<	<	0.2	0.1	0.2	3.1	<	0.1	0.4	4.5	93.3%
5	5FF2B	0.6	0.1	10.6	8.0	0.8	1.6	9.9	5.0	3.4	283.0	8.2	0.8	30.6	362.6	85.5%

Note: "<" represent results below the detection limits. DLs can be found in Appendix 4.

Just over ninety-three percent (93.3%) and eighty-five percent (85.5%) of the total (40) PFAS were comprised of the original 13 PFAS, for samples 5FF2A and 5FF2B, respectively. The sample 5FF2B from the eastern fork included 13 additional PFAS in varying amounts, to make up the total PFAS for that subsampling station.

<u>Foam</u>

Increased PFAS concentrations, up to eight times the concentrations in the bulk water , have been documented in the air-water interface (Psillakis, 2009) However, foam formation above surface waters due to winds, waves, or other turbulent forces that introduce air into the water likely cause an aggregation of the dissolved phase PFAS that results in foam. These enrichment factors are highly variable.

Foam was observed on the surface of the stream collecting in a slow-moving area of the West Fork (5FF2A). One sample was collected, and 40 PFAS were quantified in ng/L in the liquid remaining after the individual films of the foam ruptured. Results can be seen in Table 25, below.

The concentration of PFAS in the foams was notably higher than the concentration in the bulk water collected from the stream. Calculated enrichment factors were especially high for two long chain PFAS, PFNA and PFOS. However, given the limited sampling and potential uncertainty in foam analysis, these values are suggestive of a source of PFAS, but not conclusive.

Table 25: Foam and Related Surface Water Sample Results

Site	5FF2A	**PFBA**	**PFBS**	**PFDA**	**PFDoA**	**PFHpA**	**PFHxA**	**PFHxS**	**PFNA**	**PFOA**	**PFOS**	**PFOSA**	**PFPeA**	**PFUnA**	**Total 13 PFAS**	**% of Total (40)**
5	Surface Water (ng/L)	56.1	31.9	6.7	<	27.4	39.3	26.4	11.6	66.7	72.9	<	41.7	<	380.7	98.9
5	Foam (ng/L)	17.8	<	332.0	9.9	91.9	28.9	40.1	1310.0	1670.0	7430.0	8.5	21.0	212.0	11172.0	99.1
	Enrichment	*0.3*		*49.3*		*3.4*	*0.7*	*1.5*	*112.9*	*25.0*	*101.9*		*0.5*			
		8:2 FTS	**PFDS**	**PFHpS**	**PFNS**	**PFPeS**	**PFTrDA**								**Total 40**	
		<	<	<	<	4.2	<								384.9	
		47.3	5.3	30.7	16.4	<	4.3								11276.0	

Note: "<" represent results below the detection limits. DLs can be found in Appendix 4.

<u>Summary and recommendations for Site 5 Monmouth County Fire Academy</u>

There are notable levels of multiple PFAS in both the East Fork and the West Fork subsamples. The subsampling location on the East Fork appears to be more impacted. An aerial view of the site suggests contribution from stormwater runoff to the East Fork from paved area in the northeast corner of the site that appears darkened beyond ambient conditions. See Figure 32, below.

5:3 FTCA was found at elevated levels in the sediment samples collected from the East Fork. 5:3 FTCA is known to originate from landfill leachate, and it is also a known transformation product of the modern fluorotelomer AFFFs. The detection of 5:3 FTCA at 22 ng/g in the East Fork subsample was the only detection of this PFAS reported in the 36 (out of 64 total) sediment samples in which this compound was quantified in this study, and it represented a significant amount of the total PFAS quantified in that sediment sample. The level of PFOS, at 283.0 ng/g, was also the highest level of PFOS in sediment found in this study.

Figure 32: Aerial of Monmouth County Training Area

Results reported here are from one grab sample at each subsampling location and are intended to provide initial screening only. These results found here indicate a likely source that is releasing PFAS to the environment, and additional follow-up samples on- and off-site are recommended.

Somerset County Fire Training (Site 7)

The Somerset County Fire Training facility is located along Roycefield Road in Hillsborough Township (see Figure 7). Access to a subsampling station was difficult, and only one sample of both surface water and sediment could be collected, and only at a location upstream of the site. Therefore, the sample that was collected is likely not to be representative of any potential release from the intended site.

Surface Water and Sediment

The analysis included a total of 33 analytes, but all PFAS that had detectable levels in the Site 7 samples were among the original 13 PFAS. The results of the surface water and sediment sample can be seen in Table 26, below.

Table 26:Site 7, Somerset Surface Water and Sediment PFAS Results

Media	PFBA	PFBS	PFDA	PFDoA	PFHpA	PFHxA	PFHxS	PFNA	PFOA	PFOS	PFOSA	PFPeA	PFUnA	Total of 13 PFAS
Surface Water (ng/L)	7.83	2.46	<	<	3.96	5.03	4.53	2.55	19.8	13	<	6.29	<	65.45 ng/L
Sediment (ng/g)	<	<	<	<	<	<	<	<	<	0.252	<	<	<	0.252 ng/g

Note: "<" represent results below the detection limits. DLs can be found in Appendix 4.

Summary and Recommendations for Site 7, Somerset County Training Academy

The sampling location did not ultimately capture potential source contributions from the training site, so no conclusion or recommendation can be made regarding that site. However, the PFAS detected in the surface water at this location may indicate another local source. A review of existing data in this area could be reviewed to determine next steps.

Bergen County Electrical Station Fire (Site 8)

The 2015 electrical station fire, where an estimated 15,000 gallons of AFFF were used for fire suppression, was located at the headwaters of the Smokis Val Brook, a tributary to the Hohokus Brook in Waldwick, Bergen County (as seen in Figure 10). This tributary is ephemeral and was dry at the time of sampling. Therefore, surface water and sediment samples were collected from Whites Pond (FF6), an impoundment above the location where the tributary enters the Hohokus Brook and from a location downstream of this confluence (FF5).

Surface Water

Thirty-two analytes were quantified in the surface water samples. One hundred percent of all detectable PFAS in the surface water samples was from the 13 original PFAS, with three of those 13- PFDA, PFDoA and PFUnA- reported as below the detection limit. The 10 PFAS detected in the surface water samples are shown in Table 27, below.

Table 27: Site 8, Bergen Electrical Fire Site Surface Water PFAS

Site	AQUEOUS (ng/L)	PFBA	PFBS	PFHpA	PFHxA	PFHxS	PFNA	PFOA	PFOS	PFOSA	PFPeA	Total of 13 PFAS	% 13 of total (32)
8	FF5 (DS)	3.92	2.6	4.18	5.36	4.23	1.51	16.4	11.4	0.844	4.67	55.1	100.0%
8	FF6 (Whites Pond)	4.16	3.16	3.67	5.84	3.23	1.75	15.1	6.31	<	5.41	48.6	100.0%

Note: "<" represent results below the detection limits. DLs can be found in Appendix 4.

Sediment

Thirty-two analytes were also quantified for the sediment samples with 100% of PFAS in the downstream sample (FF5) and 61.5% in the Whites Pond sample (FF6) coming from the original 13 PFAS. In addition to the original 13, small percentages of PFAS came from PFDS, PFTeDA and PFTrDA in the Whites Pond sediment sample. There were no detections of PFBA, PFBS, PFDA, PFHpA, PFHxA, or PFHxS, which are among the original 13, but are more soluble than longer chain PFAS and did not partition to detectable levels to the sediment solids at this site.

Concentrations of the detectable PFAS can be seen in Table 28, below.

Table 28: Site 8, Bergen Electrical Fire Site Sediment PFAS

	SOLID (ng/g (dry weight basis))	PFDoA	PFDS	PFNA	PFOA	PFOS	PFOSA	PFTeDA	PFTrDA	PFUnA	Total of 13 PFAS	% 13 of total (32)
8	FF5 (DS)	0.085	<	<	0.155	0.324	0.212	<	<	0.098	0.9	100.0%
8	FF6 (Whites Pond)	0.231	0.12	0.128	0.15	0.547	<	0.2	0.56	0.35	1.4	61.5%

Note: "<" represent results below the detection limits. DLs can be found in Appendix 4.

Fish Tissue

Three individual fish of each of three species, Bluegill, Largemouth bass, and Common carp, (nine total) were collected from Whites Pond, upstream of the confluence with the impacted tributary, for analysis of 40 PFAS. Although this subsampling location is not likely to represent direct impact from the unique source event to the tributary, potential long-term impact from groundwater exchange is possible, and the waterbody is commonly used for recreational fishing. For these reasons, the waterbody was assessed.

The proportion of the total PFAS that was comprised of the original 13 was much lower than has been found in surface water and sediments. Total PFAS (sum of 40 compounds that had measurable detections) ranged from 12.5 ng/g in one Common Carp to 25.6 ng/g in one Largemouth bass. The percent of the total of the original 13 PFAS to the total of 40 ranged from 16.5% in a Common carp to 84.9% in a Largemouth bass. The PFAS that making the largest contributions to the total, other than the original 13, were PFDS, PFTeDA, and PFTrDA, a 10 carbon perfluoroalkyl sulfonate, a 14 carbon perfluoroalkyl carboxylate, and a 13 carbon perfluoroalkyl carboxylate, respectively. These longer chain PFAS are often part of a product mixture used in AFFF and other products containing PFAS, and they are known to preferentially partition to proteins more strongly than the shorter chain perfluoroalkyl acids. N-EtFOSE was another PFAS found in all but one of the fish, ranging from 1.07 to 3.24 ng/g. N-EtFOSE was reportedly used to produce surface coatings for textiles and paper products (Zaggia and Ameduri, 2012).

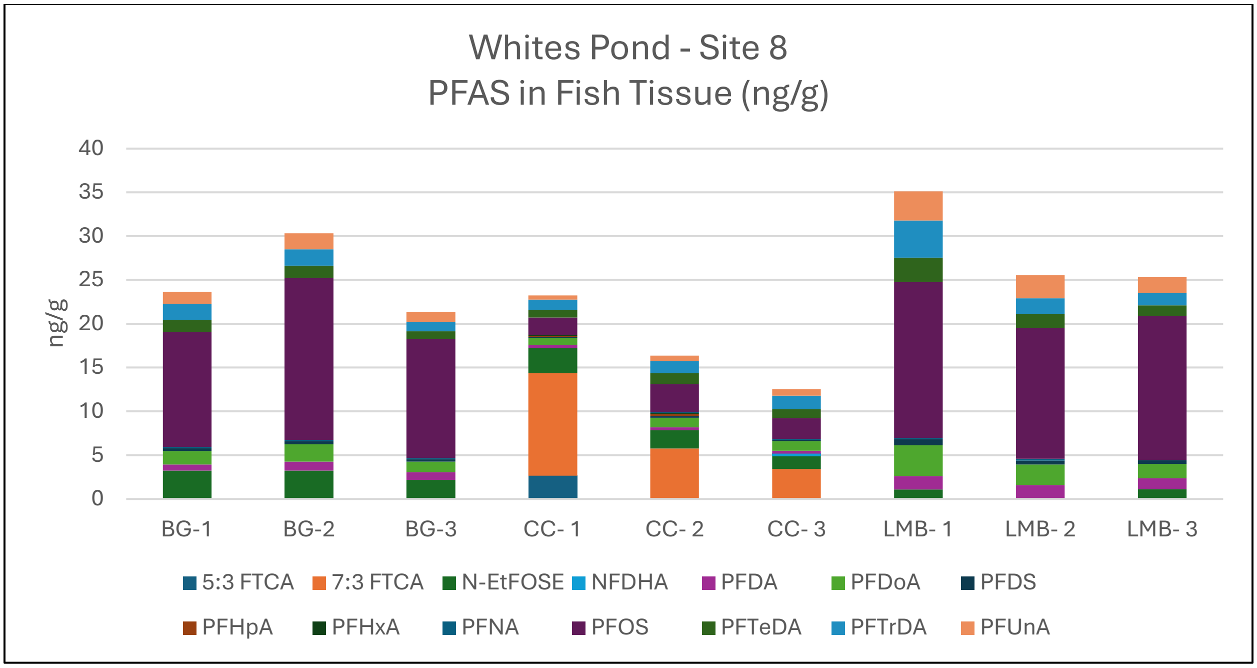

Figure 33: Whites Pond- Site 8 PFAS in Fish Tissue (ng/g)
Note: BG-Bluegill, CC- Common Carp, and LMB- Largemouth bass

Given the relatively low water solubility and high bioaccumulative potential long chain PFAS such as N-EtFOSE, PFDS, PFTeDA, and PFTrDA, it is possible for them to be detected in fish tissue but not detected in the water column, as is the case for this site.

Concentrations of 7:3 FTCA in the Common cod collected from Whites Pond are notable and should be further considered. Reported sources of 7:3 FTCA include degradation of fluorotelomer alcohols that have been released, possibly related to polymer manufacturing and/or AFFF releases.

Every fish also contained detectable levels of PFUnA, the eleven carbon perfluorocarboxylic acid which is one of the original 13 PFAS, ranging from 0.5 to 3.3 ng/L.

<u>Summary and Conclusions for Site 8</u>

The downstream subsampling locations had levels that were shown to be somewhat elevated above the water collected from Whites Pond, which may signify a source input. However, the values are too close to determine that with any level of certainty but could suggest that follow up sampling could provide better characterization.

The evaluation of the fish tissue from Whites Pond shows the bioaccumulation of PFOS and PFUnA at levels that could trigger consumption advisories. However, concentrations of other PFAS such as N-EtFOSE, PFDS, PFTeDA, and PFTrDA found here could be used to guide future efforts in evaluating the human impacts of those PFAS or combinations of PFAS detected in the environment. Since 7:3 FTCA detected in the Common Cod from Whites Pond has been reported as one of the three most common compounds found in a study of polar bears (Spaan, 2020), the occurrence of the release of this compound or its precursors should be considered in the future.

Joint Base McGuire-Dix-Lakehurst (Site 16)

The Joint Base McGuire-Dix-Lakehurst, located in both Burlington and Ocean Counties, is under active investigation by federal authorities for the impact of historic releases of AFFF used in firefighting training on this site. This investigation includes an evaluation of off-site impacts, and the data presented here is a small complement to that effort.

Five subsampling locations were included for this project. As can be seen in Figure 8, there are two subsampling locations on the northeastern boundary on the Ridgeway Branch (FSW3 upstream, and FSW2 downstream at the outer boundary of the base), an impoundment adjacent to the southern border of the base, upgradient of the Manapaqua Brook (FSW1), Horicon Lake (16-X3), and Pine Lake (16-X1).

Surface water and sediment grab samples were collected once from FSW1, FSW2, and FSW3, and analyzed for 33 PFAS. Two samples were collected from Horicon Lake on two different dates (one to more closely coincide with the collection of fish tissue), and the samples were analyzed for 40 PFAS.

Fish tissue was collected from three locations around the base, including the impoundment upgradient of Manapaqua Brook, Horicon Lake, and Pine Lake. Fish were the only environmental medium collected from Pine Lake. All fish tissue filets were analyzed for 40 PFAS.

Subsampling locations FSW1, FSW2, and FSW3 had 33 PFAS quantified in the surface water and sediment analyses, while analysis of the Horicon Lake surface water samples and all fish tissue samples also included the additional seven to report results on 40 PFAS. None of the additional seven PFAS had detectable levels in any of the samples in which they were analyzed.

Surface Water

Between 93 and 100 percent of the PFAS detected in the five surface water samples were from the list of the 13 original PFAS, with PFOS making up a large share of the total. The 13 PFAS (a mix of the PFAS in the original 13 list and others) that had detectable levels are shown in Figure 34, below.

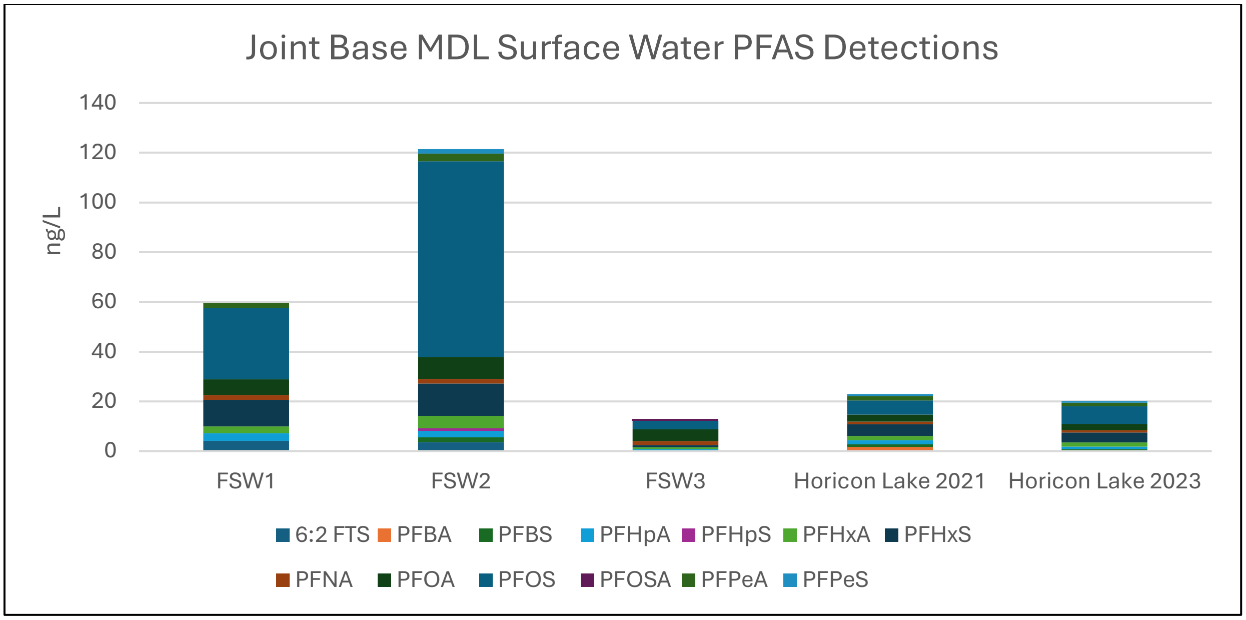

Figure 34: Site 16 Detectable Surface Water PFAS Detections in ng/L

FSW2, the subsampling station on Ridgeway Branch located on the north-eastern corner of the base, reported the highest levels of total PFAS at 121.4 ng/L, with 78.7 ng/L being PFOS. FSW1, the impoundment on the southern boundary of the base before Manapaqua Brook, also had elevated levels of PFOS at 28.6 ng/L. The two surface water samples collected from Horicon Lake two years apart show little difference in PFAS concentrations.

Foam

Foam was observed accumulating immediately after the outfall of Horicon Lake. Samples of the foam and related surface water were collected and sent for analysis. PFAS are known to have surfactant properties, and when PFAS are present in surface water at sufficient concentrations, foams can form with wind action or other turbulence. Evaluating enrichment factors can be helpful in understanding how PFAS moves to foam from the water column, along with the potential detection of PFAS that concentrate in foam but that may not be at detectable levels in the water column.

Total PFAS in the water column sample was 22.9 ng/L, while the total PFAS in the foam was 173.45 ng/L. A 15-fold enrichment was shown to occur for PFNA and PFOS, while PFUnA and PFDA were detected in the foam while being below the detection limit in the water samples.

Sediment

Thirteen PFAS (a mix of the original and others) were found at detectable levels in the sediments collected at the five subsampling stations around the Joint Base MDL (Figure 35). Total PFAS was less than 1 ng/g at all subsampling locations and ranged from 0.28 ng/g in the FSW1 sample from the southern impoundment to 0.561 ng/g in FSW2, the sample collected on the Ridgeway Branch at the north-east corner of the base. PFOS dominated the mass making up the total PFAS for these subsampling locations, with one hundred percent (100%) of PFAS in the FSW1 and FSW2 samples coming from PFOS.

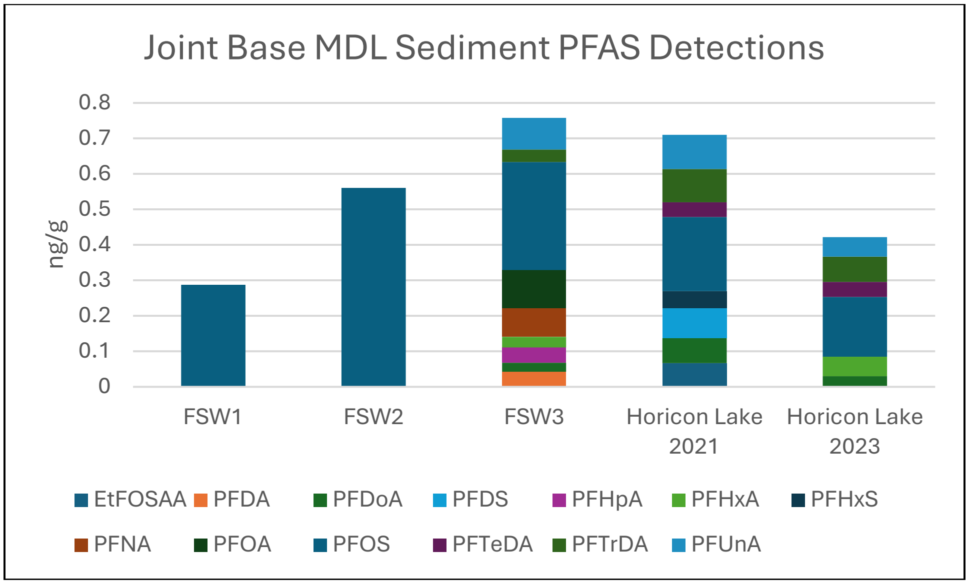

Figure 35 Site 16 Sediment PFAS Detections (ng/g)

The largest number of individual PFAS were detected in the FSW3 sediment sample, with nine PFAS making up the total PFAS at that subsampling location, albeit at relatively low levels.   Long chain carboxylic acids were detected in both of the Horicon Lake samples, in addition to the FSW3 sample.

<u>Fish Tissue</u>

Three fish each of different species that were present, were collected from three waterbodies around the Joint Base. Two species, Chain pickerel and Bluegill, were collected from the Manapaqua Impoundment, while four species, Bluegill, Chain pickerel, Brown bullhead, and American eel, were collected from Pine Lake, and two species, Chain pickerel and Yellow bullhead, were collected from Horicon Lake.

The eight chain sulfonate PFOS generally dominated the PFAS detected in the fish tissue, but relatively smaller levels of many other types of PFAS were quantified in these samples. Notable levels of 5:3 and 7:3 FTCA, were found American eel, contributing to the PFAS total in the fish collected from Pine Lake

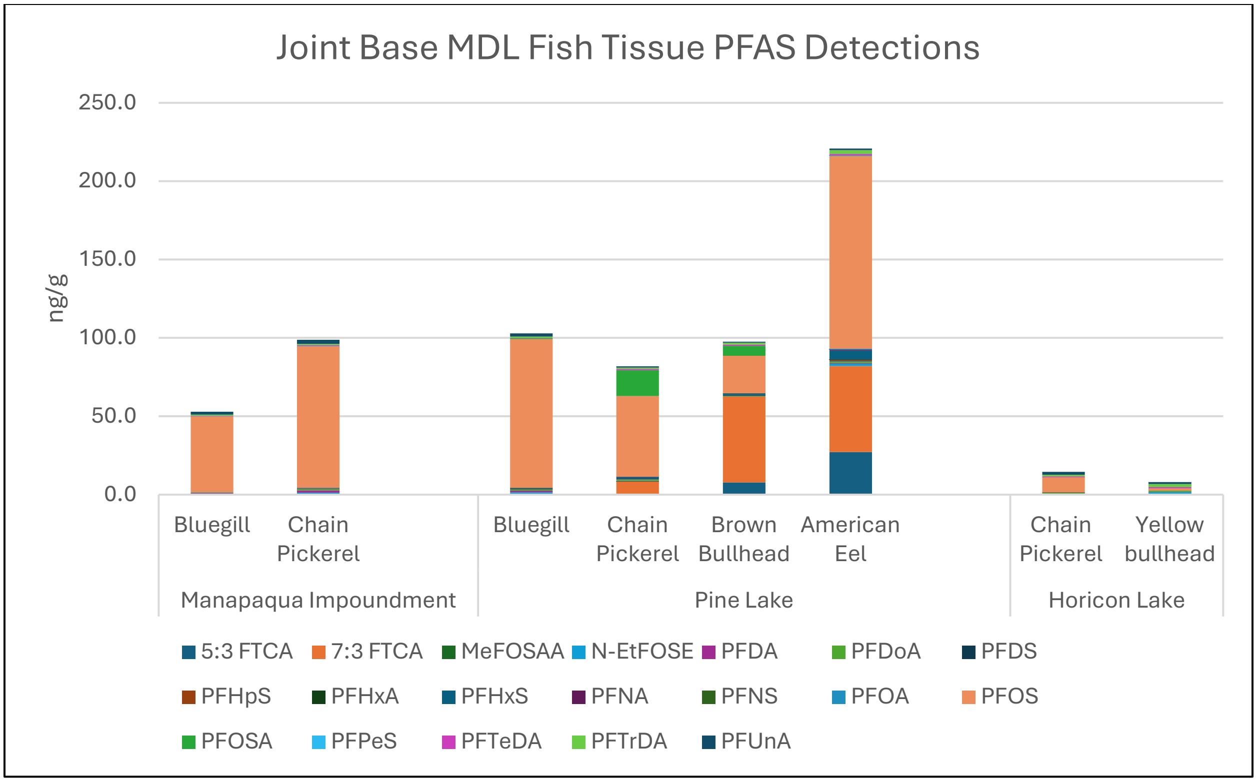

Figure 36: Site 16 Fish Tissue PFAS Detections (n=3 for each site-species)

PFOS ranged from 0.72 ng/g in a Horicon Lake Yellow bullhead up to 138 ng/g in the Pine Lake American eel.

PFOSA, a precursor to PFOS, was elevated at some subsampling locations, being detected in the Pine Lake Chain pickerel up to 25.0 ng/g. This level of an analyte that could contribute to the concentration of a PFAS with assigned trigger values may need to be considered to add protection.

The PFAS fingerprint in the Pine Lake fish tissue differed somewhat from the other fish in this area. The Pine Lake fish had notable levels of 5:3 and 7:3 FTCA, fluorotelomer carboxylic acids formed through the biotransformation of fluorotelomer alcohols. These FTCAs have been found in areas of landfill leachate release and have the potential to transform to the more recalcitrant perfluoroalkyl acids.

<u>Site Summary and Recommendations</u>

The distribution of the various analytes in the surface water and sediment indicates the samples collected from FSW1 and FSW2 have similar fingerprints that are likely related to the release of AFFF. The sample collected at FSW3 also contains a comparable fingerprint, with a few additional analytes detected. This may indicate an additional contributing source to this area.

5:3 and 7:3 FTCA were detected in the fish tissue at levels that could indicate the need for additional review of the potential impact to human health or the ecological system.

Levels of PFOSA detected in the fish tissue could transform and add to the total PFOS burden over time.

Pine Lake, along with Oakford Lake, had the highest PFOS levels in this study, and PFOS levels were higher than any fish tissue samples collected for the EPA study of PFAS in the fish of the Great Lakes.

Atlantic City (Site 17)

The subsampling locations for Site 17 covered both the potential AFFF source of the Atlantic City Airport (subsamples from upstream to downstream, ATLSU3, ATLSBA, and ATLMST) and an industrial area (subsamples ATLPC1 and ATLPC2). The industrial area results will be discussed in the Industrial Facilities section below. Surface water and sediment grab samples were collected at each of the subsampling locations, with an additional sediment sample collected at ATLSBA to evaluate heterogeneity. No fish tissue was collected in the area.

Surface Water

The surface water concentrations for the Atlantic City AFFF area can be seen in Figure 37. Total PFAS was made up of largely PFAS from list of the original 13, with larger proportions coming from PFOS, PFOA, and PFHxS. The sample ATLSU3, collected near the headwaters of the South Branch Absecon, was reported to contain only low levels of PFOA. ATLSBA6, collected at the outfall of the Atlantic City Reservoir, had total PFAS of 278.8 ng/L, with 112 ng/L from PFOS and 55.1 ng/L from PFHxS. Further downstream at ATLMST (mainstem), concentrations of PFAS dropped but still were reported to contain a total of 120.9 ng/L PFAS.

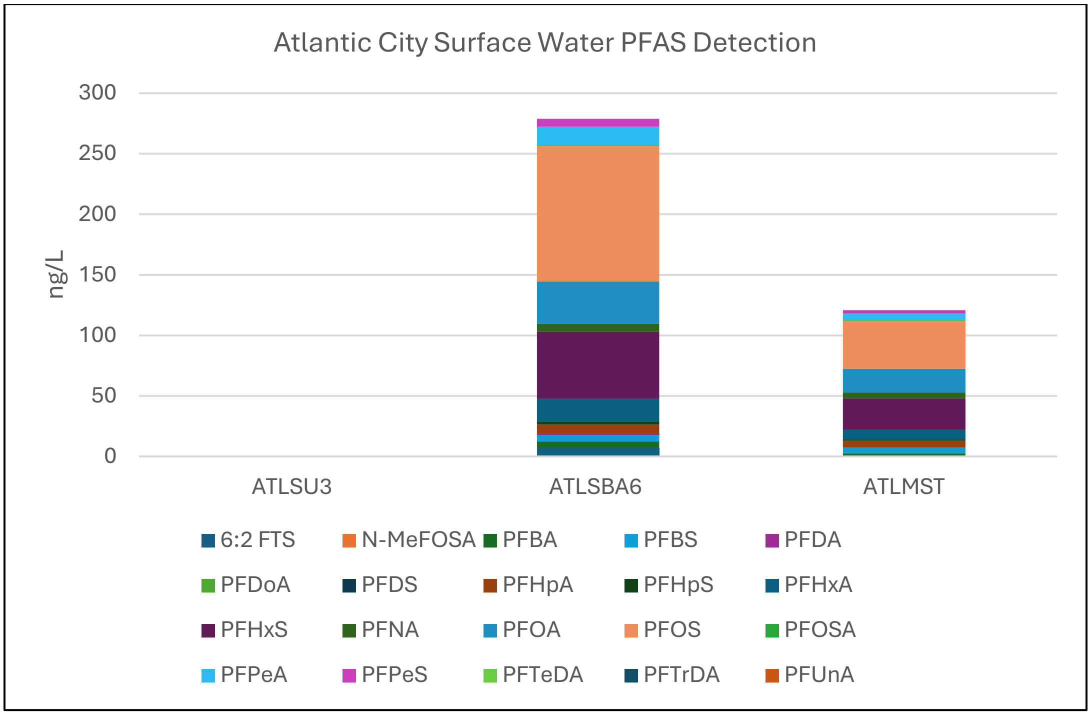

Figure 37: Site 17 Atlantic City AFFF Surface Water PFAS Detections

Sediment

One grab sample was collected at each of the three subsampling locations, with an additional grab sample collected at the outlet of the reservoir given the large range of sediment heterogeneity observed during sample collection.

Levels of various PFAS detected in the four samples can be seen in Figure 38, below.

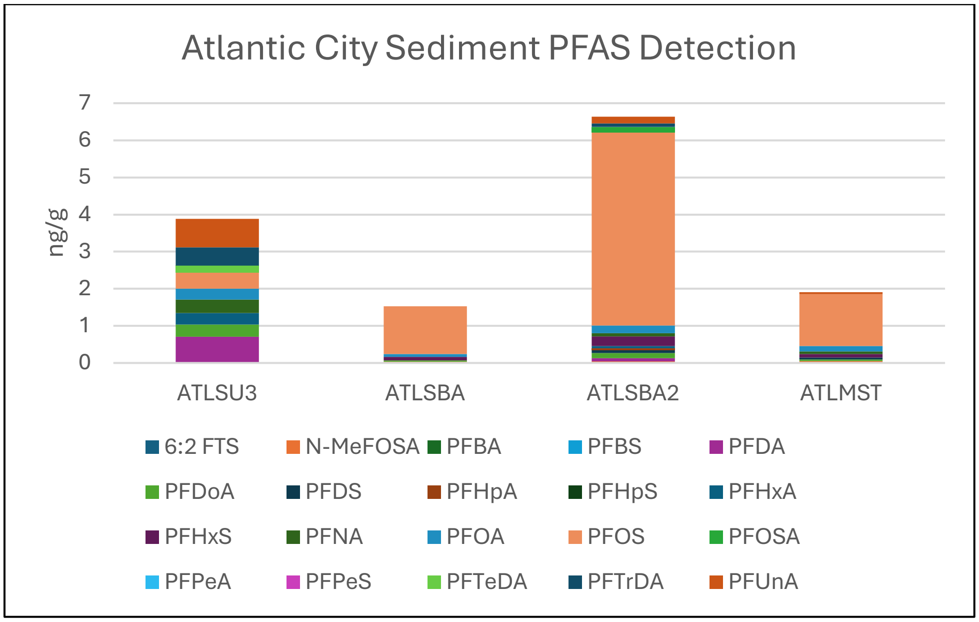

Figure 38: Atlantic City Site 17 PFAS Detection in Sediment

All subsampling locations had total PFAS greater than 1 ng/g (1 ppb), with the most upstream sample, ATLSU3 containing a wide variety of PFAS and the other three samples collected after the reservoir being dominated by PFOS. Variations in the PFAS levels between the two samples collected at the outlet of the reservoir (ATLSBA) represent the partitioning of PFAS to different types of sediments (not characterized here) and, potentially, the different locations of the two sediment samples.

Summary and Recommendations for Site 17

The levels of PFAS detected at the subsampling location just downstream of the reservoir discharge should be confirmed and investigated. The levels and types of PFAS present strongly indicate impact from an AFFF release.

Car Washes

Two locations were selected to represent the potential impact of car washes. Although car washes in New Jersey typically recycle their water and then discharge to wastewater treatment plants, one car wash, Tiny Bubbles in New Hanover (Site 6, see Figure 16), was permitted to discharge to groundwater. The other car wash, Surf City Car Wash in Toms River (Site 10, see Figure 17), was in an area where foam was observed on a downgradient waterbody and so was suspected of being a potential source.

New Hanover Car Wash (Site 6)

The Tiny Bubbles Car Wash in New Hanover is located north of the Joint Base McGuire-Dix-Lakehurst boundary to the west of Oakford Lake. As can be seen in Figure 16, four of the subsampling locations are on four tributaries to Oakford Lake. CW4 is on Beaverdam Brook flowing west to join Jumping Brook, just downstream of where the subsample CW3.1 was collected. CW3 is on the North Run flowing east to Oakford Lake, and CW2 is on a Crosswick's Creek tributary flowing east to Oakford Lake. The last subsampling location, CW1, is located at the downgradient section of the lake before discharge. Note: CW3.1 on Jumping Brook was selected to evaluate the potential contribution from the Joint Base.

Surface Water

Surface water collected at CW3.1 had a reported 51.2 ng/L of PFOS, 31.6 ng/L of PFHxS, and 7.06 ng/L of PFHpA. These three PFAS are known to be present in the mixture of PFAS found in some AFFF formulations. Sediment samples collected at CW3.1 also show elevated levels of PFOS, at 2.92 ng/g. The levels of PFOS in the surface water and sediment indicate a potential impact from sources related to the Joint Base and should be further investigated (see Figure 39).

CW1 is the subsampling station that would be expected to indicate groundwater or surface water that was impacted by the car wash discharge. However, the discharge from Oakford Lake at CW1 appears to relate to the subsampling station PFAS concentrations on the southeast tributary, CW3.1, which appears to be impacted by the Joint Base. The levels of PFHxS and PFOS, as well as 6:2 FTS, all indicators of AFFF contamination, are found at this outlet sampling station and do not appear significantly increased due to potential contributions from the site of the car wash.

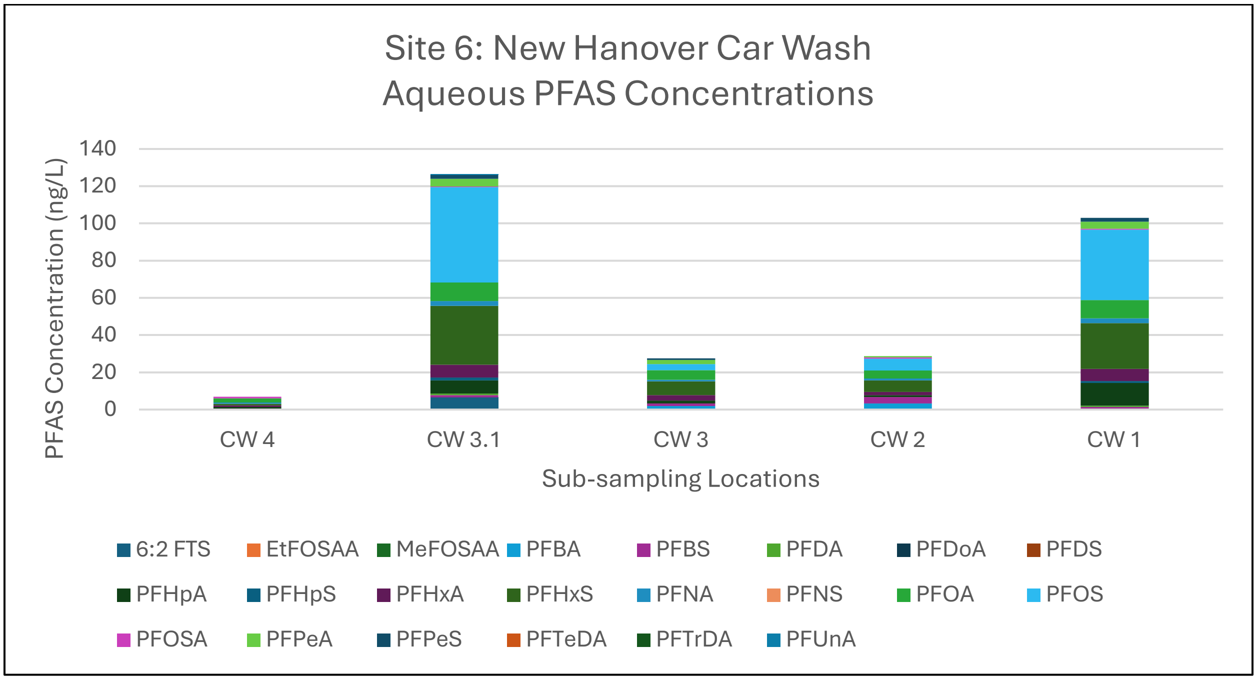

Figure 39: Site 6 New Hanover Car Wash Surface Water PFAS Concentrations

Sediment

Sediment concentrations showed a similar pattern as the PFAS concentrations in the surface water showed around the subsampling locations, with the highest concentrations including a total PFAS in sediment at the Jumping Brook CW3.1 subsampling location of 8.2 ng/g and the outlet of Oakford Lake (CW1) containing 5.1 ng/g, with large contributions from PFOS and PFDA. Sediment concentrations for the New Hanover car wash site can be seen in Figure 40, below.

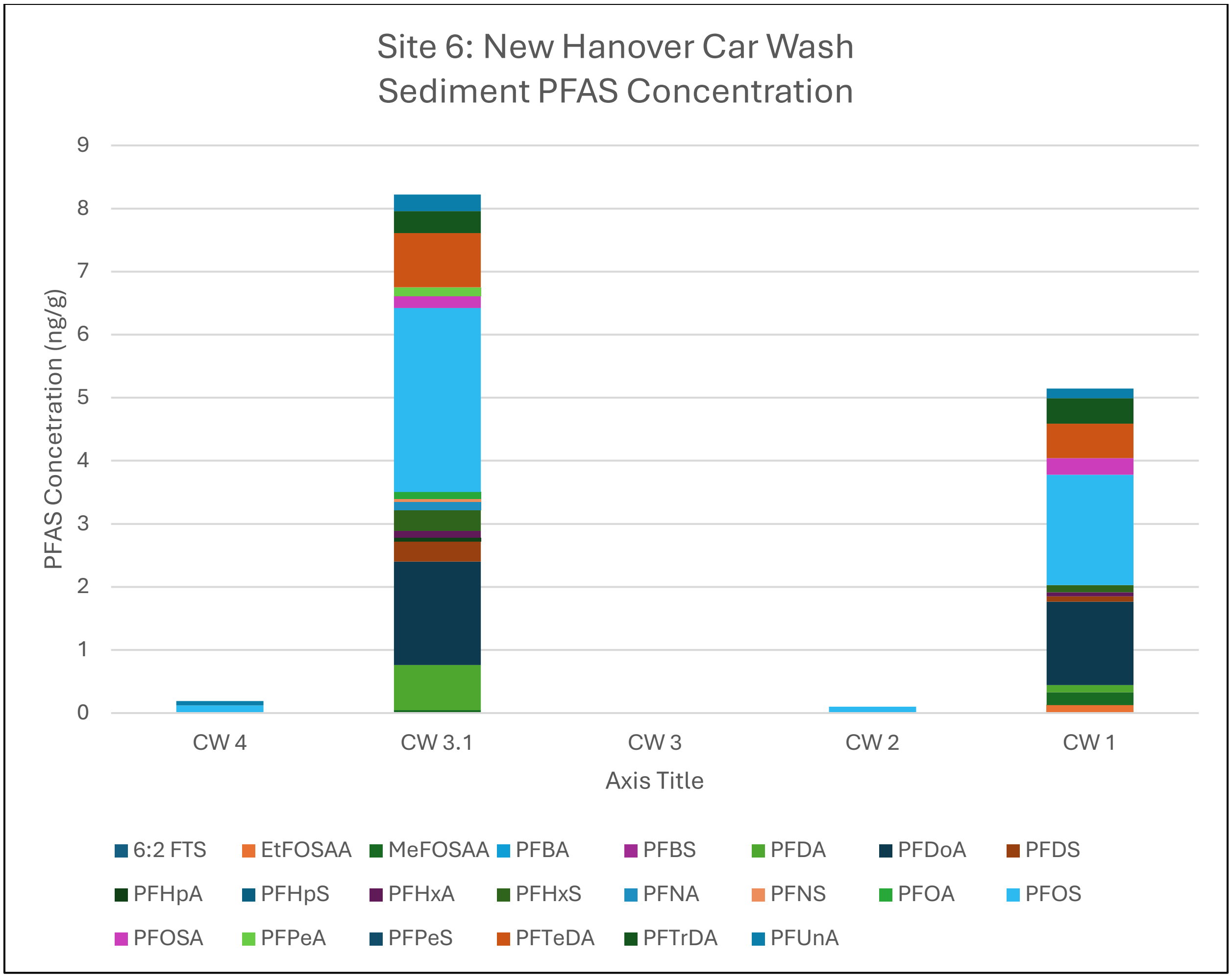

Figure 40: Site 6 New Hanover Car Wash Sediment PFAS Concentration

Fish from Site 6- Oakford Lake

Twelve fish were collected from Oakford Lake- three Brown bullhead, three Bluegill, three Common carp, and three Largemouth bass. The levels of PFOS ranged from 6.63 ng/g in a Brown bullhead to 249 ng/g in a Largemouth bass.

The levels of certain analytes found in all of the fish tissue samples indicate a similar pattern of PFAS that are commonly considered to originate in certain PFAS-containing AFFF products. PFOS dominates the input to the total PFAS. PFOS, PFHxS, along with the elevated levels of the fluorotelomer carboxylic acids, suggest that the Joint Base may be the origin of the PFAS contamination at this site. Table 29 and Figure 41 show the levels of various PFAS found in the fish tissue collected at Oakford Lake.

Table 29: Oakford Lake PFAS in Fish Tissue

TISSUE (ng/g (wet weight basis))	6:2 FTS	EtFOSAA	MeFOSAA	N-EtFOSE	NFDHA	PFBA	PFBS	PFDA	PFDoA	PFDoS	PFDS	PFHpA	PFHpS
BB1	<	<	0.373	1.36	<	<	<	0.277	2.64	<	0.313	<	<
BB2	<	<	0.368	<	<	<	<	0.349	3.07	<	0.334	<	<
BB3	<	<	0.231	<	<	<	<	0.316	2.85	<	0.311	<	<
BG1	<	<	0.158	1.29	<	<	<	1.56	6.67	<	1.34	<	0.115
BG2	<	<	0.339	2	<	<	<	1.09	3.84	<	0.79	<	0.142
BG3	<	<	0.146	2.24	<	<	<	1.03	3.63	<	0.845	<	<
CC1	<	0.123	0.197	1.98	0.816	<	<	1.62	6.34	0.118	1.05	<	0.317
CC2	<	<	0.335	3.11	<	<	<	1.09	3.6	<	0.701	<	0.17
CC3	<	<	0.308	2.4	0.583	<	<	1.17	4.47	<	1.18	0.133	0.362
LMB1	<	<	0.499	<	<	<	<	3.4	12.5	0.227	2.7	<	0.328
LMB2	<	<	0.197	<	<	<	<	1.33	6.51	<	1.11	<	0.137
LMB3	<	<	0.184	<	<	<	<	1.55	6.04	<	1.04	<	0.209

	PFHxA	PFHxS	PFNA	PFNS	PFOA	PFOS	PFOSA	PFPeA	PFPeS	PFTeDA	PFTrDA	PFUnA
BB1	<	0.201	<	<	<	6.63	0.914	<	<	4.56	1.38	0.503
BB2	<	0.251	<	<	<	10.8	1.21	<	<	3.62	1.6	0.854
BB3	<	0.426	<	<	<	8.04	1.54	<	<	2.65	1.31	0.699
BG1	<	0.406	<	0.233	<	142	0.624	<	<	4	3.17	3.15
BG2	<	0.361	<	0.113	<	68.9	0.759	<	<	2.14	1.34	2.17
BG3	<	0.269	<	0.131	<	94	0.405	<	<	2.04	1.46	1.61
CC1	<	2.49	0.212	0.139	0.112	58.8	0.642	<	<	4.15	2.36	1.54
CC2	<	1.33	0.149	<	<	43.8	0.726	<	<	2.12	1.51	1.43
CC3	<	3.19	0.205	0.107	<	47.6	1.05	<	<	4.22	2.38	1.54
LMB1	<	0.809	0.147	0.469	<	249	0.253	<	<	6.16	4.35	4.34
LMB2	<	1.07	<	0.162	<	76.2	0.212	<	<	3.64	2.9	2.09
LMB3	<	0.957	0.188	0.163	<	87.3	0.185	<	<	3.65	2.37	2.1

Note: Abbreviations for fish samples: BB= Brown bullhead, CC= Common carp, LMB= Largemouth bass, BG= Bluegill. "<" denotes value less than detection limit. Detection limits can be seen in Appendix 4.

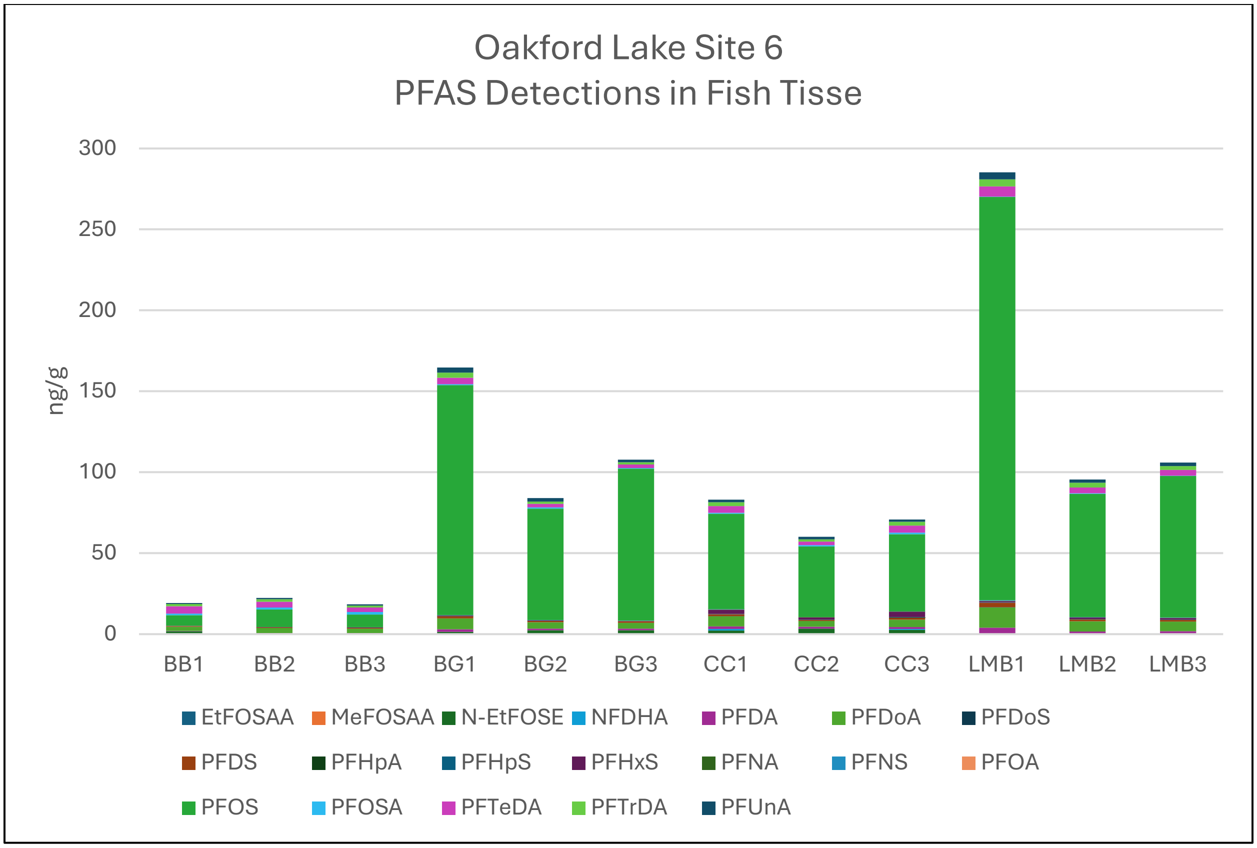

Figure 41: Oakford Lake PFAS in Fish Tissue (ng/g)

Toms River Car Wash (Site 10)

Surface Water

The identified car wash is located on Route 37/Locust Street in Toms River. The subsampling locations were selected along a tributary to the Barnegat Bay that would likely include a groundwater-to-surface water connection. The first subsampling station, CW6, was intended to represent the upstream location that would not likely be impacted by a release at the car wash, CW7 was located downstream on the same tributary, Long Swamp Creek, and the last subsampling location, CW5, was located in the bay directly where the forked tributary discharges and where foam was originally reported by a local resident.

Low levels of perfluorocarboxylic acids, such as PFBA (3.84 to 4.47 ng/L) and PFOA (5.98 to 6.02 ng/L), and perfluorosulfonic acids such as PFBS (4.82 to 5.58 ng/L) and PFOS (4.54 to 4.96) were detected in the surface water samples, CW6 and CW7, with no notable differences between the two samples. The single surface water grab sample collected at the mouth of the tributary where mixing is occurring with Barnegat Bay showed a higher PFOS level of 20 ng/L, with PFOA detected at 5.18 ng/L and PFHxS at 5.51 ng/L. See Figure 42 for PFAS detections at the Toms River car wash site.

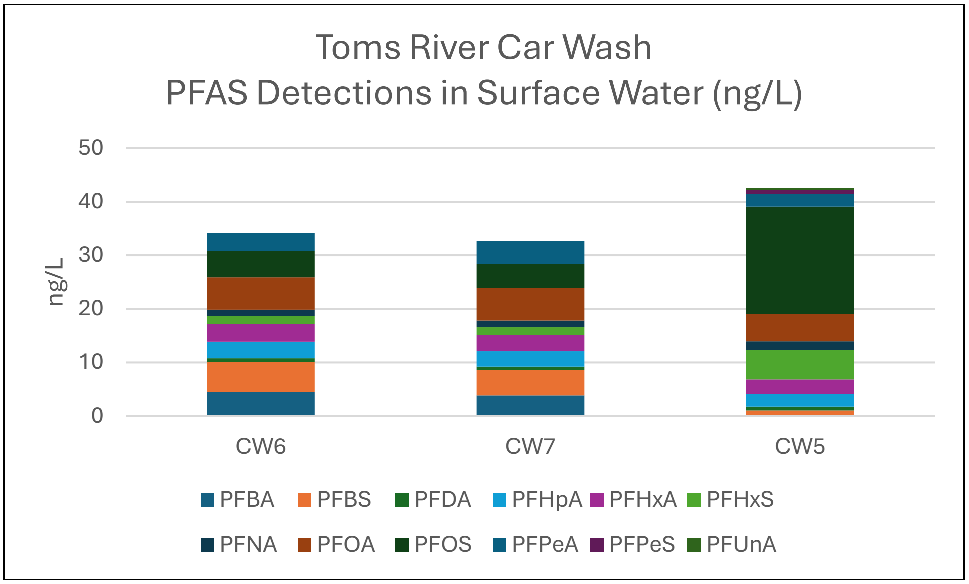

Figure 42: Toms River Car Wash PFAS Detections in Surface Water (ng/L)

Sediment

Sediment samples collected at the same two upstream subsampling locations also showed low levels of several PFAS, with the highest detectable level of 0.582 ng/g of PFOS at the downgradient subsampling location (Figure 43). A sediment sample was not collected at the mouth of the stream emptying into Barnegat Bay since only sandy, unconsolidated materials were present and therefore were not expected to represent a valid partitioning when analyzed under dry weight conditions.

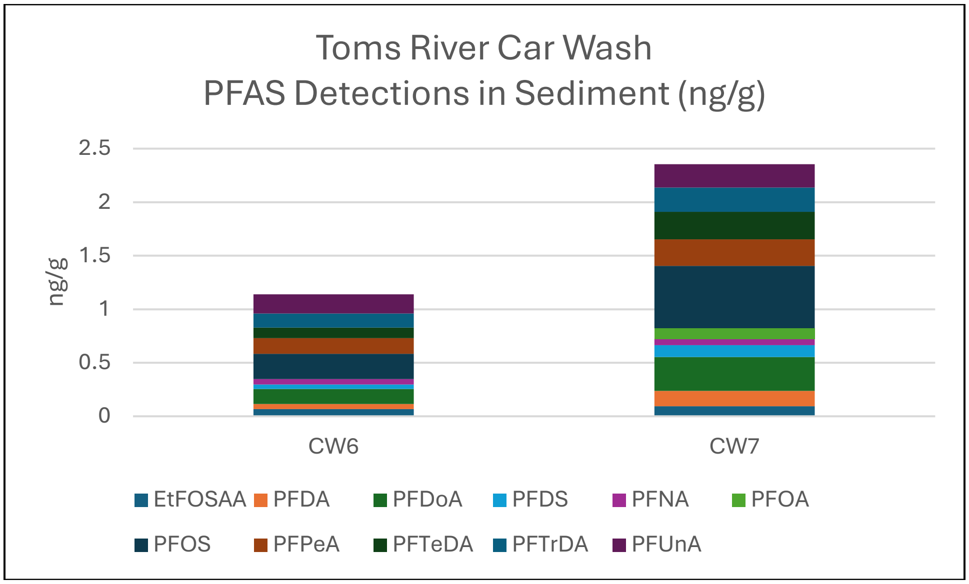

Figure 43: Toms River Car Wash PFAS Detections in Sediment (ng/g)

Fish

No fish were collected at this site.

Summary and Recommendations for Car Wash Sites (6 and 10)

Other states have reported discharges of PFAS from car washes impacting the surrounding environment. The two NJ sites selected for this evaluation, based on past permitting or reports of nearby foam, did not show obvious signs of impact from potential releases. However, PFAS levels in the surface water and sediment do indicate that PFAS has entered the system by some pathway. Levels here are relatively low, but may indicate the need for additional characterization, particularly around Barnegat Bay.

The state of New Jersey no longer has car wash facilities that have a permit to discharge to groundwater. Car washes now either discharge to the sanitary sewer where the water will be treated at a wastewater treatment facility, or the car wash water is recycled on site.

Due to the past report of foam/suds in this area, and the level of PFAS detected in the Barnegat Bay surface water sample, additional monitoring could be considered to properly understand the PFAS burden this area.

Industrial Facilities

Three sites included in this study were selected to evaluate the potential release of PFAS from nearby industrial activities that could be suspected of releasing PFAS during their use or manufacturing process. These sites include Site 9 in Bethlehem Township in Hunterdon County (plastics facilities), the southern portion of Site 17 near Atlantic City (plastics facility), and Site 18 in Ringwood, Passaic County (located by the Ford waste disposal site). Three other locations were evaluated with a single sample, allowing for a general follow up to the earlier NJDEP PFAS study (Goodrow, et al., 2020) (Site 19, Forge Pond) or to complement fish tissue evaluation for an initial PFAS characterization of the location (Site 20, Deal Lake, and Site 21, Timber Swamp Brook at outfall of Manasquan Reservoir).

Bethlehem (Site 9), Atlantic City (Site 17), and Ringwood (Site 18)

Bethlehem (Site 9, Figure 11), Atlantic City (Site 17, Figure 9), and Ringwood (Site 18, Figure 12) will be reviewed together here to get the benefit of comparison between the potential localized impact of industrial sites. The Bethlehem and Atlantic City (Egg Harbor) sites had not been previously evaluated for PFAS occurrence and were targeted to be a part of this study solely because a facility near these sites was thought to work with chemical compounds potentially related to PFAS. The Ringwood site had some previous reports of low level PFAS on the site, which was delineated for the disposal of paint sludge and related contaminants (Li, et al., 2024). In this study, researchers from the New Jersey Institute of Technology reported levels of PFAS detected in the sample collected from a nearby stream with foam located outside of the delineated boundaries for remediation.

Surface Water

Seven PFAS were not quantified by the earlier analytical method used for the two Site 9 samples, but none of those PFAS were detected in the other samples from the six subsampling locations at Sites 17 and 18 that were analyzed later. The original 13 PFAS comprises between 85 and 100% of the PFAS detected in the samples, with 6:2 FTS detected in Ringwood, upstream of the Wanaque

Reservoir, and 3:3 FTCA notably quantified at 5.33 ng/L in the Atlantic City subsampling station located closest to the targeted industrial facility. Levels of PFAS detected in the surface water samples collected at Sites 9, 17, and 18 can be seen in Figure 44, below.

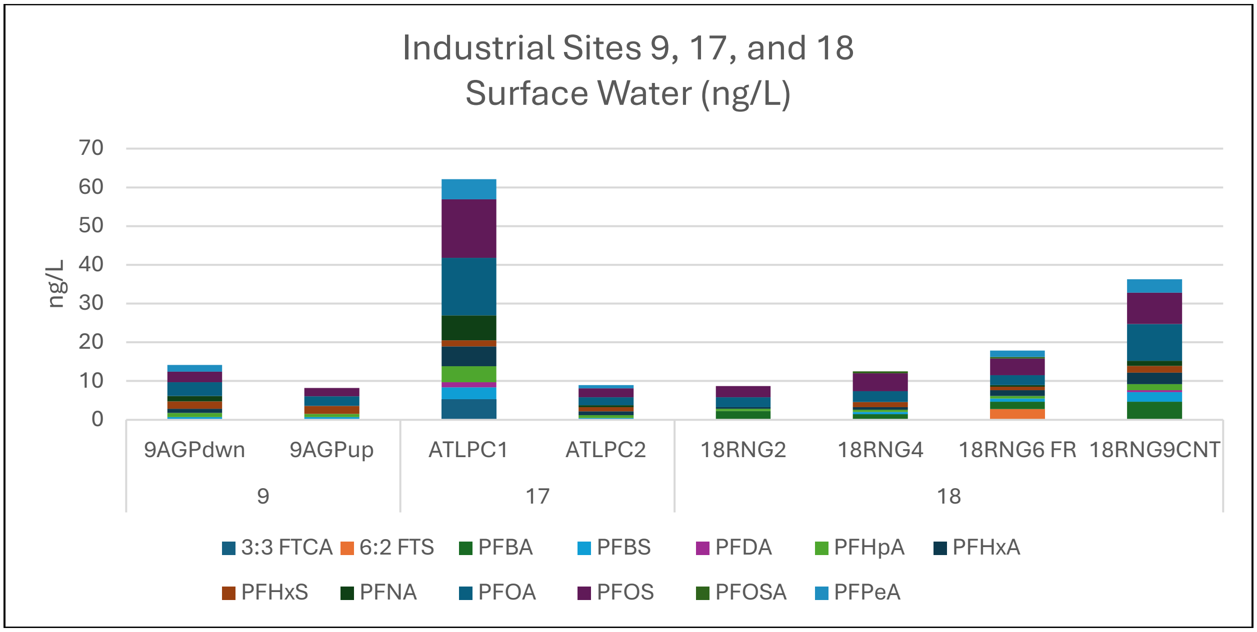

Figure 44: PFAS levels in sediment samples from industrial sites 9, 17, and 18

Sediment

Seven PFAS were not quantified in Site 9 samples, but as was the case with the surface water samples, none of those PFAS were detected in samples from any of the other six subsampling locations. The specific PFAS that made up the total PFAS in the sediments included a larger array than just the original 13, with notable levels of EtFOSAA, N-MeFOSA, and the long chain PFAS PFTeDA and PFTrDA, in addition to higher levels of PFUnA than were detected in the surface water. Detection of these various PFAS in the sediments may give an indication of source type and partitioning capacity. Levels of PFAS detected in the sediment samples collected at Sites 9, 17, and 18 can be seen in Figure 45, below.

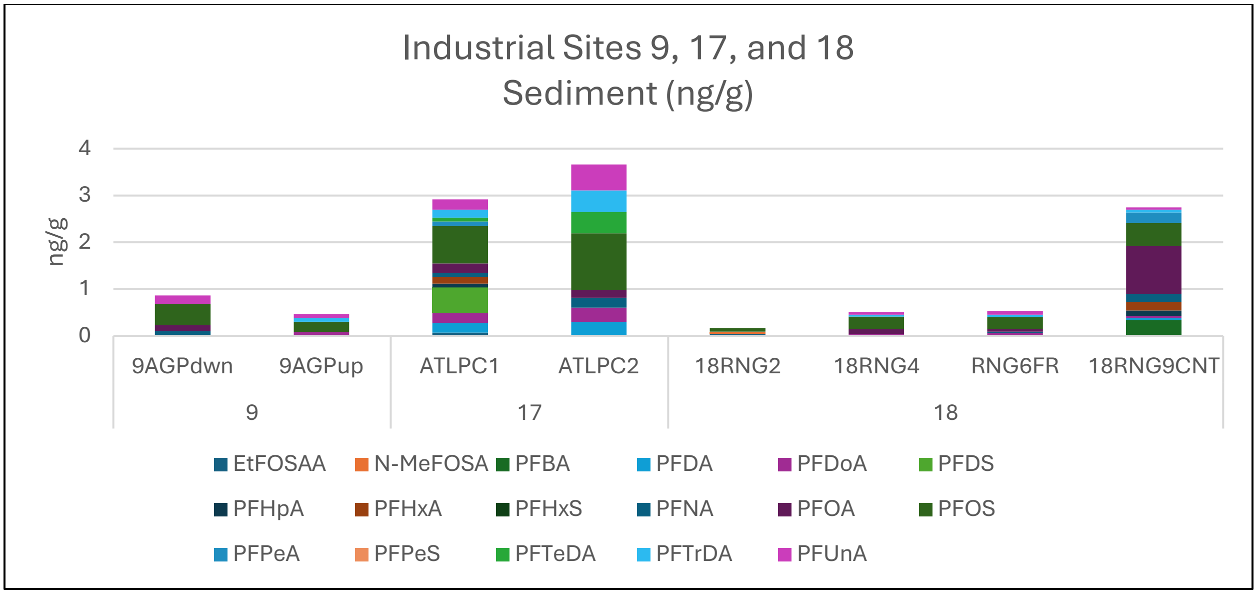

Figure 45: PFAS levels in sediment samples from industrial sites 9, 17, and 18

Forge Pond (Site 19), Deal Lake (Site 20), and Timber Swamp Brook (Site 21)

Forge Pond (Site 19), Deal Lake (Site 20) and Timber Swamp Brook (Site 21) were evaluated with a single surface water and sediment sample collected at each site, and the evaluation of the site also includes fish tissue collected from the main waterbody.

Surface Water

PFAS concentrations at Forge Pond (19) and Deal Lake (20) were elevated (> 40 ng/L) compared to the sample from Timber Swamp Brook, with PFOA and PFOS playing a dominant role in the total PFAS. Timber Swamp Brook (21), at the outlet of the Manasquan Reservoir, reported lower levels of total PFAS, under 20 ng/L. See Figure 46, below.

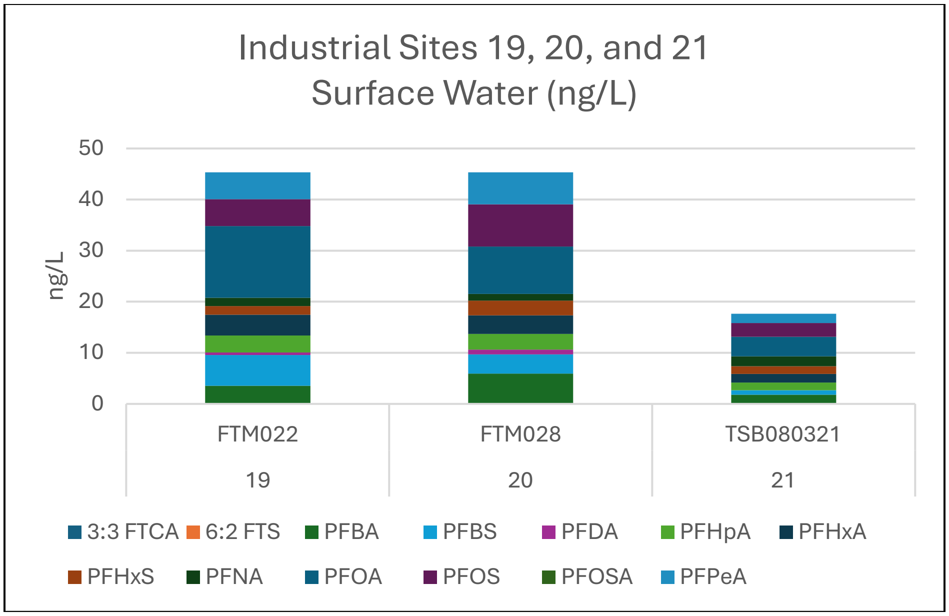

Figure 46:PFAS levels in surface water samples from industrial sites

<u>Sediment</u>

Total PFAS in the sediment samples collected at the Forge Pond (19) and Deal Lake (20) sites were under 1 ng/g (1 ppb) and contained only low levels of a limited number of PFAS. The sample collected at the Timber Swamp Brook site (21) reported nine different PFAS for a total of just under 2.0 ng/g. See Figure 47, below.

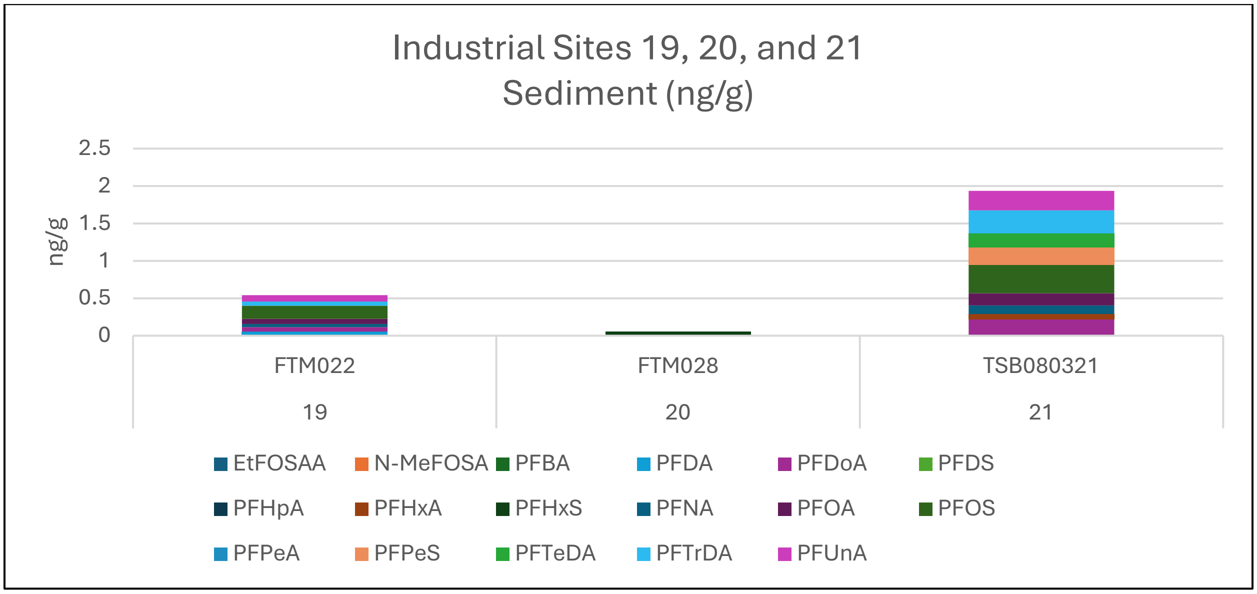

Figure 47: PFAS levels in sediment samples from industrial sites 19, 20, and 21

Fish Tissue

The fish tissue collected at the three industrial-related sites 19, 20, and 21, show some levels of PFAS. All fish contained levels of PFOS 0.56 ng/g, which is the level above which unlimited (daily) consumption is not recommended (NJDEP Toxics in Biota Risk Subcommittee, 2018) and three fish had greater than 17 ng/g, the level above which consumption more frequently than once a month is not recommended for the general population value and no consumption is recommended for the high risk population.

It is evident looking at the analyte distribution as shown in Figure 48, that PFOS is the dominant analyte, while up to 17 other analytes appear to have bioaccumulative potential and could present a possible burden to these systems.

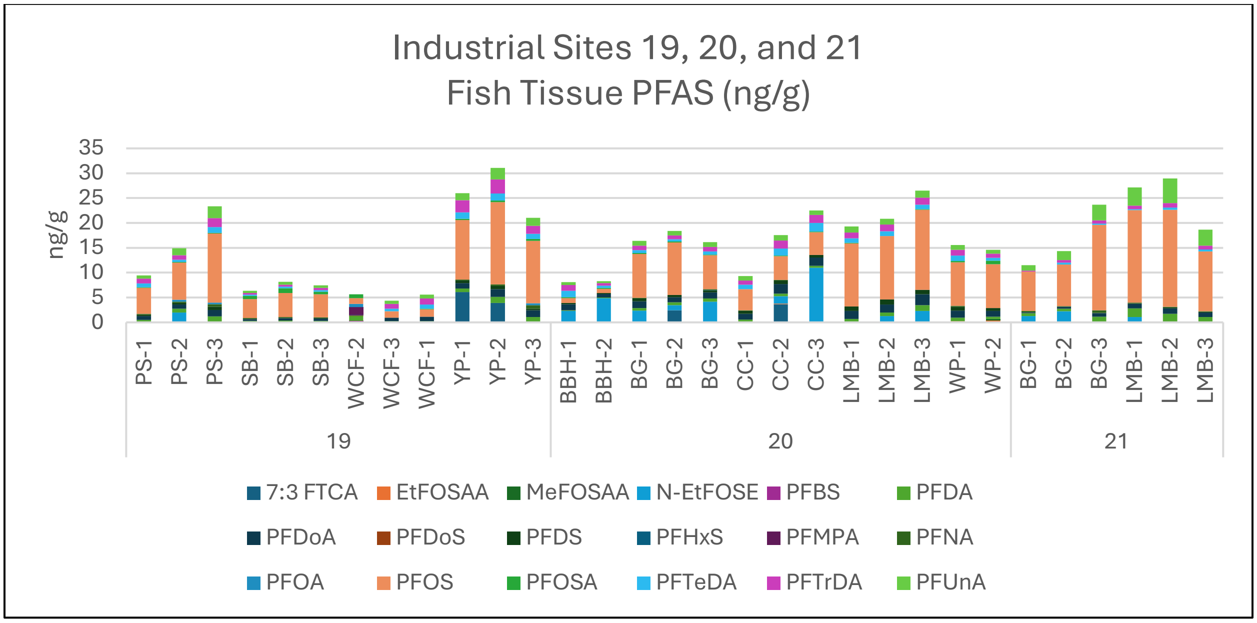

Figure 48: Industrial Sites 19, 20, and 21 Fish Tissue PFAS (ng/g)

Summary and Recommendation for Industrial Sites

The six industrial or industrial-adjacent sites detailed here have been impacted by PFAS sources, although it is not clear if the PFAS detected would be directly related to any industrial activity.

Wastewater Treatment Plants

Eight surface water and eight sediment samples were collected in the areas of five wastewater treatment plants. The intention was to collect samples upstream and downstream of discharge points, but limited access deterred some planned sampling. Samples collected in the earlier phase of this project (Sites 11, 12, 14, and one sample from Site 15) did not include analysis for several analytes such as 3:3, 5:3, and 7:3 FTCA, but all analytes not quantified in earlier analyses were reported as below the detection limit in later samples collected for the evaluation of wastewater discharge areas.   Therefore, these PFAS were not determined to be of concern at this time.

Wastewater treatment plants do not produce or use PFAS, but they accumulate various PFAS from domestic sources as well as landfill leachate and discharge from industrial sources that they may receive. Many PFAS that enter the wastewater treatment system are considered precursors and will transform to the more recalcitrant perfluoroalkyl acids (PFAAs). The longer chain PFAAs may preferentially partition to the residuals within the wastewater treatment plant, which may be removed to another location for disposal or destruction, or be discharged in the wastewater treatment effluent. Traditional wastewater treatment does not remove PFAS from these waste streams and can increase the levels of the more recalcitrant perfluoroalkyl acids due to the transformation of precursors that enter the system within the treatment plant.

The mass of PFAS contained in the water discharge from these systems could impact the receiving water body. PFAS concentrations observed in the upstream waters indicate other anthropogenic sources and may be higher or lower than downstream concentrations due to mass input and flow dilution. Although the water discharged has the potential to dilute PFAS concentrations already present in the streams, any mass release of PFAS into an aquatic system provides a source for long-term accumulation.

Surface Water

The concentration of total PFAS in the surface water samples collected from the eight sampling stations located around the wastewater treatment sites ranged from 36.4 ng/g at the downstream subsampling location of Site 15, Wayne Sheffield, to 83.1 ng/g at the downstream subsampling location of Two Bridges Sewage Authority. Surface water samples collected downstream of a discharge point should be considered representative of a mixed area and it is not a quantification of PFAS in the direct effluent. The various PFAS making up the total PFAS for these eight samples was largely driven by the original 13 PFAS, making up from 91% of the total at Two Bridges to 100% of the total at three subsampling locations. PFPeA, PFOS, and PFOA all contributed notable levels to the total PFAS measured in the eight samples. In addition to the original 13, the short chain sulfonate, PFPeS, contributed to five samples, and 6:2 FTS contributed to two samples (Sites 12 and 13). Detectable levels of PFAS found in the surface water at the eight subsampling locations at five sites can be seen in Figure 49, below.

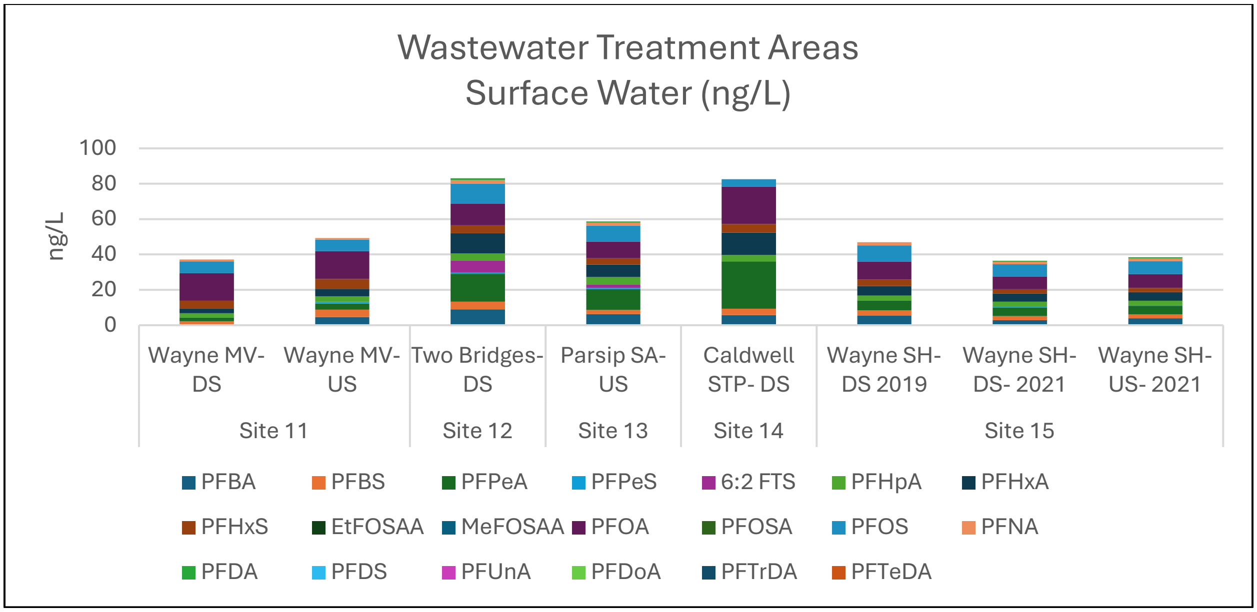

Figure 49: PFAS Concentrations at Wastewater Treatment Areas (ng/L)

Sediment

Out of the 33 PFAS quantified in the wastewater treatment area sediments, only 18 PFAS had detectable levels reported, and of those, five PFAS had only a single detection in any of the eight samples. Levels can be seen in Figure 50, below. PFOS was detected in seven of the eight samples, and PFTrDA and PFDoA were detected in six of the eight samples. Sediments from two of the subsampling locations contained less than 1 ng/g (1 ppb), while sediments from five subsampling locations were between 1.18 and 3.39 ng/g. The sediment collected where the Caldwell STP discharges contained the highest level of total PFAS, 6.6 ng/g.

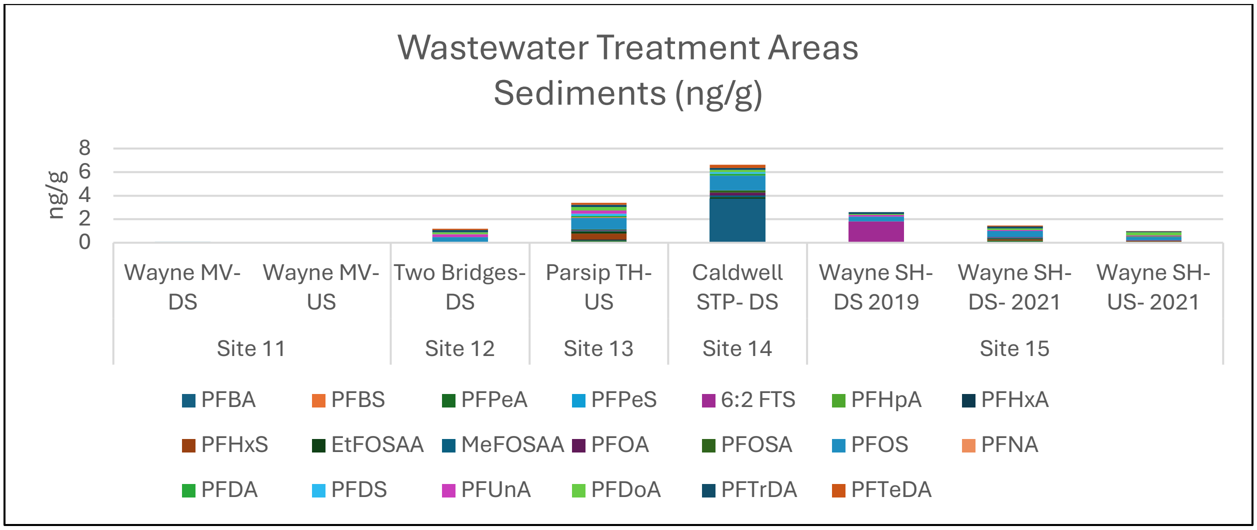

Figure 50: PFAS in Sediments in Wastewater Treatment Sites

Fish Tissue

Of the five sites selected for analysis in the wastewater category, only one site had accessible fishing options. This location was in the area of the Two Bridges Sewage Authority. Many of the individual fish collected in this area of wastewater discharge contained total levels of PFAS above those detected in the industrial site samples. The levels bioaccumulation between fish species can be seen with the lower concentrations of PFAS in the Yellow bullhead, and relatively higher concentrations in the Bluegill and Common carp that were present in the same waters. The total PFAS in all fish was largely dominated by PFOS.

Notable levels of 5:3 FTCA and 7:3 FTCA were detected in the carp. One sample of carp tissue showed a level of 6:2 FTS of 10.8 ng/g. These unique fingerprints found in the fish tissue should be confirmed but may assist source identification.

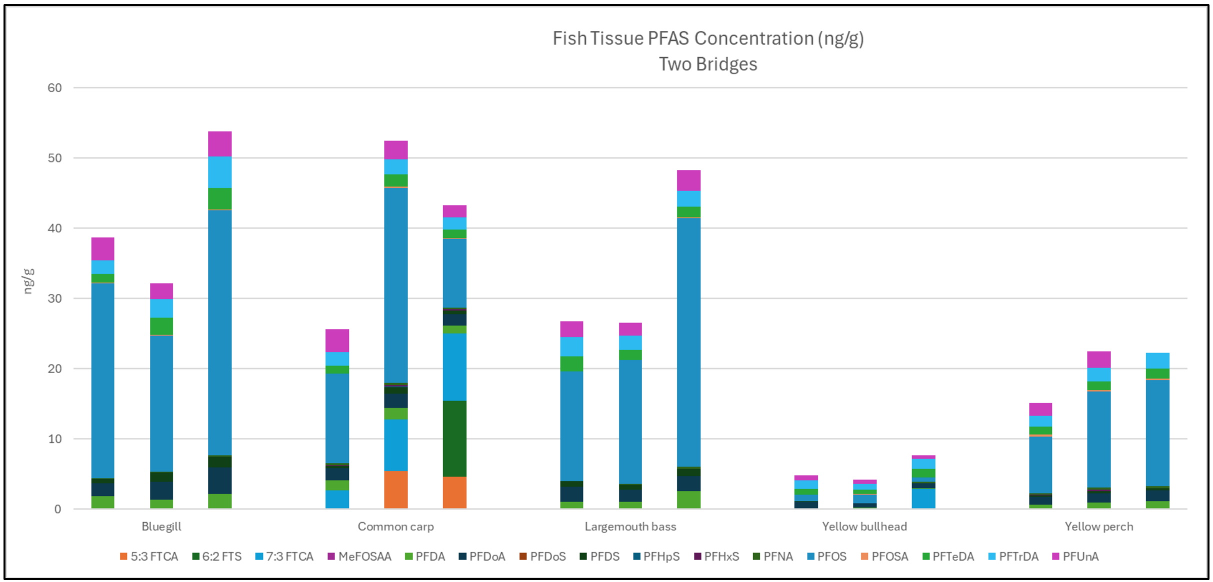

Figure 51: Fish Tissue PFAS Concentrations Two Bridges (ng/g)

Ponds

Five samples of both sediment and surface water were collected from four ponds. These ponds were selected to represent impoundments located at some distance from recognized anthropogenic activities and were assumed to have limited impact from overland migration of contaminants. The sample collection locations can be seen in Figure 24.

Surface Water

The original 13 PFAS comprised between 95.7% (Butterworth Pond) and 100% (Cedar Pond and both Echo Lake samples). The only PFAS with detectable levels that was not in the original 13 was HFPO-DA, which was detected at Butterworth Pond (0.705 ng/L) and Locustwood Pond (0.583 ng/L).

Locustwood Pond, a pond in an urbanized landscape, had the highest total PFAS, almost 40 ng/L, notably containing levels of PFNA (10 ng/L), PFOA (6.96 ng/L), and PFUnA (1.81 ng/L). The elevated levels and unique fingerprint of PFAS found at Locustwood Pond may suggest a contribution from a local source. Additional sampling is advised.

Butterworth and Cedar Ponds contained total PFAS of 16.4 and 15.1 ng/L, respectively. The specific PFAS were distributed between the short chain PFAS, PFPeA and PFBA, and low levels of the longer chain PFAS, PFNA, PFOA, and PFOS.

Total PFAS detected in both surface water samples collected from Echo Lake were less than 2 ng/L. Low levels of PFOS were detected in both samples, and the Echo Lake #2 sample included low levels of PFNA and PFOA.

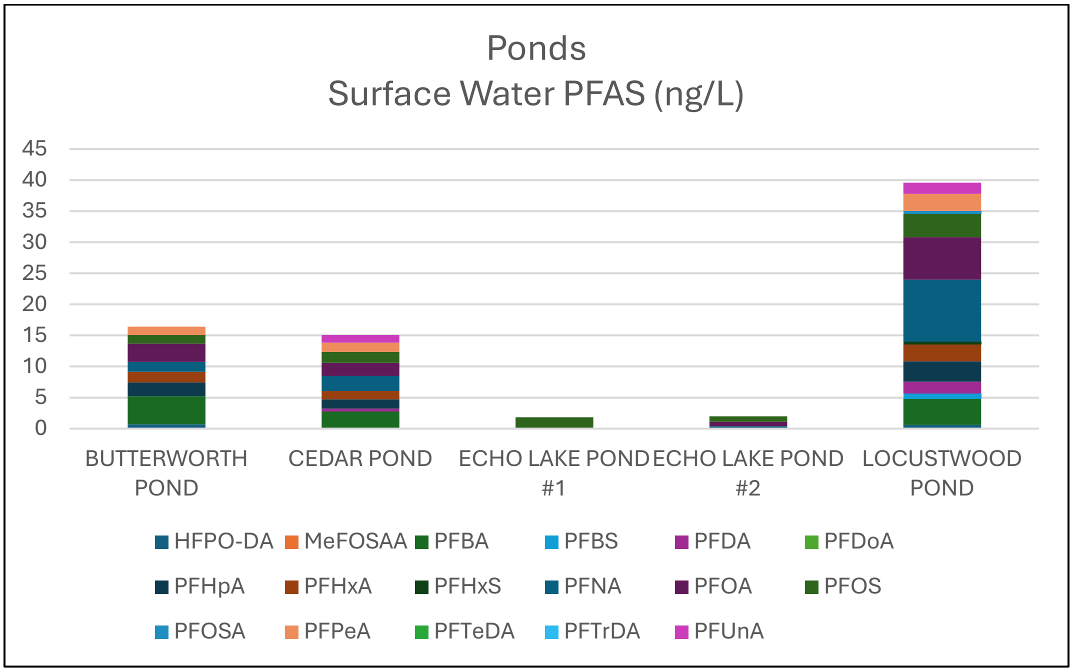

Figure 52: Ponds Surface Water PFAS (ng/L)

Sediment

Total PFAS quantified in the sediment samples ranged from 1.86 ng/g in the Butterworth Pond sample to 8.06 ng/g in the Locustwood Pond sample (see Figure 53). The original 13 PFAS made up between 72 (Echo Lake #2) and 94% (Cedar Pond) of the total PFAS detected. Two long-chain PFAS, PFTeDA and PFTrDA, were detected in three and four samples, respectively.

As the long chain PFAS preferentially partition to sediments, the detectable levels of PFAS found in the sediment of these isolated ponds were dominated by the long chain PFAS, including PFDA, PFDoA, PFNA, PFOS, and PFUnA. Lower levels of the short chain PFAS, PFPeA, PFHxA, and PFHpA were detected in fewer samples.

The sediment sample collected from Locustwood Pond, like the related surface water sample, contained elevated levels of PFNA and PFUnA, particularly when compared to the other pond samples. PFOA was in a similar range to the other samples.

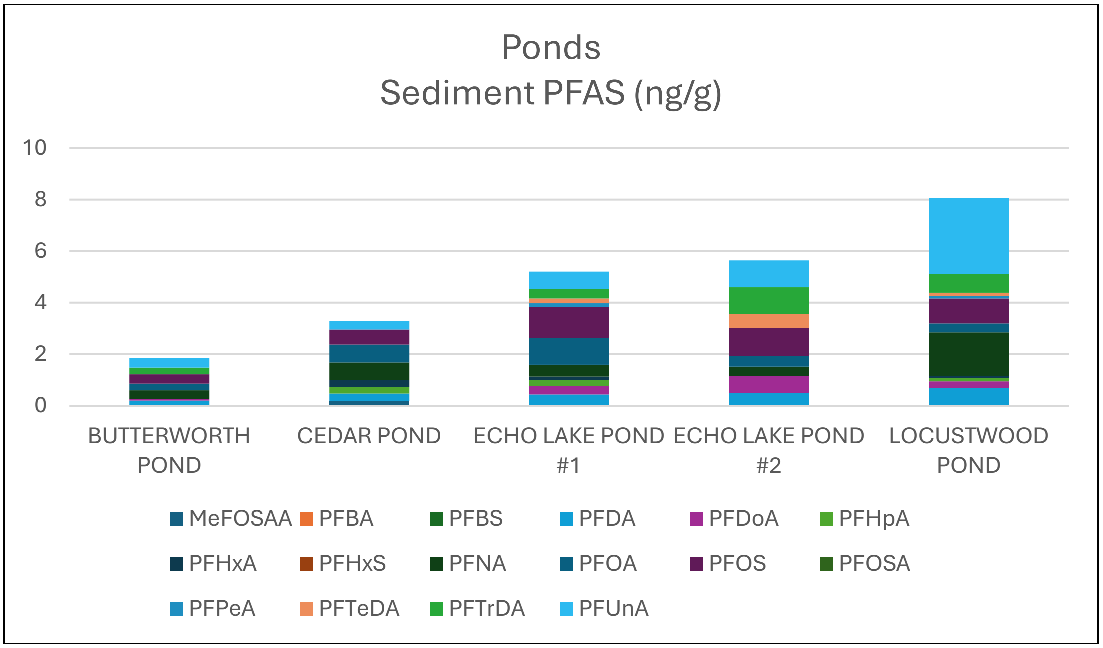

Figure 53: Ponds Sediment PFAS (ng/g)

Conclusion on PFAS Study

This report characterizes locations with potential PFAS source contributions to the environment.

On occasion, an initial sample will provide valuable details that may indicate a type of PFAS source, as when PFOS, PFHxS, and certain fluorotelomer are detected that could point to an AFFF source. Similar fingerprints at various sites could show relationships among the sites and indicate a similar source contribution. However, all data collected for this study should be considered a preliminary evaluation that requires confirmatory sampling before firm conclusions can be made.

There is much to learn from both detections of specific analytes in a sample and from lack of detection of other analytes that are quantified by the analytical method that was used. In addition to PFAS such as PFOA, PFOS, and PFNA that are well known to have been used and released for many years, many other PFAS that are less well known have also been widely used and released and may be present in the environment at levels of potential concern. This is one reason to expand the number of analytes in the analytical methods used and learning that certain analytes are not detected in multiple samples can provide limited assurance that this analyte may not be of concern.

There were no detections of nine analytes in the three media that were evaluated at most sites included in this study. These analytes can be seen in Table 30, below. It is likely that environmental contamination from these nine PFAS is not a general concern for the state.

Table 30: Analytes with no detectable levels reported

No detectable levels found in any of the three media evaluated								
11Cl-PF3OUdS	4:2 FTS	9Cl-PF3ONS	ADONA	N-EtFOSA	N-MeFOSA	N-MeFOSE	PFEESA	PFMBA

Note: Detection levels found in Appendix 4.

In other cases, an analyte might not have been detected in one or two media. In these cases, it could mean that the analyte is preferentially partitioning to another media, such as with PFBA only being found in surface water. PFBA is quite water soluble and would not readily partition to sediment or fish tissue. Having a detection in only one or two media may indicate that the PFAS is present in the system and is preferentially partitioning to that specific media. This unique fingerprint of the analytes and media could also provide details on potential sources of the PFAS. The PFAS that were detected in only one or two media can be seen in Table 31.

Table 31: PFAS Only detected in one or two media

PFAS Only Detected in one or two media							
Two media	3:3 FTCA	8:2 FTS	N-EtFOSE	NFDHA	PFMPA		
One media	5:3 FTCA	7:3 FTCA	HFPO-DA	PFBA	PFDoS	PFNS	PFPeA

Note: Detection levels found in Appendix 4.

Fish Tissue

The longer chain carboxylates, from 10 to 14 carbons, were detected between 97 (PFUnA) and 100% (PFDA) of fish tissue samples. There is limited or no toxicology information for some of these longer chain carboxylates that are frequently detected in fish tissue, and obtaining information on health effects of these compounds should be a consideration for further research.

It should also be mentioned that PFNS, not detected at all in surface water, and only twice in sediments, was detected in 18 individual fish tissue samples, or 15% of all individual samples collected in this study.

Other Emerging Contaminants

This study included a preliminary evaluation of certain emerging contaminants in several samples of fish tissue, surface water, and sediments, collected for this PFAS study or for a closely related study investigating the impacts of CECs on levels of intersex in fish. Four types of CECs were evaluated- polybrominated diphenyl ethers (PBDEs), chlorinated paraffins (CPs), pharmaceuticals and personal care products (PPCPs), and pesticides.

A list of all emerging contaminants quantified for this study, along with their related detection limits, can be seen in Appendix 8.

Polybrominated diphenyl ethers (PBDEs)

Background

Polybrominated diphenyl ethers (PBDEs), a class of organobromine chemicals, have been used in many products to impart flame retardant capabilities. PBDEs were marketed in the U.S. under several trade names as products that were formulated as mixtures of PBDE congeners. Therefore, when released to the environment, they present with varying levels of multiple PBDE congeners.

Sampling and Analysis

The design of sample collection and preparation was intended to allow for leveraging analytical results on the impacts of potential endocrine disrupting compounds, for which data are not included in this report, on the occurrence of intersex in fish.

Fish were collected from six sites, shown in Figure 54 below, in areas with angling pressures and varying levels of urbanization, with those along the Passaic and Raritan Rivers, and the most downstream site located on the Delaware River, expected to exhibit the most intense pressures from urbanization. Sites characteristics are provided in Table 32, below.

Six composite samples were composed of tissue taken from the fillets of three Largemouth bass collected at each site. Six composite fish tissue samples were analyzed for PBDEs using U.S. EPA Method 1614/1614A. Forty-six PBDE congeners were quantified for each composite sample. The analytical method produced a range of detection limits, noted here and in Appendix 8 as the "minimum detection level" and the "maximum detection level". The maximum detection level was the high end of the range of detection levels determined during analysis.

Results were reported in units of pg/g (ppt). The majority of congeners had maximum detection levels that were less than 1 pg/g (ppt), but others were less than 2 pg/g (ppt). There was only one congener that had a higher maximum detection level of 8.36 ppt. Detection levels for all congeners can be seen in Appendix 8.

Table 32: Six Fish Tissue Collection Sites for CECs

Site Name	Site Location	Easting	Northing
DR Phillipsburg	Delaware River at Phillipsburg above Lehigh River	297120.73	678115.51
DR Montague	Delaware River at Montague	410879.03	901811.08
DR Lambertville	Delaware River at STP at Lambertville NJ	367988.55	556603.54
PR Elmwood	Passaic River at Elmwood Park	593926.92	755969.81
PR Two Bridges	Passaic River at Two Bridges	553761.58	752014.34
RR Millstone	Raritan River at Millstone: Launch into Millstone	467975.79	618441.34

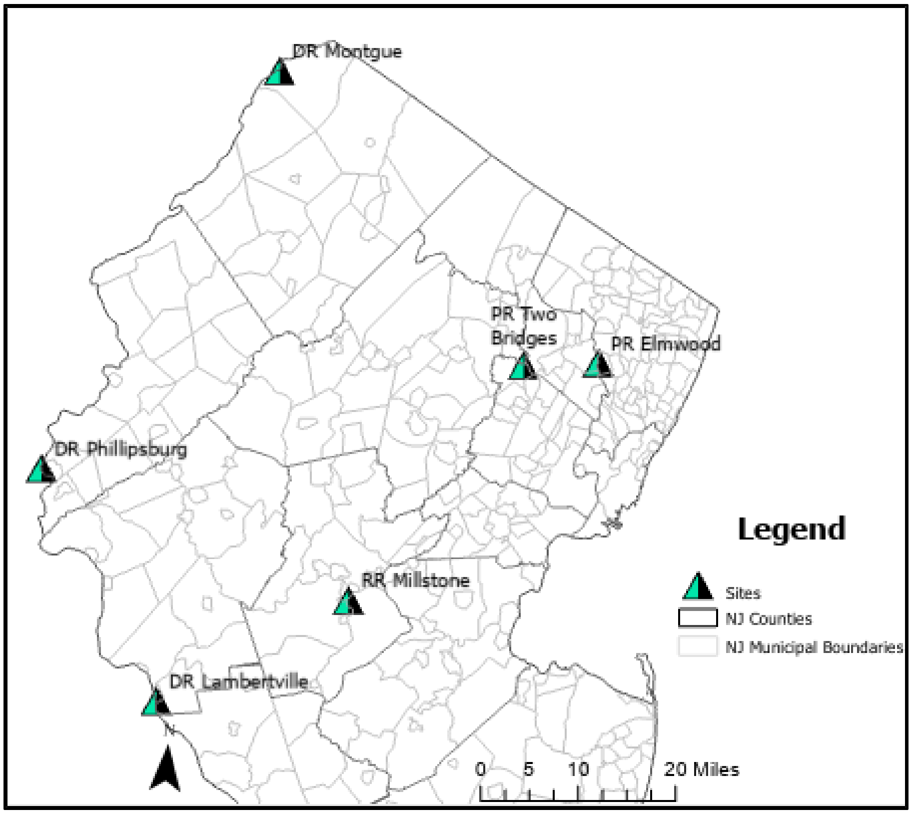

Figure 54: Six Fish Tissue Collection Sites for CECs

Results

All composite fish tissues samples contained multiple PBDE congeners. Out of the 46 congeners quantified in the analysis, 30 congeners were detected in one or more of the composite samples. Total PBDEs ranged from 2,702 ppt (pg/g) at the Delaware River at Montague site which was the most upstream and remote site, to 10,029 ppt at the site on the Passaic River at Elmwood Park.

The number of congeners in each of the composite samples ranged from 24 in the Delaware at Montague sample to 28 in the Passaic River at Elmwood Park sample.

PBDE congener concentrations in the composite samples of fish collected at the six sites can be seen in Figure 55, below. PBDE congeners are denoted by their IUPAC numbers, which are shown in Appendix 8.

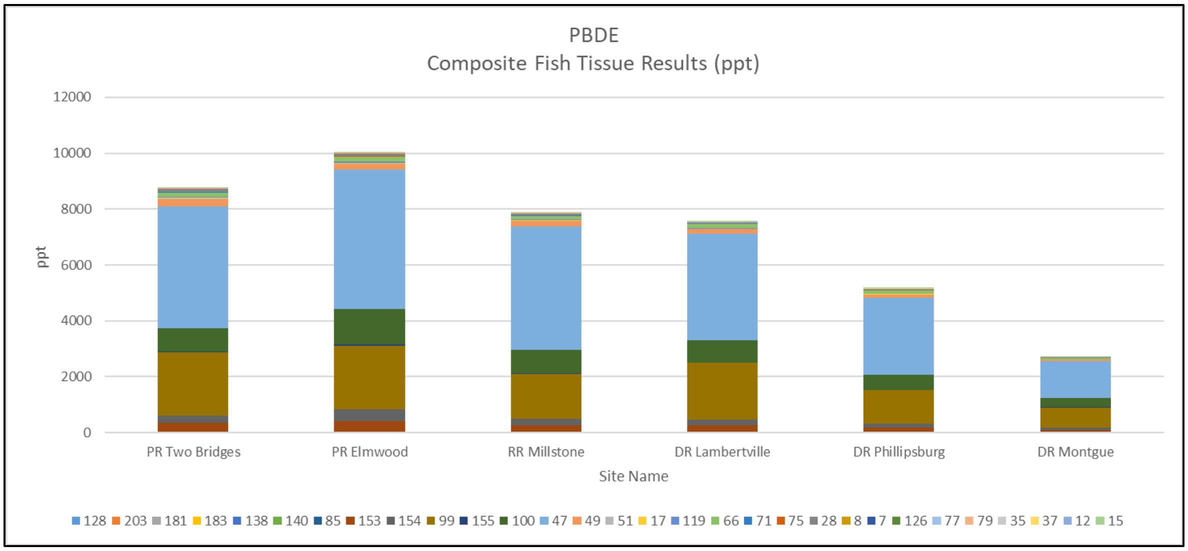

Figure 55: PBDE concentrations in six composite fish samples (IUPAC numbers defined in EPA Method 1614A, 2010)

As can be seen in Figure 55, three congeners dominate the makeup of the total PBDE concentration. These three congeners include IUPAC/Congener Number BDE-99 (brown block), IUPAC/Congener Number BDE-100 (green block), and IUPAC/Congener Number BDE-47 (blue block). Congeners 99 and 100 are penta-brominated diphenyl compounds, whereas number 47 is a tetra-bromo diphenyl ether.

Discussion

Congeners BDE- 99 and 47 are among the four most commonly detected PBDEs and U.S. EPA has developed have Reference Doses, exposure levels at which health effects are not expected, of 0.1 µg/kg body weight/day (equivalent to 100,000 pg/kg body weight/day) for them. The states of Maine and Washington have set restrictions on the use and release of PBDEs. Canada has added PBDEs to the list of substances for which the manufacture, use, or sale is prohibited (ECCC, 2022). Various manufacturers in the United States have voluntarily stopped production of certain types of PBDEs and other indicate that they expect to phase out some forms of the products.

The state of California set fish contaminant goals and advisory levels for PBDEs in 2011 (OEHHA, 2011). Advisory levels ranged from </= 100 ng/g for three 8-ounce servings per week, to >630 ng/g for a do not consume advisory. The total PBDEs found in the NJ fish ranged between 2.7 ng/g (2702.2 pg/g) and 10.02 ng/g (10,029.9 pg/g).

Although the levels of PBDEs in fish tissue determined in this study appear to fall below the levels of concern set in other states, PBDEs appear to be widely present in environmental matrices, and given their persistence in the environment, management of this contamination would benefit by up to date human and ecological health evaluations and frequent occurrence levels studies.

Chlorinated Paraffins (CPs)

Background

Chlorinated paraffins (CPs) are a series of *n*-alkanes that appear in products as complex mixtures. They have been widely used in various industrial activities since the 1930s as flame retardants in plastics and sealants and as additives in lubricants. An estimated 33 million metric tons of CP have been produced and used globally, according to recent flow analysis models (Chen, 2022). CPs are often divided into three groups: short-chain CPs (SCCPs) having 10-13 carbon atoms, medium-chain CPs (MCCPs) having 14 to 17 carbon chain atoms, and long-chain CPs (LCCPs) with 18 or more carbon chain atoms. They are considered persistent organic pollutants (POPs) able to undergo long-range transport.

Sampling and Analysis

Fish tissue, sediment, and surface water samples were collected at several sites around New Jersey to be analyzed for CPs, PPCPs, or both (see Table 33). Samples that were analyzed for chlorinated paraffins were collected at locations designated as “CP” only or “PPCP and CP” in Figure 56, below. Twelve samples of fish tissue (12 samples of individual fish- nine Largemouth bass and three Common carp), three each from four sites, and one sample run as a duplicate), seven sediment samples, and eight surface water samples were collected and analyzed.

All samples were analyzed using a proprietary analytical procedure, SGS Axys Method MLA-117 Version 03 which uses UPLC (Ultra-performance liquid chromatography) LC-MS/MS. Use of this type of method using UPLC is generally reserved for highly sensitive and specific analysis of complex mixtures.

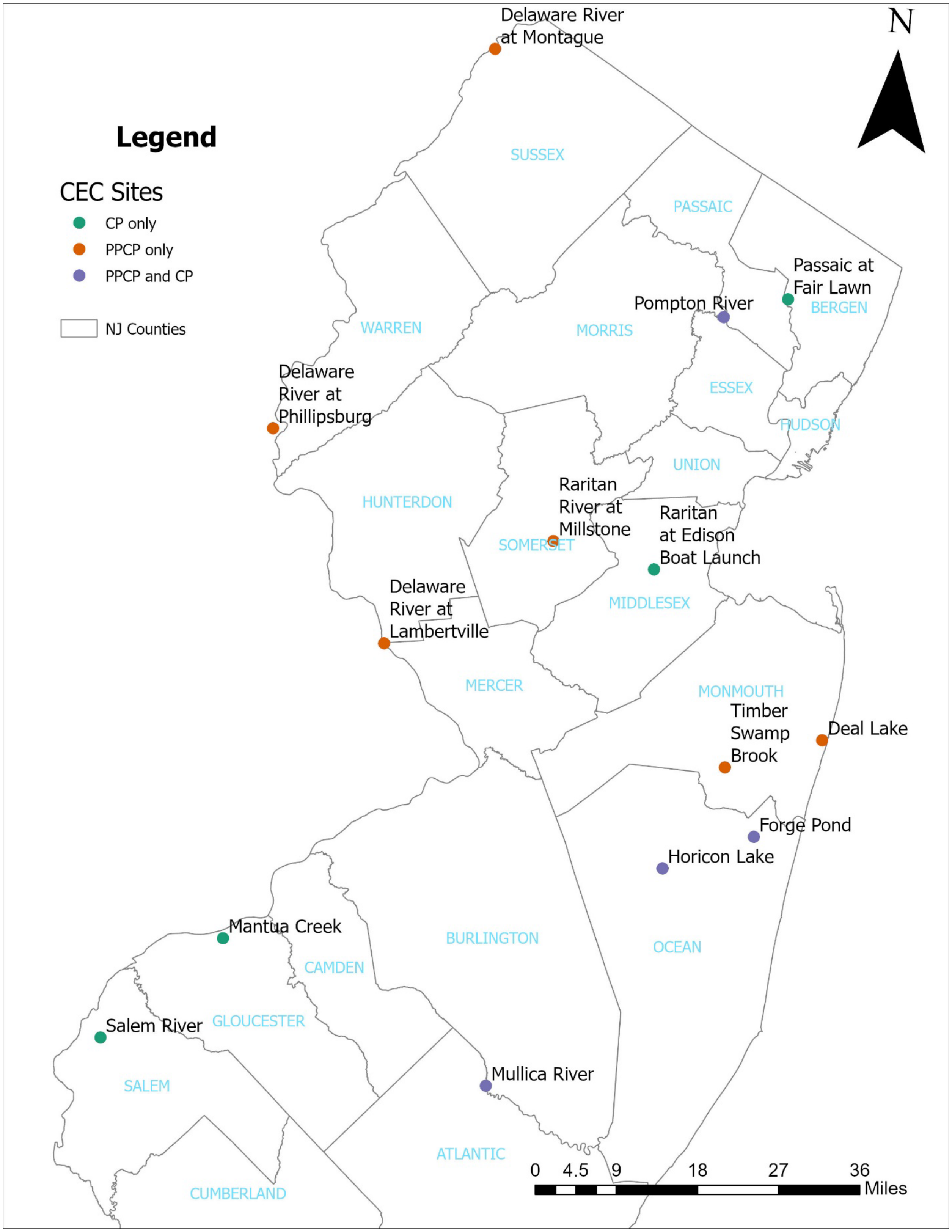

Figure 56: Locations where samples were analyzed for CPs, PPCPs, or both

Table 33: CP SW and Sed Sample Types and Locations

Waterbody	Surface Water	Sediment	Easting	Northing
Mantua Creek	X	X	294547	357324.4
Mullica River	X	X	447225.8	293954.2
Raritan River	X	X	524399	602625.4
Forge Pond	X	X	593386.4	450615.1
Horicon Lake	X	X	542444.6	428179.1
Passaic River	X	X	591811.5	765367.8
Pompton River	X	X	554995	752269.6
Salem River	X	X	220177.6	306093.9
Timber Swamp Brook		X	577495	490301

Results

Fish Tissue

There were no quantified, reportable results in any fish tissue sample. Detectable levels of any CP in fish tissue were only reported in a single duplicate sample. Since the reported levels in the duplicate were not replicative of the original sample (all reported as non-detect), the reported values for the replicate were considered unreliable. Detection limits were not different between the original sample and the duplicate. Detection limits varied widely across congeners, from a low of 0.491ng/g for C17Cl11 up to a maximum detection limit of 65.1 ng/g for C18Cl16. The analytical method produced a range of detection limits, noted in Appendix 8 as the minimum detection level and the maximum detection level.

Surface Water

There were no quantified, reportable results in any surface water sample. All results reported levels below the detection limit that was provided for that analysis. Some congeners had a respective detection limit as low as 5.85 ng/L, while other detection limits were reported in the hundreds of ng/L, up to 919 ng/L for C18Cl16.

Sediment

Three sediment samples contained notable levels of chlorinated paraffins. The samples collected from the Pompton, Passaic, and Timber Swamp Brook reported detectable levels of medium and long chain CPs, but no detections of short chain CPs. The sample collected from the Passaic River had the highest levels reported, with levels of the medium chain CPs reported to be 358 ng/g. See Figure 57 for results.

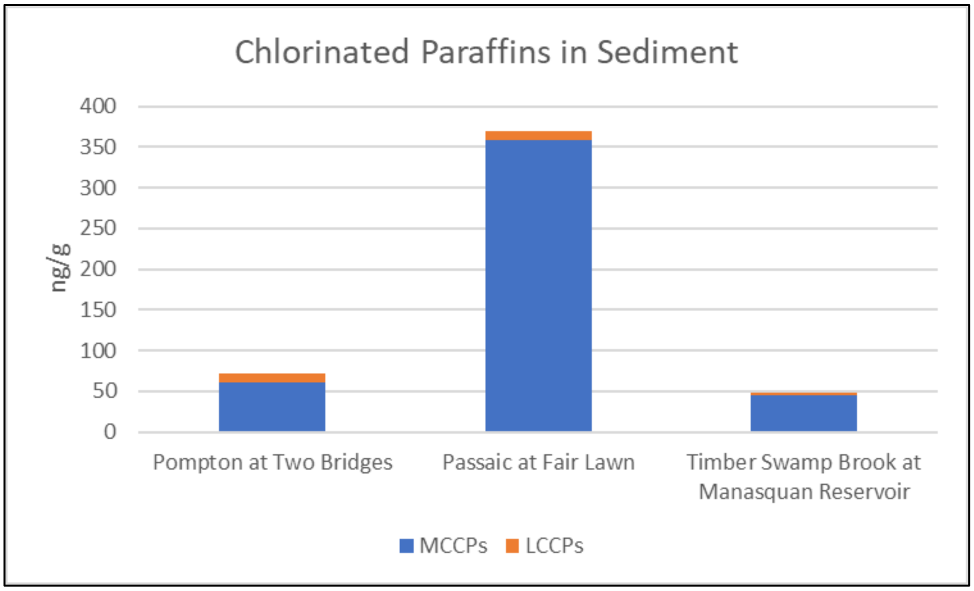

Figure 57: Sediment Concentrations of CPs at three sites

Discussion

Given the limited number of samples and potentially high detection limits, any conclusion would be suggestive. For instance, it could be surmised that CPs do not readily bioaccumulate in fish tissue, or it could be that the levels in the fish tissue are below detection. Chlorinated paraffins are generally insoluble in water and this may be why no CPs were detected in the surface water samples. However, detection limits were often relatively high and may impact results. Medium and long-chain CPs were detected in three sediment samples, suggesting that this might be a preferential pattern for partitioning.

The data were limited, and the current analysis may not capture environmentally relevant concentrations of CPs. Given the use and past industrial releases from facilities similar to those located near the sites that were sampled, it seems likely that the environmental occurrence of CPs may be underrepresented in our assessment. Additional occurrence data should be collected, and the analytical method should be further evaluated and refined to lower detection limits and assure precision and accuracy.

Pharmaceuticals and Personal Care Products (PPCPs)

Background

Pharmaceuticals and Personal Care Products (PPCPs) are a large and diverse group of chemicals that include those used in human and veterinary medicine, and in cosmetic care. Typical treatment for wastewater often does not fully remove PPCPs, and PPCPs can be released to the environment through discharges of wastewater. Many PPCPs are recalcitrant to degradation and could carry some level of impact on biota.

Sampling and Analysis

Fish tissue (22 samples of various species), sediment (7 samples), and/or surface water (6 samples) were collected at the 14 waterbodies as listed in Table 36. The locations for these sites can be seen in Figure 56. The samples of fish tissue (composite or individual), sediment, and surface water were analyzed using SGS Axys Method MLA-075, "*Analysis of Pharmaceutical and Personal Care Products and Hormones in Solid, Aqueous, Tissue and POCIS Samples*." SGS Axys developed and validated EPA Method 1694 for PPCPs in 2007 and has included several updates used in this method, including the addition of several target analytes. The method has also evolved to now use the latest Ultra Performance Liquid Chromatography (UPLC) MS/MS. The target analyte list is dependent on the pH of the extraction techniques and the subsequent analysis. For this project, samples were acid extracted and run in positive electrospray ionization (ESI), referred to here as "APOS," and negative ESI, referred to here as "ANEG." The full analyte list for APOS and ANEG can be seen in Table 34 and Table 35, below. Detection limits can be found in Appendix 8.

Table 34: List of 32 Analytes Quantified by APOS

10-hydroxy-amitriptyline	Fluocinonide	Promethazine
Alprazolam	Fluticasone propionate	Propoxyphene
Amitriptyline	Hydrocortisone	Propranolol
Amlodipine	Meprobamate	Sertraline
Benzoylecgonine	Methylprednisolone	Simvastatin
Benztropine	Metoprolol	Theophylline
Betamethasone	Norfluoxetine	Trenbolone
Cocaine	Norverapamil	Trenbolone acetate
DEET	Paroxetine	Valsartan
Desmethyldiltiazem	Prednisolone	Verapamil
Diazepam	Prednisone	

Table 35: List of 12 Analytes Quantified by ANEG

2-Hydroxy-ibuprofen	Hydrochlorothiazide
Bisphenol A	Ibuprofen
Furosemide	Naproxen
Gemfibrozil	Triclocarban
Glipizide	Triclosan
Glyburide	Warfarin

Table 36: PPCP Sample Types and Locations

Waterbody	Site ID	Surface Water	Sediment	Fish Tissue	Easting	Northing
Mullica River	MULL	X	X	X	447225.8	293954.2
Raritan River @ Millstone: Launch into Millstone	RR ML			X	524399	602625.4
Forge Pond	FORGE	X	X	X	593386.4	450615.1
Horicon Lake	HORICON	X	X	X	542444.6	428179.1
Pompton River (at Two Bridges)	POMP	X	X	X	554995	752269.6
Timber Swamp Brook/Manasquan Reservoir	MANA	X	X	X	577495	490301
Deal Lake	DEAL	X	X	X	632104.8	509425.9
Mantua Creek	MANT		X		294547	357324.4
Union Lake	UNI			X	330433.47	224435.25
Passaic River @ Elmwood Park	PR-EP			X	593926.92	755969.81
Delaware River at STP at Lambertville NJ	DR-LM			X	367988.55	556603.54
Passaic River @ Two Bridges	PR-TB			X	553761.58	752014.34
Delaware @ Phillipsburg above Lehigh River	DR-PL			X	297120.73	678115.51
Delaware River at Montague	DR-MT			X	410879.03	901811.08

Results

Fish Tissue

There were no detectable levels of any analytes evaluated in the ANEG method. Out of the 32 analytes quantified by the APOS method, nine analytes were detectable in fish tissue. The nine PPCPs that were detected in fish tissue can be seen in Table 37.

Table 37: Analytes detected in fish tissue through use of APOS analytical method

PPCP	Description
Cocaine	Highly addictive stimulant
DEET (N,N-diethyl-meta-toluamide)	Commercial insect repellent
Hydrocortisone	Steroid that provides anti-inflammatory and immunosuppressant properties
Theophylline	Medication used to treat respiratory conditions, such as asthma
Desmethylditiazem	Benzodiazepine derivative that provides amnesic, anticonvulsant, muscle relaxant, and sedative properties
Norfluoxetine	Selective serotonin reuptake inhibitor (SSRI) used to treat a variety of mental health conditions
Sertraline	Selective serotonin reuptake inhibitor (SSRI) used to treat a variety of mental health conditions
10-hydroxy-amitriptyline	Metabolite of the tricyclic antidepressant amitriptyline
Benzoylecgonine	Metabolite of cocaine

The levels of these nine PPCPs varied across sites and species. Levels of the chemical compounds detected in fish tissue can be seen in Figure 58, below.

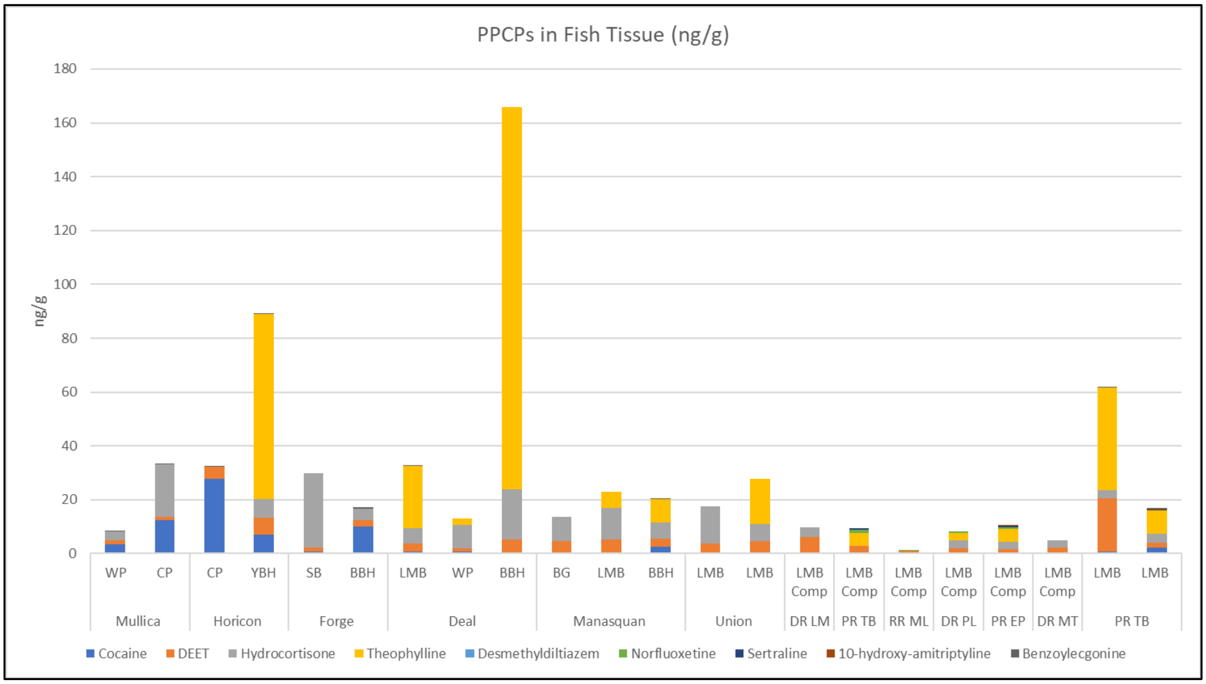

Figure 58: PPCP Concentrations in Fish Tissue.

[Note: Figure shows detections for single fish unless denoted by "Comp" meaning it is a composite sample of three fish. Fish abbreviations: LMB= Largemouth bass; BBH=Brown bullhead; BC=Black crappie; PS=Pumpkinseed; YP=Yellow perch; CC= Common carp; BG=Bluegill; YBH=Yellow bullhead; CP=Chain pickerel; SB=Striped bass; WCF=White catfish; and WP=White perch. Site abbreviations: DR= Delaware River, LM= Lambertville, P= Phillipsburg, MT=Montague, PR=Passaic River, TB= Two Bridges, EP= Elmwood Park, RR= Raritan River, ML= Millstone.]

Surface Water and Sediment

Twenty different PPCPs were detected in surface water, sediment, or both. The seven sediment samples and six surface water samples all contained varying levels of DEET. The number of samples with detectable levels of each PPCP can be seen in Table 38, below.

Table 38: Number of Detection of PPCP in Surface Water and Sediment Samples and Analyte Description

Method	Compound	Description	# SW detects (out of 6)	# SED detects (out of 7)
APOS	DEET	Commercial insect repellent	6	7
APOS	Theophylline	Medication used to treat respiratory conditions, such as asthma	6	0
ANEG	Bisphenol A	Chemical used in the production of plastics	3	3
ANEG	2-Hydroxy-ibuprofen	Metabolite of the non-steroidal anti-inflammatory pain reliever, ibuprofen	3	0
APOS	Benzoylecgonine	Metabolite of cocaine	3	0
APOS	Cocaine	Highly addictive stimulant	2	2
APOS	Meprobamate	Medication used to treat anxiety disorders	2	0
ANEG	Naproxen	Pain reliever	2	0
APOS	Metoprolol	Beta blocker mediation used to treat various cardiovascular conditions	1	1
APOS	Propranolol	Medication used for various cardiovascular conditions	1	1
ANEG	Gemfibrozil	Medication used to treat high triglycerides	1	0
ANEG	Hydrochlorothiazide	Medication used to treat high blood pressure	1	0
ANEG	Ibuprofen	Non-steroidal anti-inflammatory pain reliever	1	0
APOS	Valsartan	Medication to treat high blood pressure	1	0
ANEG	Triclocarban	Antibacterial and antifungal drug	0	3
APOS	Amitriptyline	Medication used to treat depression	0	1
APOS	Benztropine	Medication used to treat movement disorders	0	1
APOS	Norverapamil	Calcium channel blocker drug used to treat certain cardiac conditions and high blood pressure	0	1
APOS	Sertraline	Selective serotonin reuptake inhibitor (SSRI) used to treat a variety of mental health conditions	0	1
APOS	Verapamil	Calcium channel blocker drug used to treat certain cardiac conditions, high blood pressure, and migraines	0	1

Concentrations of detectable PPCPs in surface water can be seen in Figure 59, below.

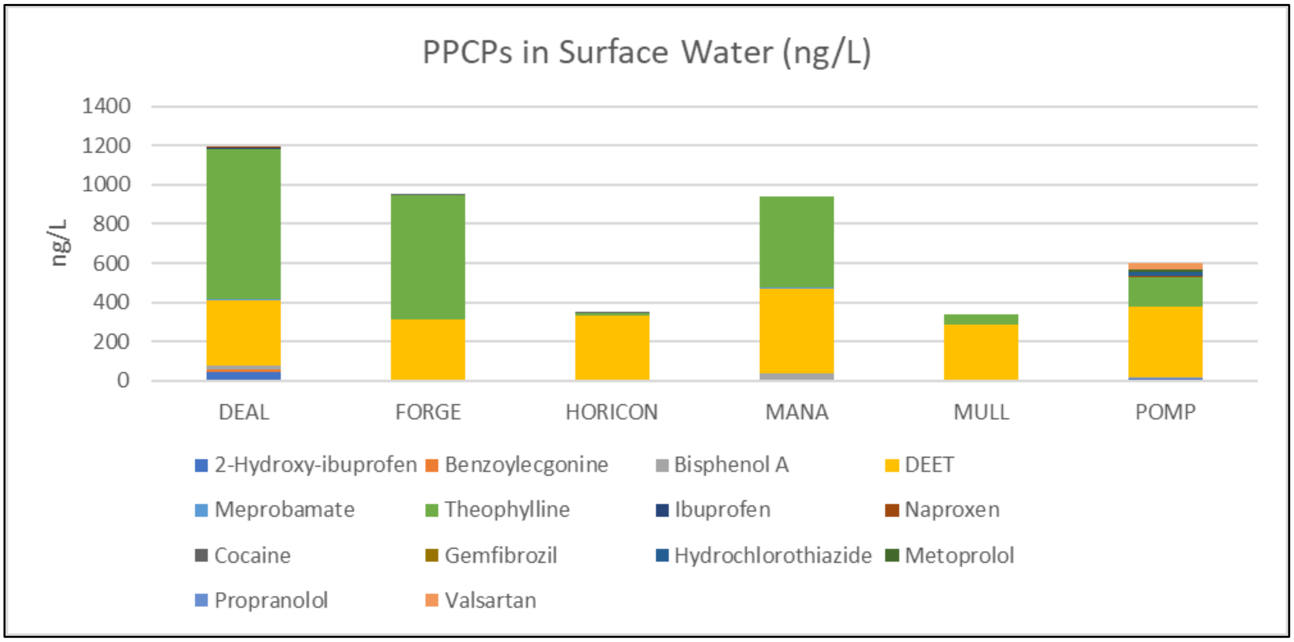

Figure 59: PPCP concentrations detected in surface water

Concentrations of detectable PPCPs in sediments can be seen in Table 39, below.

Table 39: PPCP concentrations detected in sediment

PPCP in Sediment (ng/g)	DEAL	FORGE	HORICON	MANA	MANT	MULL	POMP
Amitriptyline	<	<	<	<	<	<	1.92
Benztropine	<	<	<	<	<	<	0.74
Bisphenol A*	<	<	1000	7.85	<	<	144
Cocaine	<	<	0.158	<	<	<	0.17
DEET	1.44	1.81	1.27	1.68	2.03	1.86	2.43
Metoprolol	<	<	<	<	<	<	5.40
Norverapamil	<	<	<	<	<	<	0.31
Propranolol	<	<	<	<	<	<	1.86
Sertraline	<	<	<	<	<	<	10
Triclocarban	<	2.3	<	<	<	2.01	37
Verapamil	<	<	<	<	<	<	0.62

**Note: The result for Bisphenol A in the Horicon Lake sample is exceedingly high and should be retested to confirm that the result is representative of concentrations found at this location, as it is possible that this sample could have been influenced by cross contamination during sampling or analysis. "<" in table are non-detects. Detection limits can be seen in Appendix 8.*

Discussion

The distribution of the seven PPCPs detected in the fish tissue would be representative of PPCPs that were present in the system and that would have the capacity to accumulate in the tissues. The high number of detections of DEET and cocaine indicate a notable presence of these compounds, possibly due to use, discharge, and/or persistence. These levels suggest that a future investigation into potential impacts to human health or ecosystems may be warranted.

DEET was not only detected in 100% of all fish tissue samples, but it was also detected in 100% of all sediment and surface water samples, with some of these levels being notably above the detection limit, and some other levels closer to the detection limit.

Theophylline was found in notable concentrations above the detection limit in both fish tissue and surface water but was not detected in sediments. The physical-chemical characteristics may play a role in this distribution, but the higher detection limits from the sediment analysis may be a contributing factor.

Pesticides

Background

Sampling and Analysis

As discussed for PBDEs above, the fish were collected from six sites in areas with angling pressures and varying levels of urbanization, with those along the Passaic and Raritan Rivers, and the most downstream site located on the Delaware River, expected to exhibit the most intense pressures from urbanization. Sites characteristics are provided in Table 32, and site locations can be seen in Figure 54.

Six composite samples were composed of tissue taken from the fillets of three Largemouth bass collected at each site listed in Table 32. As with the results from the PBDE analysis, the design of sample collection and preparation was intended to allow for leveraging analytical results on the impacts of potential endocrine disrupting compounds in level of intersex in fish. This report will only address the occurrence levels and will leave full evaluation of impacts of endocrine disruption to future reports.

The six composite fish tissue samples were analyzed for pesticides using SGS AXYS Method MLA-028, “Analytical Procedure for Organochlorine Pesticides by Isotope Dilution HRMS by EPA Method 1699”. This targeted method quantified 28 different pesticides or degradation products of pesticides (see Table 40). Results were reported in ng/g (ppb) for the fish tissue and sediment samples, and ng/L (ppt) for the surface water samples. Detection limits were generally near 0.001 ng/g for the sediment and fish tissue samples, and 0.011 ng/L for the surface water samples.

Table 40:The twenty-eight pesticides evaluated

2,4'-DDD	2,4'-DDE	2,4'-DDT	4,4'-DDD	4,4'-DDE	4,4'-DDT	Aldrin	alpha-Endosulphan	beta-Endosulphan	Chlordane, alpha (cis)
Chlordane, gamma (trans)	Chlordane, oxy-	Dieldrin	Endosulphan Sulphate	Endrin	Endrin Aldehyde	Endrin Ketone	HCH, alpha	HCH, beta	HCH, delta
HCH, gamma	Heptachlor	Heptachlor Epoxide	Hexachlorobenzene	Methoxychlor	Mirex	Nonachlor, cis-	Nonachlor, trans-		

Results

Fish Tissue

The results for the pesticides detected in fish tissue can be seen in Table 41. 4,4-DDE, a break-down product of the pesticide DDT, was notably present in all composite samples tested. Nonachlor, both cis- and trans- isomers, was present in notable concentrations in all samples. Nonachlor is a component of the pesticide chlordane. Although all samples reported detections of all pesticides, many detections were very near the detection limit. The pesticides with such low-level detections include aldrin, endrin, and methoxychlor.

Table 41:Pesticides detected in fish tissue (ng/g)

Pesticide in Fish Tissue (ng/g)

Site Name	2,4'-DDD	2,4'-DDE	2,4'-DDT	4,4'-DDD	4,4'-DDE	4,4'-DDT	Aldrin	alpha-Endosulphan	beta-Endosulphan	Chlordane, alpha (cis)
PR Two Bridges	0.371	0.092	0.092	2.48	10.9	0.554	0.006	0.017	0.006	2.7
PR Elmwood	0.289	0.075	0.072	1.97	7.42	0.245	0.007	0.012	0.005	2.72
RR Millstone	0.31	0.085	0.097	2.64	14	0.377	0.005	0.007	0.0005	1.39
DR Lambertville	0.196	0.034	0.09	1.44	5.46	0.475	0.005	0.003	0.0005	0.177
DR Phillipsburg	2.23	0.126	0.428	4.7	5.86	0.833	0.005	0.002	0.0005	0.205
DR Montague	0.019	0.014	0.024	0.235	4	0.068	0.004	0.002	0.0005	0.047

Pesticide in Fish Tissue (ng/g)

Site Name	Chlordane, gamma (trans)	Chlordane, oxy-	Dieldrin	Endosulphan Sulphate	Endrin	Endrin Aldehyde	Endrin Ketone	HCH, alpha	HCH, beta	HCH, delta
PR Two Bridges	0.836	2.55	2.62	0.034	0.017	0.012	0.011	0.002	0.004	0.002
PR Elmwood	0.987	2.37	3.03	0.033	0.019	0.015	0.014	0.001	0.004	0.005
RR Millstone	0.455	1.27	2.33	0.006	0.019	0.013	0.011	0.002	0.005	0.005
DR Lambertville	0.064	0.281	0.528	0.004	0.013	0.011	0.002	0.002	0.004	0.001
DR Phillipsburg	0.064	0.247	0.576	0.002	0.012	0.028	0.003	0.001	0.003	0.003
DR Montague	0.016	0.098	0.048	0.001	0.001	0.033	0.0005	0.0005	0.004	0.006

Pesticide in Fish Tissue (ng/g)

Site Name	HCH, gamma	Heptachlor	Heptachlor Epoxide	Hexachlorobenzene	Methoxychlor	Mirex	Nonachlor, cis-	Nonachlor, trans-
PR Two Bridges	0.008	0.03	1.33	0.13	0.0005	0.456	3.12	10
PR Elmwood	0.007	0.013	1.04	0.135	0.0005	0.238	2.55	8.42
RR Millstone	0.008	0.011	0.487	0.122	0.0005	0.534	1.37	4.43
DR Lambertville	0.005	0.002	0.084	0.073	0.0005	0.023	0.306	1.05
DR Phillipsburg	0.005	0.001	0.072	0.09	0.0005	0.019	0.224	0.795
DR Montague	0.005	0.0005	0.012	0.097	0.0005	0.021	0.141	0.438

Sediment

The results for pesticides detected in the sediment can be seen in Table 42. 4,4-DDT was present in all sediment samples, with higher levels found in PR Elmwood sample (85.7 ng/g). Given that this level is substantially higher than in other samples, a confirmatory sample would be recommended. Chlordane (alpha, gamma, and oxy- isomers) was also notably high at this site. Levels of all pesticides were comparatively low at the RR Millstone and DR Montague sites.

Table 42: Pesticides detected in sediments (ng/g)

Pesticide in Sediments (ng/g)

	2,4'-DDD	**2,4'-DDE**	**2,4'-DDT**	**4,4'-DDD**	**4,4'-DDE**	**4,4'-DDT**	**Aldrin**	**alpha-Endosulphan**	**beta-Endosulphan**	**Chlordane, alpha (cis)**
DR Lambertville	0.601	0.043	0.569	1.43	1.92	2.21	0.021	0.005	<	0.382
DR Phillipsburg	0.598	0.067	0.572	2.41	4.04	12.1	0.056	0.041	0.033	1.3
DR Montague	0.02	0.003	0.023	0.078	0.192	0.097	0.008	<	<	0.02
PR Elmwood	3.19	0.41	2.09	17.8	11.4	85.7	0.85	0.093	0.047	22.5
PR Two Bridges	0.733	0.117	0.26	2.59	3.7	1.21	0.207	0.037	0.065	3.8
RR Millstone	0.031	0.004	0.026	0.072	0.121	0.082	0.008	<	<	0.04

Pesticide in Sediments (ng/g)

	Chlordane, gamma (trans)	**Chlordane, oxy-**	**Dieldrin**	**Endosulphan Sulphate**	**Endrin**	**Endrin Aldehyde**	**Endrin Ketone**	**HCH, alpha**	**HCH, beta**	**HCH, delta**
DR Lambertville	0.349	0.04	0.227	0.018	0.081	0.033	<	0.009	0.007	0.002
DR Phillipsburg	1.45	0.131	1.16	0.08	0.041	<	0.034	0.009	0.008	<
DR Montague	0.021	0.003	0.008	0.002		0.014	<	<	<	<
PR Elmwood	25.5	1.39	4.74	0.145	0.122	<	0.098	0.018	0.03	<
PR Two Bridges	3.34	0.196	0.703	0.115	0.031	<	<	0.004	0.006	<
RR Millstone	0.041	0.003	0.025	<	0.003	0.013	0.001	<	<	<

Pesticide in Sediments (ng/g)

	HCH, gamma	**Heptachlor**	**Heptachlor Epoxide**	**Hexachlorobenzene**	**Methoxychlor**	**Mirex**	**Nonachlor, cis-**	**Nonachlor, trans-**
DR Lambertville	0.01	0.009	0.043	0.135	0.167	0.011	0.122	0.4
DR Phillipsburg	0.01	0.028	0.194	0.319	0.554	0.038	0.396	1.27
DR Montague	<	<	0.002	0.021	0.05	0.002	0.009	0.033
PR Elmwood	0.029	0.347	1.73	1	2.73	0.415	7.44	23.9
PR Two Bridges	0.012	0.033	0.204	0.241	0.358	0.052	1.35	3.56
RR Millstone	<	<	0.004	0.011	0.014	0.001	0.012	0.039

"<" in table are non-detects. Detection limits can be seen in Appendix 8.

Surface Water

The results for the pesticides found in the surface water samples can be seen in Table 43. The sample collected from the PR Elmwood site has among the higher reported levels of many pesticides as compared to the other five sites. Chlordane, heptachlor, and dieldrin have levels notably higher than the other sites.

Three pesticides were not detected in any of the surface water samples: 2,4-DDE, HCH-delta, and Mirex. Others, such as 2,4-DDT and methoxychlor were found in a lower number of samples and at levels near the detection limit.

Table 43: Pesticides in Surface Water (ng/L)

Pesticides in Surface Water (ng/L)

	2,4'-DDD	2,4'-DDE	2,4'-DDT	4,4'-DDD	4,4'-DDE	4,4'-DDT	Aldrin	alpha-Endosulphan	beta-Endosulphan	Chlordane, alpha (cis)
DR Lambertville	0.015	<	0.012	0.025	0.033	0.028	0.119	<	<	0.069
DR Phillipsburg	<	<	<	0.011	0.024		0.119	<	<	0.02
DR Montague	<	<	<	<	0.015		0.124	<	<	
PR Elmwood	0.051	<	0.025	0.142	0.106	0.064	0.143	<	<	0.539
PR Two Bridges	0.016	<		0.039	0.044	0.018	0.116	0.025	0.028	0.128
RR Millstone	0.02	<	0.012	0.032	0.043	0.024	0.109	<	<	0.039

Pesticides in Surface Water (ng/L)

	Chlordane, gamma (trans)	Chlordane, oxy-	Dieldrin	Endosulphan Sulphate	Endrin	Endrin Aldehyde	Endrin Ketone	HCH, alpha	HCH, beta	HCH, delta
DR Lambertville	0.063	<	0.1	<	<	0.081	0.016	<	0.017	<
DR Phillipsburg	0.021	<	0.156	<	0.012	0.095	0.021	<	0.011	<
DR Montague	<	<	0.016	<	<	0.091	<	<	<	<
PR Elmwood	0.396	0.048	1.52	0.053	0.029	0.111	0.125	0.013	0.028	<
PR Two Bridges	0.084	0.02	0.426	0.111	0.014	0.091	0.046	<	0.011	<
RR Millstone	0.029	0.01	0.159	<	0.017	0.08	0.015	0.013	0.013	<

Pesticides in Surface Water (ng/L)

	HCH, gamma	Heptachlor	Heptachlor Epoxide	Hexachlorobenzene	Methoxychlor	Mirex	Nonachlor, cis-	Nonachlor, trans-
DR Lambertville	0.014	<	0.035	<	<	<	0.018	0.056
DR Phillipsburg	0.011	<	0.039	0.014	<	<	<	0.015
DR Montague	0.02	<	<	<	<	<	<	
PR Elmwood	0.053	0.013	0.802	0.025	0.014	<	0.105	0.241
PR Two Bridges	0.023	0.011	0.33	0.016	<	<	0.038	0.066
RR Millstone	0.02		0.062	0.016			0.013	0.028

"<" in table are non-detects. Detection limits can be seen in Appendix 8.

Discussion

The presence of various pesticides in surface water, sediments, and fish tissue has been shown at six sites around New Jersey. Levels of pesticides that have long been banned, such as DDT (banned for most uses since 1972) and chlordane (banned for most uses in 1983 and totally banned in 1988), continue to be detected along with transformation products DDE and DDD.   More heavily urbanized sites, such as the Passaic River at Elmwood Park, have generally higher levels of pesticides reported.

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Appendix 1: Site and Sample Details

Main Site Id	Name	Sample ID	Category	Sample Collection Date	Municipality	County	SPCS X-coordinate	SPCS Y-coordinate
1	Pemberton LLAMA 1	LL1A	Biosolid Application Site	9/24/2018	Pemberton Township	Burlington	439754.1	421202.2
1	Pemberton LLAMA 2	LL1B	Biosolid Application Site	9/24/2018	Pemberton Township	Burlington	443462.5	421944.9
1	Pemberton LLAMA 3	LL1C	Biosolid Application Site	9/24/2018	Pemberton Township	Burlington	447825.2	420700.6
1	Pemberton LLAMA 4	LL1D	Biosolid Application Site	9/24/2018	Pemberton Township	Burlington	445509.3	419058.4
2	Landis LLAMA 5	LL2A	Biosolid Application Site	7/21/2021	Pittsgrove Township	Salem	324803.7	232556.8
2	Landis LLAMA 6	LL2B	Biosolid Application Site	7/21/2021	Vineland City	Cumberland	330365.3	224367.1
2	Landis LLAMA 7	LL2C	Biosolid Application Site	7/21/2021	Vineland City	Cumberland	331543.4	226894.7
2	Landis LLAMA 8	LL2D	Biosolid Application Site	7/21/2021	Vineland City	Cumberland	335638.3	228847.3
2	Landis LLAMA 9	LL2E	Biosolid Application Site	7/21/2021	Vineland City	Cumberland	335697.4	231205.9
2	Landis LLAMA 10	LL2F	Biosolid Application Site	7/21/2021	Vineland City	Cumberland	330671.7	236724.9
2	Landis LLAMA 11	LL2G	Biosolid Application Site	7/21/2021	Vineland City	Cumberland	330170.7	230441.1
2	Union Lake 12	LL2H	Biosolid Application Site	7/21/2021	Millville	Cumberland	332814.9	213496.2
2	Landis open sapce NJDEP	LL4	Biosolid Application Site	7/21/2021	Millville	Cumberland	322148.0	219681.0
3	Ash Lane LLAMA 13	LL3A	Biosolid Application Site	7/21/2021	Alloway Township	Salem County	264620.8	251986.4
3	Ash Lane LLAMA 14	LL3B	Biosolid Application Site	7/21/2021	Alloway Township	Salem County	264239.5	256012.8
3	Ash Lane at Swing Road	LL3CSW	Biosolid Application Site	7/21/2021	Alloway Township	Salem County	269692.9	254022.6
4	Ocean Cty Fire Training	FF1A	AFFF Use	7/8/2021	Ocean Township	Ocean	577721.6	351864.5
4	Ocean Cty Fire Traiing	FF1B	AFFF Use	7/8/2021	Ocean Township	Ocean	577506.9	349657.6
5	Monmouth Cty FF dwnstrm	5FF2A	AFFF use	8/3/2021	Howell Township	Monmouth	575228.4	508512.5
5	Monmouth Cty FF dwstrm	5FF2B	AFFF use	8/3/2021	Howell Township	Monmouth	575819.3	508234.6
6	Car Wash Upstream	CW1	Car Wash or Suds	11/1/2018	Plumstead Township	Ocean	483384.7	449365.4
6	Car Wash Impoundment	CW2	Car Wash or Suds	11/1/2018	N.Hanover Township	Burlington	477567.5	445583.2
6	Car Wash Downstream	CW3	Car Wash or Suds	11/1/2018	N.Hanover Township	Burlington	474597.5	442992.8

Main Site Id	Name	Sample ID	Category	Sample Collection Date	Municipality	County	SPCS X-coordinate	SPCS Y-coordinate
6	Car Wash other trib	CW4	Car Wash or Suds	11/1/2018	Plumstead Township	Ocean	483607.1	442140.0
6	Car Wash trib from base	CW3.1	Car Wash Trib near base	11/1/2018	N.Hanover Township	Burlington	481009.0	441640.8
7	Somerset FF training &GAS	7SFF	AFFF use	10/7/2019	Hillsborough Township	Somerset	453220.4	622410.5
8	dstrm of trib with fire	FF5	AFFF use	10/7/2019	Waldwick Borough	Bergen	594932.6	792754.2
8	upstream of trib with fire	FF6	AFFF use	10/7/2019	Waldwick Borough	Bergen	594000.2	794967.1
9	Allgrind Plastics and Ico	AG_up	Commercial Facility	10/7/2019	Bethlehem Township	Hunterdon	341733.7	666782.3
9	Allgrind Plastics and Ico	AG_dwn	Commercial Facility	10/7/2019	Bethlehem Township	Hunterdon	340134.9	668669.6
10	Car Wash toms river	CW5	Car Wash or Suds	7/28/2021	Toms River Township	Ocean	588026.1	405657.9
10	Car Wash Toms River	CW7	Car Wash or Suds	7/28/2021	Toms River Township	Ocean	585834.8	408258.6
10	Car Wash Toms River	CW6	Car Wash or Suds	7/28/2021	Toms River Township	Ocean	585736.2	409088.2
11	Wayne Mountain View	POTW11 dwn	Wastewater Treatment Plant	10/15/2019	Wayne Township	Passaic	561251.6	753442.2
11	Upstream st gobain Wayne MW	POTW11 up	Wastewater Treatment Plant	10/15/2019	Wayne Township	Passaic	563335.4	758422.7
12	Two Bridges Sewage Auth	POTW8	Wastewater Treatment Plant	10/7/2019	Wayne Twp/ Lincoln Park Boro	Morris/ Passaic	555020.6	752123.8
13	ParsippanyTroyHills Maj Dis	13-WHIP	Wastewater Treatment Plant	8/2/2021	Parsippany Troy Hills	Morris	535244.9	733172.9
14	Caldwell Discharge Point	POTW14dwn	Wastewater Treatment Plant	10/15/2019	West Caldwell Township	Essex	543549.4	730776.7
15	Wayne Sheffield	POTW6	Wastewater Treatment Plant	10/7/2019	Wayne Township	Passaic	552575.2	777617.2
15	Wayne Sheffield	POTW6_15UP	Wastewater Treatment Plant	10/7/2019	Wayne Township	Passaic	552236.0	778258.7
15	Wayne Sheffield	POTW6_15DS	Wastewater Treatment Plant	10/7/2019	Wayne Township	Passaic	552582.4	777219.7
16	Former Cranberry Bog	FSW1	AFFF use	5/22/2019	Manchester Township	Ocean	534350.3	433402.0
16	Ridgeway Br at 547	FSW2	AFFF use	5/22/2019	Manchester Township	Ocean	546509.8	436921.0
16	Ridgeway Br at Highbridge Rd	FSW3	AFFF use	5/22/2019	Jackson Township	Ocean	532781.3	444926.6
16	Horicon Lake	16-X3	AFFF use	8/12/2021	Lakehurst Borough	Ocean	542506.4	428645.8
16	Pine Lake	16-X1	AFFF use	8/12/2021	Manchester Township	Ocean	561691.4	428272.4

Main Site Id	Name	Sample ID	Category	Sample Collection Date	Municipality	County	SPCS X-coordinate	SPCS Y-coordinate
17	Atlantic Plastic Container 2	ATLPC2	Commercial Facility	5/31/2023	Egg Harbor Township	Atlantic	461154.5	208444.9
17	Atlantic Plastic Container 1	ATLPC1	Commercial Facility	5/31/2023	Egg Harbor Township	Atlantic	460751.0	212991.1
17	outflow from reservoir	ATLMST	AFFF use	5/31/2023	Absecon	Atlantic	492469.9	214783.2
17	upstream at English Creek Road	ATLSU3	AFFF use	5/31/2023	Egg Harbor Township	Atlantic	464167.1	223555.7
17	Absecon Creek after discharge from reservoir	ATLSBA6	AFFF use	5/31/2023	Absecon	Atlantic	486346.8	217319.7
18	upper Ringwood Creek Tributary	RNG2	Commercial Facility	11/2/2022	Ringwood Borough	Passaic	556203.9	842532.0
18	lower Ringwood Creek Tributary	RNG4	Commercial Facility	11/2/2022	Ringwood Borough	Passaic	558584.7	839639.8
18	Ringwood Creek at Farm Road	RNG6FR	Commercial Facility	11/2/2022	Ringwood Borough	Passaic	558128.7	838276.3
18	outflow_reservoir_Chestnut	RNG9CNT	Commercial Facility	11/2/2022	Wanaque Borough	Passaic	552459.5	810212.8
19	Forge Pond	FTM022	Commercial Facility	8/12/2021	Brick Township	Ocean	593166.7	450567.4
20	Deal Lake	FTM028	Commercial Facility	8/10/2021	Howell Township	Monmouth	631583.6	509355.6
21	Timber Swamp Brook at Outfall to Manasquan Reservoir	TSB	Commercial Facility	8/3/2021	Howell Township	Monmouth	577947.5	490395.3
21	Manasquan Reservoir	FTM030	Commercial Facility	10/28/2021	Howell Township	Monmouth	574887.5	490240.2
Pond	Echo Spring North	Echo_1	Pond	4/24/2018	West Milford	Passaic	519540.0	804997.3
Pond	Echo Spring South	Echo_2	Pond	4/24/2018	West Milford	Passaic	519391.6	802556.5
Pond	Butterworth Pond	Butterworth	Pond	6/27/2018	Woodland	Burlington	486716.9	386333.9
Pond	Locustwood Pond	Locustwood	Pond	4/24/2018	Cherry Hill	Camden	346151.6	397498.6
Pond	Cedar Pond	Cedar	Pond	6/27/2018	Buena Vista	Atlantic	384622.1	256026.4

Appendix 2: QAPP

July 28, 2021

NEW JERSEY DEPARTMENT OF ENVIRONMENTAL PROTECTION
DIVISION OF SCIENCE and RESEARCH
428 EAST STATE STREET, TRENTON, NEW JERSEY 08625

QUALITY ASSURANCE PROJECT PLAN

Multimedia Investigation of PFAS and other Emerging Contaminants in New Jersey Fish Tissue, Sediment, and Surface Water

(Phase II +)

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Contents

Project Name:
Multimedia Investigation of PFAS and other Emerging Contaminants in New Jersey Fish Tissue, Sediment, and Surface Water (Phase II +)

1. **Project Request/ Lead Agency**:
   The Division of Science and Research (DSR)

2. **Date of Request**:
   May 2021

3. **Date of Project Initiation**:
   July 2021

4. **Project Fiscal Information**:
   Job Numbers: 7MULTIBX, 7W106CXX, 33030000, 7W106EXX. DSR will authorize payments to contract laboratory upon receipt and validation of data submitted.

5. **Field Coordinators**:
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   Lee Lippincott, Ph.D., Research Scientist 1
   NJDEP, Division of Science and Research

6. **Project Officers**:
   Dr. Sandra Goodrow, Research Scientist 1
   Bruce Ruppel, Environmental Specialist 4
   NJDEP, Division of Science and Research

7. **Quality Assurance Officers**:
   Ryan Compton, Research Scientist 2
   NJDEP, Office of Quality Assurance (OQA)

   Melissa Hornsby, Administrative Analyst 3
   NJDEP, Office of Quality Assurance (OQA)

8. **Special Training Needs/Certifications**

   Field sampling personnel for the project will be trained in the operation and use of all sampling equipment including the proper safety and handling procedures for electroshocking equipment.

   The sampling and processing of the samples will be carried out by trained personnel aware of the risk of sample contamination posed by incorrect handling. This will include but not limited to avoiding all contact of samples with Teflon, sticky labels, and adhesive tape (bottles should be written on with a permanent marker, sealed then stored in a Ziploc or whirlpak type bag for secondary containment). Sampling personnel should also avoid wearing jackets or other outer clothing that is new or that has not been washed few times (See Appendix E for Collection and Handling of Samples for Analysis of PFOA, PFOS and Other Fluorinated Compounds). Laboratory items and sampling materials used for the collection of samples intended to be analyzed for PFAS, will be free of fluorinated compounds, as demonstrated by lab validation, peer reviewed study or equipment blanks.

9. Project Description

Background

The current "PFAS Phase II+" project builds off a project completed in 2018 that collected surface water, sediment, and fish tissue at eleven (11) targeted sites across the state. Poly- and per-fluoroalkyl substances (PFAS, also known as poly- and per- fluorinated compounds (PFCs)) are synthetic compounds used in a large variety of industrial processes and have been detected in various environmental matrices including water, sediment, air, and biota. The most commonly detected PFAS, perfluorooctane sulfonate (PFOS) has been classified as a persistent and bioaccumulative substance and has been detected in environmental media including water, sediment, and fish. Both PFOS and perfluorooctanoic acid (PFOA) have been detected at elevated concentrations in groundwater near firefighting training areas because of the repeated application of aqueous film forming foam (AFFF) which is known to contain many classes of PFAS. AFFFs have been commercially available for fire-fighting applications since their development by the United States Navy and 3M Company in the mid-1960s. Elevated concentrations of multiple PFAS compounds have also been detected in surface and groundwater in areas near fluoropolymer manufacturing facilities, textile manufacturers and electroplating facilities, among others. Environmental and Human Health Assessments in other states have revealed that exposure to elevated levels of PFAS from AFFF and other products released to the environment are potentially hazardous to human health. Exposure of PFAS has been found to be largely through direct ingestion of drinking water and consumption of fish from contaminated surface water. Several studies conducted by the Department have identified elevated levels of perfluorinated substances in source water and drinking water throughout the state. In a study performed by DSR published in July of 2018, multiple fish species were collected in targeted areas (Goodrow et al., 2020). The results of this study, known within NJDEP as "Phase I," was the basis for this current project, as it identified areas in New Jersey that have been impacted by these emerging contaminants.

Objectives

This project will focus primarily on the issue of PFAS contamination that has become an overwhelming concern, being found in multiple environmental media at levels thought to impact human health and the environment. This project will continue NJDEP's efforts to identify potential sources of PFAS contamination through multimedia sampling, which will promote remediation of water and land. Multimedia sampling will include surface water, sediment, and fish tissue.

The scope of work presented here is a continuation of the first phase of the project "PFAS in Fish Tissue, Sediment, and Surface Water in New Jersey". The initial project, now called "Phase I" focused on PFAS contamination in targeted areas where known industrial facilities existed (Goodrow et al., 2018; Goodrow et al., 2020). The current project will evaluate additional classifications of potential PFAS sources and increase sample numbers that would contribute to more robust partitioning analysis. By adding the analysis of select samples for pesticides and pharmaceuticals and personal care products (PPCPs), other emerging compounds will be quantified for the evaluation of occurrence and potential impact.

The additional source classifications include waterways downstream of the discharge from wastewater treatment plants (WWTPs), waters near biosolid application sites, waters near areas of aqueous film forming foam (AFFF) use and industrial manufacturing, and waters near car washes that potentially discharged directly to the environment in the past. In addition, there are several sites of specific industrial activity or military use that requires follow up from Phase I. The outcomes for these objectives are 1) to detect previously unknown sources of PFAS in the environment of New Jersey, 2) to continue to collect data that contributes to the assessment of the extent of PFAS in NJ's environment, and 3) identify sites and media for remediation to protect human health and the environment. This is known as Phase II. Additional analytical procedures for quantifying pesticides and PPCPs will be performed on samples collected at select sites. This will be known as, "+".

In the portion of the project denoted as "+", fish tissue samples collected for two other projects will be leveraged for evaluation of PFAS and other identified emerging compounds of concern. The levels of PFAS, pesticides, and pharmaceuticals and personal care products (PPCPs) will be evaluated in select fish tissue collected during 2021 at sites in the New Jersey 2021 Routine Monitoring Network and fish tissue collected for the 2021 New Jersey Intersex Fish Investigation. Samples of sediment and surface water will be added to these sites to evaluate partitioning of these compounds.

The benefits of the Multimedia Investigation of PFAS and other Emerging Contaminants in New Jersey Fish Tissue, Sediment, and Surface Water (Phase II +) will provide valuable data on the levels of PFAS and other select emerging contaminants in fish tissue, water column and sediment concentrations. The determination of levels of PFAS and other select emerging contaminants in fish tissue will give the Department:

- Needed data to determine the potential health risk to the fish consuming public and whether consumption advisories are warranted.
- A clearer understanding of human exposure and associated ambient environmental levels in various media, and
- An understanding of whether further investigations into other water bodies of the state is warranted to inform fish consumption use assessments.

9.3 Study Design- Phase II + (Phase II, Toxics in Biota, and Intersex Sites)

The study design includes the collection of fish, water, and sediment samples from select sampling locations throughout New Jersey. These sample locations were chosen to reflect areas where potential sources may be emitting to the environment and includes areas where fish may be taken for consumption or where other emerging contaminants might be present potentially impact human health and/or the environment. These potential source locations include areas where aqueous film forming foam (AFFF) is thought to have been used in large amounts or where there are industrial areas where the manufacturing or use of products containing per- and polyfluoroalkyl substances have been identified. Fish collection at these locations will primarily be carried out with the use of electroshocking, although netting, trot line or other methods will also be employed when needed (e.g., when waterbody is inaccessible to boat access or in the case of equipment failure when staff is field deployed). When possible, water and sediment samples will be taken at the same time as the fish species are collected. However, sediment and water samples can be collected from that site at an alternate time if holding times for these samples do not match available analytical service capacity or fish collection strategy.

Additional sample results from two other projects will be leveraged for this project. The Bureau of Freshwater and Biological Monitoring are collecting fish tissue samples for the Routine Toxics in Biota program where fish consumption advisories are set according to contaminant concentrations. The Intersex Fish project is collecting smallmouth bass to evaluate the health of reproductive tissues in the fish. The ability to leverage these samples (to use the sample media and the analytical results to pair with and evaluate alongside) collected in other projects will allow for the expansion of evaluating potential impacts. Using fish tissue sample results from the Fish Tissue Monitoring Program will allow us to incorporate the levels of PCBs and total mercury (determined under a separate QAPP) alongside additional analytical parameters into our evaluation of total contaminants stress at these locations. Including fish collected from the Intersex Project, the scientists within the Division of Science and Research intend to investigate not only levels of intersex occurrence but begin to ascertain environmental conditions that may contribute to the intersex condition.

Sediments and surface water samples will be collected at all sites to evaluate for PFAS concentrations, in accordance with document procedures found either in the NJDEP Field Sampling Procedures Manual (August 2005), or the Michigan Department of Environmental Quality Sampling Guidance for PFAS (2018, Appendix E). The NJDEP Field Sampling Procedures manual will be the primary source of procedures, with the Michigan document providing content not covered in the current NJDEP document. The primary goal of collecting these media will be to provide a greater understanding of PFAS partitioning in the environment, between sediments, surface water, and fish tissue. The sediment and surface water samples from twelve sites determined in the Intersex project will also be analyzed for pesticides, PPCPs, and/or PCBs to aid in characterizing impact to the system.

9.3.1 Site Selection of Sampling Locations

Phase II: Several studies were used to select the locations for sampling for Phase II PFAS Evaluation. The initial studies that were used included an investigation into the PFAS analytical data collected from a statewide source water investigation by the NJDEP/DSR and the Division of Water Supply and Geosciences, Safe Drinking Water Program. This study was used to prioritize the sampling site selection process by DSR Project Officers. Using the Department's GIS data layers, sampling locations were selected based on the identification of one or more PFAS concentrations over detectable levels in the surface water where recreational fishing is permitted within proximity to known or suspected PFAS sources, such as firefighting facilities, airports, PFAS manufacturers or facilities that use such products (e.g., fabric and carpeting manufacture or treatment, metal plating) as well as a background site for comparison. Samples of surface water and sediment will be collected from sixteen locations throughout the State, with fish tissue being collected at seven of those sixteen sites, as available (See Table 1, Appendix A). Additional sediment and surface water samples (noted in Table 1 as those >1) will be collected in locations that provide a characterization of the hydrology and source apportionment of the area. Preliminary locations for these additional sites can be found in Appendix A maps.

The fish species collected will depend on the occurrence of the fish in that system at time of collection but will attempt to prioritize those fish categorized as high bioaccumulators of PFAS determined during the Phase I PFAS project. Those fish are as follows: White Perch>LMB>Bluegill>Carp>Brown Bullhead>Sunfish>Chain Pickerel. All attempts will be made to retrieve fish large enough to be considered as typical for human consumption.

Table 1: Phase II Sites and Media

Site ID		Site Description		Media			Location		
ID	ID_1	Name	Category	Fish	Sed	SW	Lat	Long	
1	LL1A	Pemberton LLAMA 1	Biosolid	0	4	4	39.9894	-	74.6811
2	LL2B	Landis LLAMA 6/Union Lake	Biosolid	9	9	9	40.7594	-	72.3368
3	LL3A	Ash Lane LLAMA 13	Biosolid	0	3	3	39.5225	-	75.3064
4	FF1	Ocean Cty Fire Fight	AFFF use	0	2	2	39.7996	-	74.2056
5	FF2	Monmouth Cty FF	AFFF use	0	2	2	40.2304	-	74.2009
6	CW2	Car Wash Impoundment/Oakford Lake	Car Wash or Suds	9	5	5	40.0540	-	74.5413
7	FF4	Somerset FF training	AFFF use	0	2	2	40.5408	-	74.6229
8	FF5	Firefighting Event/Whites Pond	AFFF use	9	3	3	41.0156	-	74.1310
9	FSW5	Allgrind Plastics	Industrial	0	1	1	40.6629	-	75.0427
10	CW5	Car Wash/Toms River at Long Swamp Creek	Car Wash or Suds	9	3	3	39.9497	-	74.1656
11	POTW7	Wayne Mountain View	WWTP	0	1	1	40.9077	-	74.2469
12	POTW8	Two Bridges Sew Auth/Pompton River	WWTP	9	1	1	40.8996	-	74.2729
13	POTW5	ParsippanyTH/Pine Brook	WWTP	9	1	1	40.8461	-	74.3416
14	POTW9	Caldwell	WWTP	0	2	2	40.8399	-	74.3129
15	POTW6	Wayne Sheffield	WWTP	0	2	2	40.9673	-	74.2807
16	FSW1	Joint Base LDM/Cranberry Bog	Military Base	6	3	3	40.0229	-	74.3475

**Coordinates denote fish collection site and/or main site of concern where fish = 0.*

Intersex Sites Under a DSR proposal entitled, "Evaluation of Site-Specific Intersex in New Jersey Surface Waters", fish tissue has been collected from six sites around New Jersey (Table 2). The intent of that project was to evaluate the reproductive tissue to determine prevalence and severity of intersex in male smallmouth bass. The fish fillets collected in this effort, along with sediment and surface water samples collected under this QAPP, will be sent for analysis for quantification of estrogen disrupting compounds, including PFAS, PPCPs, and pesticides. The PFAS concentrations from the fish tissues, sediments, and surface waters will also allow the calculation of partitioning coefficients that are the focus of the Phase II goals. This leveraging of samples collected for an alternative project will allow funding for analytical services to be maximized.

Target fish collected for this effort will be smallmouth bass only.

Table 2: Sites for Intersex Study and Media

Site ID		Location		Media		
Waterbody	Site Name	Lat	Long	Fish	Sed	SW
Delaware River	Delaware River at Montague	41.3088	-74.7972	3-6	1	1
Delaware River	Delaware River at STP at Lambertville NJ	40.3606	-74.9455	3-6	1	1
Delaware River	Delaware @ Phillipsburgh above Lehigh River	40.6924	-75.2033	3-6	1	1
Raritan River	Raritan River@ Millstone: Launch into Millstone	40.5310	-74.5874	3-6	1	1
Passaic River	Passaic River@ Two Bridges	40.8978	-74.2799	3-6	1	1
Passaic River	Passaic River@ Elmwood Park	40.9098	-74.1318	3-6	1	1

<u>Toxics in Biota Sites Selected for Leveraged Data Analysis</u> DSR and the Bureau of Freshwater and Biological Monitoring coordinate efforts to collect fish tissue that will be analyzed to support fish consumption advisories based on toxin concentration. The fish are collected and analyzed for the Toxics in Biota effort under another QAPP entitled, "Fish Tissue Monitoring Program- Round 2 Atlantic Targeted Region 2021 and Probabilistic Monitoring". The Toxics in Biota plan has focused on mercury and PCB concentrations but will also include select sites where PFAS in the collected fish tissue will be quantified by the analytical laboratory (Table 3). To leverage these data, this QAPP covers the additional samples of surface water and sediments collected at sites where PFAS will be measured in the fish tissue to increase paired data used for partitioning evaluation.

Table 3: Sites from Routine Monitoring and Media

Site Identification		Location			Media						
Site ID	Waterbody	County	Lat	Long	LMB	CP	WP	BH	Carp	Sed	SW
FTM018	Mullica River	BURLINGTON	39.6396	-74.6592	3		3			1	1
FTM020	Horicon Lake	OCEAN	40.0072	-74.3220		3		3		1	1
FTM022	Forge Pond	OCEAN	40.0664	-74.1342	3		3		3	1	1
FTM028	Deal Lake	MONMOUTH	40.2296	-74.0094	3		3		3	1	1
FTM030	Manasquan Reservoir	MONMOUTH	40.1743	-74.2002	3					1	1

Note: LMB=Largemouth Bass; CP=Chain Pickerel; BH= Bullhead; WP=White Perch

Location verification: All sampling locations will be established using an approved global positioning system (GPS) device (Trimble GeoExplorer 2008 or newer model). Subsequently, all sampling locations will be verified by sampling staff during each sampling event using an approved GPS device.

Fish Collection: Targeted species of largemouth bass (*Micropterus salmoides*), chain pickerel (*Esox niger*), White Perch (*Morone americana*), common carp (*Cyprinus carpio*), or Brown Bullhead (*Ameiurus nebulosus*) will be prioritized for collection. Depending on availability, Sunfish (*Lepomis spp*.), catfish (*Ictalurus punctatus, Ameiurus spp*.) or American eel (*Anguilla rostrata*) can be collected in place of those prioritized. These species are shown to accumulate PFAS/PFOS, are common in the lakes and rivers to be sampled and are expected to be available for a site comparison.

9.3.2 Field Collection

9.3.2.1 Fish

Fish will be collected through Direct Current (DC) electrofishing; however, other methods will be used if necessary (e.g., traps, nets, trot line or angling). Other methods will be used only in areas where boat access is limited, or in the event of equipment failure during staff deployment. Electrofishing is inherently dangerous and therefore team leaders must be trained in safe electrofishing techniques and practices to ensure safe working conditions for themselves and the field staff (AFS Professional Safety Committee 2008). Exposure to low electrical current (like that used in electrofishing) may cause death due to respiratory arrest or cardiac fibrillation (AFS Professional Safety Committee 2008). Due to these dangers, the field team leader must be trained in CPR and AED procedures. All crew members are required to wear a Coast Guard approved personal flotation device (PFD), rubber boots with non-slip soles, and electrician gloves rated at 7,500 watts.

In addition to electrofishing, baited hoop nets, gill nets, trot line or hook and line may be employed to ensure the collection of all specimens. All nets will be set in the evening near the appropriate habitat and will be checked early the following morning to minimize mortality of incidental catch. Sampling gear and crew size is at the discretion of the field coordinator. A checklist of sampling and safety equipment will be prepared prior to field work.

It is highly desirable to collect live, intact fish that have not been mutilated by the collection gear and that do not have any skin lacerations or fin deterioration that would allow body fluids to leak out of the specimen or contaminants to pass into the specimen after collection. EPA recommends that fish captured in passive collection devices not remain in the water for more than 24 hours after the passive collection device is first deployed and that specimens that show any skin or fin deterioration or external lacerations of any kind not used for chemical analysis. In addition, some fish collected by electroshocking methods may have ruptured organs due to the electroshocking procedure. Fish that are found floating dead at a site should not be used for sample analysis for human risk assessments.

For sites included in Phase II and routine monitoring, three (3) specimens of each species will be collected. These include largemouth bass (*Micropterus salmoides*), chain pickerel (*Esox niger*), white perch (*Morone americana*), or brown bullhead (*Ameiurus nebulosus*). Depending on availability, sunfish (*Lepomis spp*.), common carp (*Cyprinus carpio*), catfish (*Ictalurus punctatus, Ameiurus spp*.) or American eel (*Anguilla rostrata*) can be collected in place of those prioritized. If any of these species cannot be collected, an appropriate alternate species will be collected at the discretion of the Project Field Coordinator through consultation with the Project Officer for the day. Alternate species can be substituted for a targeted species if targeted species sample quantity is not completed at the time of collection. (See Table 4: Alternate Fish List). Additional fish samples and or species may be collected at the discretion of the Project Field Coordinator through consultation with the Project Officer for the day. Captured specimens will be held in a livewell until sampling is complete. The three (3) targeted specimens of the similar size will be removed for analysis and the remaining fish returned to the waterbody.

Table 4: Alternate Fish List

Common Name	Scientific Name
Rock bass	*Ambloplites rupestris*
Northern Pike	*Esox lucius*
Striped Bass	*Morone saxatilis*
Striped x White Bass hybrid	*Morone saxatilis x chrysops*
Yellow Perch	*Perca flavescens*
Black Crappie	*Pomoxis nigromaculatus*
White Crappie	*Pomoxis annularis*
Walleye	*Sander vitreus*
White Sucker	*Catostomus commersonii*

Length and Weight Measurements

Specimens sampled will be measured in the field to the nearest millimeter for total length using a standard measuring board and weighed to the nearest 0.02 lb. using a portable electronic scale (Brecknell ElectroSamson).

Fish specimens will remain whole and placed in zip-lock bags with labels inside and outside the bag (Post-It Notes will not be used), then into a large polypropylene/polyethylene bag, and then placed in a plastic tray that is set on top of wet ice until return to the operational facility. The processed samples that are bagged in Ziploc type (plastic) are then labeled and stored frozen until picked up for transport to the contract analytical laboratory. At no time should the fish specimen come into contact with containers that include any fluorinated polymers (e.g. polytetrafluoroethylene (PTFE)). The fish storage containers will avoid all possible routes of contamination with fluoropolymer products. Within 1 year of capture, specimens will be transported to the laboratory on either wet or dry ice, with complete sample documentation. Specimens will be held frozen until thawed for laboratory preparation. All persons handling the fish during collection shall wear new nitrile gloves. All environmental information, collection data, and sampling conditions, including identification numbering for the processed specimens will be recorded and provided to the Project Officers.

9.3.2.2 Water

One surface water grab or point sample will be taken at each of the fish sampling locations with the option of additional samples being taken if field resources and analytical costs allow (to be determined during projects as subtotals of analysis are processed). Water samples will be collected as per "NJDEP Field Sampling Procedures Manual", 2005. Where possible, samples will be taken from a depth of between six (6) to eighteen (18) inches below the surface or at a depth so as not to be in contact with bottom sediments. Samples should be taken in an area where water is flowing or circulating in the generalized manner expected in said waterbody. Locations will be chosen to reflect water representative of waterbody. The procedure for choosing the location of the grab sample will be to use professional judgement as to where the water appears as well mixed as is the larger proportion of water in that water body. Samples will be collected using either a grab or point sample technique by field sampler. If the sample is taken by grab or point sample directly into sampling container, a field blank will not be necessary.

Sample containers for water samples that will be provided by the contract laboratory and be in strict compliance with operating procedures that avoid cross contamination with all fluropolymer products. (See Appendix E, Collection and Handling of Samples for Analysis of PFOA, PFOS and Other Fluorinated Compounds). Sample bottles will be 500 mL or 1.0 L, as supplied by the lab. Sample containers shall be dedicated as <u>Single Use</u>. Samples that will be sent for PFAS and PPCP analysis shall be collected in polyethylene (HDPE) wide-mouth bottles fitted with an unlined (no Teflon), polyethylene screw cap. Polypropylene products shall not be used. Samples that will be analyzed for pesticides will be collected in glass, preferably brown/amber glass, provided by the lab. The HDPE sample bottles shall be labeled only by writing directly on bottle with permanent marker and shall not have a sticky label attached. The bottles will then be sealed individually in a Ziploc or whirlpak type bag and stored in a cooler at 4°C. Water samples and any field blanks should be shipped to the laboratory in a cooler on packaged wet ice or with sufficient blue ice packs to maintain the cool temperature.

9.3.2.3 Sediment

At least one sediment sample will be taken at each of the fish sampling locations. Sediment samples will be collected at a location of sediment deposition and where possible within the area of fish collection. Exact location of sediment samples will be determined at the time of collection and will be determined using best professional judgement of location where sediment is likely to accumulate. Ideal locations for sediment collection will include areas downstream of suspected sources or identified industrial areas where the velocity of the water slows due to changes in direction of the stream or due to dams. These locations can be tentatively identified prior to field work. If two samples of sediment will be obtained from same site location, the downstream location will be obtained first.

Appropriate GPS location information will be collected at the time of sampling. The collection of surface sediment samples (sampler to record estimated depth of sample, aiming to acquire top 5-20 cm) will be performed as a grab sample via the use of petite ponar sampler. Sediments will be collected with the ponar from the surface of the sediment bed and placed into a stainless-steel tray. The sample in the tray will not be homogenized, but a subsample taken from the surficial sediments will be transferred by stainless steel spoon/scoop or nitrile-gloved hand directly in laboratory provided HDPE jars.

Field equipment used for sediment sampling will be decontaminated prior to initial site. The decontamination of the field equipment (ponar, tray and scoop) will include a five-step procedure: 1) detergent wash 2) tap water rinse, 3) acetone rinse, 4) deinonized (or Pico © water) rinse, and 5) air drying. Equipment should be wrapped in untreated aluminum foil for transport.

If two or more sediment samples are to be taken in a single sampling day, precaution for cross contamination will require attention. If sediments samples are taken from the same river, the ponar can be rinsed with ambient water for a minimum of two minutes. If sediment samples are taken from different water bodies in the same day, the ponar will be cleaned in between sites using the five-step method outlined above.

Sample containers for sediment samples will be provided by the contract laboratory and be in strict compliance with operating procedures that avoid contamination. Sediment samples for PFAS and PPCP analysis will be collected in 250 mL HDPE jars, ensuring that no Teflon material is used during the collection process. Polypropylene products will not be used. Sediment samples for pesticide analysis will be collected in a glass, preferably a brown/amber, glass vessel provided by the lab. Sample containers shall be dedicated, Single Use. Once filled, each jar of sample should be sealed individually in a Ziploc or whirlpak type bag and frozen or stored in a cooler at 4°C. Sediment samples should be shipped to the laboratory in a cooler on packaged wet ice or with sufficient blue ice packs to maintain the cool temperature.

9.4 **Laboratory Analysis**

The Division of Science and Research is tasked with determining the occurrence, fate, and transport of emerging compounds that may impact the human health and the environment of New Jersey. Often, the analytical methods for these compounds are not fully certified according to state requirements specified in the NJDEP Office of Quality Assurance Regulations. The results are not used for compliance purposes but are applied to the base knowledge to determine future actions. The results are also used to apply fish consumption advisories, which are also not regulatory in nature. Therefore, many analytical procedures are evaluated by the scientists of the Division and deemed to be appropriate for the research study documented here.

DSR stipulates that the contract laboratory shall have previous analytical research in multimedia investigations for the state of New Jersey and provide the most recent analytical procedures to quantify required analytes. The contract laboratory procured for this project has been determined through review of proposals after the posting of a "Request for Proposals" in March of 2021. SGS Axys Analytical (CANA005) was selected to perform the necessary analysis of the collected media to quantify the per- and polyfluoroalkyl substances present, pesticides, and PPCPs. SGS Axys Analytical will be responsible to conduct all water, sediment and fish tissue analyses for the samples submitted under this research project. The processing of the samples will include the fish sample preparation (i.e., skinning, filleting, and homogenizing) of all fish samples submitted to maintain sample integrity.

Samples of fish shall be stored frozen until processing in the laboratory. Samples of water and sediment shall also be stored frozen. The maximum holding time for intact whole fish will be 1 year. The maximum holding time for water samples is ninty (90) days. All transfers of samples will be properly documented throughout transport and analysis (internal lab chain of custody). All laboratory equipment will be properly calibrated as per each method completed. Careful cleaning of all laboratory equipment and instruments using the appropriate soaps, solvents, acids, and double deionized water (DDW) will be employed.

Fish tissue samples may be composited for analysis. All specimens will be filleted by the contracted laboratory using clean methods as outlined in EPA's "Guidance for Assessing Chemical Contaminant data for Use in Fish Advisories Vol 1 Fish Sampling and Analysis" 2000. The decision to composite tissue samples or to analyze individual samples will be made in coordination with maximizing budget allocations while supporting optimal data to evaluate for end purposes.

All samples collected will be analyzed for at least 40 PFAS, based on methods below. Select samples will also be analyzed for pesticides and/or PPCPs. The details of sample collection and analytical methods are provided below.

9.4.1 PFAS

SGS Axys Method MLA-110 uses solid phase extraction before analysis by liquid chromatography/tandem mass spectrometry (LC-MS/MS). Quantitation is achieved with isotopically labeled standards using the isotope dilution technique. Typical Method Reporting Limits (RLs) vary by media and PFAS structure and are presented (on page 1) in the method summary document, "Summary of SGS Axys Method MLA-110 Rev 02 Ver 11: Analytical Procedure for the Analysis of Per- and Polyfluoroalkyl Substances (PFAS) in Aqueous Samples, Solids, Tissues, AFFF Products, Blood/Serums and Solvent Extracts by LC-MS/MS" provided in Appendix B for details. Reporting Levels, which are required and acceptable for the purposes of this study, are provided for fish tissue, water, and sediments in the method summary. The specifications for sample collection are noted in the following table (Table 5).

Table 5: PFAS Sample Collection Specifications

Method:	**MLA-110 PFAS by UPLC-MS/MS**		
Target Compounds:	40 PFAS including PFAAs, Fluorotelomer sulfonates, HFPO-DA, ADONA, and others		
Matrix:	**Water/Effluent**	**Sediment**	**Tissue**
Units/Sample Size:	ng/L based on 0.5 L sample	ng/g based on 5 g sample	ng/g based on 2 g sample
Default Extract Volume:	4000 uL	4000 uL	4000 uL
Sample Preservation:	None	None	None
Bottle Type:	HDPE	HDPE	HDPE
Sample Holding Time:	90 days	1 year	1 year
Extract Holding Time	30 days	30 days	30 days

9.4.2 Pesticides

SGS Axys Method MLA-028 Rev 06 Ver 14 is the analytical procedure used to detect organochlorine pesticides by isotope dilution HRGC/HRMS and meets the performance and quality procedures requirements of EPA Method 1699. Refer to summary document, “SGS Axys Method MLA-028 Rev 06 Ver 14: Analytical Procedures for Organochlorine pesticides by isotope dilution HRGC/HRMS by EPA Method 1699” included in Appendix C. Results will include both E1 and E2 analytes. Typical sample specific detection limits, which are required and acceptable for the purposes of this study, are provided for fish tissue, water, and sediments (on page 13) in the method summary include in Appendix C. The specifications for sample collection are noted in Table 6, below.

Table 6: Pesticides Sample Collection Specifications

Method:	**MLA-028: OC Pesticides by GC/HRMS**		
Target Compounds:	30 OC pesticides, includes endrin aldehyde		
Matrix:	**Water/Effluent**	**Sediment**	**Tissue**
Units/Sample Size:	ng/L based on 1 L sample	ng/g based on 10 g sample	ng/g based on 10 g samples
Default Extract Volume:	200 uL	200 uL	200uL
Sample Preservation:	<4 °C, no preservation	<4 °C, no preservation	<4 °C, no preservation
Bottle Type:	Glass (amber/brown)	Glass (amber/brown)	N/A

Sample Holding Time:	7 days	1 year	1 year
Extract Holding Time	1 year	1 year	1 year

9.4.3 Pharmaceuticals and Personal Care Products

SGS Axys Method MLA-075 Rev 09 Ver 01 is the analytical procedure used to detect pharmaceutical and personal care products and hormones by LC-MS/MS. Refer to summary document, “SGS Axys Method MLA-075 Rev 09 Ver 01: Summary of SGS Axys Method MLA-075 Rev 09 Ver 01: Analysis of Pharmaceutical and Personal Care Products and Hormones in Solid, Aqueous, Tissue and POCIS Samples by LC-MS/MS” in Appendix D. Analysis requested (analyte lists) will be dependent on site characteristics and funding. Sample specific reporting limits, which are required and acceptable for the purposes of this study, are provided for fish tissue, water, and sediments in the method summary. The specifications for sample collection are noted in Table 7, below.

Table 7: PPCPs Sample Collection Specifications

Method:	**MLA-075/ EPA 1694 PPCPs by LC-MS/MS**			
Target Compounds:	Hormones, Pharmaceutical and Personal Care Products			
Matrix:	**Water/Effluent**	**Sediment**	**Tissue- Acid Extraction**	**Tissue- Basic extraction**
Units/Sample Size:	ng/L based on 0.5 L sample	ng/g based on 0.5 (dry) g but not more than 2.5 g wet sample	ng/g based on 1.25 (wet) g	ng/g based on 0.5 (wet) g
Default Extract Volume:	2000uL	2000uL	2000uL	2000uL
Sample Preservation:	None	None	None	None
Bottle Type:	Amber glass or HDPE	Amber glass or HDPE	Amber glass or HDPE	Amber glass or HDPE
Sample Holding Time:	7 days for < 4 ºC storage	7 days for -20 ºC storage	7 days for -20 ºC storage	7 days for -20 ºC storage
Extract Holding Time	40 days	40 days	40 days	40 days

10 Schedule of Tasks and Products

Project Requested: March 2021
Work/Quality Assurance Plan: June 2021
Sampling Activities: July 2021 through September 2022
Laboratory Activities: July 2021 through October 2022
Data Reports: data tables and Level IV Data Packages (electronic) as laboratory results become available.

11 **Quality Assurance**

QA/QC with be the responsibility of the project quality assurance officer. Any deviations from the QA/QC measures detailed in the Summary SOPs will require approval by project management. As part of quality assurance and quality control (QA/QC), Standard Reference Material (SRM) will be analyzed by the analytical laboratory (SGS Axys, Certification ID CANA005). This material will be obtained from the National Institute of Standards and Technology (NIST) or equivalent agency and may consist of DORM 1, EPA SRS903, SRM 1974 (or equivalent if available). All sample spikes will use specimens from this project only. Also, duplicate samples will be analyzed to help assess laboratory variations in the analysis of fish tissue.

12.1 Sample Retention

All samples must be retained by the laboratory until such time that the Project Manager from the DSR approves the reported results.

12.2 Blanks

Equipment blanks will be collected with each use of equipment that comes into contact with sample before that sample is deposited in the ultimate sample collection vessel. For instance, if a dredge ponar is used to collect sediment, an equipment blank of lab verified water will be rinsed over the ponar before use.

Field blanks will be collected every five sampling events.

Analytical blanks will be performed by the lab, one for each batch of analysis performed.

13 **Data Quality Requirements**

Analytical samples will be evaluated by the methods specified in this QAPP and for which the laboratory has provided method summaries and acceptable method reporting and detection limits. Quality control procedures (including required calibrations, equipment cleaning, and other quality control procedures required by regulation or by the method shall be defined in SGS AXYS' QA/QC document "Quality Assurance/Quality Control (QA/QC) Policies and Procedures Manual, QDO-001 Revision No. 33, 01-June-2021, SGS AXYS Analytical Services Ltd." This document defines and encompasses all aspects of SGS AXYS technical and managerial processes including sample receiving, sample analysis, acceptability criteria, data validation and monitoring. Specific method details and quality control elements are described in the laboratory's Standard Operating Procedures (SOPs) and Method Summaries.

14 Sample Custody Procedures

Chain of custody will be required for all samples as per N.J.A.C. 7:18-9.3(b). SGS Axys will provide chain of custody forms.

15 Data Validation

The Project Managers are responsible for all initial data validation. If apparent anomalous data are suspected, the Project Managers will review the sampling procedures with the Project Field Coordinator or field team leader to make sure the proper collection and preservation procedures were followed. If the data is still suspect, the laboratory will be contacted. An internal review of their laboratory procedures and/or calculations used in the analysis of the suspect sample, with special emphasis on transcription of data to assure that no transposition of figures occurred will be conducted. The laboratory will be asked to check on equipment calibration. They may be further requested to reanalyze the retained portion of the sample. If no problems are found in the analytical laboratory procedures, the data may then be compared to any historical data that might have been collected at the same site prior to the most recent sampling event to see if similar anomalies might have been found previously. The suspect data may also be compared to literature values or standard analytical treatises to verify whether the results are within the limits of accuracy of the test method.

If no obvious problems are found after these reviews, the complete data set will be reported with the suspect data identified as such. The DSR will then conduct its own review of the data, as it relates to the objectives(s) and data accuracy required in this project.

16 Performance System Audits

This project plan could be audited by the Office of Quality Assurance (OQA) to ensure compliance with stated procedures.

The analytical methods used for this project are not covered by a NJ certification (at the time of this writing) and since they are not certified, they would not be subject to OQA audits for these methods. However, NJ certified laboratories are subject to audits and to the requirements of the OQA Laboratory Certification Program as well as internal performance evaluations for methods where they do hold certification. Given SGS Axys holds certification for analytes not covered by this QAPP, this lab would be subject to those reviews.

17 Data Reporting

17.1 Reporting of Data

Final analytical data will be reported to the DSR, from the laboratory employed for this project, in the form of electronic and/ or hard copies of full Level IV Data Package within 6-

8 weeks from receipt of sample. All data shall be reported in a complete and concise fashion and shall meet the reporting requirements of NJAC 7:18. Routine quality control results must be retained on file for review by the DSR and the OQA.

18 Data Storage and Distribution

Sampling results will be stored locally in a Microsoft Access or Excel database. Data will be entered into New Jersey's Water Quality Data Exchange (WQDE) and USEPA STORET Data Warehouse by the DSR by June of the following year it is verified. Data will be available through STORET. All data records shall be maintained for a period of no less than five years.

19 Assessment, Oversight and Response

The DSR Project Officers will be responsible for the overall design of the project, field collection of sediment and water, processing and shipping of water and sediment samples to the laboratory, data review and entering data into WQDE and STORET. The BFBM Field Coordinator will oversee all fish tissue field operations, collections, processing, and shipping of fish tissue samples to the laboratory and serve as a liaison to help coordinate BFBM support to assist DSR with field collection of sediment and water. The Project Officers will participate in field collection functions, assess, and make corrections when necessary to maintain the data accuracy as defined in this plan. If any major changes or significant modifications are made to this plan regarding data collection, laboratory analysis or data quality assurance as it relates to the objectives(s) and data accuracy required in this project, all original signees of the QAPP will be notified.

References

EPA "Guidance for Assessing Chemical Contaminant data for Use in Fish Advisories Vol 1 Fish Sampling and Analysis" 2000. EPA-823-B-00-007. United States Environmental Protection Agency, Office of Water, Washington, D.C.

Goodrow, S. M, B. Ruppell, R. L. Lippincott, G. B. Post. 2018. Investigation of Levels of Perfluorinated Compounds in New Jersey Fish, Surface Water, and Sediment. NJ Department of Environmental Protection. Trenton, NJ.

Goodrow, S. M, B. Ruppell, R. L. Lippincott, G. B. Post, and N. A. Procopio. 2020. Investigation of levels of perfluoroalkyl substances in surface water, sediment and fish tissue in New Jersey, USA. Science of the Total Environment. 729:138839. https://doi.org/10.1016/j.scitotenv.2020.138839

Michigan Department of Environmental Quality PFAS Sampling Guidance, Revised 10/16/2018, https://www.michigan.gov/pfasresponse/0,9038,7-365-88059_91297---,00.html Accessed 7/6/2021.

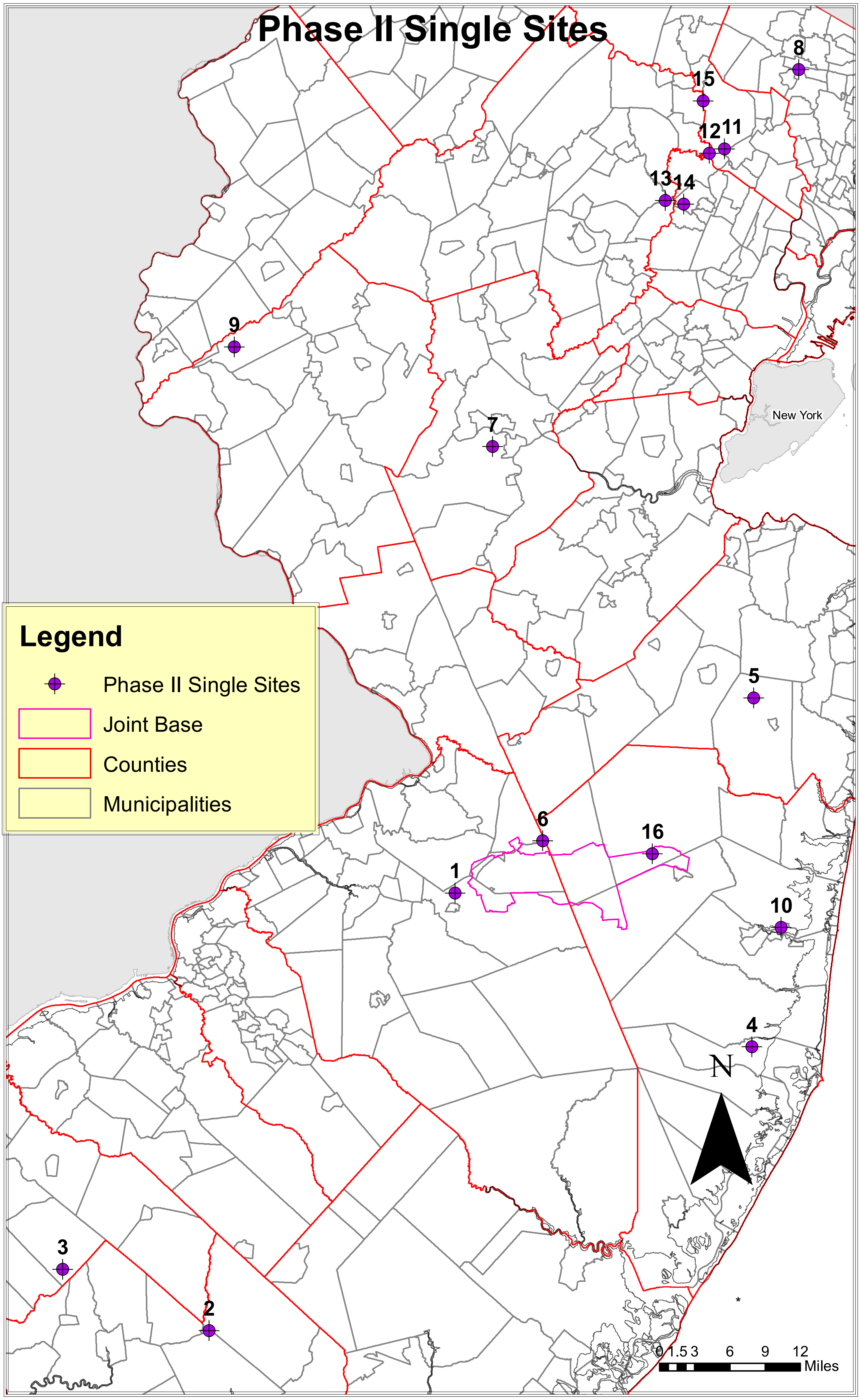
NJDEP Field Sampling Procedures Manual, August 2005:
http://www.state.nj.us/dep/srp/guidance/fspm/pdf/fsmp2005.pdf
Accessed online June 30, 2021.

EPA “Guidance for Assessing Chemical Contaminant data for Use in Fish Advisories Vol 1 Fish Sampling and Analysis” 2000. EPA-823-B-00-007. United States Environmental Protection Agency, Office of Water, Washington, D.C.

Appendix A:

Map of PFAS II+ Sample Sites

Phase II Single Sites



4/11/2019 sg

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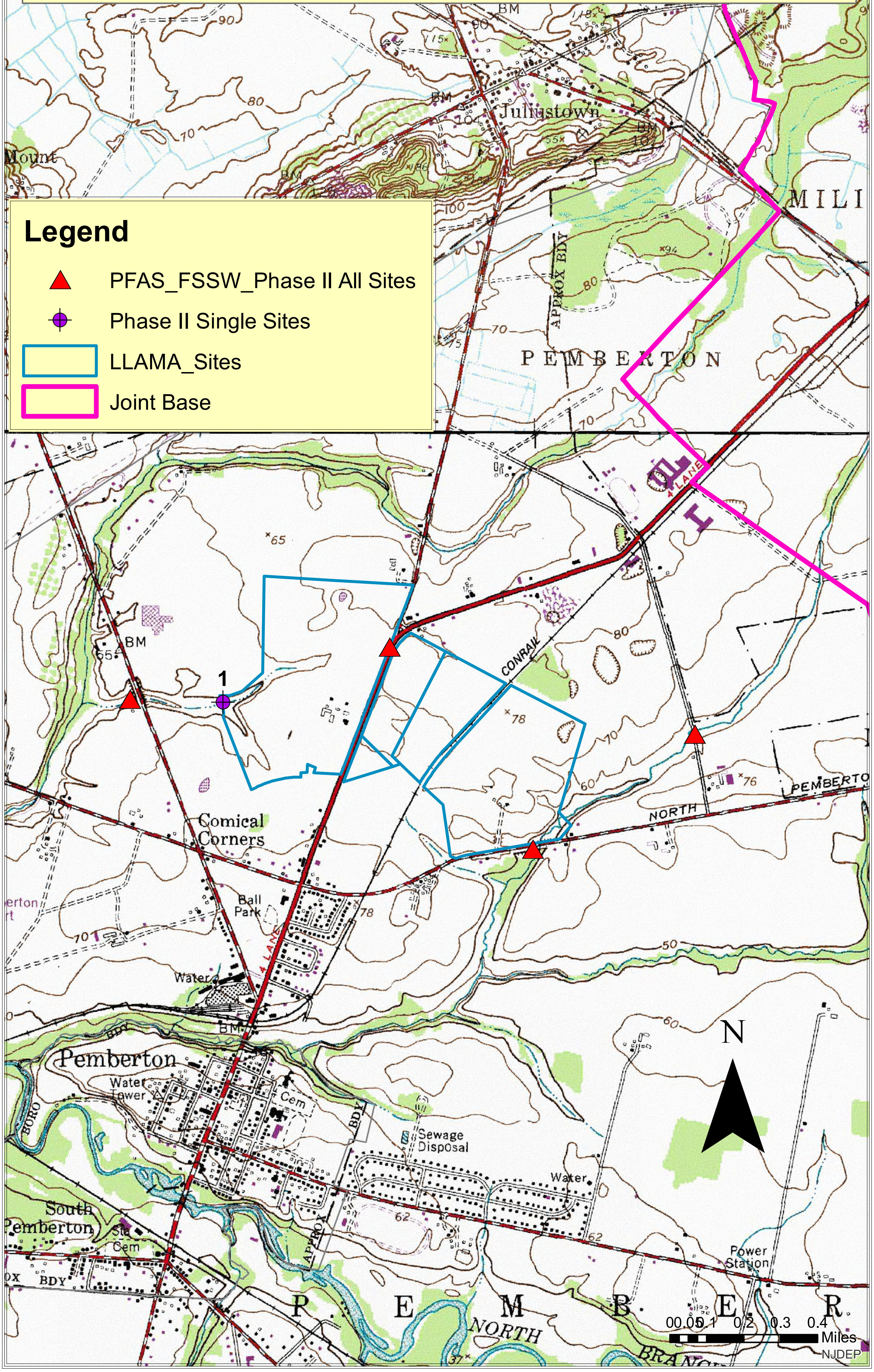
Phase II: Site 1 and four sub-sampling stations



4/11/2019 sg

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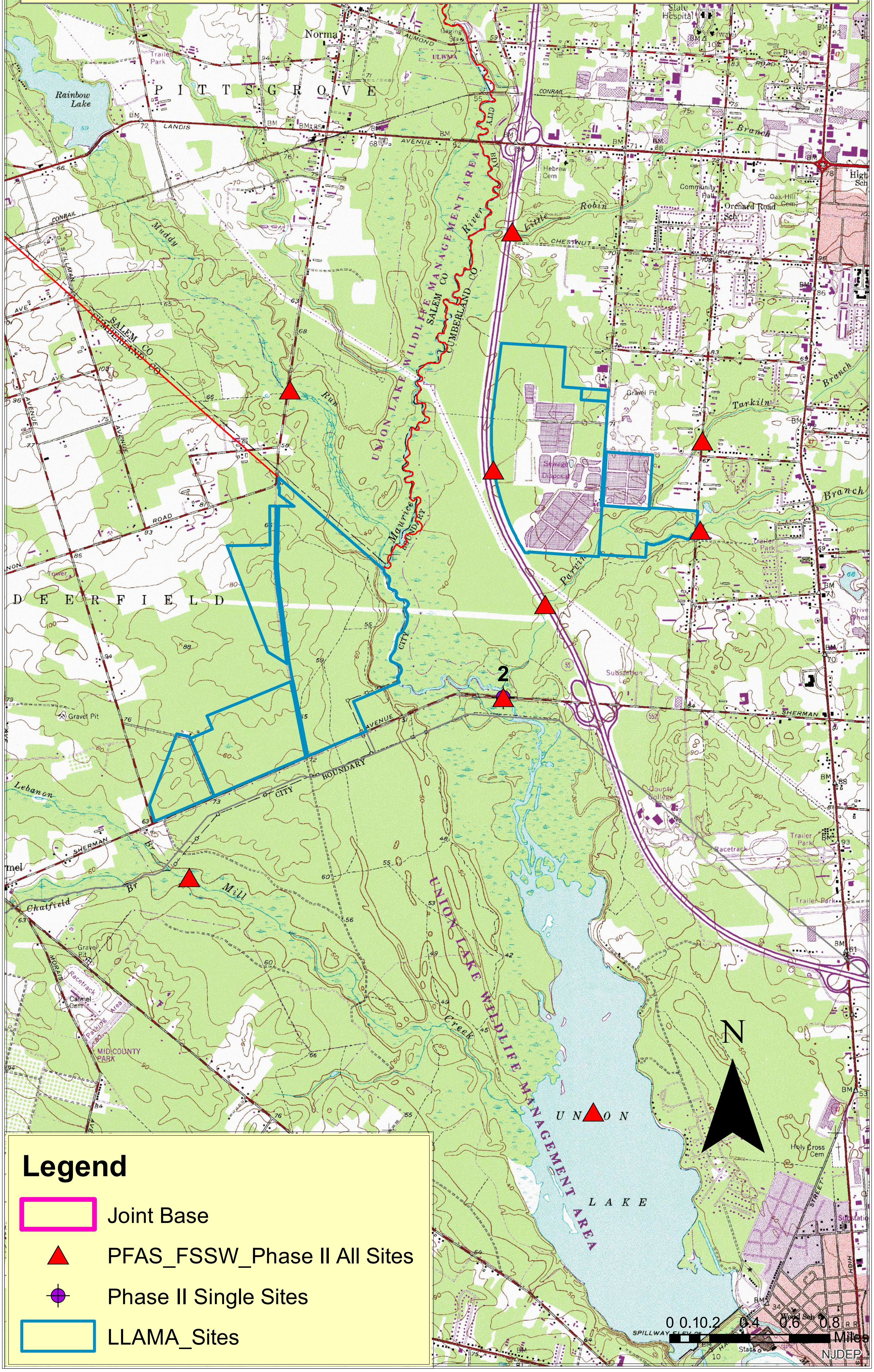
Phase II: Site 2 and nine sub-sampling stations



4/11/2019 sg

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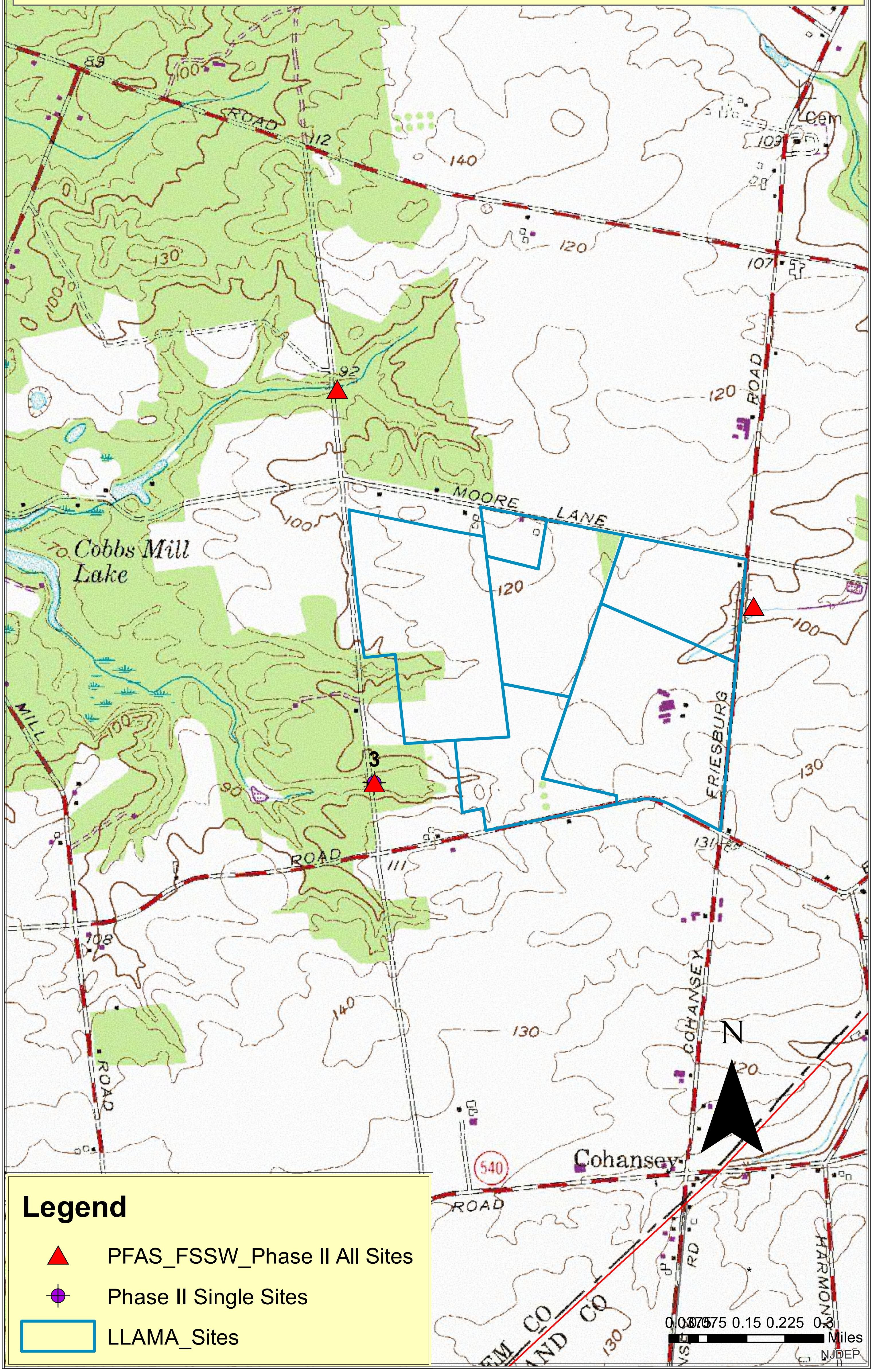
Phase II: Site 3 and three sub-sampling stations



4/11/2019 sg

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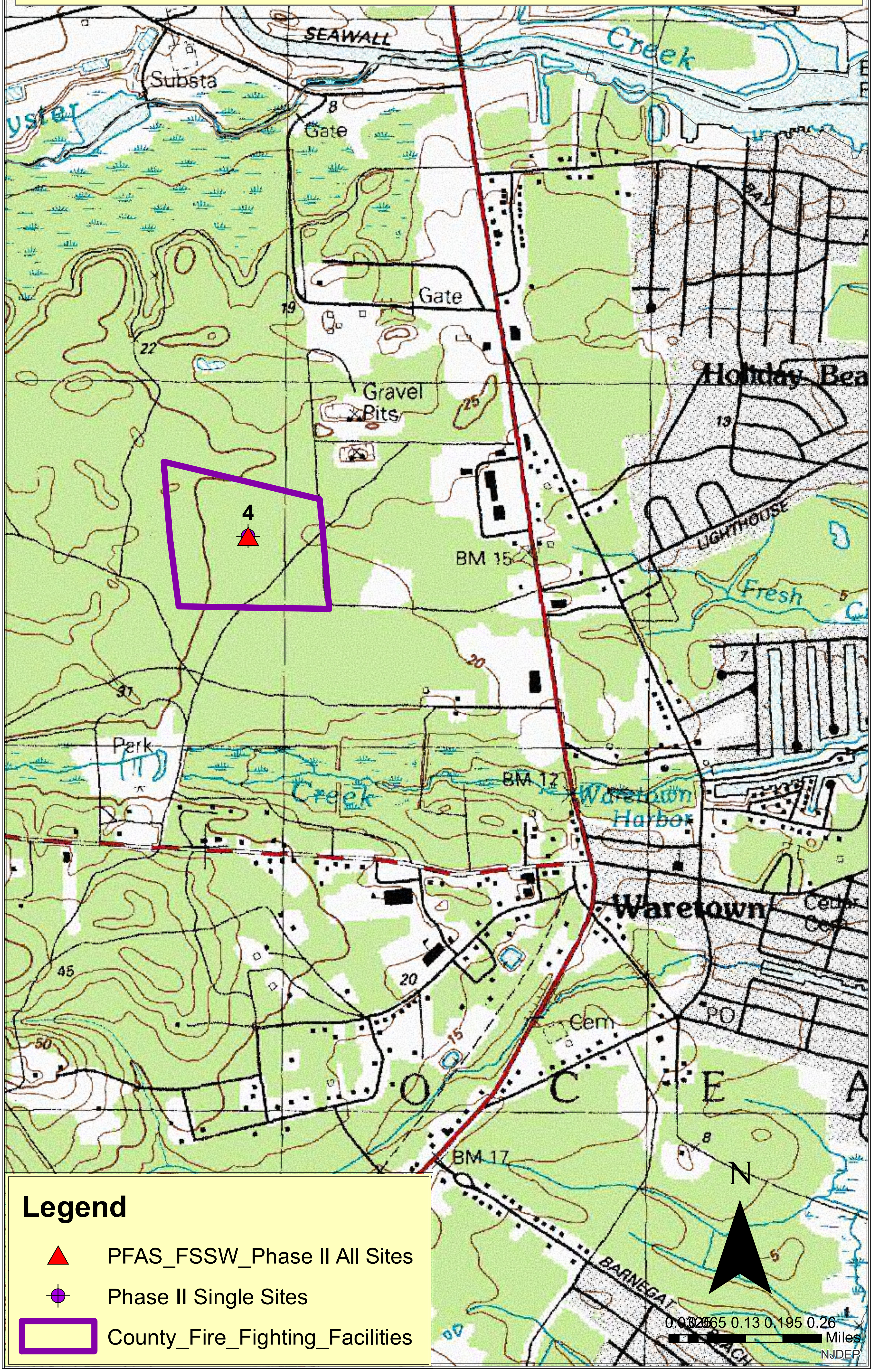
Phase II: Site 4 and single sub-sampling station



4/11/2019 sg

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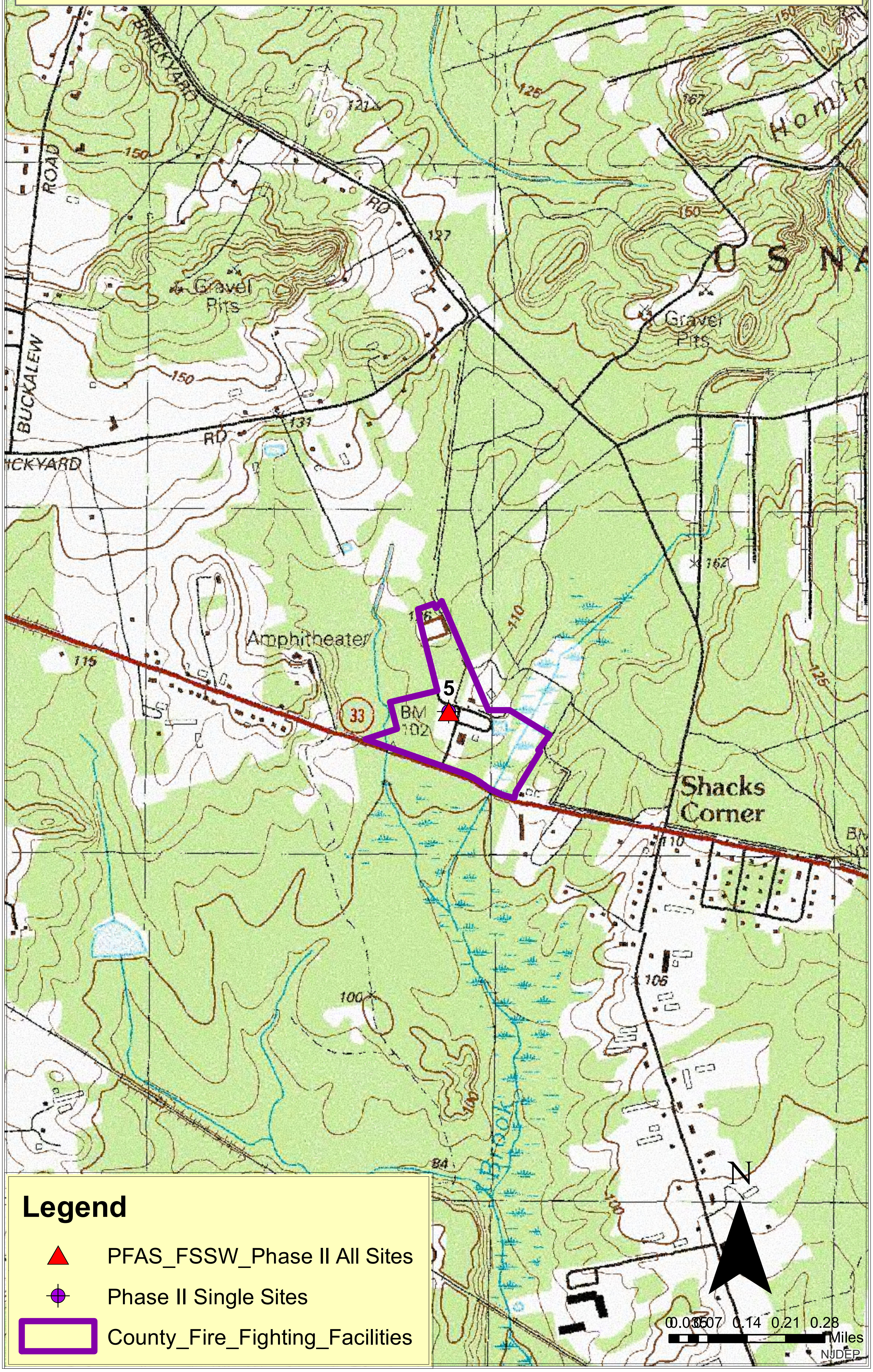
Phase II: Site 5 and single sub-sampling station



4/11/2019 sg

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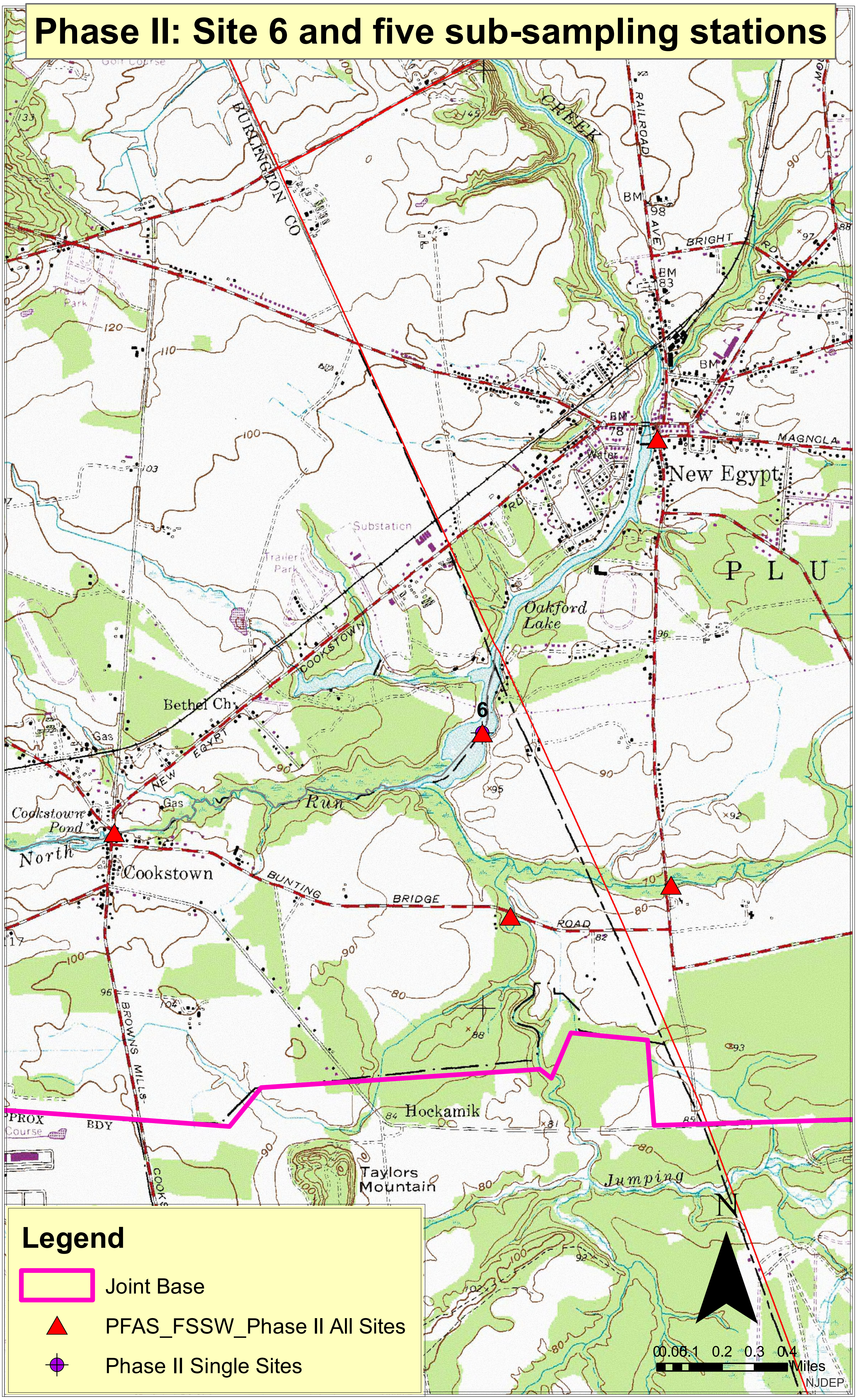
Phase II: Site 6 and five sub-sampling stations



4/11/2019 sg

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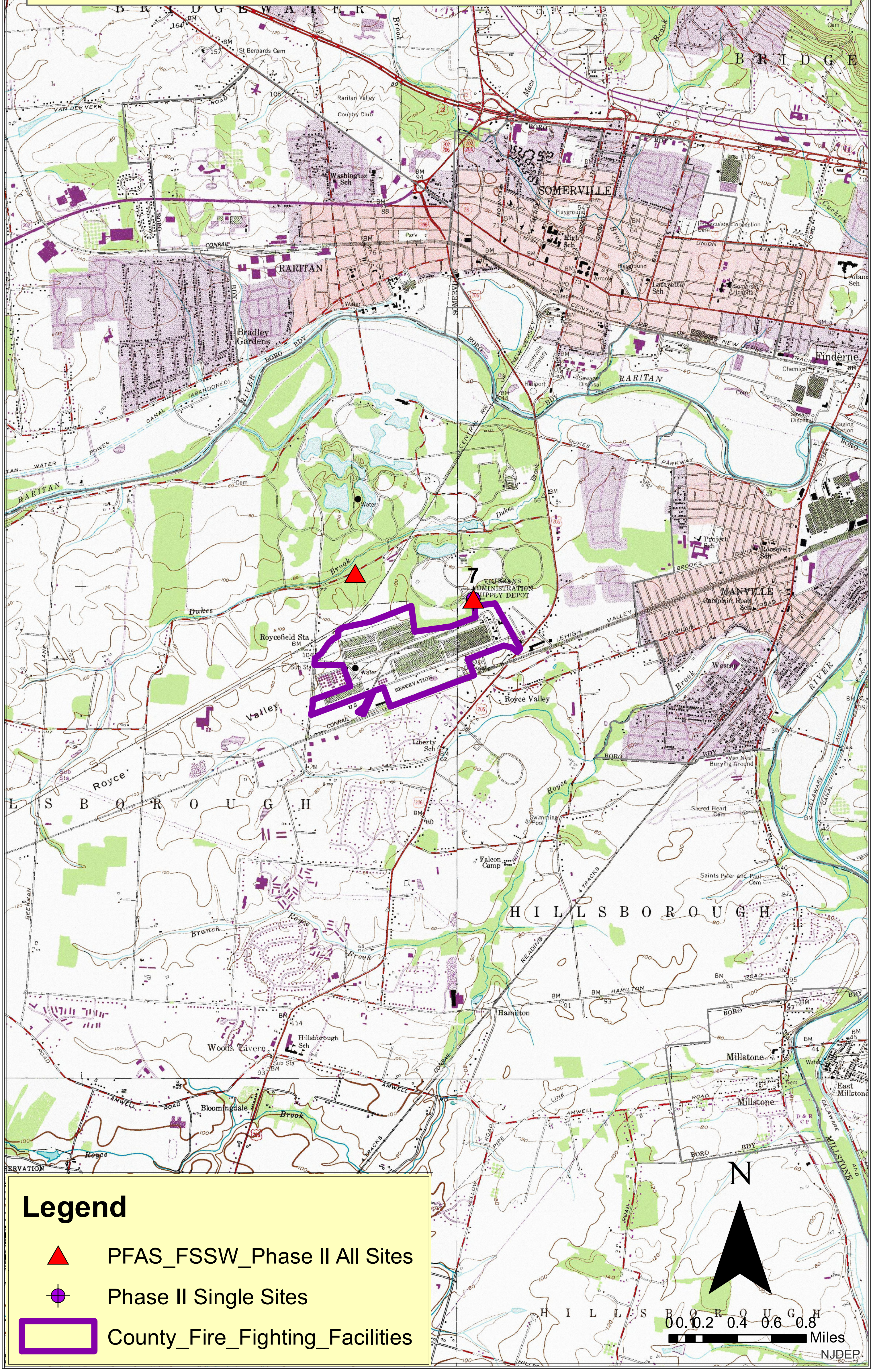
Phase II: Site 7 and two sub-sampling stations



4/11/2019 sg

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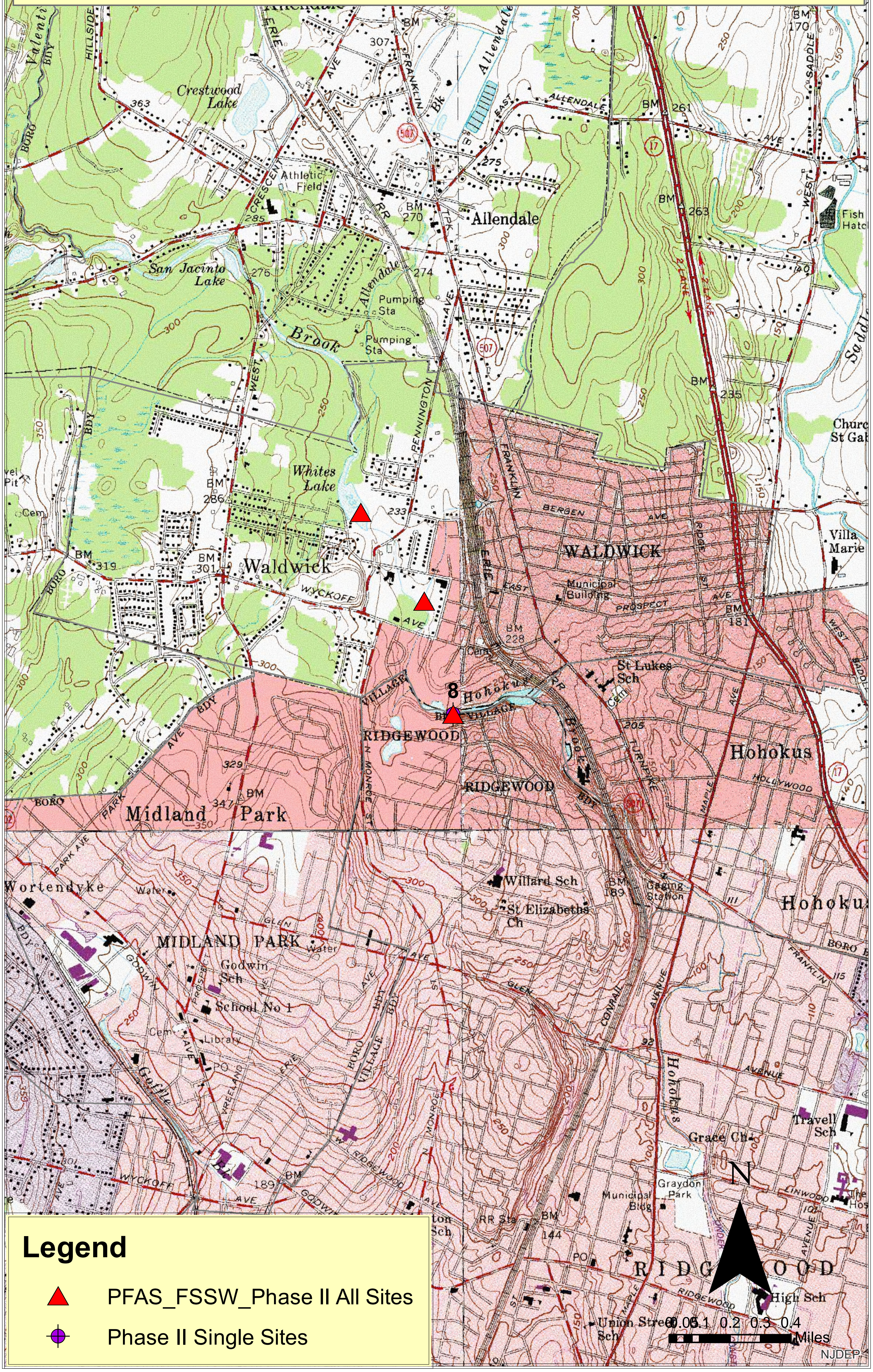
Phase II: Site 8 and three sub-sampling stations



4/11/2019 sg

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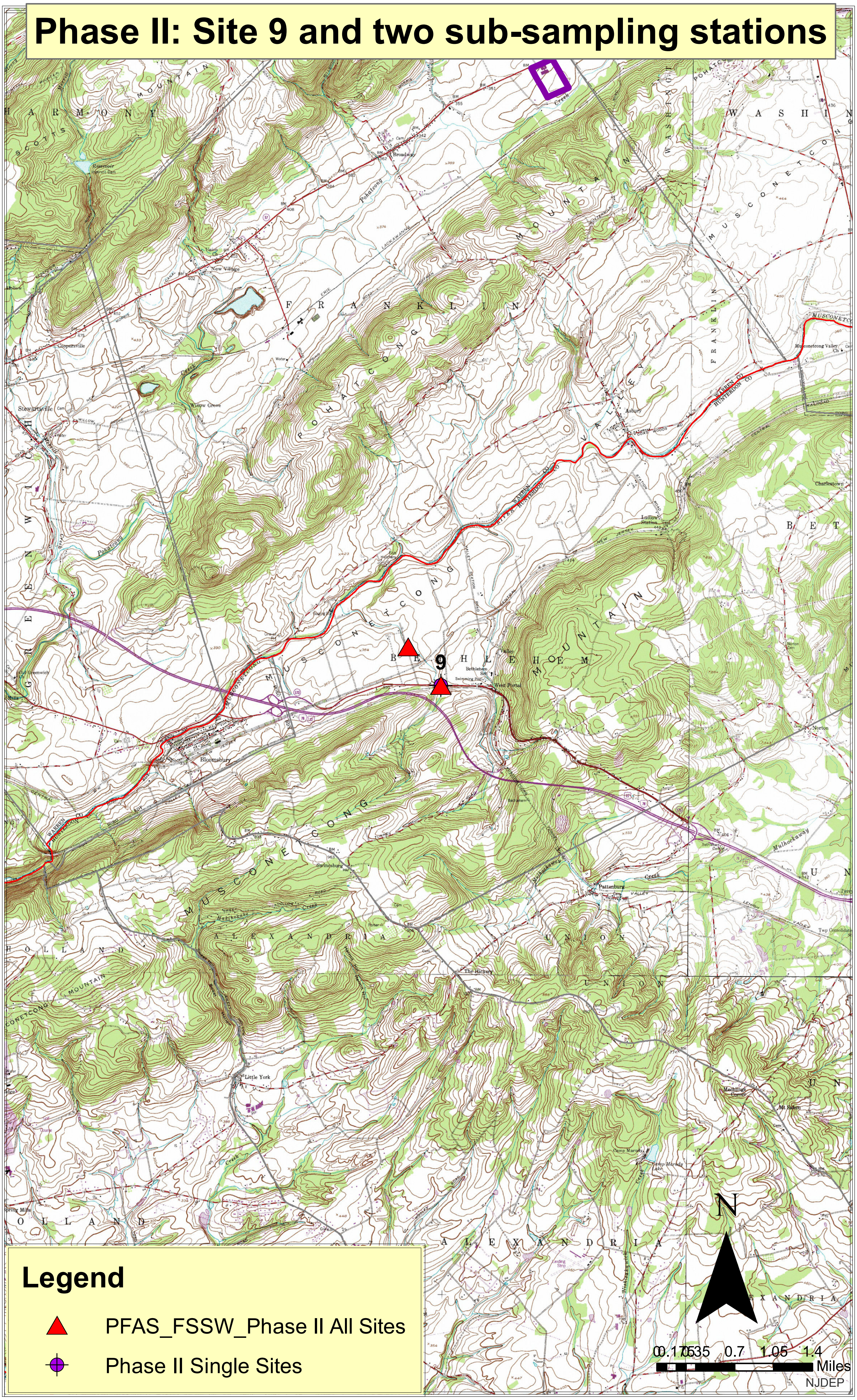
Phase II: Site 9 and two sub-sampling stations



Legend

▲ PFAS_FSSW_Phase II All Sites

⊕ Phase II Single Sites

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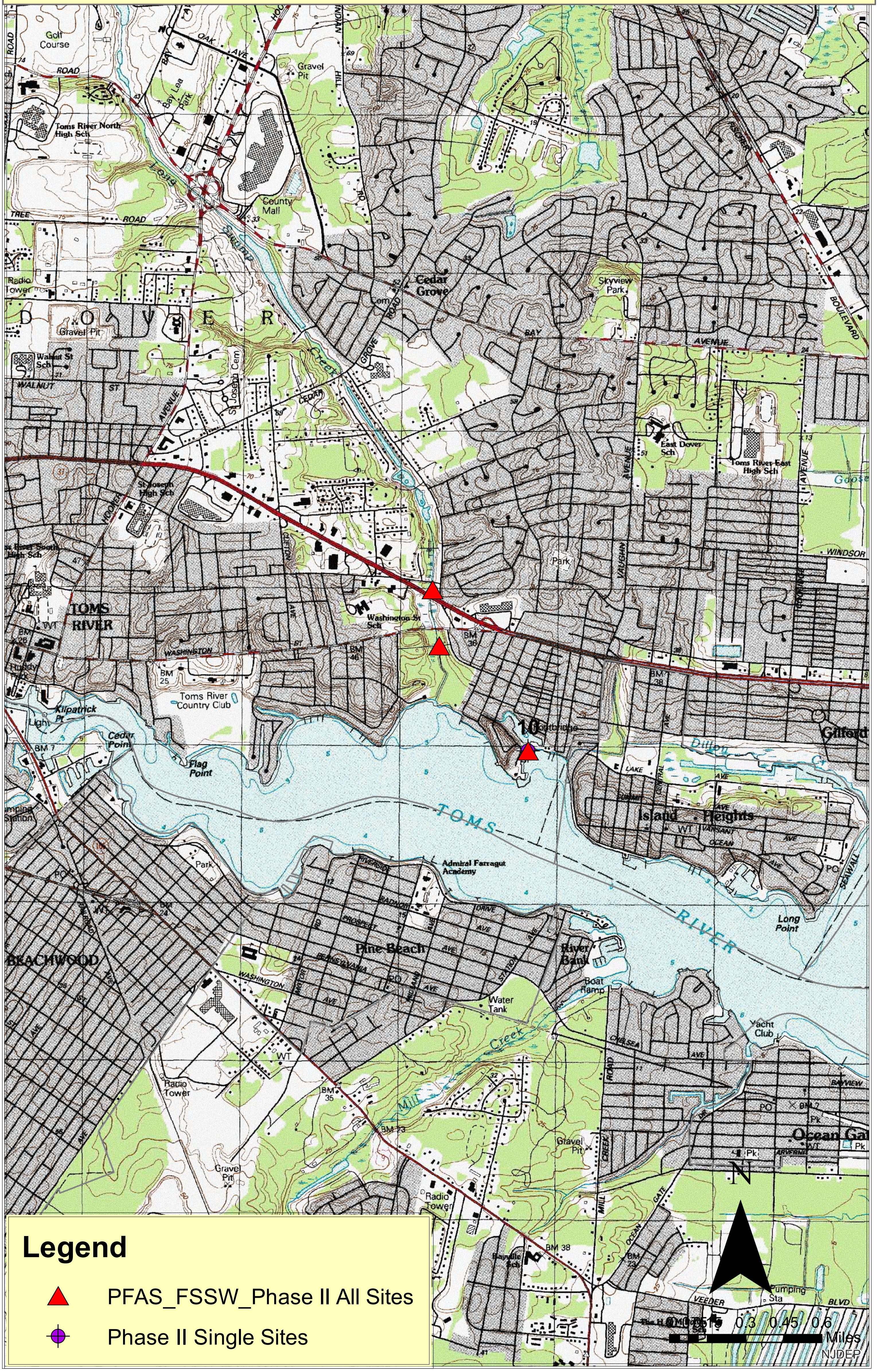
Phase II: Site 10 and three sub-sampling stations



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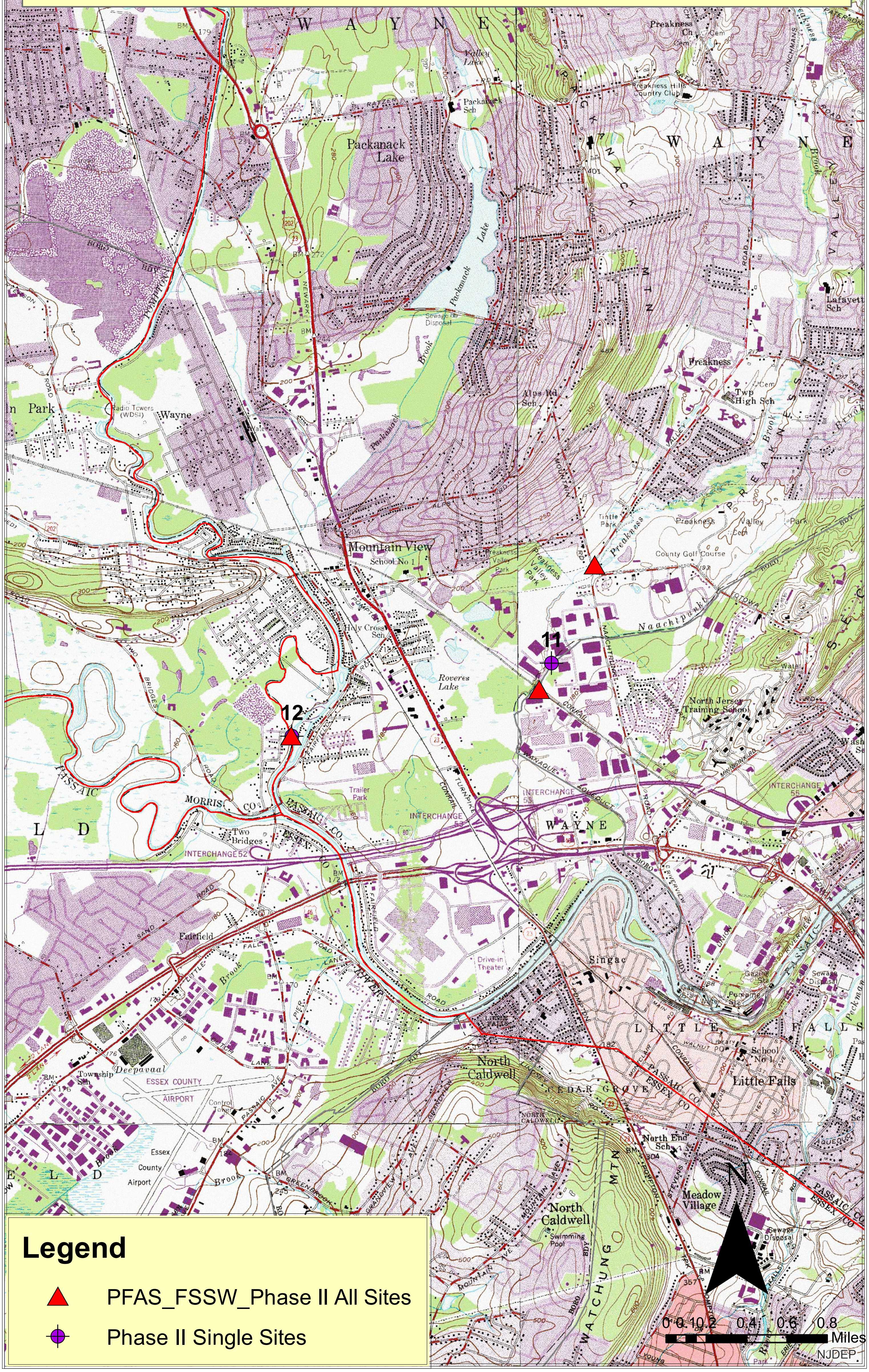
Phase II: Sits 11 and 12 and three sub-sampling stations



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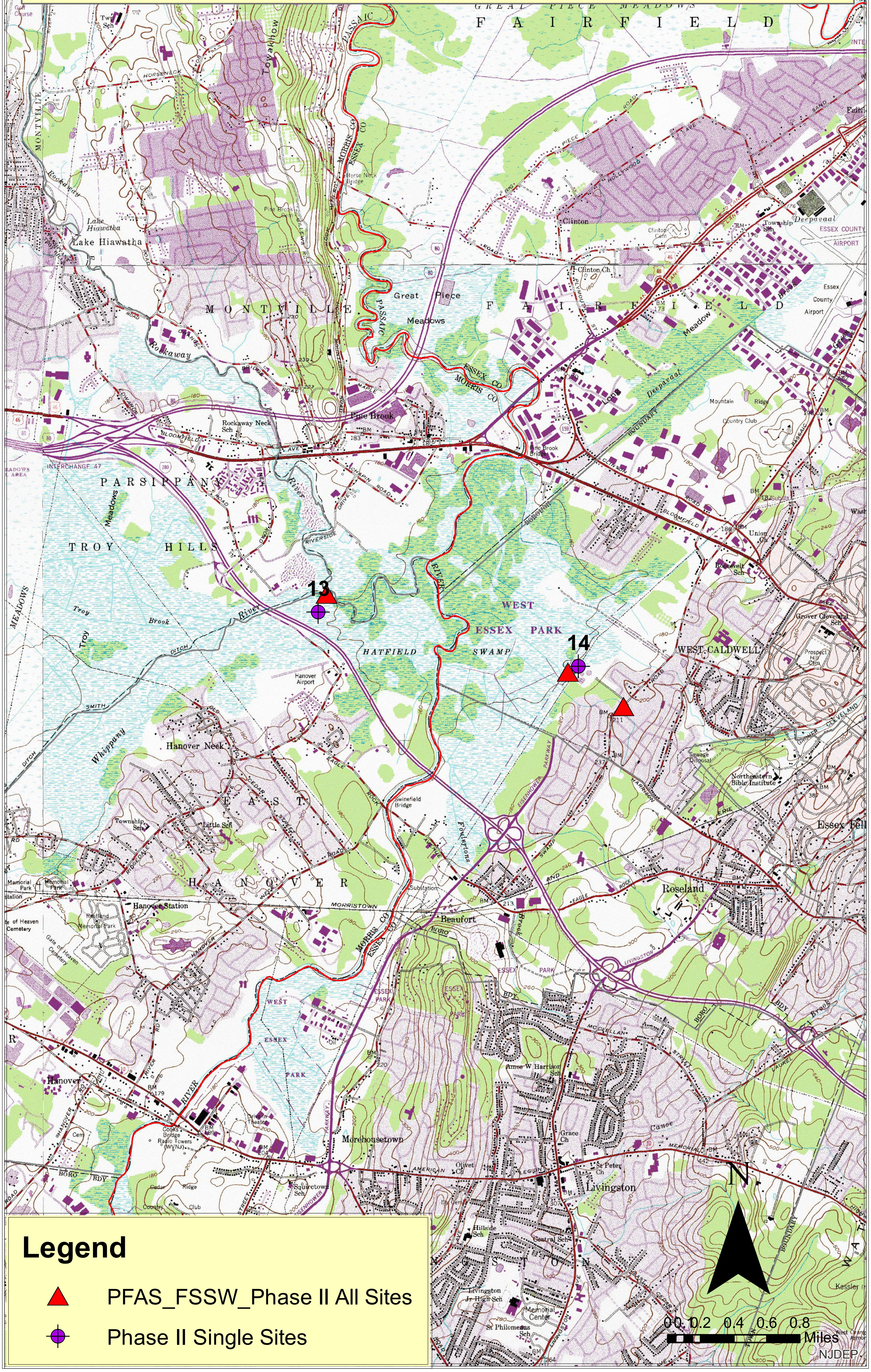
Phase II: Sits 13 and 14 and three sub-sampling stations



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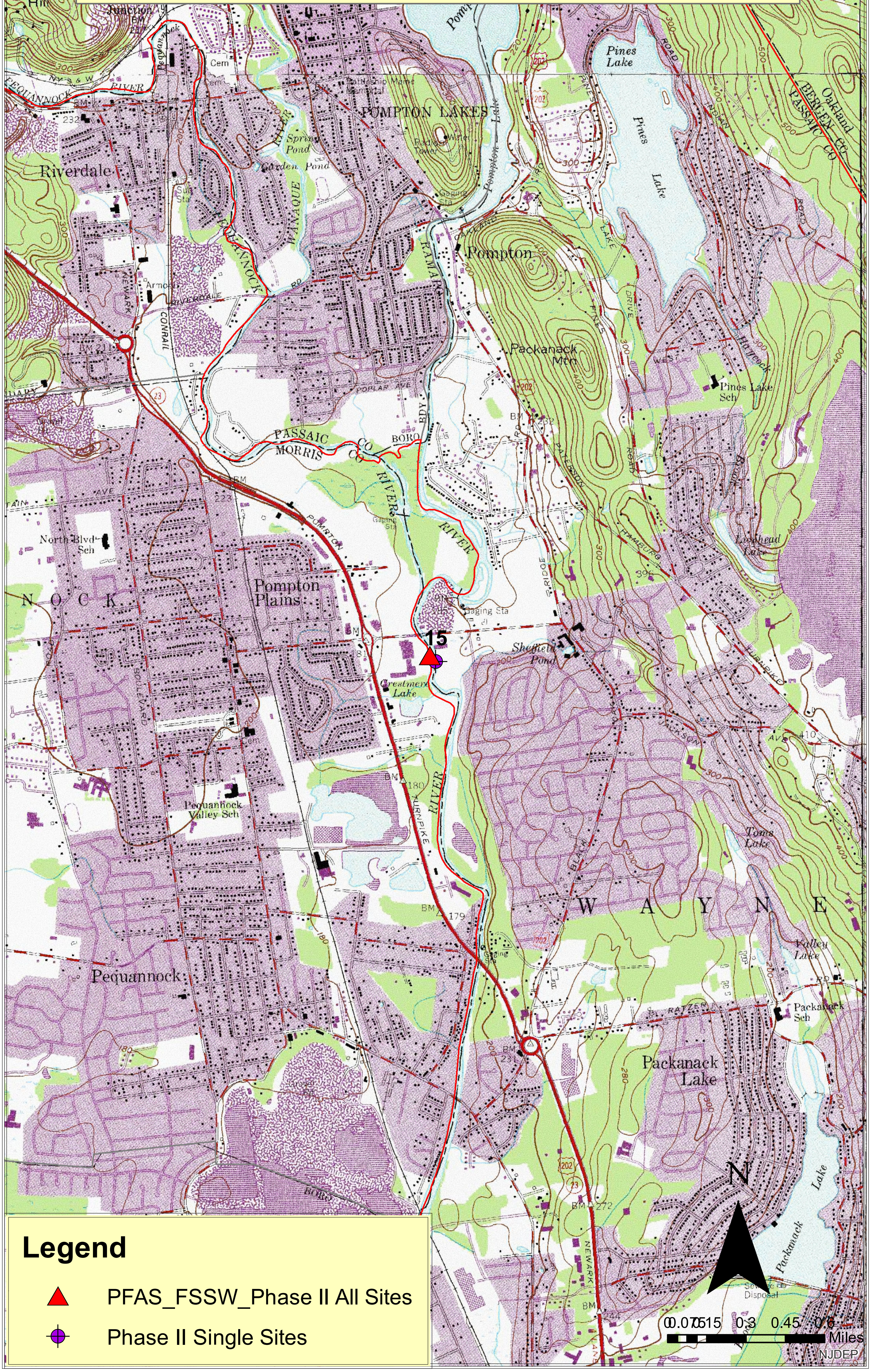
Phase II: Site 15 and single sub-sampling station



Legend

- 🔺 PFAS_FSSW_Phase II All Sites
- ● Phase II Single Sites

4/11/2019 sg

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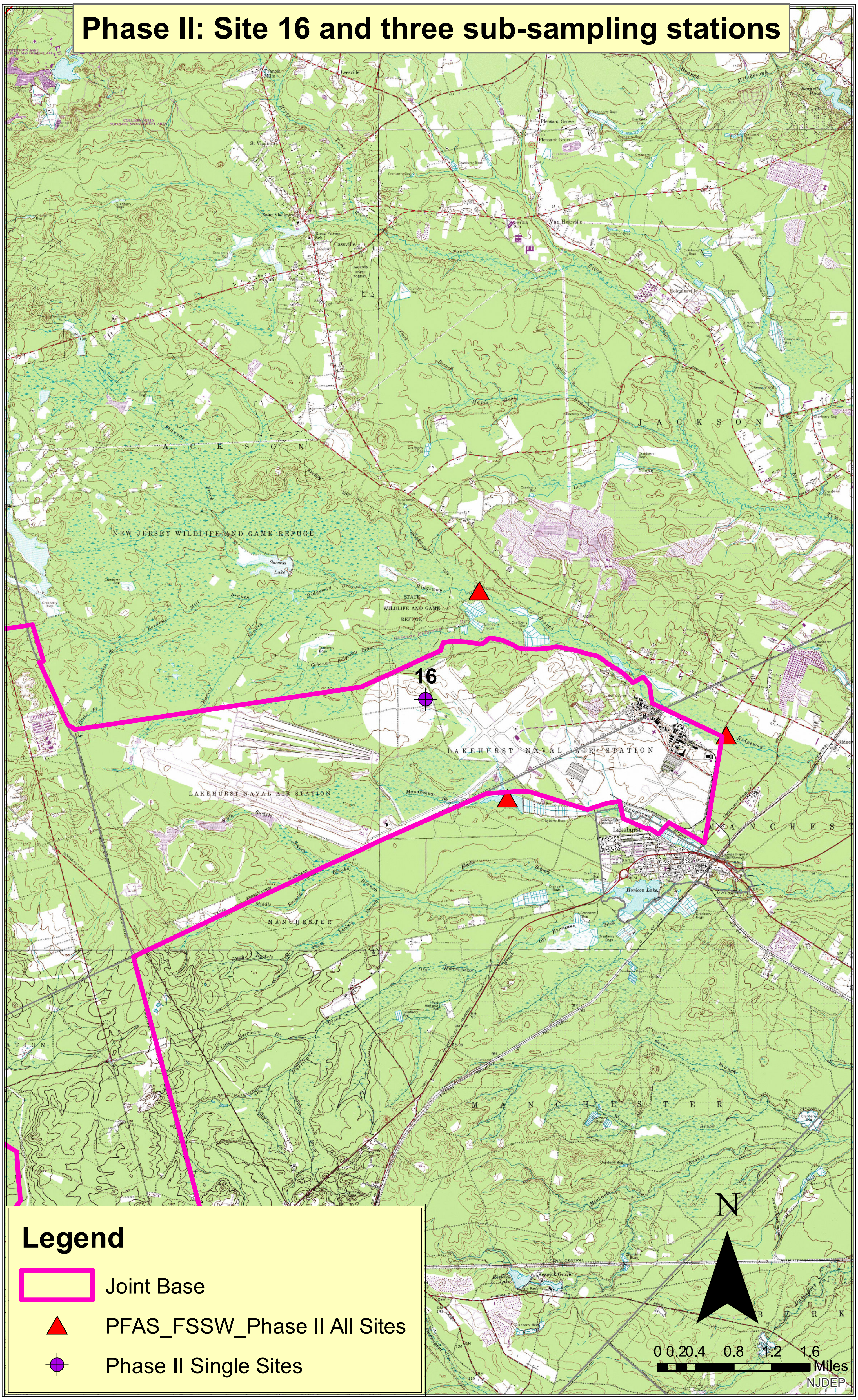
Phase II: Site 16 and three sub-sampling stations



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Appendix B:

SGS Axys PFAS Method Summary

SGS AXYS Analytical Services Ltd.

SUMMARY OF SGS AXYS METHOD MLA-110 REV. 02 VER. 11

(AMENDMENT 2, 25-MAR-2021)

Analytical Procedure for the Analysis of Per- and Polyfluoroalkyl Substances (PFAS) in Aqueous Samples, Solids, Tissues, AFFF Products, Blood/Serums and Solvent Extracts by LC-MS/MS

This method describes the analysis of per- and polyfluoroalkyl substances (PFAS) in aqueous, solid, biosolid, tissue, blood/serum and AFFF product samples and solvent extracts, determined as the total of linear and branched isomers. After spiking with isotopically labeled surrogate standards samples are extracted and cleaned up by Solid Phase Extraction (SPE). The extracts are then analyzed by liquid chromatography/mass spectrometry (LC-MS/MS). Final sample concentrations are determined by isotope dilution/internal standard quantification.

Typical reporting limits, based on the A CAL, are shown below, for the method default sample sizes:

Analyte groups	Aqueous sample	Extract	Solid	Biosolid	Tissues	AFFF Products	Blood, Serum
Typical sample size	0.5 L	0.75 mL	5 g^3	0.5 g^3	2.0 g^3	0.02 g	2 mL
Units	ng/L	ng/mL	ng/g	ng/g	ng/g	ng/g	ng/g^1
Perfluoroalkyl carboxylates							
PFBA	1.6	1.1	0.16	1.6	0.4	40	0.4
PFPeA	0.8	0.53	0.08	0.8	0.2	20	0.2
PFHxA	0.4	0.27	0.04	0.4	0.1	10	0.1
PFHpA	0.4	0.27	0.04	0.4	0.1	10	0.1
PFOA	0.4	0.27	0.04	0.4	0.1	10	0.1
PFNA	0.4	0.27	0.04	0.4	0.1	10	0.1
PFDA	0.4	0.27	0.04	0.4	0.1	10	0.1
PFUnA	0.4	0.27	0.04	0.4	0.1	10	0.1
PFDoA	0.4	0.27	0.04	0.4	0.1	10	0.1
PFTrDA	0.4	0.27	0.04	0.4	0.1	10	0.1
PFTeDA	0.4	0.27	0.04	0.4	0.1	10	0.1
Perfluoroalkyl sulfonates							
PFBS	0.4	0.27	0.04	0.4	0.1	10	0.1
PFPeS	0.4	0.27	0.04	0.4	0.1	10	0.1

Analyte groups	Aqueous sample	Extract	Solid	Biosolid	Tissues	AFFF Products	Blood, Serum
Typical sample size	0.5 L	0.75 mL	5 g[3]	0.5 g[3]	2.0 g[3]	0.02 g	2 mL
Units	ng/L	ng/mL	ng/g	ng/g	ng/g	ng/g	ng/g[1]
PFHxS	0.4	0.27	0.04	0.4	0.1	10	0.1
PFHpS	0.4	0.27	0.04	0.4	0.1	10	0.1
PFOS	0.4	0.27	0.04	0.4	0.1	10	0.1
PFNS	0.4	0.27	0.04	0.4	0.1	10	0.1
PFDS	0.4	0.27	0.04	0.4	0.1	10	0.1
PFDoS	0.4	0.27	0.04	0.4	0.1	10	0.1
Fluorotelomer sulfonates							
4:2 FTS	1.6	1.1	0.16	1.6	0.4	40	0.4
6:2 FTS	1.6	1.1	0.16	1.6	0.4	40	0.4
8:2 FTS	1.6	1.1	0.16	1.6	0.4	40	0.4
Fluorotelomer carboxylates							
3:3 FTCA	1.6	1.1	0.16	1.6	0.4	40	0.4
5:3 FTCA	10	6.7	1	10	2.5	250	2.5
7:3 FTCA	10	6.7	1	10	2.5	250	2.5
Perfluorooctane sulfonamides							
PFOSA	0.4	0.27	0.04	0.4	0.1	10	0.1
N-MeFOSA	0.4	0.27	0.04	0.4	0.1	10	0.1
N-EtFOSA	0.4	0.27	0.04	0.4	0.1	10	0.1
Perfluorooctane sulfonamidoacetic acids							
N-MeFOSAA	0.4	0.27	0.1	0.4	0.1	10	0.1
N-EtFOSAA	0.4	0.27	0.1	0.4	0.1	10	0.1
Perfluorooctane sulfonamide ethanols							
N-MeFOSE	4	2.7	1	4	1[2]	100	1
N-EtFOSE	4	2.7	1	4	1[2]	100	1
Per- and polyfluoroether carboxylates							
HFPO-DA	1.6	1.1	0.16	1.6	0.4	40	0.4
ADONA	1.6	1.1	0.16	1.6	0.4	40	0.4

Analyte groups	Aqueous sample	Extract	Solid	Biosolid	Tissues	AFFF Products	Blood, Serum
Typical sample size	0.5 L	0.75 mL	5 g[3]	0.5 g[3]	2.0 g[3]	0.02 g	2 mL
Units	ng/L	ng/mL	ng/g	ng/g	ng/g	ng/g	ng/g[1]
NFDHA	0.8	0.53	0.08	0.8	0.2	20	0.2
PFMBA	0.8	0.27	1	0.8	0.1	10	0.1
PFMPA	1.6	0.53	2	1.6	0.2	20	0.2
Ether sulfonates							
9Cl-PF3ONS	1.6	1.1	0.16	1.6	0.4	40	0.4
11Cl-PF3OUdS	1.6	1.1	0.16	1.6	0.4	40	0.4
PFEESA	0.4	0.27	0.04	0.4	0.1	10	0.1

[1] Serum reporting unit default is ng/g, but units may be converted to ng/mL upon client request by using serum density.

[2] Low surrogate recoveries in tissues of D_7-N-MeFOSE D_9-N-EtFOSE typically increase the reporting limits for N-MeFOSE and N-EtFOSE by a factor of 5.

[3] Upper weight limits for dry samples. 2 g is the upper weight limit for wet tissue samples

Refer to Appendix B for sample storage conditions.

PFAS Target Analytes

Perfluoroalkyl carboxylates

Perfluorobutanoic acid (PFBA, Perfluorobutanoate)
Perfluoropentanoic acid (PFPeA, Perfluoropentanoate)
Perfluorohexanoic acid (PFHxA, Perfluorohexanoate)
Perfluoroheptanoic acid (PFHpA, Perfluoroheptanoate)
Perfluorooctanoic acid (PFOA, Perfluorooctanoate)
Perfluorononanoic acid (PFNA, Perfluorononanoate)
Perfluorodecanoic acid (PFDA, Perfluorodecanoate)
Perfluoroundecanoic acid (PFUnA, Perfluoroundecanoate)
Perfluorododecanoic acid (PFDoA, Perfluorododecanoate)
Perfluorotridecanoic acid (PFTrDA, Perfluorotridecanoate)
Perfluorotetradecanoic acid (PFTeDA, Perfluorotetradecanoate)

Perfluoroalkyl sulfonates

Perfluorobutanesulfonic acid (PFBS, Perfluorobutanesulfonate)
Perfluoropentanesulfonic acid (PFPeS, Perfluoropentanesulfonate)

Perfluorohexanesulfonic acid (PFHxS, Perfluorohexanesulfonate)

Perfluoroheptanesulfonic acid (PFHpS, Perfluoroheptanesulfonate)

Perfluorooctanesulfonic acid (PFOS, Perfluorooctanesulfonate)

Perfluorononanesulfonic acid (PFNS, Perfluorononanesulfonate)

Perfluorodecanesulfonic acid (PFDS, Perfluorodecanesulfonate)

Perfluorododecanesulfonic acid (PFDoS, Perfluorododecanesulfonate)

Fluorotelomer sulfonates

1H, 1H, 2H, 2H-perfluorohexane sulfonic acid (4:2 FTS, 1H, 1H, 2H, 2H-perfluorohexane sulfonate)

1H, 1H, 2H, 2H-perfluorooctane sulfonic acid (6:2 FTS, 1H, 1H, 2H, 2H-perfluorooctane sulfonate)

1H, 1H, 2H, 2H-perfluorodecane sulfonic acid (8:2 FTS, 1H, 1H, 2H, 2H-perfluorodecane sulfonate)

Fluorotelomer carboxylates

2H, 2H, 3H, 3H-perfluorohexanoic acid (3:3 FTCA, 2H, 2H, 3H, 3H-perfluorohexanoate)

2H, 2H, 3H, 3H-perfluorooctanoic acid (5:3 FTCA, 2H, 2H, 3H, 3H-perfluorooctanoate)

2H, 2H, 3H, 3H-perfluorodecanoic acid (7:3 FTCA, 2H, 2H, 3H, 3H-perfluorodecanoate)

Perfluorooctane sulfonamides

Perfluorooctanesulfonamide (PFOSA) [1]

N-Methylperfluorooctanesulfonamide (N-MeFOSA)

N-Ethylperfluorooctanesulfonamide (N-EtFOSA)

Perfluorooctane sulfonamidoacetic acids

N-Methylperfluoro-1-octanesulfonamidoacetic acid (N-MeFOSAA, N-Methylperfluoro-1-octanesulfonamidoacetate)

N-Ethylperfluoro-1-octanesulfonamidoacetic acid (N-EtFOSAA, N-Ethylperfluoro-1-octanesulfonamidoacetate)

Perfluorooctane sulfonamidoethanols

N-Methylperfluoro-1-octanesulfonamidoethanol (N-MeFOSE)

N-Ethylperfluoro-1-octanesulfonamidoethanol (N-EtFOSE)

Ether carboxylates

2,3,3,3-Tetrafluoro-2-(1,1,2,2,3,3,3-heptafluoropropoxy)propionic acid (HFPO-DA, 2,3,3,3-Tetrafluoro-2-(1,1,2,2,3,3,3-heptafluoropropoxy)propionoate)

Decafluoro-3H-4,8-dioxanonoate (ADONA, DONA, Decafluoro-3H-4,8-dioxanonoic acid)

Perfluoro-3,6-dioxaheptanoate (NFDHA, Perfluoro-3,6-dioxaheptanoic acid)

Perfluoro-3-methoxypropanoate (PFMPA, Perfluoro-3-methoxypropanoic acid)

Perfluoro-4-methoxybutanoate (PFMBA, Perfluoro-4-methoxybutanoic acid)

Ether sulfonates

9-chlorohexadecafluoro-3-oxanonane-1-sulfonic acid (9Cl-PF3ONS, 9-chlorohexadecafluoro-3-oxanonane-1-sulfonate)

11-chloroeicosafluoro-3-oxaundecane-1-sulfonic acid (11Cl-PF3OUdS, 11-chloroeicosafluoro-3-oxaundecane-1-sulfonate)
Perfluoro(2-ethoxyethane)sulfonic acid (PFEESA, Perfluoro(2-ethoxyethane)sulfonate

[1] PFOSA also called FOSA

The carboxylic and sulfonic acid analyte concentrations can be reported as either the acid or the anion forms. The anion and corresponding acid forms and their CAS Registry Numbers are shown in Appendix A of this summary.

1.0 EXTRACTION AND CLEANUP PROCEDURES

Aqueous samples size may be up to 1000 mL for aqueous samples analyzed by this method, and up to 0.75 mL for extracts/solvents. Leachate samples are typically 120 mL. The OPR and BLK (reagent water) must be of the same volume as a typical sample in the batch. Samples are stored in HDPE (high density polyethylene) containers. All samples are spiked with surrogate standards. Aqueous samples are extracted by solid phase extraction (SPE) using weak anion exchange cartridges; wash and elution procedures are chosen to meet various analysis requirements. Sample extracts are then treated with carbon powder, spiked with recovery standards and analyzed by LC-MS/MS. For aqueous samples that clog the SPE cartridge during loading, the project manager may approve matters related to change in sample size and the spiking of MS/MSD samples. A second, non-standard procedure may be used to filter samples using the entire sample bottle. This option measures dissolved PFAS. It does not include PFAS bound to particulate. It does not give equivalent results to the standard procedure above.

Extract/solvent samples don't undergo solid phase extraction. The samples are spiked with surrogate and recovery standards, and analyzed by LC-MS/MS.

The sample sizes may be up to 5 g dry weight or up to 10 g wet weight for solid samples and up to 0.5 g dry weight or up to 5 g wet weight for biosolid samples. After addition of isotopically labelled surrogate standards the sample is extracted by shaking three times with methanolic ammonium hydroxide solution, each time collecting the supernatants. The supernatants are combined, treated with ultra pure carbon powder and evaporated to remove methanol. The resulting solution is diluted with water and cleaned up by solid phase extraction (SPE) using disposable cartridges containing a weak anion exchange sorbent. The eluate is spiked with recovery standards and analyzed by LC-MS/MS.

Tissue sample size may be up to 2 g wet weight. After addition of isotopically labelled surrogate standards the sample is extracted with methanolic potassium hydroxide solution, with acetonitrile, and finally with methanolic potassium hydroxide solution, each time collecting the supernatants. The supernatants are combined, treated with ultra pure carbon powder and evaporated to remove methanol. The resulting solution is diluted with water and cleaned up by solid phase extraction (SPE) on a weak anion exchange sorbent. The eluate is spiked with recovery standards and analyzed by LC-MS/MS.

All AFFF samples are pre-screened before analysis to determine the appropriate amount of sample to analyze. A suitable subsample is dissolved in water, spiked with surrogate standards and extracted by solid phase extraction (SPE). The extracts are treated with carbon powder, spiked with recovery standards and analyzed by LC-MS/MS.

Blood/Serum sample size may be up to 2 mL. After the addition of isotopically labelled surrogate standards the sample is extracted with 50% formic acid. The resulting solution is cleaned up by solid phase extraction (SPE) on a weak anion exchange sorbent. The eluent tubes are spiked with recovery standards and then eluent collected and analyzed by LC-MS/MS.

2.0 INSTRUMENTATION

Analysis of the sample extract is performed on a UPLC-MS/MS (ultrahigh performance liquid chromatography) reversed phase C18 column using a solvent gradient. The column is coupled to a triple quadrupole mass spectrometer run at unit mass resolution in the Multiple Reaction Monitoring (MRM) in negative electrospray ionization mode.

3.0 CALIBRATION

Initial calibration of the UPLC-MS/MS instrument is performed by the analysis of five or more calibration solutions. A mid-level calibration standard is analyzed to verify the initial calibration and injected after:

- at least every 12 hours.
- For DoD accredited work after every 10 client samples or every 12 hours, whichever occurs first, and at the end of the instrumental run sequence.

List of Surrogate and Recovery Standards

Surrogate Standards	Recovery Standards
$^{13}C_4$-PFBA	$^{13}C_3$-PFBA
$^{13}C_5$-PFPeA	$^{13}C_2$-PFHxA
$^{13}C_5$-PFHxA	$^{13}C_4$-PFOA
$^{13}C_4$-PFHpA	$^{13}C_5$-PFNA
$^{13}C_8$-PFOA	$^{13}C_2$-PFDA
$^{13}C_9$-PFNA	$^{18}O_2$-PFHxS
$^{13}C_6$-PFDA	$^{13}C_4$-PFOS
$^{13}C_7$-PFUnA	
$^{13}C_2$-PFDoA	
$^{13}C_2$-PFTeDA	
$^{13}C_3$-PFBS	
$^{13}C_3$-PFHxS	
$^{13}C_8$-PFOS	
$^{13}C_2$-4:2 FTS	

$^{13}C_2$-6:2 FTS $^{13}C_2$-8:2 FTS	
$^{13}C_8$-PFOSA	
D_3-N-MeFOSA D_5-N-EtFOSA	
D_3-N-MeFOSAA D_5-N-EtFOSAA	
D_7-N-MeFOSE D_9-N-EtFOSE	
$^{13}C_3$-HFPO-DA	

4.0 QUANTIFICATION PROCEDURES

4.1 Calculations

Target compounds are quantified using the isotope dilution/internal standard method, comparing the area of the primary (quantifying) transition product ion of the target compound to that of the ^{13}C-labelled or deuterium labeled standard and correcting for response factors. Mean relative response factors (RRF), determined from the multi-level initial calibration series are used to convert raw peak areas in sample chromatograms to final concentrations as follows:

$$\text{Concentration of Target} = \left(\frac{\text{area of Target}}{\text{area of Qt Std}}\right) \times \left(\frac{\text{weight of Qt Std}}{\text{RRF}}\right) \times \left(\frac{1}{\text{weight of sample}}\right)$$

where $\text{RRF} = \left(\frac{\text{area of Target}}{\text{area of Qt Std}}\right) \times \left(\frac{\text{weight of Qt Std}}{\text{weight of Target}}\right)$

and the Qt Std is either the surrogate or the internal standard

The isotopically labeled analog of an analyte (surrogate) is used for quantitation (Isotope Dilution Quantitation). If a labeled analog is not commercially available, a surrogate with chemical similarity and close retention time is used for quantitation (internal standard quantitation). Final target concentrations are recovery corrected by this method of quantification.

4.2 Reporting Limits

Sample Specific Detection Limits (SDL) are determined by converting the area equivalent to 3.0 times the estimated chromatographic noise height to a concentration in the same manner that target peak responses are converted to final concentrations. The SDL accounts for any effect of matrix on the detection system and for recovery achieved through the analytical work-up.

Results are reported to the greater of the SDL or the concentration equivalent to CAL A. The lowest method calibration limit (LMCL) is CAL B. Results below the LOQ are J flagged.

Analytes and Quantification References

Target analytes	Quantified using
PFBA	$^{13}C_4$-PFBA
PFPeA	$^{13}C_5$-PFPeA
PFHxA	$^{13}C_5$-PFHxA
PFHpA	$^{13}C_4$-PFHpA
PFOA	$^{13}C_8$-PFOA
PFNA	$^{13}C_9$-PFNA
PFDA	$^{13}C_6$-PFDA
PFUnA	$^{13}C_7$-PFUnA
PFDoA	$^{13}C_2$-PFDoA
PFTrDA	avg. $^{13}C_2$-PFTeA and $^{13}C_2$-PFDoA
PFTeDA	$^{13}C_2$-PFTeDA
PFBS	$^{13}C_3$-PFBS [1]
PFPeS	$^{13}C_3$-PFHxS
PFHxS	$^{13}C_3$-PFHxS
PFHpS	$^{13}C_8$-PFOS
PFOS	$^{13}C_8$-PFOS
PFNS	$^{13}C_8$-PFOS
PFDS	$^{13}C_8$-PFOS
PFDoS	$^{13}C_8$-PFOS
4:2 FTS	$^{13}C_2$-4:2 FTS
6:2 FTS	$^{13}C_2$-6:2 FTS
8:2 FTS	$^{13}C_2$-8:2 FTS
PFOSA	$^{13}C_8$-PFOSA
N-MeFOSA	D_3-N-MeFOSA
N-EtFOSA	D_5-N-EtFOSA
N-MeFOSAA	D_3-N-MeFOSAA
N-EtFOSAA	D_5-N-EtFOSAA
N-MeFOSE	D_7-N-MeFOSE
N-EtFOSE	D_9-N-EtFOSE
HFPO-DA	$^{13}C_{-3}$-HFPO-DA
ADONA	$^{13}C_{-3}$-HFPO-DA
9Cl-PF3ONS	$^{13}C_{-3}$-HFPO-DA
11Cl-PF3OUdS	$^{13}C_{-3}$-HFPO-DA
3:3 FTCA	$^{13}C_5$-PFPeA
5:3 FTCA	$^{13}C_5$-PFHxA

Target analytes	Quantified using
7:3 FTCA	$^{13}C_5$-PFHxA
PFMPA	$^{13}C_5$-PFPeA
PFMBA	$^{13}C_5$-PFPeA
NFDHA	$^{13}C_5$-PFHxA
PFEESA	$^{13}C_5$-PFHxA

[1] If the quantifying standard $^{13}C_3$-PFBS is unavailable PFBS may optionally be quantified against $^{13}C_3$-PFHxS.

Surrogate standards	Quantified using
$^{13}C_4$-PFBA	$^{13}C_3$-PFBA
$^{13}C_5$-PFPeA	$^{13}C_2$-PFHxA
$^{13}C_5$-PFHxA	$^{13}C_2$-PFHxA
$^{13}C_4$-PFHpA	$^{13}C_4$-PFOA ($^{13}C_2$-PFHxA for AFFF products)
$^{13}C_8$-PFOA	$^{13}C_4$-PFOA
$^{13}C_9$-PFNA	$^{13}C_5$-PFNA
$^{13}C_6$-PFDA	$^{13}C_2$-PFDA
$^{13}C_7$-PFUnA	$^{13}C_2$-PFDA
$^{13}C_2$-PFDoA	$^{13}C_2$-PFDA
$^{13}C_2$-PFTeDA	$^{13}C_2$-PFDA
$^{13}C_3$-PFBS	$^{18}O_2$-PFHxS
$^{13}C_3$-PFHxS	$^{18}O_2$-PFHxS
$^{13}C_8$-PFOS	$^{13}C_4$-PFOS
$^{13}C_2$-4:2 FTS	$^{18}O_2$-PFHxS
$^{13}C_2$-6:2 FTS	$^{18}O_2$-PFHxS
$^{13}C_2$-8:2 FTS	$^{18}O_2$-PFHxS
$^{13}C_8$-PFOSA	$^{13}C_4$-PFOS
D_3-N-MeFOSA	$^{13}C_4$-PFOS
D_5-N-EtFOSA	$^{13}C_4$-PFOS
D_3-N-MeFOSAA	$^{13}C_4$-PFOS
D_5-N-EtFOSAA	$^{13}C_4$-PFOS
D_7-N-MeFOSE	$^{13}C_4$-PFOS
D_9-N-EtFOSE	$^{13}C_4$-PFOS
$^{13}C_3$-HFPO-DA	$^{13}C_2$-PFHxA
Recovery standards	**Quantified using**
$^{13}C_3$-PFBA	External

$^{13}C_2$-PFHxA	External
$^{13}C_4$-PFOA	External
$^{13}C_5$-PFNA	External
$^{13}C_2$-PFDA	External
$^{18}O_2$-PFHxS	External
$^{13}C_4$-PFOS	External

5.0 QUALITY ACCEPTANCE CRITERIA

Samples are analyzed in batches consisting of a maximum of twenty samples, one procedural blank and one spiked matrix (OPR) sample. A duplicate is analyzed, provided there is sufficient sample, with batches containing 7-20 samples. Matrix spike/matrix spike duplicate (MS/MSD) pairs may be analyzed on an individual contract basis. The batch is carried through the complete analytical process as a unit. For sample data to be reportable, the batch QC data must meet the established acceptance criteria presented on the analysis reports.

QC Specification Table for Aqueous, Solid, AFFF and Tissue Samples: Procedural Blank Levels and OPR Recovery Ranges, includes DoD QSM Recovery Ranges

Compound	Procedural Blank Level (ng/sample)	OPR Recovery Range for Aqueous, Solid and AFFF Samples (%)	OPR Recovery Range for Tissue Samples (%)	OPR Recovery Range for Blood/ Serum Samples (%)
PFBA	≤ 1.6	73-129	70-130	70-130
PFPeA	≤ 0.8	72-129	70-130	70-130
PFHxA	≤ 0.4	72-129	70-130	70-130
PFHpA	≤ 0.4	72-130	70-130	70-130
PFOA	≤ 0.4	71-130	70-130	70-130
PFNA	≤ 0.4	72-129	70-130	70-130
PFDA	≤ 0.4	71-129	60-130	70-150
PFUnA	≤ 0.4	70-130	70-140	70-140
PFDoA	≤ 0.4	72-130	70-130	70-140
PFTrDA	≤ 0.4	65-130	70-130	70-130
PFTeDA	≤ 0.4	71-130	70-130	70-130
PFBS	≤ 0.4	72-128	60-130	70-130
PFPeS	≤ 0.4	73-127	70-130	70-130
PFHxS	≤ 0.4	70-130	70-130	70-130
PFHpS	≤ 0.4	70-130	70-130	70-130
PFOS	≤ 0.4	70-130	70-140	70-130

PFNS	≤ 0.4	70-127	60-150	40-140
PFDS	≤ 0.4	70-130	40-150	70-130
PFDoS	≤ 0.4	60-130	70-140	70-130
4:2 FTS	≤ 1.6	70-130	40-150	70-130
6:2 FTS	≤ 5	70-130	70-130	70-130
8:2 FTS	≤ 1.6	70-130	70-170	70-130
PFOSA	≤ 0.4	70-130	70-130	70-130
N-MeFOSA	≤ 0.4	70-130	50-140	70-130
N-EtFOSA	≤ 0.4	70-130	70-130	70-130
N-MeFOSAA	≤ 0.4	70-130	60-140	70-150
N-EtFOSAA	≤ 0.4	70-130	60-140	60-130
N-MeFOSE	≤ 4	70-130	70-150	70-130
N-EtFOSE	≤ 4	70-130	70-130	70-160
HFPO-DA	≤ 1.6	70-130	70-130	70-130
ADONA	≤ 1.6	70-130	70-130	70-130
9Cl-PF3ONS	≤1.6	70-130	70-130	60-130
11Cl-PF3OUdS	≤ 1.6	70-130	60-130	70-130
3:3 FTCA	≤ 1.6	65-130	70-130	n.a.
5:3 FTCA	≤ 10	70-130	70-180	n.a.
7:3 FTCA	≤ 10	70-130	70-130	n.a.
PFMPA	≤ 0.8	70-130	70-130	n.a.
PFMBA	≤ 0.4	70-130	70-130	n.a.
NFDHA	≤ 0.8	65-140	60-130	n.a.
PFEESA	≤ 0.4	70-130	70-130	n.a.

Marginal exceedance allowance – results for one compound may fall outside of these limits by a maximum of 10% of the value. Note that for AFFF products, these are interim specifications and data outside the specifications may be acceptable based on application and professional judgment. DoD specification for procedural blanks: No analytes detected >½ LOQ or > 1/10th the amount measured in any sample or 1/10th the regulatory limit, whichever is greater.

QC Specifications for Aqueous, Solid, AFFF, Tissue and Blood/Serum Samples: Surrogate Standard Recoveries, OPR and Samples

Surrogate Standard	OPR and Sample Recovery Range [1] for Aqueous, Solid and AFFF Samples (%)	OPR and Sample Recovery Range [1] for Tissue Samples (%)	OPR and Sample Recovery Range [1] for Blood/Serum Samples (%)
$^{13}C_4$-PFBA	50-150	50-150	50-150
$^{13}C_5$-PFPeA	50-150	50-150	50-150
$^{13}C_5$-PFHxA	50-150	50-150	50-150

$^{13}C_4$-PFHpA	50-150	50-150	50-150
$^{13}C_8$-PFOA	50-150	50-150	50-150
$^{13}C_9$-PFNA	50-150	50-150	50-150
$^{13}C_6$-PFDA	50-150	50-180	50-150
$^{13}C_7$-PFUnA	50-150	50-150	50-160
$^{13}C_2$-PFDoA	50-150	50-150	50-150
$^{13}C_2$-PFTeDA	50-150	50-150	40-150
$^{13}C_3$-PFBS	50-150	50-150	50-150
$^{13}C_3$-PFHxS	50-150	50-150	50-150
$^{13}C_8$-PFOS	50-150	50-150	50-150
$^{13}C_2$-4:2 FTS	50-150	50-220	50-150
$^{13}C_2$-6:2 FTS	50-150	50-180	30-180
$^{13}C_2$-8:2 FTS	50-150	50-300	30-180
$^{13}C_8$-PFOSA	50-150	50-150	50-150
D_3-N-MeFOSA	30-150	5-150[2]	50-150
D_5-N-EtFOSA	20-150	10-150[2]	50-150
D_3-N-MeFOSAA	50-150	50-180[2]	40-200
D_5-N-EtFOSAA	50-150	50-250[2]	40-200
D_7-N-MeFOSE	30-150	2-150[2]	50-150
D_9-N-EtFOSE	30-150	2-150[2]	30-150
$^{13}C_3$-HFPO-DA	50-150	50-150	50-150

[1] Lower surrogate recoveries may be accepted based on application and professional judgment. Note that for AFFF products, these are interim specifications and data outside the specifications may be acceptable based on application and professional judgment.

[2] These surrogates used only to quantify the analogous native compounds. Surrogate recoveries that fall within method specifications are considered reliable. Surrogate recoveries falling outside of method specifications may result in the qualification of analyte data, if surrogate recoveries are below 10% or in analyte data being non-quantifiable if below 1%. Professional judgement applies in all cases.

QC Specification Table: Other Parameters

QC Parameter	**Specification**[1]
MS Acquisition Rate	Minimum acquisition rate for every native analyte and labeled compound peak: At least 10 data points per peak.
Mass Calibration	Instrument must have a valid mass calibration following the manufacturer specified procedure prior to any sample analysis. The mass calibration is updated on an as-needed basis (e.g. QC failures, ion masses fall outside of the ±0.5 amu of the true value, major instrument maintenance, or if the instrument is moved.) Refer to SIN-033. The entire range (bracketing all the masses of the target analytes) must be mass calibrated. The maximal allowed residual error is ≤ 0.1 Da for each mode with no more than two calibration points missed.
Mass Calibration Verification	Mass calibration must be verified after each mass calibration, prior to any sample analysis. Mass calibration must be performed per the instrument manufacturer's instructions. A mass calibration verification must be performed using standards whose mass range brackets the masses of interest (quantitative and qualitative ions).
Initial Calibration (I-CAL)	Run initially, and as required to maintain compliance with calibration verification and instrument sensitivity. The isotopically labeled analog of an analyte (surrogate standard) must be used for quantitation if commercially available (Isotope Dilution Quantitation). If a labeled analog is not commercially available, the surrogate standard with the closest retention time or chemical similarity to the native standard must be used for quantitation. Quantification is achieved by the constant RRF method. The I-CAL specifications (CAL B to CAL J) for the RRF are <20% RSD of mean RRFs and 70-130% recovery of analytes and surrogates at each concentration level from Cal C and above. CAL B to CAL J must meet a 3:1 S/N specification in the quantification ion and a 3:1 S/N in the confirmation ion. The A CAL (sensitivity CAL) must achieve 3:1 S/N for the quantification ion. Peak Asymmetry, SGS AXYS guidance: 0.8-1.5 for PFBA and PFPeA measured in CAL E (mid cal point) at 10% of the peak height. If this is not achieved, perform instrument maintenance and re-run I-CAL.
Initial Calibration Verification (ICV):	After each Initial Calibration (I-CAL) and prior to sample analysis; analyze a second source standard (similar concentration to the CAL E); quantify against I-Cal, results must meet Cal/Ver accuracy specifications of 70% to 130%.

Retention Time (RT) window	Relative retention times (RRT) for linear and branched isomers vs, the surrogates are determined from the qualitative standard run every 12 hours (see Table 12). Maximum RRTs windows are 0.1 minutes.
Surrogate Standards	Must be added to every field sample, standard, blank, and QC sample. Recoveries of the surrogate standard analytes (EPA term for Surrogate Standard is Extracted Internal Standard-EIS; EPA term for Recovery Standard is Injection Internal Standard-IIS) are calculated by internal standard quantification against the IIS using an average RRF. Recovery criteria for surrogate standard analytes in instrument blanks and standards is 70% to 130%. Recovery criteria for surrogate standard in field samples and preparatory QC samples are listed in Table 7.
Recovery Standards	Must be added to every prepared field sample, standard, blank, and QC sample prior to instrumental analysis. Recovery standard analyte recovery is calculated by external standards with an SGS AXYS specification of 50% to 200%. Professional judgement applies.
Calibration Verification (Cal/Ver or CCV)	CAL E. Prior to sample analysis and at the end of the analytical sequence, or every 12 hours. Quantify against I-CAL. Native standard analyte and surrogate standard concentrations must be within ±30% of their true value. *Additional requirement for DoD: Run Cal/Ver every 10 client samples or every 12 hours, whichever occurs first, and at the end of the analytical sequence; quantify against I-CAL*. For internal purposes monitor Peak Asymmetry for every Cal/Ver
Instrument Sensitivity Check (ISC)	Run every 12 hours. CAL A – S:N ≥ 3:1 for quantification ion. Cal C- S:N ≥ 3:1 for quantification and confirmation ion. Native compound recoveries 70-130%.
Instrument Background	For DoD work an instrument blank containing surrogates is run immediately after every Initial Calibration (highest standard) and Calibration Verification and daily thereafter. The concentration of each analyte in the instrument blank must be ≤ ½ of the LOQ (C CAL). In this case (assuming Cal J would fail this test) the instrument blank can be run after CAL I and before the CAL J. Cal J is not included in the ICAL for DoD samples but can be included for other work. If any sample concentrations exceed the highest allowed standard and the sample(s) following exceed this acceptance criteria (>½ LOQ), they must be reanalyzed.
Instrument Carryover	The specification is ≤ 0.3 % carryover from the Cal Ver standard into following instrument blank or from any sample into the following injection.
Duplicate Samples	If conc. ≥ 5 times R.L., RPD ≤ 40% If conc. < 5 times R.L., guideline RPD ≤ 100%

Ongoing Precision and Recovery (OPR or LCS)	Ongoing Precision and Recovery (OPR) or Laboratory Control Sample (LCS) is CAL E. Refer to Table 6 for native analytes for OPRs and Table 7 for OPR surrogate acceptance limits.
Method Blank (MB)	One per preparatory batch. No analyte can be detected > ½ LOQ or >1/10th the amount measured in field samples in the batch, whichever is greater.
Limit of Quantitation (LOQ) (or EPA LLOQ) Verification	Run quarterly for each method/matrix/analyte combination on each instrument used for sample analysis during that quarter. Native standard concentration must be spiked at concentrations equivalent to the LOQ. Recovery criteria for native standards in the LOQ/LLOQ are set at 70-130%. Ion ratios must be within 50-150% (except PFNS which is 50-200%) of the ratios determined from I-CAL CAL E. *Additional requirement for DoD: run LOQ Verification with every batch of samples. The LOQ verification must be run at a concentration between 1 and two times the LOQ (CAL C).*
MS/MSD (required for DOD, optional for others)	Native standard concentration must be spiked at concentrations ≥ LOQ and ≤ the mid-level calibration concentration. If an MS/MSD cannot be used, an OPR duplicate must be run instead. This is optional for non-DoD work. *Additional requirement for DoD: For all matrices analyzed by SPE one Matrix Spike and one Matrix Spike Duplicate shall be included with every analysis batch. MS/MSD recoveries are evaluated against project limits if prescribed by the client, otherwise MS/MSD recoveries are evaluated against the DOD specific acceptance ranges for OPRs listed in Table 6 of this document, or against the MLA-110 OPR method recovery limits for analytes not listed in Table 6. RPDs are evaluated against project limits if prescribed by the client, otherwise RPDs are evaluated against the DoD specific limit of ≤30%. Ion ratios must be within 50-150% (except PFNS which is 50-200%) of the ratios determined from I-CAL CAL E.*

[1] Corrective actions stated in the current version of DoD QSM, Appendix B, Table B-15 must be utilized when QC parameter fails to meet the specification

APPENDIX A: NAMING CONVENTION AND CAS NUMBERS

PFAS: Naming Convention and CAS Numbers

Abbreviation	***Name - Anion Form***	***CAS#***
PFBA	Perfluorobutanoate	45048-62-2
PFPeA	Perfluoropentanoate	45167-47-3
PFHxA	Perfluorohexanoate	92612-52-7
PFHpA	Perfluoroheptanoate	120885-29-2
PFOA	Perfluorooctanoate	45285-51-6
PFNA	Perfluorononanoate	72007-68-2
PFDA	Perfluorodecanoate	73829-36-4
PFUnA	Perfluoroundecanoate	196859-54-8
PFDoA	Perfluorododecanoate	171978-95-3
PFTrDA	Perfluorotridecanoate	862374-87-6
PFTeDA	Perfluorotetradecanoate	365971-87-5
PFBS	Perfluorobutanesulfonate	45187-15-3
PFPeS	Perfluoropentanesulfonate	175905-36-9
PFHxS	Perfluorohexanesulfonate	108427-53-8
PFHpS	Perfluoroheptanesulfonate	146689-46-5
PFOS	Perfluorooctanesulfonate	45298-90-6
PFNS	Perfluorononanesulfonate	474511-07-4
PFDS	Perfluorodecanesulfonate	126105-34-8
PFDoS	Perfluorododecanesulfonate	343629-43-6
4:2 FTS	4:2 fluorotelomersulfonate	414911-30-1
6:2 FTS	6:2 fluorotelomersulfonate	425670-75-3
8:2 FTS	8:2 fluorotelomersulfonate	481071-78-7
3:3 FTCA	3:3 perfluorohexanoate	1169706-83-5
5:3 FTCA	5:3 perfluoroctanoate	1799325-94-2
7:3 FTCA	7:3 perfluorodecanoate	n.a.
N-MeFOSAA	N-Methylperfluorooctanesulfonamidoacetate	n.a.
N-EtFOSAA	N-Ethylperfluorooctanesulfonamidoacetate	n.a.
HFPO-DA	2,3,3,3-Tetrafluoro-2-(1,1,2,2,3,3,3-heptafluoropropoxy)propanoate	122499-17-6
ADONA	Dodecafluoro-3H-4,8-dioxanonanoate	2127366-90-7

PFAS: Naming Convention and CAS Numbers

9Cl-PF3ONS	9-chlorohexadecafluoro-3-oxanonane-1-sulfonate	1621485-21-9
11Cl-PF3OUdS	11-chloroeicosafluoro-3-oxaundecane-1-sulfonate	2196242-82-5
NFDHA	Perfluoro-3,6-dioxaheptanoate	39187-41-2
PFMPA	Perfluoro-3-methoxypropanoate	n.a.
PFMBA	Perfluoro-4-methoxybutanoate	n.a.
PFEESA	Perfluoro(2-ethoxyethane)sulfonate	n.a.

Abbreviation	***Name - Acid Form***	***CAS#***
PFBA	Perfluorobutyric acid	375-22-4
PFPeA	Perfluoropentanoic acid	2706-90-3
PFHxA	Perfluorohexanoic acid	307-24-4
PFHpA	Perfluoroheptanoic acid	375-85-9
PFOA	Perfluorooctanoic acid	335-67-1
PFNA	Perfluorononanoic acid	375-95-1
PFDA	Perfluorodecanoic acid	335-76-2
PFUnA	Perfluoroundecanoic acid	2058-94-8
PFDoA	Perfluorododecanoic acid	307-55-1
PFTrDA	Perfluorotridecanoic acid	72629-94-8
PFTeDA	Perfluorotetradecanoic acid	376-06-7
PFBS	Perfluorobutanesulfonic acid	375-73-5
PFPeS	Perfluoropentanesulfonic acid	2706-91-4
PFHxS	Perfluorohexanesulfonic acid	355-46-4
PFHpS	Perfluoroheptanesulfonic acid	375-92-8
PFOS	Perfluorooctanesulfonic acid	1763-23-1
PFNS	Perfluorononanesulfonic acid	68259-12-1
PFDS	Perfluorodecanesulfonic acid	335-77-3
PFDoS	Perfluorododecanesulfonic acid	79780-39-5
4:2 FTS	4:2 fluorotelomersulfonic acid	757124-72-4
6:2 FTS	6:2 fluorotelomersulfonic acid	27619-97-2
8:2 FTS	8:2 fluorotelomersulfonic acid	39108-34-4
3:3 FTCA	3:3 perfluorohexanoic acid	356-02-5
5:3 FTCA	5:3 perfluorooctanoic acid	914637-49-3

PFAS: Naming Convention and CAS Numbers

7:3 FTCA	7:3 perfluorodecanoic acid	812-70-4
N-MeFOSAA	N-Methylperfluorooctanesulfonamidoacetic acid	2355-31-9
N-EtFOSAA	N-Ethylperfluorooctanesulfonamidoacetic acid	2991-50-6
HFPO-DA	2,3,3,3-Tetrafluoro-2-(1,1,2,2,3,3,3-heptafluoropropoxy)propanoic acid	13252-13-6
ADONA	Dodecafluoro-3H-4,8-dioxanonanoic acid	919005-14-4
9Cl-PF3ONS	9-chlorohexadecafluoro-3-oxanonane-1-sulfonic acid	756426-58-1
11Cl-PF3OUdS	11-chloroeicosafluoro-3-oxaundecane-1-sulfonic acid	763051-92-9
NFDHA	Perfluoro-3,6-dioxaheptanoic acid	151722-58-6
PFMPA	Perfluoro-3-methoxypropanoic acid	377-73-1
PFMBA	Perfluoro-4-methoxybutanoic acid	863090-89-5
PFEESA	Perfluoro(2-ethoxyethane)sulfonic acid	113507-82-7
Abbreviation	***Name - Sulfonamide***	***CAS#***
PFOSA	Perfluorooctanesulfonamide	754-91-6
N-MeFOSA	N-Methylperfluorooctanesulfonamide	31506-32-8
N-EtFOSA	N-Ethylperfluorooctanesulfonamide	4151-50-2
Abbreviation	***Name - Sulfonamidoethanol***	***CAS#***
N-MeFOSE	N-Methylperfluorooctanesulfonamidoethanol	24448-09-7
N-EtFOSE	N-Ethylperfluorooctanesulfonamidoethanol	1691-99-2

APPENDIX B: SAMPLE STORAGE CONDITIONS

Sample Storage Requirements

Matrix	Sample Size (per analysis)	Sample Container [1]	Sample Condition Upon Receipt	Storage Condition [2]	Sample Hold Time [3]	Extract Hold Time [4]	Preservation
Aqueous	Up to 1000 mL, but typically 500 mL or less (max. 50 mg solids) Leachate samples are 100-120 mL	High density polyethylene (HDPE)	0-4°C, dark	≤ -20ºC, dark	90 days	30 days	None Required
Solvent extracts[7]	Typically 0.75 mL	High density polyethylene (HDPE)	0-4°C, dark	0-4°C, dark	60 days	30 days	None Required
Solid[8]	Up to 5 g dry but not more than 10 g wet.	High density polyethylene (HDPE)	0-4°C, dark	≤ -20ºC, dark	1 Year	30 days	None required
Biosolid[9]	Up to 0.5 g dry but not more than 5 g wet.	High density polyethylene (HDPE)	0-4°C, dark	≤ -20ºC, dark	1 Year	30 days	None required
Tissue[10]	Up to 2 g (wet)	High density polyethylene (HDPE) or amber glass jar	0-4°C, dark	≤ -20ºC, dark	1 Year	30 days	None Required
AFFF	Up to 0.02 g	High density polyethylene (HDPE)	Room temperature.	0-4°C, dark	90 days	30 days	None Required
Whole Blood [5] or Serum[6]	Up to 2 mL	HDPE or Glass, not polypropylene.	0-4°C, dark	≤ -20ºC, dark	1 Year	30 days	None Required

[1] HDPE containers are preferred; amber glass containers are also acceptable. All containers should be organically clean; i.e. solvent-rinsed or purchased 'certified' clean. All containers should be tightly sealed with screw cap lids.

[2] Storage temperatures quoted are nominal temperatures.

[3] Hold times are from time of sampling. Client negotiated requests for specific holding times or other method-specific holding times are adhered to. This 90-day holding time on freezing of aqueous samples is based on SGS AXYS storage stability studies.

[4] Hold times for sample extracts are from time of extraction with storage at 4°C. This 30-day holding time is a guideline, longer hold times may be accepted based on professional judgement.

[5] Whole blood must be treated with anticoagulant such as heparin or sodium citrate at time of collection.

[6] Glass vacutainers should be used for blood or serum collection. Plastic vacutainers (such as PET plastic) are not suitable as long chain PFAS compounds may be adsorbed onto the vacutainer surface.

[7] Detection limits for extract samples depend on the samples size processed. The reporting limits provided here are based on the typical extract sample size listed above.

[8] A maximum of 10 g wet, or 5 g dry, solid may be analyzed.

[9] A maximum of 5 g wet, or 0.5 g dry, biosolid may be analyzed.

[10] A maximum of 2 g of tissue may be analyzed.

SGS AXYS Analytical Services Ltd.

Stability of PFAS in Aqueous Samples

SGS AXYS has completed an extended-time storage study for the 29 PFAS listed in the table below. The study was conducted in reagent water, surface water and two wastewater treatment plant (WWTP) effluents. PFAS concentrations were measured at 0, 7, 14, 30, 90, and 180-day timepoints with data analysis of a 180-day timepoint in progress. Data analysis up to the 90-day timepoint shows that precursors present in samples containing matrix and biological activity can transform under room temperature and cold storage conditions within 7 days. At this time, only freezing of aqueous samples was demonstrated to stabilize the analytes over a period of up to 90 days. We are recommending freezing non-potable aqueous samples as soon as practicable if not analyzed within 3-4 days.

Summary of Analyte Stability in Aqueous Samples by Storage Condition

	Stability (days)		**Remarks**
Analyte	**4°C**	**-20°C**	
C_4-C_{14} Perfluorinated carboxylates including PFOA	90	90	
C_4-C_{10} perfluorinated sulfonates including PFOS	90	90	
PFDoS	90	90	
4:2 FTS	90	90	
6:2 FTS	90	90	
8:2 FTS	14	90	Decreasing trend seen from day 7 at 4°C
PFOSA	14	90	Increasing trend seen day 14 onwards at 4°C
N-MeFOSA	7	90	Decreasing trend seen at first stability point 4°C
N-EtFOSA	7	90	Decreasing trend seen at first stability point 4°C
N-MeFOSAA	<7	90	Increase from transformation of MeFOSE
N-EtFOSAA	7	90	Increase from transformation of EtFOSE
N-MeFOSE	<7	90	Loss seen at first stability point 4°C
N-EtFOSE	<7	90	Loss seen at first stability point 4°C

Appendix C:

SGS Axys Pesticide Method Summary

SGS AXYS Analytical Services Ltd.

SUMMARY OF SGS AXYS METHOD MLA-028 REV 06 VER 14:

ANALYTICAL PROCEDURES FOR ORGANOCHLORINE PESTICIDES BY ISOTOPE DILUTION HRGC/HRMS BY EPA METHOD 1699

SGS AXYS Method MLA-028 covers determination of organochlorine pesticides in water, solids (soil, sediment, biosolids, etc.), tissue (including whole blood, plasma, serum and milk), aqueous samples, XAD-2 water sampling columns, air samples and solvent extracts by capillary gas chromatography and high-resolution mass spectrometry.

This method also allows for the optional analysis of technical Toxaphene and Toxaphene congeners (Parlars). $^{13}C_{12}$-PCB 159 is used as the surrogate standard for both of these analyses, but different ions are monitored.

MLA-028 performance and quality procedures meets the requirements of EPA 1699 for analysis of selected organochlorine pesticides.

Target Analytes

Compound	CAS No.
E1 Pesticides	
1,3-dichlorobenzene (1,3-Cl_2-BZ) [1,2]	541-73-1
1,4-dichlorobenzene (1,4-Cl_2-BZ) [1,2]	106-46-7
1,2-dichlorobenzene(1,2-Cl_2-BZ) [1,2]	95-50-1
1,2,4-trichlorobenzene (1,2,4-Cl_3-BZ) [1,2]	120-82-1
1,2,3-trichlorobenzene (1,2,3-Cl_3-BZ) [1,2]	87-61-6
1,3,5-trichlorobenzene (1,3,5-Cl_3-BZ) [1,2]	108-70-3
1,2,4,5-tetrachlorobenzene (1,2,4,5-Cl_4-BZ) [1,2]	95-94-3
1,2,3,5-tetrachlorobenzene (1,2,3,5-Cl_4-BZ) [1,2]	634-90-2
1,2,3,4-tetrachlorobenzene (1,2,3,4-Cl_4-BZ) [1,2]	634-66-2
Pentachlorobenzene (Cl_5-BZ) [1]	608-93-5
Hexachlorobenzene (HCB)	118-74-1
alpha-HCH	319-84-6
beta-HCH	319-85-7
gamma-HCH	58-89-9
Heptachlor	76-44-8
Aldrin	309-00-2
Oxychlordane	27304-13-8
trans-Chlordane	5103-74-2
cis-Chlordane	5103-71-9
o,p'-DDE	3424-82-6
p,p'-DDE	72-55-9
trans-Nonachlor	39765-80-5
cis-Nonachlor	5103-73-1

o,p'-DDD	53-19-0
p,p'-DDD	72-54-8
o,p'-DDT	789-02-6
p,p'-DDT	50-29-3
Mirex	2385-85-5
Hexachlorobutadiene [1]	87-68-3
Octachlorostyrene [1]	29082-74-4
Technical Toxaphene [1]	8001-35-2
Toxaphene Congeners/Parlars [1]	
E2 Pesticides	
cis-Heptachlor epoxide	1024-57-3
alpha-Endosulfan	959-98-8
delta-HCH	319-86-8
Dieldrin	60-57-1
Endrin	72-20-8
Methoxychlor	72-43-5
Endosulfan sulfate	1031-07-8
Endrin ketone	53494-70-5
beta-Endosulfan	33213-65-9
Endrin aldehyde	7421-93-4
Oxadiazon [1]	19666-30-9
Dacthal [1]	1861-32-1

[1] Non-routine compounds that must be specifically requested by clients if required.

[2] Recoveries of dichlorobenzenes, trichlorobenzenes and tetrachlorobenzenes may be low due to loss through volatilization during the analytical work-up. These are ‘compounds of opportunity’ that may be reported when recoveries are judged adequate for quantification. Formal recovery acceptance limits have not been established.

1.0 Extraction and cleanup procedures

All samples are spiked with ^{13}C-labeled surrogate standards prior to extraction and extracted as per the table below.

Matrix	Extraction
Aqueous	Liquid-liquid extraction with dichloromethane. If more than 1% suspended solids are present, the sample is centrifuged prior to extraction, and the particulate fraction is separately extracted by Soxhlet extraction. The two extracts are then combined.
Solid (sediment, soil, sludge, pulp, ash)	Soxhlet extraction with dichloromethane (or optionally with hexane/acetone).
Tissue	Soxhlet extraction with dichloromethane

Whole blood / plasma / serum	Liquid-liquid extraction with ethanol:hexane:saturated ammonium sulfate.
Milk	Liquid-liquid extraction with acetone and hexane.
XAD-2 column and filter	XAD-2 adsorbent is dried and Soxhlet extracted with dichloromethane. The filter is extracted by Dean-Stark Soxhlet extraction using toluene.
Ambient air (PUF and filter)	The PUF and filter(s) are Soxhlet extracted together using dichloromethane.

Extracts are cleaned up on a Florisil column for which column cutpoints are regularly determined. The following fractions are eluted from the Florisil column:

- E1 contains non-polar and moderately polar chlorinated pesticides, chlorobenzenes, and toxaphene.
- E2 contains the more polar chlorinated pesticides.

Additionally, tissue, milk and certain blood/plasma/serum extracts are cleaned up by chromatography on a Biobead column.

2.0 INSTRUMENTATION

Instrumental analysis is performed on a DB-5 capillary chromatography column coupled to a high-resolution mass spectrometer (HRMS). The HRMS is operated at a static (8000) mass resolution (10% valley) in the electron ionization (EI) mode using multiple ion detection (MID). Selected PFK ions are used as a reference for mass lock. Two masses from the molecular ion cluster are used to monitor each of the target analytes and ^{13}C-labeled surrogate standards.

3.0 Calibration

Initial calibration is performed using at least a five-point calibration series of solutions that encompass the working instrumental concentration range. Initial calibration solutions contain the suite of labeled surrogate and recovery standards and authentic target pesticides. Calibration is verified at least once every 12 hours by analysis of a mid-level calibration solution. Calibration procedures use the mean RRFs determined from the initial calibration to calculate analyte concentrations.

Alternatively, the client may request that bracketing calibration protocols are followed. Bracketing calibration procedures use mean RRFs from the analysis of the mid-level calibration solution prior to and after samples, to calculate analyte concentrations.

Concentrations of E1 Chlorobenzenes and Pesticides Calibration Standard Solutions

Compound Name	Level A CS-0 (Instrument Spec)	Calibration Standards				
		Level B CS-1	Level C CS-2	Level D CS-3 (Mid-Level)	Level E CS-4	Level F CS-5
1,4-dichlorobenzene	2	10	40	80	400	2000
1,3-dichlorobenzene	2	10	40	80	400	2000
1,2-dichlorobenzene	2	10	40	80	400	2000
1,2,4-trichlorobenzene	2	10	40	80	400	2000
1,2,3-trichlorobenzene	2	10	40	80	400	2000
1,3,5-trichlorobenzene	2	10	40	80	400	2000
1,2,4,5-tetrachlorobenzene	2	10	40	80	400	2000
1,2,3,5-tetrachlorobenzene	2	10	40	80	400	2000
1,2,3,4-tetrachlorobenzene	2	10	40	80	400	2000
Pentachlorobenzene	2	10	40	80	400	2000
Hexachlorobenzene	2	10	40	80	400	2000
alpha-HCH	4	20	80	160	800	4000
beta-HCH	4	20	80	160	800	4000
delta-HCH	4	20	80	160	800	4000
gamma-HCH	4	20	80	160	800	4000
Heptachlor	2	10	40	80	400	2000
Aldrin	4	20	80	160	800	4000
Octachlorostyrene	2	10	40	80	400	2000
Oxychlordane	4	20	80	160	800	4000
cis-Chlordane	4	20	80	160	800	4000
trans-Chlordane	4	20	80	160	800	4000
o,p'-DDE	2	10	40	80	400	2000
p,p'-DDE	2	10	40	80	400	2000
cis-Nonachlor	4	20	80	160	800	4000
trans-Nonachlor	4	20	80	160	800	4000
o,p'-DDD	2	10	40	80	400	2000
p,p'-DDD	2	10	40	80	400	2000
o,p'-DDT	2	10	40	80	400	2000
p,p'-DDT	2	10	40	80	400	2000
Mirex	2	10	40	80	400	2000
Labeled Surrogates						
$^{13}C_6$-1,4-dichlorobenzene	80	80	80	80	80	80
$^{13}C_6$-1,2,3-trichlorobenzene	80	80	80	80	80	80
$^{13}C_6$-1,2,3,4-tetrachlorobenzene	80	80	80	80	80	80
$^{13}C_6$-pentachlorobenzene	80	80	80	80	80	80
$^{13}C_6$-hexachlorobenzene	80	80	80	80	80	80

Compound Name	Level A CS-0 (Instrument Spec)	Calibration Standards				
		Level B CS-1	Level C CS-2	Level D CS-3 (Mid-Level)	Level E CS-4	Level F CS-5
$^{13}C_6$-beta-HCH	80	80	80	80	80	80
$^{13}C_6$-delta-HCH	80	80	80	80	80	80
$^{13}C_6$-gamma-HCH	80	80	80	80	80	80
$^{13}C_{10}$-Heptachlor	80	80	80	80	80	80
$^{13}C_{12}$-Aldrin	80	80	80	80	80	80
$^{13}C_{10}$-Oxychlordane	80	80	80	80	80	80
$^{13}C_{10}$-trans-Chlordane	80	80	80	80	80	80
$^{13}C_{12}$-o,p'-DDE	80	80	80	80	80	80
$^{13}C_{12}$-p,p'-DDE	80	80	80	80	80	80
$^{13}C_{10}$-trans-Nonachlor	80	80	80	80	80	80
$^{13}C_{10}$-cis-Nonachlor	80	80	80	80	80	80
$^{13}C_{12}$-p,p'-DDD	80	80	80	80	80	80
$^{13}C_{12}$-o,p'-DDT	80	80	80	80	80	80
$^{13}C_{12}$-p,p'-DDT	80	80	80	80	80	80
$^{13}C_{10}$-Mirex	80	80	80	80	80	80
Recovery Standards						
$^{13}C_{12}$-PCB 52	50	50	50	50	50	50
$^{13}C_{12}$-PCB 138	50	50	50	50	50	50

Concentrations of Calibration Solutions for Technical Toxaphene

Native Compound Name	Calibration Standard Concentration
Toxaphene	5000 ng/mL
Surrogate Standard	
$^{13}C_{12}$-PCB-159	100 ng/mL
Recovery Standard	
$^{13}C_{12}$-PCB-138	50 ng/mL

E2 Pesticides - Nominal Concentrations of Calibration Solutions (ng/mL)

Compound Name	Level A CS-0 (Instrument Specif.)	Calibration Standards						
		Level B CS-1	Level C CS-2	Level D CS-3 (Mid-Level)	Level E CS-4	Level F CS-5	Level G CS-6	Level H CS-7
delta-HCH	2	4	10	20	40	50	80	200
cis-Heptachlor epoxide	8	16	40	80	160	200	320	800
alpha-Endosulfan	8	16	40	80	160	200	320	800
Dieldrin	4	8	20	40	80	100	160	400
Endrin	8	16	40	80	160	200	320	800
Methoxychlor	4	8	20	40	80	100	160	400
Endosulfan sulfate	8	16	40	80	160	200	320	
Endrin ketone	8	16	40	80	160	200	320	800
beta-Endosulfan	16	32	80	160	320	400	640	
Endrin aldehyde	8	16	40	80	160	200	320	800
beta-HCH [1]	4	8	20	40	80	100	160	400
Dacthal				100				
Oxadiazon				100				
Surrogates								
$^{13}C_6$-delta-HCH	80	80	80	80	80	80	80	80
$^{13}C_{12}$-Dieldrin	80	80	80	80	80	80	80	80
$^{13}C_{12}$-Endrin	80	80	80	80	80	80	80	80
$^{13}C_{12}$-Endrin aldehyde	80	80	80	80	80	80	80	80
$^{13}C_{10}$-cis-Heptachlor epoxide	80	80	80	80	80	80	80	80
$^{13}C_{12}$-Methoxychlor	80	80	80	80	80	80	80	80
$^{13}C_9$-alpha-Endosulfan	80	80	80	80	80	80	80	80

Compound Name	**Level A CS-0 (Instrument Specif.)**	**Calibration Standards**						
		Level B CS-1	**Level C CS-2**	**Level D CS-3 (Mid-Level)**	**Level E CS-4**	**Level F CS-5**	**Level G CS-6**	**Level H CS-7**
$^{13}C_9$-beta-Endosulfan	80	80	80	80	80	80		
$^{13}C_{12}$-Endrin ketone [2]	80	80	80	80	80	80	80	80
$^{13}C_6$-beta-HCH [1]	80	80	80	80	80	80	80	80
Recovery Standard								
$^{13}C_{12}$-PCB 153	50	50	50	50	50	50	50	50

[1] Beta-HCH and $^{13}C_6$-beta-HCH are normally quantified in cleanup fraction E1, but are also included in fraction E2 standards for diagnostic purposes or for custom quantification with Client Services' approval.

[2] $^{13}C_{12}$-Endrin ketone is included for diagnostic purposes or for custom quantification with Client Services' approval.

4.0 QUANTIFICATION PROCEDURES

The response for any component is taken as the sum of the integrated peak areas for the two characteristic masses for that compound. Quantification is by the isotope dilution method. Target concentrations are determined with respect to labeled surrogate standards. Mean relative response factors (RRF), determined from the multi-level initial calibration series are used to convert raw peak areas in sample chromatograms to final concentrations as follows:

$$\text{Concentration of Target} = \left(\frac{\text{area of Target}}{\text{area of Qt Std}}\right) \times \left(\frac{\text{weight of Qt Std}}{\text{RRF}}\right) \times \left(\frac{1}{\text{weight of sample}}\right)$$

where $\text{RRF} = \left(\frac{\text{area of Target}}{\text{area of Qt Std}}\right) \times \left(\frac{\text{weight of Qt Std}}{\text{weight of Target}}\right)$

and the Qt Std is either the surrogate or the internal standard

Those compounds quantified against a labeled standard added at the beginning of the analysis procedure are recovery corrected by the method of quantification. Surrogate recoveries are determined similarly against the recovery (internal) standard and are used as general indicators of overall analytical quality.

4.1 Reporting Limits

Concentrations and detection limits for the pesticides are reported. Typical reporting units for all data are ng/g, ng/L, or ng/sample. Concentrations for solids are reported on a dry weight basis. Concentrations in tissues (including blood and milk) are reported on a wet weight basis and/or on a lipid weight basis when requested. Concentrations in aqueous samples are reported on a volume basis. Concentrations in XAD-2 resin and filter samples are reported on a per sample basis or a per volume basis. Concentrations in particulate filters are reported on a per sample basis.

The following are commonly requested reporting limits:

Sample Specific Detection Limit or Sample Detection Limit (SDL) – determined individually for every sample analysis run by converting the area equivalent of 3 times the estimated chromatographic noise height to a concentration in the same manner that target peak responses are converted to final concentrations. SDLs must be prorated depending on sample size, extract dilution/split and final extract volume. The SDL accounts for any effect of matrix on the detection system and for recovery achieved through the analytical work-up. Equivalent term(s): Estimated Detection Limit (EDL) from EPA method 8290.

Method Detection Limit (MDL) - determined as specified by US Fed. Reg. 40 CFR Part 136, Appendix B, rev.2, Aug 2017. The 99% confidence level MDL is determined based on analysis of blanks and a minimum of 7 replicate matrix spikes fortified at 1-10 times the estimated detection limit. MDL is determined as required based on accreditation, contract and workload requirements.

Lower Method Calibration Limit (LMCL) - determined by prorating the concentration of the lowest calibration limit for sample size and extract volume. The following equation is used. ((lowest level cal conc.) x (extract volume))/sample size. Typical extract volume for pesticides is 20 µL.

The Minimum Reporting Limit practice is to never report lower than 1/5 of the typical detection limits stated in the tables in the QA/QC section below. Assuming the nominal final extract volume fv=200 µL the Minimum Reporting Limit in this method is defined as the sample concentration equivalent to 0.04 ng for E1 pesticides and 0.1 ng for E2 pesticides. If other sample sizes or other final extract volumes than the nominal are used, the Minimum Reporting Limit must be prorated accordingly. Prorating is also necessary if extract dilution or split occurred.

Reporting limit practice is to use the highest of the SDL or the Minimum Reporting Limit.

Analyte Ions Monitored, Surrogates Used and RRF Determination for E1 Pesticides

(No entry in the "RRF Determination" field designated an RRF derived from that same compound.)

Analyte Name	Quantification and Retention Time Reference	RRF Determination	Default GC Program Retention Time	Longer GC Program Retention Time	RT Win. (sec)	Acquired in Function (Default/ Long)		mass1	mass2	m1/m2 ratio	Ion Ratio Tolerance (+/- %)
Non-routine analytes acquired only with the longer GC program											
1,3-Cl$_2$-BZ	$^{13}C_6$-1,4-Cl$_2$-BZ			9:05	-5,5		1	145.969	147.966	1.56	20
1,4-Cl$_2$-BZ	$^{13}C_6$-1,4-Cl$_2$-BZ			9:10	-1,3		1	145.969	147.966	1.56	20
1,2-Cl$_2$-BZ	$^{13}C_6$-1,4-Cl$_2$-BZ			9:31	-5,5		1	145.969	147.966	1.56	20
1,3,5-Cl$_3$-BZ	$^{13}C_6$-1,2,3-Cl$_3$-BZ			11:04	-5,5		1	179.93	181.927	1.03	20
1,2,4-Cl$_3$-BZ	$^{13}C_6$-1,2,3-Cl$_3$-BZ			11:47	-5,5		1	179.93	181.927	1.03	20
1,2,3-Cl$_3$-BZ	$^{13}C_6$-1,2,3-Cl$_3$-BZ			12:21	-1,3		1	179.93	181.927	1.03	20
1,2,4,5/3,5-Cl$_4$-BZ	$^{13}C_6$-1,2,3,4-Cl$_4$-BZ			14:14	-5,5		2	213.891	215.888	0.77	20
Hexachlorobutadiene	$^{13}C_6$-1,2,3-Cl$_3$-BZ	1,2,3-Cl$_3$-BZ		12:20	-5,5		1	189.872	187.875	1.32	20
1,2,3,4-Cl$_4$-BZ	$^{13}C_6$-1,2,3,4-Cl$_4$-BZ			15:02	-1,3		2	213.891	215.888	0.77	20
Cl$_5$-BZ	$^{13}C_6$-Cl$_5$-BZ			17:27	-1,3		2	247.852	249.849	0.62	20
Routine analytes acquired with both the default and longer GC programs											
HCB	$^{13}C_6$-HCB		15:10	20:55	-1,3	1	3	283.81	285.807	1.25	20
alpha-HCH	$^{13}C_6$-gamma-HCH		14:54	20:36	-5,5	1	3	216.915	218.912	0.77	20
beta-HCH	$^{13}C_6$-beta-HCH		15:48	21:39	-1,3	1	3	216.915	218.912	0.77	20
gamma-HCH	$^{13}C_6$-gamma-HCH		16:00	21:48	-1,3	1	3	216.915	218.912	0.77	20
Delta-HCH [1]	$^{13}C_6$-delta-HCH		16:48	22:43	-1,3	1	3	216.915	218.912	0.77	20
Heptachlor	$^{13}C_{10}$-Heptachlor		18:32	24:25	-1,3	1	3	271.81	273.81	1.24	20
Aldrin	$^{13}C_{12}$-Aldrin		19:47	25:43	-1,3	1	3	262.857	264.854	1.55	20
Octachlorostyrene	$^{13}C_{10}$-trans-Chlordane		21:07	27:05	-5,5	1	3	270.844	272.841	0.62	20
Oxychlordane	$^{13}C_{10}$-Oxychlordane		21:19	27:16	-1,3	1	3	262.857	264.854	1.55	20
trans-Chlordane	$^{13}C_{10}$-trans-Chlordane		22:09	28:06	-1,3	2	4	271.81	273.807	1.24	20
cis-Chlordane	$^{13}C_{10}$-trans-Chlordane		22:43	28:41	-5,5	2	4	271.81	273.807	1.24	20
o,p-DDE	$^{13}C_{12}$-o,p-DDE		22:20	28:19	-1,3	2	4	246	247.997	1.56	20
p,p-DDE	$^{13}C_{12}$-p,p-DDE		23:30	29:29	-1,3	2	4	246	247.997	1.56	20
trans-Nonachlor	$^{13}C_{10}$-trans-Nonachlor		22:54	28:52	-1,3	2	4	271.81	273.807	1.24	20
cis-Nonachlor	$^{13}C_{10}$-cis-Nonachlor		25:14	31:12	-1,3	3	5	271.81	273.807	1.24	20

Analyte Name	Quantification and Retention Time Reference	RRF Determination	Default GC Program Retention Time	Longer GC Program Retention Time	RT Win. (sec)	Acquired in Function (Default/ Long)		mass1	mass2	m1/m2 ratio	Ion Ratio Tolerance (+/- %)
o,p-DDD	$^{13}C_{12}$-p,p-DDD		23:49	29:49	-5,5	3	5	235.008	237.005	1.56	20
p,p-DDD	$^{13}C_{12}$-p,p-DDD		25:04	31:03	-1,3	3	5	235.008	237.005	1.56	20
o,p-DDT	$^{13}C_{12}$-o,p-DDT		25:11	31:10	-1,3	3	5	235.008	237.005	1.56	20
p,p-DDT	$^{13}C_{12}$-p,p-DDT		26:16	32:24	-1,3	3	5	235.008	237.005	1.56	20
Mirex	$^{13}C_{10}$-Mirex		28:39	34:54	-1,3	3	5	269.813	271.81	0.52	20
Non-routine surrogates acquired only with the longer GC program											
$^{13}C_6$-1,4-Cl_2-BZ	$^{13}C_{12}$-PCB-52			9:10	-10,10		1	151.989	153.986	1.56	20
$^{13}C_6$-1,2,3-Cl_3-BZ	$^{13}C_{12}$-PCB-52			12:20	-10,10		1	185.95	187.947	1.03	20
$^{13}C_6$-1,2,3,4-Cl_4-BZ	$^{13}C_{12}$-PCB-52			15:02	-10,10		2	221.908	223.905	2.08	20
$^{13}C_6$-Cl_5-BZ	$^{13}C_{12}$-PCB-52			17:26	-10,10		2	255.869	257.866	1.55	20
Routine surrogates acquired with both the default and longer GC programs											
$^{13}C_6$-HCB	$^{13}C_{12}$-PCB-52		15:09	20:55	-10,10	1	3	289.83	291.828	1.25	20
$^{13}C_6$-beta-HCH	$^{13}C_{12}$-PCB-52		15:47	21:38	-10,10	1	3	222.935	224.932	0.78	20
$^{13}C_6$-gamma-HCH	$^{13}C_{12}$-PCB-52		15:59	21:47	-10,10	1	3	222.935	224.932	0.78	20
$^{13}C_6$-delta-HCH	$^{13}C_{12}$-PCB-52		16:47	22:42	-10,10	1	3	222.935	224.932	0.78	20
$^{13}C_{10}$-Heptachlor	$^{13}C_{12}$-PCB-52		18:31	24:24	-10,10	1	3	276.827	278.824	1.24	20
$^{13}C_{10}$-Aldrin	$^{13}C_{12}$-PCB-52		19:47	25:41	-10,10	1	3	269.88	271.877	1.55	20
$^{13}C_{10}$-Oxychlordane	$^{13}C_{12}$-PCB-52		21:18	27:15	-10,10	1	3	269.88	271.877	1.55	30
$^{13}C_{10}$-trans-Chlordane	$^{13}C_{12}$-PCB-138		22:08	28:05	-10,10	2	4	276.827	278.824	1.24	20
$^{13}C_{10}$-trans-Nonachlor	$^{13}C_{12}$-PCB-138		22:53	28:51	-10,10	2	4	276.827	278.824	1.24	20
$^{13}C_{10}$-cis-Nonachlor	$^{13}C_{12}$-PCB-138		25:13	31:11	-10,10	3	5	276.827	278.824	1.24	20
$^{13}C_{12}$-o,p-DDE	$^{13}C_{12}$-PCB-138		22:19	28:18	-10,10	2	4	258.041	260.038	1.56	20
$^{13}C_{12}$-p,p-DDE	$^{13}C_{12}$-PCB-138		23:29	29:29	-10,10	2	4	258.041	260.038	1.56	20
$^{13}C_{12}$-p,p-DDD	$^{13}C_{12}$-PCB-138		25:03	31:02	-10,10	3	5	247.048	249.045	1.56	20
$^{13}C_{12}$-o,p-DDT	$^{13}C_{12}$-PCB-138		25:11	31:09	-10,10	3	5	247.048	249.045	1.56	20
$^{13}C_{12}$-p,p-DDT	$^{13}C_{12}$-PCB-138		26:15	32:23	-10,10	3	5	247.048	249.045	1.56	20
$^{13}C_{10}$-Mirex	$^{13}C_{12}$-PCB-138		28:38	34:53	-10,10	3	5	276.827	278.824	1.25	20
Recovery Standards											
$^{13}C_{12}$-PCB-52			19:10	25:07	-40,40	1	3	301.963	303.96	0.77	20
$^{13}C_{12}$-PCB-138			26:22	32:31	-40,40	3	5	299.947	301.944	0.77	20

[1] Delta-HCH normally will elute primarily in the E2 fraction and can be quantified solely from this fraction. Recoveries of ^{13}C-delta-HCH may be reported as the sum of the E1 and E2 recoveries if significant concentrations of ^{13}C-delta-HCH are observed in the E1 fraction.

Surrogates Used and RRF Determination for Technical Toxaphene

Compound Name	Quant. Ion m/z	Conf. Ion m/z	Quant/conf ion ratio	Ion ratio tolerance (± %)	Surrogate	RRF Determined From
Toxaphene peaks T1, T2, T3, T4, T5, T6, T7	246.9244	244.9273	0.48	20	^{13}C-PCB 159	Toxaphene peaks T1, T2, T3, T4, T5, T6, T7
Surrogate Standard						
$^{13}C_{12}$-PCB 159	299.947	301.944	0.77	20	^{13}C-PCB 138	Toxaphene peaks T1, T2, T3, T4, T5, T6, T7
Recovery Standard						
$^{13}C_{12}$-PCB 138	299.947	301.944	0.77	20		

Analyte Ions Monitored, Surrogates Used and RRF Determination for E2 Pesticides

(No entry in the "RRF Determination" field designates an RRF derived from that same compound.)

Analyte Name	Quantification and Retention Time Reference	RRF Determination	Typical Retention Time	RT Win. (sec)	Acquired in Function	mass1	mass2	m1/m2 ratio	Ion Ratio Tolerance (+/- %)
delta-HCH	$^{13}C_{6}$-delta-HCH		13:13	-1,3	1	216.915	220.909	1.63	20
Dacthal	$^{13}C_{12}$-PCB-153		16:39	-10,10	1	298.884	300.881	0.78	20
cis-Heptachlor epoxide	$^{13}C_{9}$-Heptachlor-Epoxide		18:31	-1,3	2	354.841	352.844	0.8	20
alpha-Endosulfan	$^{13}C_{9}$-alpha-endosulfan		20:59	-1,3	2	264.854	262.857	0.64	20
Dieldrin	$^{13}C_{12}$-Dieldrin		23:00	-1,3	2	264.854	262.857	0.64	20
Oxadiazon	$^{13}C_{12}$-PCB-153		23:18	-10,10	2	258.033	260.030	1.58	20
Endrin	$^{13}C_{12}$-Endrin		24:22	-1,3	2	264.854	262.857	0.64	20
beta-Endosulfan	$^{13}C_{9}$-beta-Endosulfan		24:55	-1,3	2	264.854	262.857	0.64	20
Endosulfan sulfate	$^{13}C_{9}$-beta-Endosulfan		27:06	-5,5	2	264.854	262.857	0.64	20
Endrin aldehyde	$^{13}C_{12}$-Endrin aldehyde		25:56	-1,3	2	346.896	344.899	0.64	20
Endrin ketone	$^{13}C_{12}$-Endrin		29:00	-5,5	2	318.901	316.904	0.64	20
Methoxychlor	$^{13}C_{12}$-Methoxychlor		29:45	-1,3	3	228.111	227.107	0.17	20
Labeled Surrogates									
$^{13}C_{6}$-delta-HCH	$^{13}C_{12}$-PCB-153		13:12	-10,10	1	222.935	224.932	0.78	20
$^{13}C_{9}$-Heptachlor-Epoxide	$^{13}C_{12}$-PCB-153		18:30	-10,10	2	364.875	362.878	0.8	20
$^{13}C_{9}$-alpha-endosulfan	$^{13}C_{12}$-PCB-153		20:58	-10,10	2	271.877	269.88	0.64	20
$^{13}C_{12}$-Dieldrin	$^{13}C_{12}$-PCB-153		22:58	-10,10	2	271.877	269.88	0.64	20
$^{13}C_{12}$-Endrin	$^{13}C_{12}$-PCB-153		24:20	-10,10	2	271.877	269.88	0.64	20
$^{13}C_{9}$-beta-Endosulfan	$^{13}C_{12}$-PCB-153		24:55	-10,10	2	271.877	269.88	0.64	20
$^{13}C_{12}$-Endrin aldehyde	$^{13}C_{12}$-PCB-153		25:55	-10,10	2	358.936	356.939	0.64	20
$^{13}C_{12}$-Methoxychlor	$^{13}C_{12}$-PCB-153		29:45	-10,10	3	239.148	240.151	22	50
Recovery Standard									
$^{13}C_{12}$-PCB-153			26:16	-40,40	2	299.947	301.944	0.78	20

5.0 QUALITY ACCEPTANCE CRITERIA

Samples are analyzed in batches consisting of a maximum of twenty samples, one procedural blank and one spiked matrix (ongoing precision and recovery, OPR) sample. A duplicate is analyzed, provided there is sufficient sample, with batches containing 7-20 samples. Matrix spike/matrix spike duplicate (MS/MSD) pairs may be analyzed on an individual contract basis. The batch is carried through the complete analytical process as a unit. For sample data to be reportable, the batch QC data must meet the established acceptance criteria presented on the analysis reports.

QC Specification Table: Authentic and Surrogate Standard Recoveries, OPR and Samples for E1 Pesticides

Analyte	Typical Sample Specific Detection Limits					Procedural Blank Level	Acceptable Matrix Spike
	Solid	Aqueous	Tissue	Pulp	XAD-2 Column		
	ng/g	ng/L	ng/g	ng/g	ng	ng	% Recovery
Dichlorobenzenes	0.1	1.0	0.1	0.05	1.0	≤ 1.0	[3]
Trichlorobenzenes	0.1	1.0	0.1	0.05	1.0	≤ 1.0	[3]
Tetrachlorobenzenes	0.1	1.0	0.1	0.05	1.0	≤ 0.5	[3]
Pentachlorobenzene	0.01	0.1	0.01	0.05	0.1	≤ 0.1	70-130
Hexachlorobenzene	0.01	0.1	0.01	0.05	0.1	≤ 0.1	70-130
alpha-HCH	0.02	0.2	0.02	0.01	0.2	≤ 0.2	70-130
beta-HCH	0.02	0.2	0.02	0.01	0.2	≤ 0.2	70-130
gamma-HCH	0.02	0.2	0.02	0.01	0.2	≤ 0.2	70-130
delta-HCH [1]	n.a.	n.a.	n.a.	n.a.	n.a.	n.a.	n.a.
Heptachlor	0.02	0.2	0.02	0.01	0.2	≤ 0.2	70-130
Aldrin	0.02	0.2	0.02	0.01	0.2	≤ 0.2	70-130
Oxychlordane	0.02	0.2	0.02	0.01	0.2	≤ 0.2	70-130
trans-Chlordane	0.02	0.2	0.02	0.01	0.2	≤ 0.1	70-130
cis-Chlordane	0.02	0.2	0.02	0.01	0.2	≤ 0.1	70-130
o,p'-DDE	0.02	0.2	0.02	0.01	0.2	≤ 0.1	70-130
p,p'-DDE	0.02	0.2	0.02	0.01	0.2	≤ 0.1	70-130
trans-Nonachlor	0.02	0.2	0.02	0.01	0.2	≤ 0.1	70-130
cis-Nonachlor	0.02	0.2	0.02	0.01	0.2	≤ 0.1	70-130
o,p'-DDD	0.02	0.2	0.02	0.01	0.2	≤ 0.1	70-130
p,p'-DDD	0.02	0.2	0.02	0.01	0.2	≤ 0.1	70-130
o,p'-DDT	0.02	0.2	0.02	0.01	0.2	≤ 0.1	70-130
p,p'-DDT	0.02	0.2	0.02	0.01	0.2	≤ 0.1	70-130
Mirex	0.02	0.2	0.02	0.01	0.2	≤ 0.1	70-130
Technical Toxaphene	0.1	1	0.1	n.a.	1	≤ 0.5	60-150
Typical Sample Size:	10 g	1 L	10 g	20 g	1 Column		
Typical final vol., µL [2]	200	200	200	200	200		

[1] Delta-HCH normally will elute primarily in the E2 fraction and can be quantified solely from this fraction. Recoveries of delta-HCH may be reported as the sum of the E1 and E2 recoveries if significant concentrations of delta-HCH are observed in the E1 fraction.

[2] Extract volumes may range from 20 µL to 200 µL. To avoid excessive loss of volatile compounds, where recovery of volatile compounds is critical, extract volumes lower than 100 µL are not recommended.

[3] Recovery of dichlorobenzenes, trichlorobenzenes and tetrachlorobenzenes may be low due to loss through volatilization during the analytical work-up. These compounds may be reported only when recoveries are judged adequate for quantification. Formal recovery acceptance limits have not been established.

Note: Custom QC criteria protocols apply for non-routine targets (e.g. octachlorostyrene, hexachlorobutadiene, etc.) **and must be defined *a priori* in Project Notes.**

SURROGATE STANDARD RECOVERIES	**% RECOVERY RANGES (ALL MATRICES)**	
$^{13}C_6$-1,4-dichlorobenzene	[1]	
$^{13}C_6$-1,2,3-trichlorobenzene	[1]	
$^{13}C_6$-1,2,3,4-tetrachlorobenzene	[1]	
$^{13}C_6$-Pentachlorobenzene	20-150	
$^{13}C_6$-Hexachlorobenzene	20-150	
$^{13}C_6$-beta-HCH	30-150	
$^{13}C_6$-delta-HCH [2]	n.a.	
$^{13}C_6$-gamma-HCH	30-150	
$^{13}C_{10}$-Heptachlor	30-150	
$^{13}C_{12}$-Aldrin	30-150	
$^{13}C_{10}$-Oxychlordane	30-200	
$^{13}C_{10}$-trans-Chlordane	30-200	
$^{13}C_{12}$-o,p'-DDE	40-150	
$^{13}C_{12}$-p,p'-DDE	40-150	
$^{13}C_{10}$-trans-Nonachlor	30-150	
$^{13}C_{10}$-cis-Nonachlor	30-150	
$^{13}C_{12}$-p,p'-DDD	40-150	
$^{13}C_{12}$-o,p'-DDT	40-150	
$^{13}C_{12}$-p,p'-DDT	40-150	
$^{13}C_{10}$-Mirex	30-150	
$^{13}C_{12}$-PCB 159	40-130	(only when technical toxaphene is analyzed)

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[1] Recovery of dichlorobenzenes, trichlorobenzenes and tetrachlorobenzenes may be low due to loss through volatilization during the analytical work-up. These compounds may be reported only when recoveries are judged adequate for quantification. Formal recovery acceptance limits have not been established

[2] Delta-HCH normally will elute primarily in the E2 fraction and can be quantified solely from this fraction. Recoveries of ^{13}C-delta-HCH may be reported as the sum of the E1 and E2 recoveries if significant concentrations of ^{13}C-delta-HCH are observed in the E1 fraction.

SGS AXYS Analytical Services Ltd.

QC Specification Table: Instrumental Analysis and Analyte Quantification for E1 Pesticides

QC Parameter	Specification
Analysis Duplicate	Max. 40% RPD (applicable to concentrations >10 times the DL).
Procedural Blank	See the table above, or ≤10% of analyte value.
Instrument Sensitivity	S/N ≥3:1 for 2-4 pg injected for all target analytes. S/N ≥2:1 for 1.6 ng of technical Toxaphene with a minimum of 7 peaks detected.
Instrument Linearity	Linearity is demonstrated by a 5-point calibration over the working concentration range with a relative standard deviation of the RRFs ≤20% for targets with a labeled analog present, ≤35% for targets with no labeled analog present and all labeled compounds.
Bracketing Calibration	RRFs for the opening and closing calibration standards over a 12-hour period must agree to within ±25% of the mean, i.e., ≤ 50 RPD. Note that 50 RPD is equivalent to 35.4% RSD.
Continuing Cal Ver	For opening and closing Cal Vers, concentrations of native compounds must be within ±20% of expected values for targets with a labeled analog present, and within ±35% for targets with no labeled analog present. For opening Cal Vers, concentrations of labeled compounds must be within ±35% of expected values. For closing Cal Vers, concentrations of labeled compounds must be within ±50% of the expected values.
Chromatogram Quality Max Peak Width: Resolution:	 1. Separation between p,p'-DDD and o,p-DDT ≤ 35% of valley height. 2. p,p-DDT breakdown must be ≤15%.
Analyte/Surrogate Ratios	Response must be within the calibrated range of the instrument. Coders may use data from more than one chromatogram to get the responses in the calibrated range.
Retention Times	RRT windows are calculated from daily calibration verification run data using RRT references and fixed RT window brackets in seconds.
Ion Ratios	Ion ratios must fall within ±20% (±30% for $^{13}C_{10}$-Oxychlordane) of the theoretical values for positive identification of all E1 targets in the calibration standards and samples. For low analyte concentrations (within x2 method LOQ) ion ratios within 50-200% of the theoretical can be accepted with professional judgment and narration explanation with the reported data.

SGS AXYS Analytical Services Ltd.

QC Specification Table: Authentic and Surrogate Standard Recoveries, OPR and Samples for E2 Chlorinated Pesticides

Analyte:	**Typical Sample Specific Detection Limits**					**Procedural Blank Level ng**	**Acceptable Matrix Spike % Recovery [2]**
	Solid	**Aqueous**	**Tissue**	**Pulp**	**XAD-2 column**		
	ng/g	**ng/L**	**ng/g**	**ng/g**	**ng**		
Delta-HCH	0.05	0.5	0.05	0.05	0.5	≤ 0.5	60-130
cis-Heptachlor epoxide	0.05	0.5	0.05	0.05	0.5	≤ 0.5	60-130
alpha-Endosulfan	0.05	0.5	0.05	0.05	0.5	≤ 0.5	70-130
Dieldrin	0.05	0.5	0.05	0.05	0.5	≤ 0.5	60-130
Endrin	0.05	0.5	0.05	0.05	0.5	≤ 0.5	60-130
Endosulfan sulfate	0.05	0.5	0.05	0.05	0.5	≤ 0.5	70-130
Endrin ketone	0.05	0.5	0.05	0.05	0.5	≤ 0.5	60-130
beta-Endosulfan	0.05	0.5	0.05	0.05	0.5	≤ 0.5	70-130
Endrin aldehyde	0.05	0.5	0.05	0.05	0.5	≤ 0.5	50-130
Methoxychlor	0.1	1	0.1	0.1	1.0	≤ 1	60-130
Typical Sample Size:	10 g	1 L	10 g	20 g	1 column		
Typical Final Vol, µL [1]	200	200	200	200	200		

[1] Extract volumes may range from 20 µL to 200 µL. To avoid excessive loss of volatile compounds, where recovery of volatile compounds is critical, extract volumes lower than 100 µL are not recommended.

[2] Recoveries quoted are guidelines only and vary according to matrix. Consult detailed method performance data available with method documentation for specific criteria.

Note: Custom QC criteria protocols apply for non-routine targets (e.g. dacthal or oxadiazon) **and must be defined *a priori* in Project Notes.**

SURROGATE STANDARD RECOVERIES:	**% RECOVERY RANGES ALL MATRICES**
$^{13}C_6$-delta-HCH	30-150
$^{13}C_{12}$-Dieldrin	30-150
$^{13}C_{12}$-Endrin	30-150
$^{13}C_{12}$-Endrin aldehyde	30-150
$^{13}C_{10}$-cis-Heptachlor epoxide	30-150
$^{13}C_{12}$-Methoxychlor	30-150
$^{13}C_9$-alpha-Endosulfan	30-150
$^{13}C_9$-beta-Endosulfan	30-150

QC Specification Table: Instrumental Analysis, and Analyte Quantification for E2 Pesticides

QC Parameter	Specification
Analysis Duplicate	Max. 40% RPD (applicable to concentrations >10 times the DL).
Procedural Blank	See the table above, or <10% of analyte value.
Instrument Sensitivity	S/N ≥3:1 for 2-16 pg injected for all target analytes.
Instrument Linearity	Linearity is demonstrated by a 5-point calibration over the working concentration range with a relative standard deviation of the RRFs ≤20% for targets with a labeled analog present, ≤35% for targets with no labeled analog present and all labeled compounds.
Bracketing Cal Ver	RRFs for the opening and closing calibration standards over a 12 hour period must agree to within ±25% of the mean, i.e., ≤ 50 RPD. Note that 50 RPD is equivalent to 35.4% RSD.
Continuing Cal Ver	For opening and closing Cal Vers concentrations of native compounds must be within ±20% of expected values for targets with a labeled analog present, and within ±35% for targets with no labeled analog present. For opening Cal Vers concentrations of labeled compounds must be within ±35% of expected values. For closing Cal Vers concentrations of labeled compounds must be within ±50% of the expected values.
Chromatogram Quality (GC Resolution)	^{13}C-labeled endrin breakdown must be ≤20%.
Analyte /Surrogate Ratios	Response ratio must be within the calibrated range of the instrument. Coders may use data from more than one chromatogram to get the responses in the calibrated range.
Retention Times	RRT windows are calculated from daily calibration verification run data using RRT references and fixed RT window brackets in seconds.
Ion Ratios	Ion ratios must fall within ±20% of the theoretical values for positive identification of all E2 targets and surrogates (with the exception of labeled methoxychlor which is ±50%) in the calibration standards and samples. For low analyte concentrations (within x2 method LOQ) ion ratios within 50-200% of the theoretical can be accepted with professional judgment and narration explanation with the reported data.

Modifications to EPA Method 1699

	SGS AXYS MLA-028	EPA 1699
Target Compounds	30 OC pesticides, includes endrin aldehyde does not include perthane, quintozene, tecnazene, dacthal, captan	34 OC pesticides
Sample Receipt and Preservation	<4 °C, no preservation	<6 °C, no preservation
Sample Storage	Aqueous: 0-4 °C Solids, tissue: -20 °C	Aqueous: <6 °C Solids, tissue: <-10 °C
Sample Hold Time	Aqueous: 7 days Solids, tissue: 1 year	Aqueous: 7 days Solids, tissue: 1 year
Surrogate Compounds Added Prior to Extraction	27 isotopically labeled standards	20 isotopically labeled standards
Extraction / Clean-up	Florisil, optional GPC	GPC, SPE, Micro-silica plus Florisil or Alumina
Extract Hold Time, Temperature	1 year, <-10 °C	40 days, < -10 °C
GC column	DB-5 60m; 0.25mm i.d.; 0.10mm film	DB-17 60m; 0.25x0.02mm i.d.; 0.10mm film
GC Resolution	pp-DDD and op-DDT (or labeled analogues) must resolve to ≤35 % valley height	t-nonachlor and t-chlordane (or labeled analogues) must resolve to 10% valley height
GC Breakdown Calculation	Endrin breakdown is calculated using the concentrations. The denominator for endrin and DDT breakdown is the sum of the parent compound and the breakdown products. pp-DDT breakdown must be ≤15%.	Endrin breakdown is calculated using the areas. The denominator for endrin and DDT breakdown is the parent compound only. For both compounds breakdown must be ≤20%.
Ion ratio tolerances	Usually +- 20%	Usually +- 25%
m/z's	Some alternate m/z's as per EPA 1699 section 10.4.2 allowance See Table 10, MLA-028	See Table 6 EPA 1699
Calibration Verification Limit, %	80-120 for compounds with an exact labeled analogue, 65-135 for compounds without an exact labeled analogue and for labeled surrogates.	Varies by compound from 70-130 to 75-125

Quantification References	23 labeled surrogates, includes 13C-endrin aldehyde, 13C-4,4-DDD, and 13C-2,4-DDE, does not include 13C-a-HCH.	21 labeled surrogates
Recovery correction of results	yes	yes
Method Blank Limit, ng	varies by compound from 0.1 to 1.0	Less than the ML in Table 1.
IPR acceptance limit range, %	Varies by compound from 50-130 to 70-130	Varies by compound from 5-200 to 55-108
OPR acceptance limit range, %	Varies by compound from 50-130 to 70-130	Varies by compound from 5-200 to 50-120
Recovery in Samples, %	Varies by compound from 30-200 to 40-150	Varies by compound from 5-199 to 32-130

Appendix A: ANALYSIS OF TOXAPHENE BY ECNI HRGC/MS

The concentration of technical Toxaphene, and of a suite of individual Toxaphene congeners/Parlars, may be determined in the E1 extract by an additional analysis by electron capture negative ion (ECNI) HRGC/MS. $^{13}C_{12}$-PCB-159 is used as the surrogate standard and a recovery standard containing $^{13}C_{12}$-labeled PCB-153 is added prior to instrument analysis.

Sensitivity for Technical Toxaphene

The signal-to-noise ratio must be greater than or equal to 5:1 for a minimum of 5 peaks for 2.5 ng of technical Toxaphene injected.

Sensitivity for Toxaphene Congeners/Parlars

The signal to noise must be greater than or equal to 3:1 for 500 pg of Parlar 62 injected and greater than or equal to 3:1 for 25 pg of other congeners/Parlars injected.

Instrument Linearity

The linearity of the HRGC/MS is demonstrated using a series of technical Toxaphene solutions that encompass the working concentration range of the instrument. The solutions are prepared from technical Toxaphene, the surrogate standard, and the recovery standard. The percent relative standard deviation (%RSD) of the relative response factor (RRF) determined from the calibration solutions must be ≤ 35%.

Nominal Concentrations of Linearity Standard Solutions for Technical Toxaphene (ng/mL)

Compound Name	Technical Toxaphene					
	Level A (Instrument Spec)	Level B	Level C	Level D (Mid-Level)	Level E	Level F
Technical Toxaphene	2500	5000	25,000	100,000	250,000	800,000
Surrogate Standard						
$^{13}C_{12}$-PCB-159	400	400	400	400	400	400

Calibration for Technical Toxaphene

The mid-level standard solution (Level C) from the linearity series is analyzed every twelve hours, at the beginning and end of a bracket of samples. The RRFs from the beginning and end of the 12-hour bracket must agree to within 25% of the mean.

Calibration for Toxaphene Congeners/Parlars

A calibration standard containing Toxaphene congeners/Parlars, the surrogate and the recovery standard is analyzed every twelve hours, at the beginning and end of a bracket of samples. The RRFs from the beginning and end of the 12-hour bracket must agree to within 25% of the mean.

Nominal Concentration of Toxaphene congeners/Parlars Calibration Standard (ng/mL)

Compound Name	Calibration Std. Concentration (ng/mL)
2-exo,3-endo,6-exo,8,9,10-HexaCB	500
2-endo,3-exo,5-endo,6-exo,8,9,10-HeptaCB	500
2-exo,3-endo,5-exo,8,9,10,10-HeptaCB	500
2,2,5,5,8,9,10-HeptaCB and 2,2,5-endo,6-exo,8,9,10-HeptaCB	1000
2-exo,3-endo,6-exo,8,9,10,10-HeptaCB and 2-exo,3-endo,5-exo,6-exo,8,9,10-HeptaCB	1000
2-exo,5-exo,6-endo,8,9,10,10-HeptaCB	500
2-endo,3-exo,5-endo,6-exo,8,8,10,10-OctaCB	500
2,2,3-exo,5-endo,6-exo,8,9,10-OctaCB	500
2-endo,3-exo,5-endo,6-exo,8,9,10,10-OctaCB and 2-exo,3-endo,5-exo,8,9,9,10,10-OctaCB	1000
2,2,5-endo,6-exo,8,8,9,10-OctaCB	500
2-exo,5,5,8,9,9,10,10-OctaCB	500
2,2,5-endo,6-exo,8,9,10,10-OctaCB	500
2-endo,3-exo,6-exo,8,8,9,10,10-OctaCB	500
2-endo,3-exo,5-endo,6-exo,8,8,9,10,10-NonaCB	500
2,2,3-exo,5,5,9,9,10,10-NonaCB	500
2,2,3-exo,5-endo,6-exo,8,9,10,10-NonaCB and 2-exo,3,3,5-exo,6-endo,8,9,10,10-NonaCB and 2,2,5-endo, 6-exo,8,8,9,10,10-NonaCB	1500
2,2,3-exo,5,5,8,9,10,10-NonaCB	500
2,2,5-endo, 6-exo,8,9,9,10,10-NonaCB	500
2,2,5,5,8,9,9,10,10-NonaCB	500
2-exo,3-endo,5-exo,6-exo,8,8,9,10,10-NonaCB	500
2,2,5,5,6-exo,8,9,9,10,10-DecaCB	500
Surrogate Standard	
$^{13}C_{12}$-PCB-159	80
Recovery Standard	
$^{13}C_{12}$-PCB-153	200

ECNI HRGC/MS Analysis

High-resolution gas chromatography/high resolution mass spectrometry (HRGC/HRMS) analysis of the E1 fraction for Toxaphene is done on a high-resolution mass spectrometer (HRMS) equipped with a high-resolution gas chromatograph. The MS is operated at a static (5000) mass resolution in the electron capture negative ionization mode (ECNI) using multiple ion detection (MID) acquiring two characteristic ions for each target analyte and surrogate standard.

ANALYTE IDENTIFICATION

A peak is identified as a Toxaphene component only when the following criteria are met.

1. The peak must occur within 2 seconds of the retention time predicted from the calibration data and adjusted for the sample RT of the labeled retention time reference.
2. The ratio of confirming to quantification ions must be within table “Analyte Ions Monitored, Surrogates Used, and RRF Determination for Toxaphene Congeners/Parlars” limits.
3. The quantification and confirmation ions must co-maximize to within 2 seconds.

QUANTIFICATION PROCEDURES

Technical Toxaphene

Technical Toxaphene is quantified from the summed area of all individual Toxaphene component peaks in the chromatogram, using an RRF determined from the technical Toxaphene bracketing calibration standards.

The sample specific detection limit (SDL) is determined by converting the minimum detectable area to a concentration.

Toxaphene Congeners/Parlars

Individual Toxaphene congeners/Parlars are quantified, as above, using the RRFs determined from the Toxaphene congener/Parlar calibration solution.

Final concentrations of technical Toxaphene and Toxaphene congener/Parlars are recovery corrected by the method of quantification.

QUALITY ACCEPTANCE CRITERIA

Samples are analyzed in batches consisting of a maximum of twenty samples, a procedural blank, a spiked reference sample and a duplicate sample where requested. Matrix spike/matrix spike duplicate (MS/MSD) pairs may be substituted for duplicates on an individual contract basis. The batch is carried through the complete analytical process as a unit. For sample data to be reportable, the batch QC data must meet the following established acceptance criteria.

- Concentration of target analytes in the procedural blanks must be less than 2 ng.
- The percent recovery of the surrogate standards must be between 40 – 130%.
- The percent recovery of technical Toxaphene in a spiked matrix sample must be between 60 – 150%.
- The percent recovery of Toxaphene congeners/Parlars in a spiked matrix sample must be between 50 – 150%.
- The difference between target concentrations for duplicate samples must be less than 40%.

Analyte Ions Monitored, Surrogates Used, and RRF Determination for Toxaphene Congeners/Parlars

(No entry in the "RRF Used" field designates an RRF derived from that same compound.)

Analyte Name		Quantified against labeled standard	RRF Dtr'n	Typical Retention Time	RT Win. (sec)	Acquired in Function	mass1	mass2	m1/m2 ratio	on Ratio Tol'nce (+/- %)
IUPAC Name	Parlar Name & (Lab Code)									
2-exo,3-endo,6-exo,8,9,10-HexaCB	(2- Hex sed)	$^{13}C_{12}$-PCB-159		18:04	10	1	306.938	308.935	0.62	20
2-endo,3-exo,5-endo,6-exo,8,9,10-HeptaCB	(3- Hept sed)	$^{13}C_{12}$-PCB-159		19:04	10	1	340.899	342.896	0.52	40
2-exo,3-endo,5-exo,8,9,10,10-HeptaCB	(5)	$^{13}C_{12}$-PCB-159		21:13	10	2	340.899	342.896	0.52	20
2,2,5,5,8,9,10-HeptaCB and **2,2,5-endo,6-exo,8,9,10-HeptaCB** *	Parlar 32 (6)	$^{13}C_{12}$-PCB-159		21:42	10	2	340.899	342.896	0.52	20
2-exo,3-endo,6-exo,8,9,10,10-HeptaCB and 2-exo,3-endo,5-exo,6-exo,8,9,10-HeptaCB	(7)	$^{13}C_{12}$-PCB-159		21:57	10	2	340.899	342.896	0.52	20
2-exo,5-exo,6-endo,8,9,10,10-HeptaCB	(9)	$^{13}C_{12}$-PCB-159		23:24	10	2	340.899	342.896	0.52	20
2-endo,3-exo,5-endo,6-exo,8,8,10,10-OctaCB	Parlar 26 (4)	$^{13}C_{12}$-PCB-159		20:24	10	2	374.86	376.857	0.45	20
2,2,3-exo,5-endo,6-exo,8,9,10-OctaCB	Parlar 39 (10)	$^{13}C_{12}$-PCB-159		23:38	10	2	374.86	376.857	0.45	20
2-endo,3-exo,5-endo,6-exo,8,9,10,10-OctaCB and 2-exo,3-endo,5-exo,8,9,9,10,10-OctaCB	Par 40/41 (11,12)	$^{13}C_{12}$-PCB-159		23:59	10	2	374.86	376.857	0.45	20
2,2,5-endo,6-exo,8,8,9,10-OctaCB	Parlar 42a (13)	$^{13}C_{12}$-PCB-159		24:17	10	2	374.86	376.857	0.45	20
2-exo,5,5,8,9,9,10,10-OctaCB	Parlar 44 (14)	$^{13}C_{12}$-PCB-159		24:37	10	2	374.86	376.857	0.45	20
2,2,5-endo,6-exo,8,9,10,10-OctaCB	(15)	$^{13}C_{12}$-PCB-159		25:56	10	2	374.86	376.857	0.45	20
2-endo,3-exo,6-exo,8,8,9,10,10-OctaCB	(18)	$^{13}C_{12}$-PCB-159		26:49	10	2	374.86	376.857	0.45	20
2-endo,3-exo,5-endo,6-exo,8,8,9,10,10-NonaCB	Parlar 50 (16)	$^{13}C_{12}$-PCB-159		26:05	10	2	410.818	412.815	0.89	20
2,2,3-exo,5,5,9,9,10,10-NonaCB	(17)	$^{13}C_{12}$-PCB-159		26:42	10	2	410.818	412.815	0.89	20
2,2,3-exo,5-endo,6-exo,8,9,10,10-NonaCB and 2-exo,3,3,5-exo,6-endo,8,9,10,10-NonaCB and **2,2,5-endo, 6-exo,8,8,9,10,10-NonaCB** *	Parlar 56 (19/20)	$^{13}C_{12}$-PCB-159		27:49	10	2	410.818	412.815	0.89	20

2,2,3-exo,5,5,8,9,10,10-NonaCB	Parlar 58 (21)	$^{13}C_{12}$-PCB-159		28:19	10	2	410.818	412.815	0.89	20
2,2,5-endo, 6-exo,8,9,9,10,10-NonaCB	Parlar 59	$^{13}C_{12}$-PCB-159	Parlar 58	28:35	10	2	410.818	412.815	0.89	20
2,2,5,5,8,9,9,10,10-NonaCB	Parlar 62 (22)	$^{13}C_{12}$-PCB-159		29:25	10	2	410.818	412.815	0.89	40
2-exo,3-endo,5-exo,6-exo,8,8,9,10,10-NonaCB	Parlar 63	$^{13}C_{12}$-PCB-159	Parlar 58	29:48	10	2	410.818	412.815	0.89	20
2,2,5,5,6-exo,8,9,9,10,10-DecaCB	Parlar 69	$^{13}C_{12}$-PCB-159	Parlar 58	32:27	10	2	444.779	446.776	0.78	20
Labeled Compounds										
$^{13}C_{12}$-PCB-159		$^{13}C_{12}$-PCB 153		25:22	20	2	371.882	373.879	1.25	20
$^{13}C_{12}$-PCB-153				22:24	50	2	371.882	373.879	1.25	20

* Bolded IUPAC name corresponds to the Parlar number shown

Appendix D:

SGS Axys PPCP Method Summary

SGS AXYS Analytical Services Ltd.

SUMMARY OF SGS AXYS METHOD MLA-075 REV 09 VER 01:

SGS AXYS METHOD MLA-075: ANALYSIS OF PHARMACEUTICAL AND PERSONAL CARE PRODUCTS AND HORMONES IN SOLID, AQUEOUS, TISSUE AND POCIS SAMPLES BY LC-MS/MS

This method is suitable for the determination of a suite of hormones and pharmaceutical and personal care compounds in biosolid, aqueous and POCIS samples (Lists 1, 2, 3, 4, 5, 6 and hormones), soil/sediment samples (Lists 1, 3, 4, 5, 6 and hormones) and in tissue samples (Lists 1, 3, 4, 5, 6 and hormones). The analysis requires extraction at two different pH conditions: Basic extraction for analysis of List 4 analytes, and acidic extraction for the analysis of List 1, 2, 3, 5 and 6 analytes and hormones.

Target Analytes

List 1 - Aqueous, solid, tissue and POCIS samples (Acid extraction, positive ESI)	
Acetaminophen	Norfloxacin
Azithromycin	Norgestimate
Caffeine	Ofloxacin
Carbadox	Ormetoprim
Carbamazepine	Oxacillin [1]
Cefotaxime	Oxolinic acid
Ciprofloxacin	Penicillin G [1]
Clarithromycin	Penicillin V
Clinafloxacin	Roxithromycin
Cloxacillin [1]	Sarafloxacin
Dehydronifedipine	Sulfachloropyridazine
Digoxigenin	Sulfadiazine
Digoxin	Sulfadimethoxine
Diltiazem	Sulfamerazine
1,7-Dimethylxanthine	Sulfamethazine
Diphenhydramine	Sulfamethizole
Enrofloxacin	Sulfamethoxazole
Erythromycin	Sulfanilamide
Flumequine	Sulfathiazole
Fluoxetine	Thiabendazole
Lincomycin	Trimethoprim
Lomefloxacin	Tylosin
Miconazole	Virginiamycin M1

List 2 – Aqueous, biosolid (only) and POCIS samples (Tetracyclines, positive ESI)	
Anhydrochlortetracycline (ACTC)	4-Epichlortetracycline (ECTC)
Anhydrotetracycline (ATC)	4-Epioxytetracycline (EOTC)
Chlortetracycline (CTC)	4-Epitetracycline (ETC)
Demeclocycline	Isochlortetracycline (ICTC)
Doxycycline	Minocycline
4-Epianhydrochlortetracycline (EACTC)	Oxytetracycline (OTC)
4-Epianhydrotetracycline (EATC)	Tetracycline (TC)
List 3 - Aqueous, solid, tissue and POCIS samples (Acid extraction, negative ESI)	
Bisphenol A	2-hydroxy-ibuprofen
Furosemide	Ibuprofen
Gemfibrozil	Naproxen
Glipizide	Triclocarban
Glyburide	Triclosan
Hydrochlorothiazide	Warfarin
List 4 - Aqueous, solid, tissue and POCIS samples (Base extraction, positive ESI)	
Albuterol	Cotinine
Amphetamine	Enalapril
Atenolol	Hydrocodone
Atorvastatin	Metformin
Cimetidine	Oxycodone
Clonidine	Ranitidine
Codeine	Triamterene
List 5 - Aqueous, solid, tissue and POCIS samples (Acid Extraction, positive ESI)	
Alprazolam	Metoprolol
Amitriptyline	Norfluoxetine
Amlodipine	Norverapamil
Benzoylecgonine	Paroxetine
Benztropine	Prednisolone
Betamethasone	Prednisone
Cocaine	Promethazine
DEET (N,N-diethyl-m-toluamide)	Propoxyphene
Desmethyldiltiazem	Propranolol
Diazepam	Sertraline

Fluocinonide	Simvastatin
Fluticasone propionate	Theophylline
Hydrocortisone	Trenbolone
10-hydroxy-amitriptyline	Trenbolone acetate
Meprobamate	Valsartan
Methylprednisolone	Verapamil
List 6 - Aqueous, solid, tissue and POCIS samples (Acid Extraction, positive ESI)	
Amsacrine	Iopamidol
Azathioprine	Medroxyprogesterone acetate
Busulfan	Melphalan
Citalopram	Metronidazole
Clotrimazole	Moxifloxacin [2]
Colchicine	Oxazepam
Cyclophosphamide	Rosuvastatin
Daunorubicin	Tamoxifen
Diatrizoic acid	Teniposide
Doxorubicin	Venlafaxine
Drospirenone	Zidovudine
Etoposide	
HM-APOS - Aqueous, solid, tissue and POCIS samples (Acid Extraction, positive ESI)	
Allyl trenbolone	Mestranol
Androstenedione	Norethindrone
Androsterone	Norgestrel
Desogestrel	Progestrone
17beta-Estradiol 3-Benzoate (not tissues)	Testosterone
HM-ANEG - Aqueous, solid, tissue and POCIS samples (Acid Extraction, negative ESI)	
17alpha-Dihydroequilin	17beta-Estradiol
Equilenin	Estriol
Equilin	Estrone
17alpha-Estradiol	17alpha-Ethinyl estradiol

[1] Analysis result is classified as ‘information value’ of estimated concentration.

[2] Moxifloxacin in solid samples is classified as ‘information value’ of estimated concentration.

1.0 EXTRACTION AND CLEANUP PROCEDURES

The analysis requires extraction at two different pH conditions: At pH 10 for analysis of fourteen analytes (List 4); and at pH 2 for the analysis of the other analytes (Lists 1, 2, 3, 5 and 6 and hormones). Prior to extraction and/or clean-up, samples are adjusted to the required pH and spiked with surrogates.

Surrogate standards are added to all samples before extraction.

Solid and tissue samples are extracted by sonication with aqueous buffered acetonitrile and with pure acetonitrile, concentrated by rotary evaporation, and diluted with ultra-pure water to 200 mL. The acidic extract is treated with EDTA. The extracts are filtered, cleaned up by solid phase extraction (SPE), and analyzed by LC/ESI-MS/MS in positive and negative ionization modes.

POCIS samplers are solvent extracted before analysis by LC/ESI-MS/MS.

All aqueous samples are filtered and the aqueous portion is cleaned up by solid phase extraction before analysis by LC/ESI-MS/MS.

Aqueous samples with no or limited visible particulate (e.g. surface water, ground water, wastewater treatment final effluent, typically with <100 mg/L TSS) normally can be processed with up to 0.5 L samples sizes. The sample is filtered and routinely only the aqueous phase is analyzed. However, upon specific agreement a separate extraction may be performed on the solids phase. The solids extract may in this case either be carried through the analysis individually as a separate sample that is reported separately, or the aqueous extract and the solids extract may be combined just prior to clean-up and reported as a combined aqueous/solids phase result.

For mixed phase aqueous/solids samples with significant solids and distinct aqueous and solids phases such as wastewater influent or process streams the sample may either be analyzed as an aqueous phase only or as two separate samples, one aqueous and one solid.

Before analysis by LC/ESI-MS/MS all extracts are spiked with recovery standards.

2.0 INSTRUMENTATION

Analysis of the sample extract is performed on a high-performance liquid chromatograph (for Lists 1 and 2,) or on an ultra-performance liquid chromatograph (for Lists 3, 4, 5, 6 and hormones) coupled to a triple quadrupole mass spectrometer. The LC-MS/MS is run in MRM (Multiple Reaction Monitoring) mode and quantification is performed by recording the peak areas of the applicable parent ion/daughter ion transitions. Some analytes are analyzed in the ESI positive mode and some are analyzed in the ESI negative mode. Analysis of the complete list of analytes requires 8 different LC-MS/MS runs.

3.0 CALIBRATION

Initial calibration is performed using a series of calibration solutions that encompass the working concentration range. Initial calibration solutions contain the suite of labelled surrogate and recovery standards and authentic targets. The concentration of the native analytes in the solutions varies to encompass the working range of the instrument, while the concentrations of the surrogates and recovery standards remain constant. A mid-level solution is analyzed every 12 hours or every 20 samples, whichever occurs first. The List 1, List 3, List 5, List 6 and hormones calibration standards are prepared in 75:25 methanol:0.1% formic acid buffer, the List 2 calibration standards in methanol and the List 4 calibration standards in 1:1 methanol:acetonitrile.

Initial calibration for any native compound requires by defaults at least 5 consecutive calibration levels, although in some cases it may be necessary to use only 4, 5 or 6 of the calibration points to establish a linear quantification range (constant RRF method for List 3, 4, 5, 6 and hormones) or a calibration curve meeting residual specifications (Lists 1 and 2 use a linear regression calculation). All calibration solutions in the table below may be analyzed, but in certain cases only 4, 5 or 6 of the levels are used to establish the initial calibration. In the table below the calibration concentrations routinely included are printed in bold type. If the number of routinely included calibration points shown for a compound is less than five, concentrations below and/or above are added as necessary based on analyst judgement to achieve the minimum number of consecutive concentration levels. Note that reporting limits are adjusted as necessary to reflect the lowest calibration concentration included in the initial calibration.

Nominal Concentrations of Calibration Solutions

List 1 (Acid extraction, positive ESI)

Compound name	Calibration Standards List 1 (Acid extraction, positive ESI)						
	CS-1 Level A	CS-2 Level B	CS-3 Level C	CS-4 CAL VER Level D	CS-5 Level E	CS-6 Level F	CS-7 Level G
Acetaminophen	**3.75**	**12.5**	**37.5**	**187**	**625**	**2500**	**12500**
Azithromycin	**0.375**	**1.25**	**3.75**	**18.7**	**62.5**	*250*	*1250*
Caffeine	**3.75**	**12.5**	**37.5**	**187**	**625**	*2500*	*12500*
Carbadox	**0.375**	**1.25**	**3.75**	**18.7**	**62.5**	*250*	*1250*
Carbamazapine	**0.375**	**1.25**	**3.75**	**18.7**	**62.5**	**250**	*1250*
Cefotaxime	*1.5*	*5*	*15*	*75*	*250*	*1000*	*5000*
Ciprofloxacin	**1.5**	**5**	**15**	**75**	**250**	*1000*	*5000*
Clarithromycin	**0.375**	**1.25**	**3.75**	**18.7**	**62.5**	*250*	*1250*
Clinafloxacin	*1.5*	**5**	**15**	**75**	**250**	*1000*	*5000*
Cloxacillin	*0.75*	**2.5**	**7.5**	**37.5**	**125**	**500**	*2500*
Dehydronifedipine	**0.15**	**0.5**	**1.5**	**7.5**	**25**	**100**	*500*
Digoxigenin	*1.5*	**5**	**15**	**75**	**250**	**1000**	*5000*
Digoxin	**1.5**	**5**	**15**	**75**	**250**	*1000*	*5000*
Diltiazem	**0.075**	**0.25**	**0.75**	**3.75**	**12.5**	**50**	*250*
1,7-Dimethylxanthine	**15**	**50**	**150**	**750**	**2500**	**10000**	*50000*
Diphenhydramine	**0.15**	**0.5**	**1.5**	**7.5**	**25**	**100**	*500*
Enrofloxacin	**0.75**	**2.5**	**7.5**	**37.5**	**125**	*500*	*2500*
Erythromycin	**0.075**	**0.25**	**0.75**	**3.75**	**12.5**	**50**	*250*

Flumequine	**0.375**	**1.25**	**3.75**	**18.7**	**62.5**	*250*	*1250*
Fluoxetine	**0.375**	**1.25**	**3.75**	**18.7**	**62.5**	*250*	*1250*
Lincomycin	**0.75**	**2.5**	**7.5**	**37.5**	**125**	*500*	*2500*
Lomefloxacin	**0.75**	**2.5**	**7.5**	**37.5**	**125**	*500*	*2500*
Miconazole	**0.375**	**1.25**	**3.75**	**18.7**	**62.5**	*250*	*1250*
Norfloxacin	**3.75**	**12.5**	**37.5**	**187**	**625**	*2500*	*12500*
Norgestimate	**0.75**	**2.5**	**7.5**	**37.5**	**125**	*500*	*2500*
Ofloxacin	**0.375**	**1.25**	**3.75**	**18.7**	**62.5**	*250*	*1250*
Ormetoprim	**0.15**	**0.5**	**1.5**	**7.5**	**25**	*100*	*500*
Oxacillin	*0.75*	**2.5**	**7.5**	**37.5**	**125**	*500*	*2500*
Oxolinic acid	*0.15*	**0.5**	**1.5**	**7.5**	**25**	*100*	*500*
Penicillin G	**0.75**	**2.5**	**7.5**	**37.5**	**125**	*500*	*2500*
Penicillin V	*0.75*	**2.5**	**7.5**	**37.5**	**125**	**500**	*2500*
Roxithromycin	**0.075**	**0.25**	**0.75**	**3.75**	**12.5**	*50*	*250*
Sarafloxacin	**3.75**	**12.5**	**37.5**	**187**	**625**	*2500*	*12500*
Sulfachloropyridazine	**0.375**	**1.25**	**3.75**	**18.7**	**62.5**	*250*	*1250*
Sulfadiazine	**0.375**	**1.25**	**3.75**	**18.7**	**62.5**	**250**	**1250**
Sulfadimethoxine	**0.075**	**0.25**	**0.75**	**3.75**	**12.5**	**50**	*250*
Sulfamerazine	**0.15**	**0.5**	**1.5**	**7.5**	**25**	**100**	*500*
Sulfamethazine	*0.15*	**0.5**	**1.5**	**7.5**	**25**	*100*	*500*
Sulfamethizole	**0.15**	**0.5**	**1.5**	**7.5**	**25**	**100**	*500*
Sulfamethoxazole	*0.15*	**0.5**	**1.5**	**7.5**	**25**	**100**	**500**
Sulfanilamide	**3.75**	**12.5**	**37.5**	**187.5**	**625**	**2500**	*12500*
Sulfathiazole	**0.375**	**1.25**	**3.75**	**18.7**	**62.5**	*250*	*1250*
Thiabendazole	**0.375**	**1.25**	**3.75**	**18.7**	**62.5**	*250*	*1250*
Trimethoprim	**0.375**	**1.25**	**3.75**	**18.7**	**62.5**	**250**	*1250*
Tylosin	**1.5**	**5**	**15**	**75**	**250**	*1000*	*5000*
Virginiamycin M1	**0.75**	**2.5**	**7.5**	**37.5**	**125**	*500*	*2500*
Surrogate Standards							
$^{13}C_2$, ^{15}N-Acetaminophen	50	50	50	50	50	50	50
$^{13}C_3$-Caffeine	75	75	75	75	75	75	75
d_{10}-Carbamazepine	25	25	25	25	25	25	25
$^{13}C_3$, ^{15}N-Ciprofloxacin	100	100	100	100	100	100	100
$^{13}C_2$-Erythromycin	25	25	25	25	25	25	25
d_5-Fluoxetine	25	25	25	25	25	25	25
$^{13}C_6$-Sulfamethazine	25	25	25	25	25	25	25
$^{13}C_6$-Sulfamethoxazole	25	25	25	25	25	25	25
d_6-Thiabendazole	25	25	25	25	25	25	25
$^{13}C_3$-Trimethoprim	25	25	25	25	25	25	25
Recovery Standards							

$^{13}C_3$-Atrazine	50	50	50	50	50	50	50

List 2 (Tetracyclines)

Compound name	**Calibration Standards List 2 (ng/mL) (Tetracyclines)**						
	CS-1 Level A	**CS-2 Level B**	**CS-3 Level C**	**CS-4 CAL VER Level D**	**CS-5 Level E**	**CS-6 Level F**	**CS-7 Level G**
Anhydrochlortetracycline (ACTC)	*3.75*	*12.5*	**31.25**	**62.5**	**125**	*375*	*1000*
Anhydrotetracycline (ATC)	*3.75*	**12.5**	**31.25**	**62.5**	**125**	*375*	*1000*
Chlortetracycline (CTC)	*1.5*	**5**	**12.5**	**25**	**50**	**150**	*400*
Demeclocycline	**3.75**	**12.5**	**31.2**	**62.5**	**125**	**375**	**1000**
Doxycycline	**1.5**	**5**	**12.5**	**25**	**50**	**150**	**400**
4-Epianhydrochlortetracycline (EACTC)	*15*	**50**	**125**	**250**	**500**	**1500**	*4000*
4-Epianhydrotetracycline (EATC)	**3.75**	**12.5**	**31.2**	**62.5**	**125**	**375**	*1000*
4-Epichlortetracycline (ECTC)	**3.75**	**12.5**	**31.2**	**62.5**	**125**	**375**	**1000**
4-Epioxytetracycline (EOTC)	**1.5**	**5**	**12.5**	**25**	**50**	**150**	*400*
4-Epitetracycline (ETC)	*1.5*	**5**	**12.5**	**25**	**50**	*150*	*400*
Isochlortetracycline (ICTC)	**1.5**	**5**	**12.5**	**25**	**50**	**150**	**400**
Minocycline	*15*	**50**	**125**	**250**	**500**	*1500*	*4000*
Oxytetracycline (OTC)	**1.5**	**5**	**12.5**	**25**	**50**	**150**	*400*
Tetracycline (TC)	**1.5**	**5**	**12.5**	**25**	**50**	**150**	*400*
Surrogate Standards							
d_6-Thiabendazole	25	25	25	25	25	25	25
Recovery Standards							
$^{13}C_3$-Atrazine	50	50	50	50	50	50	50

List 3 (Acid extraction, negative ESI)

Compound name	**Calibration Standards List 3 (ng/mL) (Acid extraction, negative ESI)**						
	CS-1 Level A	**CS-2 Level B**	**CS-3 Level C**	**CS-4 CAL VER Level D**	**CS-5 Level E**	**CS-6 Level F**	**CS-7 Level G**
Bisphenol A	1.5	5	15	75	225	1125	3750
Furosemide	1	3.00	10	50	150	750	2500
Gemfibrozil	0.2	0.60	2	10	30.0	150	500
Glipizide	0.2	0.60	2	10	30.0	150	500
Glyburide	0.2	0.60	2	10	30.0	150	500
Hydrochlorothiazide[1]	1	3.00	10	50	150	750	2500
2-hydroxy-ibuprofen	1	3.00	10	50	150	750	2500
Ibuprofen	1	3.00	10	50	150	750	2500
Naproxen	0.5	1.50	5	25	75.0	375	1250
Triclocarban	0.1	0.30	1	5	15.0	75	250

Triclosan	1.5	4.50	15	75	225	1125	3750
Warfarin	0.1	0.30	1	5	15.0	75	250
Surrogate Standards							
d_6-Bisphenol A	100	100	100	100	100	100	100
d_5- Furosemide	50	50	50	50	50	50	50
d_6-Gemfibrozil	5	5	5	5	5	5	5
d_{11}-Glipizide	25	25	25	25	25	25	25
d_3-Glyburide	10	10	10	10	10	10	10
$^{13}C_1$-d_2-Hydrochlorothiazide	50	50	50	50	50	50	50
d_6-2-hydroxy-ibuprofen	25	25	25	25	25	25	25
$^{13}C_6$-Ibuprofen	10	10	10	10	10	10	10
d_3-Naproxen	50	50	50	50	50	50	50
$^{13}C_6$-Triclocarban	5	5	5	5	5	5	5
$^{13}C_6$-Triclosan	100	100	100	100	100	100	100
d_5-Warfarin	10	10	10	10	10	10	10
Recovery Standard							
$^{13}C_3$-Ibuprofen	50	50	50	50	50	50	50
$^{13}C_1$-d_3-Naproxen	50	50	50	50	50	50	50
$^{13}C_6$-2,4,5-Trichlorophenoxy-acetic acid ($^{13}C_6$-2,4,5-T)	50	50	50	50	50	50	50

List 4 (Base extraction, positive ESI)

Compound Name	**Calibration Standards List 4 (ng/mL) (Base extraction, positive ESI)**						
	CS-1 *Level A*	**CS-2 Level B**	**CS-3 Level C**	**CS-4 CAL VER Level D**	**CS-5 Level E**	**CS-6 Level F**	**CS-7 Level G**
Albuterol	0.075	0.25	0.75	3.75	12.5	50	250
Amphetamine	0.075	0.25	0.75	3.75	12.5	50	250
Atenolol	0.075	0.25	0.75	3.75	12.5	50	250
Atorvastatin	0.30	1.00	3.00	15	50	200	1000
Cimetidine	0.15	0.50	1.5	7.5	25	100	500
Clonidine	0.30	1.00	3.00	15.0	50	200	1000
Codeine	0.30	1.00	3.00	15.0	50	200	1000
Cotinine	0.075	0.25	0.75	3.75	12.5	50	250
Enalapril	0.075	0.25	0.75	3.75	12.5	50	250
Hydrocodone	0.30	1.00	3.00	15.0	50	200	1000
Metformin	0.075	0.25	0.75	3.75	12.5	50	250
Oxycodone	0.15	0.50	1.50	7.50	25	100	500

Ranitidine	0.15	0.50	1.50	7.50	25	100	500
Triamterene	0.075	0.25	0.75	3.75	12.5	50	250
Surrogate Standards							
d_3-Albuterol	5.0	5.0	5.0	5.0	5.0	5.0	5.0
d_5-Amphetamine	5.0	5.0	5.0	5.0	5.0	5.0	5.0
d_7-Atenolol	15	15	15	15	15	15	15
d_5-Atorvastatin	50	50	50	50	50	50	50
d_3-Cimetidine	5.0	5.0	5.0	5.0	5.0	5.0	5.0
d_4-Clonidine	15	15	15	15	15	15	15
d_6-Codeine	50	50	50	50	50	50	50
d_3-Cotinine	5.0	5.0	5.0	5.0	5.0	5.0	5.0
d_5-Enalapril	5.0	5.0	5.0	5.0	5.0	5.0	5.0
d_3-Hydrocodone	15	15	15	15	15	15	15
d_6-Metformin	5.0	5.0	5.0	5.0	5.0	5.0	5.0
d_6-Oxycodone	15	15	15	15	15	15	15
d_6-Ranitidine	5.0	5.0	5.0	5.0	5.0	5.0	5.0
d_5-Triamterene	15	15	15	15	15	15	15
Recovery standards							
d_9-Albuterol	5.0	5.0	5.0	5.0	5.0	5.0	5.0
d_3-Amitriptyline	10.0	10.0	10.0	10.0	10.0	10.0	10.0

List 5 (Acid extraction, positive ESI)

Compound name	**Calibration Standards List 5 (ng/mL) (Acid extraction, positive ESI)**						
	CS-1 Level A	**CS-2 Level B**	**CS-3 Level C**	**CS-4 CAL VER Level D**	**CS-5 Level E**	**CS-6 Level F**	**CS-7 Level G**
Alprazolam	0.075	0.25	0.75	3.75	12.5	50	150
Amitriptyline	0.075	0.25	0.75	3.75	12.5	50	150
Amlodipine	0.25	0.833	2.5	12.5	41.7	167	500
Benzoylecgonine	0.0375	0.125	0.375	1.87	6.25	25	75
Benztropine	0.075	0.25	0.75	3.75	12.5	50	150
Betamethasone	0.375	1.25	3.75	18.7	62.5	250	750
Cocaine	0.0375	0.125	0.375	1.87	6.25	25	75
DEET	0.075	0.25	0.75	3.75	12.5	50	150
Desmethyldiltiazem	0.0375	0.125	0.375	1.87	6.25	25	75
Diazepam	0.125	0.417	1.25	6.25	20.83	83.3	250
Fluocinonide	0.50	1.67	5.0	25	83.3	333	1000

Fluticasone propionate	0.50	1.67	5.0	25	83.3	333	1000
Hydrocortisone	1.50	5.0	15	75	250	1000	3000
10-hydroxy-amitriptyline	0.0375	0.125	0.375	1.87	6.25	25	75
Meprobamate	0.375	1.25	3.75	18.7	62.5	250	750
Methylprednisolone	1.00	3.33	10.0	50	167	667	2000
Metoprolol	0.125	0.417	1.25	6.25	20.83	83.3	250
Norfluoxetine	0.125	0.417	1.25	6.25	20.83	83.3	250
Norverapamil	0.0375	0.125	0.375	1.87	6.25	25	75
Paroxetine	0.25	0.833	2.5	12.5	41.7	167	750
Prednisolone	1.00	3.33	10.0	50	167	667	2000
Prednisone	1.50	5.0	15	75	250	1000	3000
Promethazine	0.075	0.25	0.75	3.75	12.5	50	150
Propoxyphene	0.075	0.25	0.75	3.75	12.5	50	150
Propranolol	0.075	0.25	0.75	3.75	12.5	50	150
Sertraline	0.075	0.25	0.75	3.75	12.5	50	150
Simvastatin	0.50	1.67	5.0	25	83.3	333	1000
Theophylline	1.50	5.0	15	75	250	1000	3000
Trenbolone	0.50	1.67	5.0	25	83.3	333	1000
Trenbolone acetate	0.075	0.25	0.75	3.75	12.5	50	150
Valsartan	1.0	3.33	10.0	50	167	667	2000
Verapamil	0.0375	0.125	0.375	1.87	6.25	25	75

List 6 (Acid extraction, positive ESI)

	Calibration Standards List 6 (ng/mL) (Acid extraction, positive ESI)						
Compound name	**CS-1 Level A**	**CS-2 Level B**	**CS-3 Level C**	**CS-4 Level D**	**CS-5 CAL VER Level E**	**CS-6 Level F**	**CS-7 Level G**
Amsacrine	0.01[1]	0.02	0.05	0.15	0.5	1.0	2.0
Azathioprine	0.25	0.5	1.25	3.75	12.5	25	50
Busulfan	0.5	1.0	2.5	7.5	25	50	100
Citalopram	0.1	0.2	0.5	1.5	5.0	10	20
Clotrimazole	0.1	0.2	0.5	1.5	5.0	10	20
Colchicine	0.2	0.4	1.0	3.0	10	20	40
Cyclophosphamide	0.1[1]	0.2	0.5	1.5	5.0	10	20
Daunorubicin	0.5[1]	1.0	2.5	7.5	25	50	100
Diatrizoic acid	3	6	15	45	150	300	600
Doxorubicin	1.5	3	7.5	22.5	75	150	300

Drospirenone	2	4	10	30	100	200	400
Etoposide	0.25 [1]	0.5	1.25	3.75	12.5	25	50
Iopamidol	20	40	100	300	1000	2000	4000
Medroxyprogesterone acetate	1	2	5	15	50	100	200
Melphalan	6	12	30	90	300	600	1200
Metronidazole	0.5	1.0	2.5	7.5	25	50	100
Moxifloxacin	1	2	5	15	50	100	200
Oxazepam	1	2	5	15	50	100	200
Rosuvastatin	1	2	5	15	50	100	200
Tamoxifen	0.1	0.2	0.5	1.5	5.0	10	20
Teniposide	1	2	5	15	50	100	200
Venlafaxine	0.1	0.2	0.5	1.5	5.0	10	20
Zidovudine	1.5	3	7.5	22.5	75	150	300
Surrogate Standards							
$^{13}C_4$-Azathioprine	15	15	15	15	15	15	15
d_8-Busulfan	75	75	75	75	75	75	75
d_6-Citalopram	1.25	1.25	1.25	1.25	1.25	1.25	1.25
d_5-Clotrimazole	10	10	10	10	10	10	10
d_6-Colchicine	10	10	10	10	10	10	10
d_4-Cyclophosphamide	5	5	5	5	5	5	5
$^{13}C_1$-d_3-Daunorubicin	50	50	50	50	50	50	50
d_6-Diatrizoic acid	250	250	250	250	250	250	250
$^{13}C_3$-Drospirenone	30	30	30	30	30	30	30
d_3-Etoposide	15	15	15	15	15	15	15
d_8-Iopamidol	500	500	500	500	500	500	500
d_6-Medroxyprogesterone acetate	30	30	30	30	30	30	30
d_8-Melphalan	250	250	250	250	250	250	250
d_4-Metronidazole	15	15	15	15	15	15	15
$^{13}C_1$-d_3-Moxifloxacin	30	30	30	30	30	30	30
d_5-Oxazepam	30	30	30	30	30	30	30
d_6-Rosuvastatin	125	125	125	125	125	125	125
d_5-Tamoxifen	2.5	2.5	2.5	2.5	2.5	2.5	2.5
d_6-Venlafaxine	2.5	2.5	2.5	2.5	2.5	2.5	2.5
d_3-Zidovudine	125	125	125	125	125	125	125
Recovery Standards							
$^{13}C_3$-Atrazine	50	50	50	50	50	50	50
d_4-Amlodipine (2-chlorophenyl-d_4)	50	50	50	50	50	50	50
d_7-Propranolol (1-methylethyl-d_7)	50	50	50	50	50	50	50

Hormones (Acid extraction)

Compound name		**Calibration Standards Hormones (ng/mL) (Acid extraction)**					
	CS-1 Level A	**CS-2 Level B**	**CS-3 Level C**	**CS-4 Level D**	**CS-5 CAL VER Level E**	**CS-6 Level F**	**CS-7 Level G**
Allyl Trenbolone	0.1	0.2	0.4	1	2.5	6	15
Androstenedione	0.25	0.5	1	2.5	6.25	15	37.5
Androsterone	5	10	20	50	125	300	750
Desogestrel	10	20	40	100	250	600	1500
17alpha-Dihydroequilin	0.5	1	2	5	12.5	30	75
Equilenin	0.1	0.2	0.4	1	2.5	6	15
Equilin	0.5	1	2	5	12.5	30	75
17alpha-Estradiol	1	2	4	10	25	60	150
17beta-Estradiol	1	2	4	10	25	60	150
17beta-Estradiol 3-benzoate	0.2	0.4	0.8	2	5	12	30
Estriol	2	4	8	20	50	120	300
Estrone	0.5	1	2	5	12.5	30	75
17alpha-Ethinyl estradiol	1.25	2.5	5	12.5	31.25	75	187.5
Mestranol	5	10	20	50	125	300	750
Norethindrone	0.25	0.5	1	2.5	6.25	15	37.5
Norgestrel	0.25	0.5	1	2.5	6.25	15	37.5
Progesterone	0.1	0.2	0.4	1	2.5	6	15
Testosterone	0.1	0.2	0.4	1	2.5	6	15
Labelled Compounds (Surrogates)							
$^{13}C_3$-Androstenedione	12.5	12.5	12.5	12.5	12.5	12.5	12.5
d_4-Androsterone	125	125	125	125	125	125	125
$^{13}C_2,d_2$-Desogestrel	350	350	350	350	350	350	350
d_3-Equilenin	7.5	7.5	7.5	7.5	7.5	7.5	7.5
d_3-17alpha-estradiol	100	100	100	100	100	100	100
$^{13}C_2$-17beta-estradiol	25	25	25	25	25	25	25
d_4-17beta-estradiol	125	125	125	125	125	125	125
d_3-beta-Estradiol 3-benzoate	10	10	10	10	10	10	10
d_3-Estriol	100	100	100	100	100	100	100
$^{13}C_2$-Estrone	25	25	25	25	25	25	25
$^{13}C_2$-17alpha-Ethinylestradiol	100	100	100	100	100	100	100
d_4-Mestranol	125	125	125	125	125	125	125

d_6-Norethindrone	25	25	25	25	25	25	25
d_6-Norgestrel	25	25	25	25	25	25	25
d_9-Progesterone	5	5	5	5	5	5	5
$^{13}C_3$-Testosterone	1.5	1.5	1.5	1.5	1.5	1.5	1.5
Labelled Injection Standards (Recovery standards)							
$^{13}C_3$-Atrazine	50	50	50	50	50	50	50
$^{13}C_6$-2,4,5-Trichlorophenoxy-acetic acid ($^{13}C_6$-2,4,5-T)	50	50	50	50	50	50	50
d_4-Estrone	25	25	25	25	25	25	25
$^{13}C_3$-17alpha-Hydroxy-progesterone	12.5	12.5	12.5	12.5	12.5	12.5	12.5

[1] May be excluded from the A-CAL

4.0 QUANTIFICATION PROCEDURES

Concentrations of the target compounds are calculated either by isotope dilution quantification against the surrogate standard or by internal standard quantification against the recovery standard.

The isotopically labeled analog of an analyte (surrogate) is used for quantitation (Isotope Dilution Quantitation). If a labeled analog is not commercially available, a surrogate with chemical similarity and close retention time is used for quantitation (internal standard quantitation). Final analyte concentrations are recovery corrected by this method of quantification

For Lists 1 and 2, calibration is done by linear regression calibration, using a 1/X weighting type, excluding origin.

General equation: Y = slope × X + intercept

Where: $Y = \text{Response ratio} = \left(\frac{\text{area Target}}{\text{area SUR}} \times \text{weight SUR spiked (ng)}\right)$

X = weight of target (ng)
SUR = the surrogate standard

The slope and intercept are used to convert raw peak areas in sample chromatograms to final concentrations as follows:

$$\text{Sample Conc.} = \left(\frac{\text{area of Target}}{\text{area SUR}} \times \text{weight SUR spiked(ng) - intercept}\right) x \left(\frac{1}{\text{slope}}\right) x \left(\frac{1}{\text{samplesize}}\right)$$

Constant RRF calibration is used for Lists 3, 4, 5, 6 and Hormones (both ESI positive and ESI negative). Relative response factors (RRF) are determined from the initial calibration and are confirmed at least every 12 hours (CAL/VER).

$$\underset{\text{(ng/g or ng/L)}}{\text{Concentration of Target}} = \left(\frac{\text{area of Target}}{\text{area of Surr Std}}\right) \times \left(\frac{\text{weight of Surr Std (ng)}}{\text{RRF}}\right) \times \left(\frac{1}{\text{weight of sample (g or L)}}\right)$$

where $\text{RRF} = \left(\frac{\text{area of Target}}{\text{area of Surr Std}}\right) \times \left(\frac{\text{concentration of Surr Std}}{\text{concentration of Target}}\right)$

and the Surr Std is the isotopically labeled quantitation reference

The recoveries of the surrogate standards are calculated by internal standard quantification against the recovery standards and monitored as an indication of overall data quality. Surrogate standard recovery is calculated as:

$$\%\,\text{surrogate recovery} = \left(\frac{\text{area of Surr}}{\text{area of Rec}}\right) \times \left(\frac{\text{weight of Rec}}{\text{weight of Surr}}\right) \times \frac{100}{\text{RRF}_S}, \text{ in the sample}$$

where Rec = the recovery standard used, and

$$RRF_S = \left(\frac{\text{area of Surr}}{\text{area of Rec}}\right) \times \left(\frac{\text{weight of Rec}}{\text{weight of Surr}}\right) \text{ ; from the calibration.}$$

4.1 Reporting Limits

Results are reported as concentrations in the samples analyzed; measurement uncertainty estimates (K=2), reported as percentage ranges of the analysis results, are upon agreement included in the final analytical report.

Sample specific detection limits (SDLs) are calculated by QuanLynx software using 3 times the signal of the noise in the target channel converted to an equivalent sample concentration by the same calculation used to convert target compounds responses to sample concentrations.

Concentrations and detection limits for the target analytes are reported. The lower reporting limit for each target compound is defined as the concentration equivalent to the lowest calibration standard analyzed or the SDL, whichever is greater. Typical reporting units for all data are ng/g or ng/L. Concentrations for solids are reported on a dry weight basis. Concentrations in aqueous samples are reported on a volume basis. Concentrations for tissues are reported on a wet weight basis.

The following are commonly requested reporting limits:

Method Detection Limit (MDL) - determined as specified by EPA Fed. Reg. 40 CFR Part 136 Appendix B (no iteration option). The 99% confidence level MDL is determined based on analysis of a minimum of 7 replicate matrix spikes fortified at 1-10 times the estimated detection limit. MDL is determined as required based on accreditation, contract and workload requirements.

Lower Method Calibration Limit (LMCL) - determined by prorating the concentration of the lowest calibration limit for sample size and extract volume. The following equation is used. ((lowest level cal conc.) x (extract volume))/sample size. The typical extract volume for PPCP is 4 mL.

For the analysis of PPCP and hormones it is SGS AXYS standard to report sample concentrations using the LMCL as the lower reporting limit. In cases where the SDL is higher than the LMCL, the SDL will be used as the lower reporting limit.

The SDL is defined as follows: *Sample Specific Detection Limit or Sample Detection Limit (SDL)* – determined individually for every sample analysis run by converting the area equivalent of 3.0 times (2.5 times for EPA 1600 series methods) the estimated chromatographic noise height to a concentration in the same manner that target peak responses are converted to final concentrations. The SDL accounts for any effect of matrix on the detection system and for recovery achieved through the analytical work-up.

Analytes, Ions and Quantification References

List 1 – Acid Extraction, Positive Electrospray Ionization (+)ESI

Target Analyte	Typical Retention Time (min)	Typical RRT	RRT Reference	Precursor Ion Mass	Product Ion Mass	Quantified against
Sulfanilamide	2.02	0.432	$^{13}C_2$,^{15}N-Acetaminophen	190.0	155.8	$^{13}C_6$-Sulfamethazine
Acetaminophen	4.68	1.000	$^{13}C_2$,^{15}N-Acetaminophen	152.2	110.0	$^{13}C_2$,^{15}N-Acetaminophen
Sulfadiazine	5.32	1.137	$^{13}C_2$,^{15}N-Acetaminophen	251.2	156.1	$^{13}C_6$-Sulfamethazine
1,7-Dimethylxanthine	7.02	0.753	$^{13}C_3$-Caffeine	181.2	124.0	$^{13}C_3$-Caffeine
Sulfathiazole	8.00	0.858	$^{13}C_3$-Caffeine	256.3	156.0	$^{13}C_6$-Sulfamethoxazole
Sulfamerazine	8.78	0.942	$^{13}C_3$-Caffeine	265.0	156.0	$^{13}C_6$-Sulfamethazine
Caffeine	9.32	1.000	$^{13}C_3$-Caffeine	195.0	138.0	$^{13}C_3$-Caffeine
Lincomycin	9.47	0.953	$^{13}C_3$-Trimethoprim	407.2	126.0	$^{13}C_3$-Trimethoprim
Trimethoprim	9.94	1.000	$^{13}C_3$-Trimethoprim	291.2	230.0	$^{13}C_3$-Trimethoprim
Sulfamethizole	10.09	0.983	$^{13}C_6$-Sulfamethazine	271.0	156.0	$^{13}C_6$-Sulfamethoxazole
Cefotaxime	10.09	1.015	$^{13}C_3$-Trimethoprim	456.4	396.1	$^{13}C_3$-Trimethoprim
Sulfamethazine	10.31	1.000	$^{13}C_6$-Sulfamethazine	279.0	156.0	$^{13}C_6$-Sulfamethazine
Ofloxacin	10.53	0.974	$^{13}C_3$,^{15}N-Ciprofloxacin	362.2	318.0	$^{13}C_3$,^{15}N-Ciprofloxacin
Carbadox	10.53	1.005	d_6-Thiabendazole	263.2	231.2	$^{13}C_3$-Trimethoprim
Ormetoprim	10.53	1.059	$^{13}C_3$-Trimethoprim	275.3	259.1	$^{13}C_3$-Trimethoprim
Norfloxacin	10.59	0.980	$^{13}C_3$,^{15}N-Ciprofloxacin	320.0	302.0	$^{13}C_3$,^{15}N-Ciprofloxacin
Thiabendazole	10.59	1.000	d_6-Thiabendazole	202.1	175.1	d_6-Thiabendazole
Ciprofloxacin	10.81	1.000	$^{13}C_3$,^{15}N-Ciprofloxacin	332.2	314.2	$^{13}C_3$,^{15}N-Ciprofloxacin
Sulfachloropyridazine	10.97	1.069	$^{13}C_6$-Sulfamethazine	285.0	156.0	$^{13}C_6$-Sulfamethazine
Lomefloxacin	11.14	1.031	$^{13}C_3$,^{15}N-Ciprofloxacin	352.2	308.1	$^{13}C_3$,^{15}N-Ciprofloxacin
Enrofloxacin	11.22	1.038	$^{13}C_3$,^{15}N-Ciprofloxacin	360.2	316.0	$^{13}C_3$,^{15}N-Ciprofloxacin
Sulfamethoxazole	11.33	1.000	$^{13}C_6$-Sulfamethoxazole	254.0	156.0	$^{13}C_6$-Sulfamethoxazole
Sarafloxacin	11.84	1.095	$^{13}C_3$,^{15}N-Ciprofloxacin	386.1	299.0	$^{13}C_3$,^{15}N-Ciprofloxacin
Clinafloxacin	12.04	1.059	$^{13}C_6$-Sulfamethoxazole	366.3	348.1	$^{13}C_3$,^{15}N-Ciprofloxacin
Digoxigenin	12.68	1.115	$^{13}C_6$-Sulfamethoxazole	391.2	355.2	$^{13}C_3$-Trimethoprim

Oxolinic Acid	13.11	0.819	$^{13}C_3$-Atrazine	262.1	244.0	$^{13}C_3$-Trimethoprim
Sulfadimethoxine	13.33	1.172	$^{13}C_6$-Sulfamethoxazole	311.0	156.0	$^{13}C_6$-Sulfamethoxazole
Azithromycin	13.55	0.846	$^{13}C_3$-Atrazine	749.9	591.6	$^{13}C_3$-Trimethoprim
Penicillin G	14.46	0.903	$^{13}C_3$-Atrazine	367.1	159.9	$^{13}C_3$-Trimethoprim
Diphenhydramine	14.57	0.910	$^{13}C_3$-Atrazine	256.2	167.0	$^{13}C_3$-Trimethoprim
Flumequine	15.25	0.953	$^{13}C_3$-Atrazine	262.0	173.7	$^{13}C_3$-Trimethoprim
Penicillin V	15.29	0.955	$^{13}C_3$-Atrazine	383.2	159.9	$^{13}C_3$-Trimethoprim
Diltiazem	15.34	0.958	$^{13}C_3$-Atrazine	415.5	178.0	$^{13}C_3$-Trimethoprim
Carbamazepine	15.38	1.007	d_{10}-Carbamazepine	237.4	194.2	d_{10}-Carbamazepine
Erythromycin [1]	15.94	1.000	$^{13}C_2$-Erythromycin	734.4	158	not quantified
Oxacillin	16.30	1.018	$^{13}C_3$-Atrazine	434.1	160.2	$^{13}C_3$-Trimethoprim
Tylosin	16.37	1.022	$^{13}C_3$-Atrazine	916.6	772.5	$^{13}C_6$-Sulfamethazine
Digoxin	16.58	1.036	$^{13}C_3$-Atrazine	798.5	651.3	$^{13}C_3$-Trimethoprim
Dehydronifedipine	16.65	0.981	d_5-Fluoxetine	345.1	284.1	$^{13}C_3$-Trimethoprim
Cloxacillin	16.82	0.991	d_5-Fluoxetine	468.1	160.1	$^{13}C_3$-Trimethoprim
Erythromycin anhydrate [1]	16.90	1.000	$^{13}C_2$-Erythromycin anhydrate	716.4	158	$^{13}C_2$-Erythromycin anhydrate
Fluoxetine	16.97	1.000	d_5-Fluoxetine	310.1	148.0	d_5-Fluoxetine
Virginiamycin M1	17.40	1.025	d_5-Fluoxetine	526.3	508.3	$^{13}C_3$-Trimethoprim
Clarithromycin	17.61	1.038	d_5-Fluoxetine	748.9	158.2	$^{13}C_6$-Sulfamethazine
Roxithromycin	17.83	1.051	d_5-Fluoxetine	837.6	679.0	$^{13}C_6$-Sulfamethazine
Miconazole	20.93	1.233	d_5-Fluoxetine	417.0	161.0	$^{13}C_3$-Trimethoprim
Norgestimate	21.80	1.285	d_5-Fluoxetine	370.5	124.0	$^{13}C_3$-Trimethoprim
Surrogate Standard						
$^{13}C_2$,^{15}N-Acetaminophen	4.68	0.292	$^{13}C_3$-Atrazine	155.2	111.0	$^{13}C_3$-Atrazine
$^{13}C_3$-Caffeine	9.32	0.582	$^{13}C_3$-Atrazine	198.0	140.0	$^{13}C_3$-Atrazine
$^{13}C_3$-Trimethoprim	9.94	0.621	$^{13}C_3$-Atrazine	294.2	233.0	$^{13}C_3$-Atrazine
$^{13}C_6$-Sulfamethazine	10.26	0.641	$^{13}C_3$-Atrazine	285.1	162.1	$^{13}C_3$-Atrazine
d_6-Thiabendazole	10.48	0.655	$^{13}C_3$-Atrazine	208.1	180.1	$^{13}C_3$-Atrazine
$^{13}C_3$,^{15}N-Ciprofloxacin	10.81	0.675	$^{13}C_3$-Atrazine	336.1	318.2	$^{13}C_3$-Atrazine
$^{13}C_6$-Sulfamethoxazole	11.37	0.710	$^{13}C_3$-Atrazine	260.0	162.0	$^{13}C_3$-Atrazine

d_{10}-Carbamazepine	15.28	0.954	$^{13}C_3$-Atrazine	247	204	$^{13}C_3$-Atrazine
$^{13}C_2$-Erythromycin [1]	15.86	0.991	$^{13}C_3$-Atrazine	736.4	160.0	monitor for less than 5%
$^{13}C_2$-Erythromycin anhydrate [1]	16.90	1.056	$^{13}C_3$-Atrazine	718.4	160.0	$^{13}C_3$-Atrazine
d_5-Fluoxetine	16.97	1.060	$^{13}C_3$-Atrazine	315.3	153.0	$^{13}C_3$-Atrazine
Recovery Standard						
$^{13}C_3$-Atrazine	16.01	1.000		219.1	176.9 (134.0)	External Standard

[1] Because of intramolecular dehydration during the analytical procedure erythromycin is quantified as the dehydration product “erythromycin – H_2O”. The peak area of the $^{13}C_2$-Erythromycin is monitored and must be less than 5% of the $^{13}C_2$-Erythromycin - H_2O peak area. If it is greater, the Erythromycin - H_2O result is flagged as ‘accuracy unknown’.

List 2 – Acid Extraction, Positive Electrospray Ionization (+)ESI

Target Analyte	Typical Retention Time (min)	Typical RRT	RRT Reference	Precursor Ion Mass	Product Ion Mass	Quantified against
Minocycline	3.45	0.739	d_6-Thiabendazole	458.0	441.0	d_6-Thiabendazole
Epitetracycline (ETC)	5.71	1.223	d_6-Thiabendazole	445.2	410.2	d_6-Thiabendazole
Epioxytetracycline (EOTC)	6.51	1.394	d_6-Thiabendazole	461.2	426.2	d_6-Thiabendazole
Oxytetracycline (OTC)	7.29	1.561	d_6-Thiabendazole	461.2	426.2	d_6-Thiabendazole
Tetracycline (TC)	7.74	1.657	d_6-Thiabendazole	445.2	410.2	d_6-Thiabendazole
Demeclocycline	9.63	0.470	$^{13}C_3$-Atrazine	465.0	430.0	d_6-Thiabendazole
Epichlortetracycline (ECTC)	9.92	0.485	$^{13}C_3$-Atrazine	479.0	444.0	d_6-Thiabendazole
Isochlortetracycline (ICTC) [1]	9.95	0.486	$^{13}C_3$-Atrazine	479.0	462.0	d_6-Thiabendazole
Chlortetracycline (CTC)	11.90	0.581	$^{13}C_3$-Atrazine	479.0	444.0	d_6-Thiabendazole
Doxycycline	14.40	0.703	$^{13}C_3$-Atrazine	445.2	428.2	d_6-Thiabendazole
Epianhydrotetracycline (EATC)	15.08	0.737	$^{13}C_3$-Atrazine	427.2	409.8	d_6-Thiabendazole
Anhydrotetracycline (ATC)	16.45	0.804	$^{13}C_3$-Atrazine	427.2	409.8	d_6-Thiabendazole
Epianhydrochlortetracycline (EACTC)	18.90	0.923	$^{13}C_3$-Atrazine	461.2	444.0	d_6-Thiabendazole
Anhydrochlortetracycline (ACTC)	20.63	1.008	$^{13}C_3$-Atrazine	461.2	444.0	d_6-Thiabendazole
Surrogate Standard						
d_6-Thiabendazole	4.67	0.228	$^{13}C_3$-Atrazine	208.0	180.0	$^{13}C_3$-Atrazine
Recovery Standard						
$^{13}C_3$-Atrazine	20.51	1.000		219.1	176.9 (134.0)	External Standard

[1] The presence of ECTC will create positive interference with ICTC due to use of a common transition ion.

List 3 – Acid Extraction, Negative Electrospray Ionization (-)ESI

Target Analyte	Typical Retention Time (min)	Primary MRM Transition	Secondary MRM Transition	Typical Transition Ratio (Secondary Product Ion/Primary Product Ion)	Quantification and RRT Reference[1]
Bisphenol A	3.9	227.0 > 212.0	227.0 > 133.0	tbd	d_6-Bisphenol A
Furosemide	2.8	328.8 > 205.0	328.8 > 285.0	tbd	d_5-Furosemide
Gemfibrozil	7.6	249.0 > 121.0	249.0 > 126.9	tbd	d_6-Gemfibrozil
Glipizide	4.0	444.0 > 319.1	444.0 > 170.0	tbd	d_{11}-Glipizide
Glyburide	5.7	492.9 > 170.0	493.8 > 172.0	tbd	d_3-Glyburide
Hydrochlorothiazide	0.6	295.9 > 269.0	295.9 > 204.9	tbd	$^{13}C_1$-d_2-Hydrochlorothiazide
2-hydroxy-Ibuprofen	3.1	221.1 > 177.1	n.a.	tbd	d_6-2-hydroxy-Ibuprofen
Ibuprofen	6.1	205.0 > 161.1	n.a.	tbd	$^{13}C_6$-Ibuprofen
Naproxen	4.2	229.0 > 170.0	229.0 > 169.0	tbd	d_3-Naproxen
Triclocarban	7.7	312.8 > 160.0	314.8 > 162.0	tbd	$^{13}C_6$-Triclocarban
Triclosan	7.8	286.8 > 34.8	288.8 > 34.8	tbd	$^{13}C_{12}$-Triclosan
Warfarin	4.5	306.9 > 161.0	306.9 > 250.0	tbd	d_5-Warfarin
Surrogate Standard					
d_6-Bisphenol A	3.9	233.0 > 215.1	233.0 > 138.0	tbd	$^{13}C_1$-d_3-Naproxen
d_5-Furosemide	2.8	333.8 > 206.0	333.8 > 290.0	tbd	$^{13}C_1$-d_3-Naproxen
d_6-Gemfibrozil	7.6	255.0 > 121.0	255.0 > 133.0	tbd	$^{13}C_3$-Ibuprofen
d_{11}-Glipizide	4.0	455.0 > 319.0	455.0 > 170.0	tbd	$^{13}C_1$-d_3-Naproxen
d_3-Glyburide	5.7	494.9 > 170.0	496.7 > 171.9	tbd	$^{13}C_3$-Ibuprofen
$^{13}C_1$-d_2-Hydrochlorothiazide	0.6	298.8 > 270.1	298.8 > 206.1	tbd	$^{13}C_1$-d_3-Naproxen
d_6-2-hydroxy-Ibuprofen	3.1	227.1 > 183.1	n.a.	tbd	$^{13}C_3$-Ibuprofen
$^{13}C_6$-Ibuprofen	6.1	211.0 > 167.1	n.a.	n.a.	$^{13}C_3$-Ibuprofen
d_3-Naproxen	4.2	232.0 > 173.1	232.0 > 171.1	tbd	$^{13}C_1$-d_3-Naproxen
$^{13}C_6$-Triclocarban	7.7	318.9 > 159.9	320.9 > 161.9	tbd	$^{13}C_3$-Ibuprofen
$^{13}C_6$-Triclosan	7.8	298.9 > 34.8	294.9 > 34.8	tbd	$^{13}C_3$-Ibuprofen

d_5-Warfarin	4.5	312.0 > 161.0	312.0 > 255.1	tbd	$^{13}C_1$-d_3-Naproxen
Recovery Standard					
$^{13}C_3$-Ibuprofen	6.1	208.0 > 163.1	n.a.	n.a.	External Standard
$^{13}C_1$-d_3-Naproxen	4.2	233.0 > 170.0	233.0 > 169.0	n.a.	External Standard

[1] The primary transition of the quantification reference is used as the quantification reference for the primary transition of each analyte: the secondary transition of the quantification reference is used as the quantification reference for the secondary transition of each analyte.

* Third MRM transitions are available for some compounds and can be used if interferences are present in either the primary or secondary transition. 297.9 > 270.9 can be used for hydrochlorothiazide and 380.8 > 272.1 can be used for $^{13}C_1$-d_2-Hydrochlorothiazide.

List 4 – Base Extraction, Positive Electrospray Ionization (+)ESI

Target Analyte	Typical Retention Time (min)	Primary MRM Transition	Secondary MRM Transition	Typical Transition Ratio (Secondary Product Ion/Primary Product Ion)	Quantification and RRT Reference[1]
Albuterol	4.47	240.1 > 148.0	240.1 > 222.2	tbd	d_3-Albuterol
Amphetamine	4.21	136.0 > 91.0	136.0 > 119.0	tbd	d5-Amphetamine
Atenolol	5.49	267.2 > 74.0	267.2 > 145.0	tbd	d7-Atenolol
Atorvastatin	1.14	559.5 > 440.3	559.5 > 250.1	tbd	d5-Enalapril
Cimetidine	2.57	252.6 > 159.1	252.6 > 117.0	tbd	d3-Cimetidine
Clonidine	2.26	230.1 > 44.0	231.5 > 44.0	tbd	d4-Clonidine
Codeine	4.85	300.2 > 215.1	300.2 > 58.0	tbd	d6-Codeine
Cotinine	1.16	177.0 > 80.0	177.0 > 98.0	tbd	d3-Cotinine
Enalapril	4.00	377.2 > 234.1	377.3 > 159.8	tbd	d5-Enalapril
Hydrocodone	4.37	300.2 > 199.1	300.2 > 127.9	tbd	d3-Hydrocodone
Metformin	5.26	130.0 > 60.0	130.0 > 71.0	tbd	d_6-Metformin
Oxycodone	3.34	316.2 > 241.1	316.2 > 298.2	tbd	d6-Oxycodone
Ranitidine	5.64	315.9 > 176.1	315.9 > 130.2	tbd	d_6-Ranitidine
Triamterene	2.59	254.2 > 104.0	254.2 > 237.1	tbd	d_5-Triamterene
Surrogate Standards					
d_3-Albuterol	4.62	243.3 > 151.1	243.3 > 225.2	tbd	d3-Amitriptyline
d_5-Amphetamine	4.21	141.0 > 92.8	141.0 > 124.0	tbd	d3-Amitriptyline
d_7-Atenolol	5.51	273.7 > 79.1	273.7 > 145.0	tbd	d3-Amitriptyline
d_5-Atorvastatin	1.14	564.4 > 440.4	564.4 > 250.1	tbd	d3-Amitriptyline
d_3-Cimetidine	2.65	255.6 > 162.1	255.6 > 120.0	tbd	d3-Amitriptyline
d_4-Clonidine	2.30	234.1 > 48.0	236.0 > 48.0	tbd	d3-Amitriptyline
d_6-Codeine	4.95	306.2 > 218.1	306.2 > 61.0	tbd	d3-Amitriptyline
d_3-Cotinine	1.17	180.1 > 80.0	180.1 > 101.0	tbd	d3-Amitriptyline
d_5-Enalapril	4.03	382.3 > 239.2	382.3 > 164.8	tbd	d3-Amitriptyline
d_3-Hydrocodone	4.47	303.3 > 199.1	303.3 > 127.9	tbd	d3-Amitriptyline
d_6-Metformin	5.28	136.2 > 59.9	136.2 > 77.1	tbd	d3-Amitriptyline
d_6-Oxycodone	3.59	322.3 > 247.2	322.3 > 304.2	tbd	d3-Amitriptyline
d_6-Ranitidine	5.74	321.2 > 176.0	321.2> 130.0	tbd	d3-Amitriptyline

d_5-Triamterene	2.59	259.2 > 108.0	259.2 > 242.0	tbd	d3-Amitriptyline
Recovery Standards					
d_9-Albuterol	4.60	249.3 > 148.0	249.3 > 231.2	tbd	External Standard
d_3-Amitriptyline	3.41	281.3 > 91.0	281.3 > 233.2	tbd	External Standard

List 5 – Acid Extraction, Positive Electrospray Ionization (+)ESI

Target Analyte	Typical Retention Time (min)	Primary MRM Transition	Secondary MRM Transition	Typical Transition Ratio (Secondary Product Ion/Primary Product Ion)	Quantification and RRT Reference[1]
Alprazolam	11.33	309.1 > 281.1	309.1 > 205.1	tbd	d_5-Alprazolam
Amitriptyline	12.01	278.2 > 105.1	278.2 > 91.1	tbd	d_6-Amitriptyline
Amlodipine	12.06	409.1 > 238.1	411.1 > 240.1	tbd	d_4-Amlodipine (2-chlorophenyl-d_4)
Benzoylecgonine	4.06	290.1 > 168.2	290.1 > 105.1	tbd	d_8-Benzoylecgonine
Benztropine	12.49	308.2 > 167.2	308.2 > 152.1	tbd	d_3-Benztropine
Betamethasone	9.89	393.2 > 373.2	393.2 > 355.2	tbd	d_5-Betamethasone
Cocaine	5.59	304.2 > 182.2	304.2 > 82.1	tbd	d_3-Cocaine
DEET	10.6	192.2 > 119.0	192.2 > 91.0	tbd	d_7-DEET
Desmethyldiltiazem	9.7	401.1 > 178.0	401.1 > 109.0	tbd	d_4-Desmethyldiltiazem
Diazepam	14.08	285.0 > 154.0	285.0 > 193.1	tbd	d_5-Diazepam
Fluocinonide	15.22	495.2 > 337.1	495.2 > 121.1	tbd	d_5-Fluticasone propionate
Fluticasone propionate	16.25	501.2 > 293.1	501.2 > 313.2	tbd	d_5-Fluticasone propionate
Hydrocortisone	8.03	363.2 > 121.1	363.2 > 91.1	tbd	d_4-Hydrocortisone
10-hydroxy-amitriptyline	6.28	294.2 > 276.2	294.2 > 58.0	tbd	d_6-Amitriptyline
Meprobamate	5.88	219.1 > 158.1	219.1 > 97.1	tbd	**d_5-Diazepam**
Methylprednisolone	9.75	375.2 > 357.1	375.2 > 161.1	tbd	d_3-Methylprednisolone
Metoprolol	4.94	268.2 > 116.1	268.2 > 72.1	tbd	d_7-Metoprolol
Norfluoxetine	12.68	296.1 > 134.1	296.1 > 30.0	tbd	d_5-Norfluoxetine
Norverapamil	11.6	441.3 > 165.1	441.3 > 150.1	tbd	d_7-Propranolol
Paroxetine	10.97	330.1 > 70.0	330.1 > 192.1	tbd	d_6-Paroxetine
Prednisolone	7.92	361.1 >147.1	361.1 >343.2	tbd	d_8-Prednisone
Prednisone	7.88	359.15 > 147.1	359.15 > 341.1	tbd	d_8-Prednisone
Promethazine	10.12	285.13 > 86.11	285.13 > 198.0	tbd	d_4-Promethazine

Propoxyphene	11.96	340.2 > 58.0	340.2 > 266.2	tbd	d$_5$-Propoxyphene
Propranolol	7.77	260.2 > 116.1	260.2 > 56.0	tbd	d$_7$-Propranolol (ring d$_7$)
Sertraline	13.28	306.07 > 159	308.07 > 161.0	tbd	d$_3$-Sertraline
Simvastatin	17.42	436.2 > 199.2	436.2 > 419.2	tbd	d$_6$-Simvastatin
Theophylline	3.32	181.01 > 124.0	181.1 > 42.0	tbd	^{13}C,^{15}N$_2$-Theophylline
Trenbolone	10.27	271.2 > 199.1	271.2 > 253.1	tbd	d$_5$-Trenbolone
Trenbolone acetate	15.8	313.2 > 253.2	313.2 > 107	tbd	**d$_5$-Trenbolone**
Valsartan	14.43	436.2 > 235.1	436.2 > 291.2	tbd	d$_3$-Valsartan
Verapamil	11.98	455.2 > 165.1	455.2 > 150.1	tbd	d$_7$-Verapamil
Surrogate Standards					
d$_5$-Alprazolam	11.25	314.1 > 286.1	314.1 > 210.1	tbd	d$_4$-Amlodipine (2-chlorophenyl-d$_4$) d$_7$-Propranolol (1-methylethyl-d$_7$)
d$_6$-Amitriptyline	12.01	284.2 > 105.0	284.2 > 90.9	tbd	d$_4$-Amlodipine (2-chlorophenyl-d$_4$) d$_7$-Propranolol (1-methylethyl-d$_7$)
d$_4$-Amlodipine (2-aminoethoxy-d$_4$)	12.04	413.1 > 239.0	414.9 > 240.0	tbd	d$_4$-Amlodipine (2-chlorophenyl-d$_4$)
d$_8$-Benzoylecgonine	4.04	298.1 > 171.1	298.1 > 110.1	tbd	d$_4$-Amlodipine (2-chlorophenyl-d$_4$) d$_7$-Propranolol (1-methylethyl-d$_7$)
d$_3$-Benztropine	12.48	311.2 > 167.1	311.2 > 152.0	tbd	d$_4$-Amlodipine (2-chlorophenyl-d$_4$) d$_7$-Propranolol (1-methylethyl-d$_7$)
d$_5$-Betamethasone	9.81	398.1 > 378.0	398.1 > 360.1	tbd	d$_4$-Amlodipine (2-chlorophenyl-d$_4$) d$_7$-Propranolol (1-methylethyl-d$_7$)
d$_3$-Cocaine	5.59	307.2 > 185.2	307.2 > 85.1	tbd	d$_4$-Amlodipine (2-chlorophenyl-d$_4$) d$_7$-Propranolol (1-methylethyl-d$_7$)
d$_7$-DEET	10.46	199.1 > 126.0	199.1 > 97.9	tbd	d$_4$-Amlodipine (2-chlorophenyl-d$_4$) d$_7$-Propranolol (1-methylethyl-d$_7$)
d$_4$-Desmethyldiltiazem	9.7	405.1 > 182.1	405.1 > 110.0	tbd	d$_4$-Amlodipine (2-chlorophenyl-d$_4$) d$_7$-Propranolol (1-methylethyl-d$_7$)
d$_5$-Diazepam	14	290.1 > 154.1	290.1 > 198.1	tbd	d$_4$-Amlodipine (2-chlorophenyl-d$_4$) d$_7$-Propranolol (1-methylethyl-d$_7$)
d$_5$-Fluticasone propionate	16.21	506.2 > 293.1	506.2 > 313.1	tbd	d$_4$-Amlodipine (2-chlorophenyl-d$_4$) d$_7$-Propranolol (1-methylethyl-d$_7$)

d_4-Hydrocortisone	8.01	367.2 > 121.1	367.2 > 91.0	tbd	d_4-Amlodipine (2-chlorophenyl-d_4) d_7-Propranolol (1-methylethyl-d_7)
d_3-Methylpredinisolone	9.72	378.2 > 360.2	378.2 > 342.1	tbd	d_4-Amlodipine (2-chlorophenyl-d_4) d_7-Propranolol (1-methylethyl-d_7)
d_7-Metoprolol	4.93	275.2 > 123.1	275.2 > 79.1	tbd	d_4-Amlodipine (2-chlorophenyl-d_4) d_7-Propranolol (1-methylethyl-d_7)
d_5-Norfluoxetine	12.67	301.1 > 139.1	301.1 > 32.0	tbd	d_4-Amlodipine (2-chlorophenyl-d_4) d_7-Propranolol (1-methylethyl-d_7)
d_6-Paroxetine	10.94	336.2 > 76.1	336.2 > 198.2	tbd	d_4-Amlodipine (2-chlorophenyl-d_4) d_7-Propranolol (1-methylethyl-d_7)
d_8-Prednisone	7.8	367.2 > 150.1	367.2 > 349.1	tbd	d_4-Amlodipine (2-chlorophenyl-d_4) d_7-Propranolol (1-methylethyl-d_7)
d_4-Promethazine	10.06	289.1 > 86.1	289.1 > 202.0	tbd	d_4-Amlodipine (2-chlorophenyl-d_4) d_7-Propranolol (1-methylethyl-d_7)
d_5-Propoxyphene	11.89	345.3 > 58.1	345.3 > 266.2	tbd	d_4-Amlodipine (2-chlorophenyl-d_4) d_7-Propranolol (1-methylethyl-d_7)
d_7-Propranolol (ring-d_7)	7.67	267.2 > 116.1	267.2 > 72.1	tbd	d_7-Propranolol (1-methylethyl-d_7)
d_3-Sertraline	13.27	309.1 > 159.0	311.1 > 160.9	tbd	d_4-Amlodipine (2-chlorophenyl-d_4) d_7-Propranolol (1-methylethyl-d_7)
d_6-Simvastatin	17.41	442.3 > 199.2	442.3 > 425.3	tbd	d_4-Amlodipine (2-chlorophenyl-d_4) d_7-Propranolol (1-methylethyl-d_7)
$^{13}C_1$,$^{15}N_2$-Theophylline	3.32	184.1 > 125.0	184.1 > 43.0	tbd	d_4-Amlodipine (2-chlorophenyl-d_4) d_7-Propranolol (1-methylethyl-d_7)
d_5-Trenbolone	10.21	276.2 > 204.1	276.2 > 258.1	tbd	d_4-Amlodipine (2-chlorophenyl-d_4) d_7-Propranolol (1-methylethyl-d_7)
d_3-Valsartan	14.41	439.1 > 235.1	439.1 > 294.2	tbd	d_4-Amlodipine (2-chlorophenyl-d_4) d_7-Propranolol (1-methylethyl-d_7)
d_7-Verapamil	11.89	462.2 > 165.1	462.2 > 150.1	tbd	d_4-Amlodipine (2-chlorophenyl-d_4) d_7-Propranolol (1-methylethyl-d_7)
Recovery Standards					
d_4-Amlodipine (2-chlorophenyl-d_4)	12.01	413.1 > 242.0	414.9 > 244.0	tbd	External Standard
d_7-Propranolol (1-methylethyl-d_7)	7.75	267.2 > 123.1	267.2 > 79.1	tbd	External Standard

PPCP List 6 – Acid Extraction, Positive Electrospray Ionization (+)ESI

Target Analyte	**Typical Retention Time (min)**	**Primary MRM Transition**	**Secondary MRM Transition**	**Typical Transition Ratio (Secondary Product Ion/Primary Product Ion)**	**Quantification and RRT Reference[1]**
Amsacrine	8.3	394.3 > 315.2	394.3 > 179.2	tbd	d_6-Citalopram
Azathioprine	5.05	278.1 > 142.1	278.1 > 232.1	tbd	$^{13}C_4$-Azathioprine
Busulfan	6.09	264.2 > 151.1	264.2 > 247.1	tbd	d_8-Busulfan
Citalopram	10.04	325.3 > 109.1	325.3 > 262.2	tbd	d_6-Citalopram
Clotrimazole	14.61	277.3 > 165.2	277.3 > 241.2	tbd	d_5-Clotrimazole
Colchicine	8.96	400.3 > 358.2	400.3 > 310.2	tbd	d_6-Colchicine
Cyclophosphamide	8.06	261.2 > 140.1	261.2 > 106.0	tbd	d_4-Cyclophosphamide
Daunorubicin	10.44	528.3 > 321.2	528.3 > 381.1	tbd	$^{13}C,d_3$-Daunorubicin
Diatrizoic acid	3.61	631.8 > 361.1	631.8 > 233.1	tbd	d_6-Diatrizoic acid
Doxorubicin	8.89	544.2 > 397.1	544.2 > 130.1	tbd	$^{13}C,d_3$-Daunorubicin
Drospirenone	14.37	367.3 > 97.1	367.3 > 91.1	tbd	$^{13}C_3$-Drospirenone
Etoposide	9.53	606.3 > 229.2	606.3 > 185.1	tbd	d_3-Etoposide
Iopamidol	3.50	794.8 > 777.9	794.8 > 558.9	tbd	d8-Iopamidol
Medroxyprogesterone acetate	16.96	387.3 > 123.1	387.3 > 327.3	tbd	d_6-Medroxyprogesterone acetate
Melphalan	8.41	305.2 > 288.1	305.2 > 246.1	tbd	d_8-Melphalan
Metronidazole	4.31	172.2 > 128.1	172.2 > 82.0	tbd	d_4-Metronidazole
Moxifloxacin	7.47	402.3 > 384.2	402.3 > 96.1	tbd	$^{13}C,d_3$-Moxifloxacin
Oxazepam	11.26	287.1 > 241.1	287.1 > 269.1	tbd	d_5-Oxazepam
Rosuvastatin	12.77	482.3 > 258.2	482.3 > 300.2	tbd	d_6-Rosuvastatin
Tamoxifen	16.48	372.4 > 72.1	372.4 > 44.2	tbd	d_5-Tamoxifen
Teniposide	12.64	674.3 > 229.1	674.3 > 383.2	tbd	d_3-Etoposide
Venlafaxine	8.27	278.4 > 58.1	278.4 > 260.3	tbd	d_5-Venlafaxine
Zidovudine	5.50	268.3 > 127.1	268.3 > 109.9	tbd	d_3-Zidovidine
Surrogate Standards					
$^{13}C_4$-Azathioprine	5.05	282.1 > 146.1	282.1 > 236.1	tbd	d_4-Amlodipine (2-chlorophenyl-d_4) d_7-Propranolol (1-methylethyl-d_7)
d_8-Busulfan	6.03	272.2 > 159.1	272.2 > 262.2	tbd	d_4-Amlodipine (2-chlorophenyl-d_4) d_7-Propranolol (1-methylethyl-d_7)

d_6-Citalopram	10.03	331.3 > 109.0	331.3 > 82.1	tbd	d_4-Amlodipine (2-chlorophenyl-d_4)
d_5-Clotrimazole	14.55	282.2 > 169.9	282.2 > 247.2	tbd	d_4-Amlodipine (2-chlorophenyl-d_4) d_7-Propranolol (1-methylethyl-d_7)
d_6-Colchicine	8.9	406.3 > 362.3	406.3 > 285.2	tbd	d_4-Amlodipine (2-chlorophenyl-d_4) d_7-Propranolol (1-methylethyl-d_7)
d_4-Cyclophosphamide	8.01	265.2 > 140.0	265.2 > 106.0	tbd	d_4-Amlodipine (2-chlorophenyl-d_4) d_7-Propranolol (1-methylethyl-d_7)
$^{13}C_1$-d_3-Daunorubicin	10.41	532.2 > 325.1	532.2 > 385.2	tbd	d_4-Amlodipine (2-chlorophenyl-d_4) d_7-Propranolol (1-methylethyl-d_7)
d_6-Diatrizoic acid	3.6	637.9 > 367.1	637.9 > 620.8	tbd	d_4-Amlodipine (2-chlorophenyl-d_4) d_7-Propranolol (1-methylethyl-d_7)
$^{13}C_3$-Drospirenone	14.37	370.2 > 97.1	370.2 > 91.2	tbd	d_4-Amlodipine (2-chlorophenyl-d_4) d_7-Propranolol (1-methylethyl-d_7)
d_3-Etoposide	9.49	609.2 > 229.1	609.2 > 185.1	tbd	d_4-Amlodipine (2-chlorophenyl-d_4) d_7-Propranolol (1-methylethyl-d_7)
d_8-Iopamidol	3.51	802.9 > 785.8	802.9 > 562.9	tbd	d_4-Amlodipine (2-chlorophenyl-d_4) d_7-Propranolol (1-methylethyl-d_7)
d_6-Medroxyprogesterone acetate	16.93	393.2 > 126.1	393.2 > 330.3	tbd	d_4-Amlodipine (2-chlorophenyl-d_4) d_7-Propranolol (1-methylethyl-d_7)
d_8-Melphalan	8.34	313.1 > 296.1	313.1 > 254.2	tbd	d_4-Amlodipine (2-chlorophenyl-d_4) d_7-Propranolol (1-methylethyl-d_7)
d_4-Metronidazole	4.29	176.2 > 128.1	176.2 > 87.1	tbd	d_4-Amlodipine (2-chlorophenyl-d_4) d_7-Propranolol (1-methylethyl-d_7)
$^{13}C_1$-d_3-Moxifloxacin	7.45	406.3 > 388.2	406.3 > 110.1	tbd	d_4-Amlodipine (2-chlorophenyl-d_4) d_7-Propranolol (1-methylethyl-d_7)
d_5-Oxazepam	11.18	292.1 > 246.1	292.1 > 274.1	tbd	d_4-Amlodipine (2-chlorophenyl-d_4) d_7-Propranolol (1-methylethyl-d_7)
d_6-Rosuvastatin	12.73	488.3 > 264.2	488.3 > 306.2	tbd	d_4-Amlodipine (2-chlorophenyl-d_4) d_7-Propranolol (1-methylethyl-d_7)
d_5-Tamoxifen	16.45	377.3 > 72.1	377.3 > 44.2	tbd	d_4-Amlodipine (2-chlorophenyl-d_4) d_7-Propranolol (1-methylethyl-d_7)

d_6-Venlafaxine	8.26	284.4 > 64.1	284.4 > 266.3	tbd	d_4-Amlodipine (2-chlorophenyl-d_4) d_7-Propranolol (1-methylethyl-d_7)
d_3-Zidovudine	5.49	271.2 > 130.1	271.2 > 126.1	tbd	d_7-Propranolol (1-methylethyl-d_7)
Recovery Standards					
d_4-Amlodipine (2-chlorophenyl-d_4)	11.85	413.1 > 242.0	414.9 > 244.0	tbd	External Standard
d_7-Propranolol (1-methylethyl-d_7)	9.04	267.2 > 123.1	267.2 > 79.1	tbd	External Standard

* = Confirmation ions in instances of interference
** = Parent ion monitored from the breakdown product

Hormones (HM-APOS) – Positive Electrospray Ionization (+)ESI

Target Analyte	Typical Retention Time (min)	Primary MRM Transition	Secondary MRM Transition	Typical Ratio (Secondary Product Ion/Primary Product Ion)	Quantification and RT Reference[1]
Allyl trenbolone	6.2	311.4 > 227.3	311.4 > 159.2	tbd	d_6-Norethindrone
Androstenedione	6.0	287.1 > 97.0	287.1 > 109.0	tbd	$^{13}C_3$-Androstenedione
Androsterone*	6.4	291.2 > 255.2	291.2 > 273.3	tbd	d_4-Androsterone
Desogestrel	10.1	311.3 > 67.0	311.3 > 135.1	tbd	$^{13}C_2$,d_2-Desogestrel
17beta-Estradiol 3-benzoate*	7.5	377.2 > 105.0	377.2 > 77.0	tbd	d_3-17beta-Estradiol 3-benzoate
Mestranol*	7.0	311.2 > 121.1	293.0 > 146.7	tbd	d_4-Mestranol
Norethindrone	5.5	299.4 > 109.1	299.4 > 91.1	tbd	d_6-Norethindrone
Norgestrel	6.2	313.4 > 109.1	313.4 > 91.1	tbd	d_6-Norgestrel
Progesterone	6.4	315.1 > 97.0	315.1 > 109.0	tbd	d_9-Progestrone

Testosterone	5.5	289.1 > 97.0	289.1 > 109.0	tbd	$^{13}C_3$-Testosterone
Surrogate or Extracted Internal Standard					
$^{13}C_3$-Androstenedione	6.0	290.5 > 100.0	290.5 > 112.0	tbd	$^{13}C_3$-17alpha-Hydroxyprogesterone
d_4-Androsterone	6.4	295.4 > 259.3	295.4 > 277.3	tbd	$^{13}C_3$-17alpha-Hydroxyprogesterone
$^{13}C_2$,d_2-Desogestrel	10.1	315.5 > 135.0	315.5 > 93.0	tbd	$^{13}C_3$-17alpha-Hydroxyprogesterone
d_3-17beta-Estradiol 3-benzoate	7.5	380.4 > 105.5	380.4 > 77.0	tbd	$^{13}C_3$-17alpha-Hydroxyprogesterone
d_4-Mestranol	7.0	315.4 > 123.1	315.4 > 161.3	tbd	$^{13}C_3$-17alpha-Hydroxyprogesterone
d_6-Norethindrone	5.5	305.4 > 86.7	305.4 > 113.3	tbd	$^{13}C_3$-17alpha-Hydroxyprogesterone
d_6-Norgestrel	6.2	319.5 > 251.4	319.5 > 91.2	tbd	$^{13}C_3$-17alpha-Hydroxyprogesterone
d_9-Progestrone	6.4	324.3 > 100.0	324.3 > 113.0	tbd	$^{13}C_3$-17alpha-Hydroxyprogesterone
$^{13}C_3$-Testosterone	5.5	292.6 > 100.0	292.6 > 112.0	tbd	$^{13}C_3$-17alpha-Hydroxyprogesterone
Recovery or Injection Internal Standard					
$^{13}C_3$-17alpha-Hydroxyprogesterone	6.1	334.3 > 100.0	n.a.		External Standard

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[1] The primary transition of the quantification reference is used as the quantification reference for the primary transition of each analyte: the secondary transition of the quantification reference is used as the quantification reference for the secondary transition of each analyte.

* Third MRM transitions are available for some compounds and can be used if interferences are present in either the primary or secondary transition. 255.0 > 198.8 can also be used for androsterone. 377.2 > 135.1 can also be used for 17beta-Estradiol 3-benzoate. 293.0 > 173.0 can also be used for mestranol. 380.4 > 138.0 can be used for d_3-17beta-Estradiol 3-benzoate.

Hormones (HM-ANEG) – Negative Electrospray Ionization (-)ESI

Target Analyte	Typical Retention Time (min)	Primary MRM Transition	Secondary MRM Transition	Typical Ratio (Secondary Product Ion/Primary Product Ion)	Quantification Reference [1]
17alpha-Dihydroequilin	5.6	269.2 > 267.3	269.2 > 211.2	tbd	d_4-17beta-Estradiol
Equilenin	5.6	264.8 > 221.3	264.8 > 249.2	tbd	d_3-Equilenin
Equilin	6.1	267.2 > 265.1	267.2 > 143.0	tbd	d_4-17beta-Estradiol
17alpha-Estradiol	6.1	271.4 > 145.1	271.4 > 183.2	tbd	d_3-17alpha-Estradiol
17beta-Estradiol	5.5	271.4 > 145.1	271.4 > 183.2	tbd	d_4-17beta-Estradiol
Estriol	4.5	287.3 > 171.0	287.3 > 145.0	tbd	d_3-Estriol
Estrone	6.2	269.4 > 145.2	269.4 > 159.2	tbd	$^{13}C_3$-Estrone
17alpha-Ethinylestradiol	6.1	295.3 > 145.0	295.3 > 159.0	tbd	$^{13}C_2$-17alpha-Ethinylestradiol
Surrogate Standard				tbd	
d_3-Equilenin	5.6	268.2 > 222.2	268.2 > 252.1	tbd	d_4-Estrone
d_3-17alpha-Estradiol	6.1	274.4 > 145.0	274.4 > 185.0	tbd	d_4-Estrone
d_4-17beta-Estradiol	5.5	275.4 > 147.1	275.4 > 187.0	tbd	d_4-Estrone
d_3-Estriol	4.5	290.2 > 173.1	290.2 > 147.1	tbd	d_4-Estrone
$^{13}C_3$-Estrone	6.2	272.3 > 148.0	272.3 > 162.0	tbd	d_4-Estrone
$^{13}C_2$-17alpha-Ethinylestradiol	6.1	297.2 >145.1	297.2 > 199.1	tbd	d_4-Estrone
Recovery Standard					
d_4-Estrone	6.2	273.2 > 147.0	n.a.		External Standard

[1] The primary transition of the quantification reference is used as the quantification reference for the primary transition of each analyte: the secondary transition of the quantification reference is used as the quantification reference for the secondary transition of each analyte.

* Third MRM transitions are available for some compounds and can be used if interferences are present in either the primary or secondary transition. 267.2 > 239.1 can be used for equilin.269.4 > 183.3 can be used for estrone.

5.0 QUALITY ACCEPTANCE CRITERIA

Samples are analyzed in batches consisting of a maximum of twenty samples, one procedural blank and one spiked matrix (OPR) sample. A duplicate is analyzed, provided there is sufficient sample, with batches containing 7-20 samples. A Matrix Spike (MS) Sample prepared by spiking target analytes into a client sample, should be performed with every soil/sediment batch containing 7 or more test samples. Matrix spike/matrix spike duplicate (MS/MSD) pairs may be analyzed on an individual contract basis. The batch is carried through the complete analytical process as a unit. For sample data to be reportable, the batch QC data must meet the established acceptance criteria presented on the analysis reports.

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QC Acceptance Limits [1], Aqueous, POCIS [5], Solid[11] and Tissue [10] Samples

	Aqueous Samples (and POCIS [5])			**Solid/Biosolid[10] Samples**			**Tissue Samples**		
	OPR recovery (% rec.)	**Surrogate recovery in samples (% rec.)**	**Blank limit [4] (ng)**	**OPR recovery (% rec.)**	**Surrogate recovery in samples (% rec.)**	**Blank limit [4] (ng)**	**OPR recovery (% rec.)**	**Surrogate recovery in samples (% rec.)**	**Blank limit [4] (ng)**
List 1 Compounds (APOS)									
Acetaminophen	70-140		≤15	70-130		≤15	70-130		≤15
Azithromycin	10-130		≤1.5	70-160		≤1.5	70-250		≤1.5
Caffeine	25-160		≤15	70-135		≤15	70-130		≤15
Carbadox	25-180		≤1.5	30-130		≤1.5	10-130		≤1.5
Carbamazepine	25-200		≤1.5	70-160		≤1.5	70-150		≤1.5
Cefotaxime	10-300		≤6	65-300		≤6	70-300		≤6
Ciprofloxacin	25-180		≤6	70-180		≤6	70-130		≤6
Clarithromycin	50-160		≤1.5	50-200		≤1.5	70-250		≤1.5
Clinafloxacin	25-300		≤6	70-180		≤6	70-200		≤6
Cloxacillin [2]	70-130		≤3	70-220		≤3	70-250		≤3
Dehydronifedipine	35-160		≤0.6	70-180		≤0.6	70-200		≤0.6
Digoxigenin	50-150		≤6	70-160		≤6	50-200		≤6
Digoxin	35-200		≤6	60-180		≤6	70-250		≤6
Diltiazem	20-160		≤0.3	70-135		≤0.3	70-200		≤0.3
1,7-Dimethylxanthine	30-300		≤60	70-180		≤60	70-250		≤60
Diphenhydramine	70-130		≤0.6	70-150		≤0.6	60-130		≤0.6
Enrofloxacin	30-220		≤3	70-150		≤3	70-130		≤3
Erythromycin - H_2O	70-130		≤0.3 [3]	70-145		≤0.3 [3]	70-130		≤0.3 [3]

	Aqueous Samples (and POCIS [5])			Solid/Biosolid[10] Samples			Tissue Samples		
	OPR recovery (% rec.)	**Surrogate recovery in samples (% rec.)**	**Blank limit [4] (ng)**	**OPR recovery (% rec.)**	**Surrogate recovery in samples (% rec.)**	**Blank limit [4] (ng)**	**OPR recovery (% rec.)**	**Surrogate recovery in samples (% rec.)**	**Blank limit [4] (ng)**
Flumequine	40-160		≤1.5	70-180		≤1.5	60-200		≤1.5
Fluoxetine	60-150		≤1.5	65-135		≤1.5	70-130		≤1.5
Lincomycin	10-300		≤3	40-250		≤3	70-300		≤3
Lomefloxacin	50-250		≤3	70-160		≤3	70-150		≤3
Miconazole	35-130		≤1.5	55-145		≤1.5	5-130		≤1.5
Norfloxacin	10-250		≤15	70-200		≤15	70-150		≤15
Norgestimate	35-130		≤3	40-130		≤3	5-130		≤3
Ofloxacin	60-250		≤1.5	70-180		≤1.5	70-200		≤1.5
Ormetoprim	70-150		≤0.6	70-145		≤0.6	70-130		≤0.6
Oxacillin [2]	20-130		≤3	70-180		≤3	70-200		≤3
Oxolinic Acid	60-150		≤0.6	70-180		≤0.6	70-130		≤0.6
Penicillin G [2]	10-130		≤3	70-200		≤3	20-130		≤3
Penicillin V	40-140		≤3	70-250		≤3	70-250		≤3
Roxithromycin	50-140		≤0.3	45-160		≤0.3	50-200		≤0.3
Sarafloxacin	50-200		≤15	70-180		≤15	50-130		≤15
Sulfachloropyridazine	60-160		≤1.5	70-200		≤1.5	70-200		≤1.5
Sulfadiazine	70-130		≤1.5	70-180		≤1.5	70-300		≤1.5
Sulfadimethoxine	35-160		≤0.3	50-130		≤0.3	70-130		≤0.3
Sulfamerazine	60-140		≤0.6	70-135		≤0.6	70-200		≤0.6
Sulfamethazine	70-130		≤0.6	70-135		≤0.6	70-130		≤0.6

	Aqueous Samples (and POCIS [5])			**Solid/Biosolid[10] Samples**			**Tissue Samples**		
	OPR recovery (% rec.)	**Surrogate recovery in samples (% rec.)**	**Blank limit [4] (ng)**	**OPR recovery (% rec.)**	**Surrogate recovery in samples (% rec.)**	**Blank limit [4] (ng)**	**OPR recovery (% rec.)**	**Surrogate recovery in samples (% rec.)**	**Blank limit [4] (ng)**
Sulfamethizole	30-140		≤0.6	55-135		≤0.6	60-130		≤0.6
Sulfamethoxazole	70-130		≤0.6	70-130		≤0.6	70-130		≤0.6
Sulfanilamide	2-160		≤15	50-150		≤15	50-300		≤15
Sulfathiazole	30-180		≤1.5	35-130		≤1.5	70-130		≤1.5
Thiabendazole	60-150		≤1.5	70-160		≤1.5	70-130		≤1.5
Trimethoprim	50-150		≤1.5	70-135		≤1.5	70-130		≤1.5
Tylosin	70-130		≤6	30-145		≤6	60-200		≤6
Virginiamycin M1	15-300		≤3	70-180		≤3	30-200		≤3
Surrogate Standard									
$^{13}C_2$, ^{15}N-Acetaminophen	30-160	30-160		50-160	30-160		30-150	30-250	
$^{13}C_3$-Caffeine	40-140	40-140		50-150	40-140		30-150	20-250	
d_{10}-Carbamazepine	40-150	40-150		50-150	40-150		30-150	30-150	
$^{13}C_3$, ^{15}N-Ciprofloxacin	7-150	7-150		40-160	7-150		30-150	30-200	
$^{13}C_2$-Erythromycin - H_2O	35-130	35-130		40-180	35-130		30-206	5-200	
d_5-Fluoxetine	10-160	10-160		50-160	10-160		30-150	20-150	
$^{13}C_6$-Sulfamethazine	30-160	30-160		40-150	30-160		30-150	30-150	
$^{13}C_6$-Sulfamethoxazole	30-140	30-140		50-180	30-140		30-150	10-150	
d_6-Thiabendazole	25-180	25-180		30-150	25-180		30-150	30-150	
$^{13}C_3$-Trimethoprim	30-140	30-140		40-150	30-140		30-150	30-200	
Recovery Standard									

	Aqueous Samples (and POCIS [5])			Solid/Biosolid[10] Samples			Tissue Samples		
	OPR recovery (% rec.)	Surrogate recovery in samples (% rec.)	Blank limit [4] (ng)	OPR recovery (% rec.)	Surrogate recovery in samples (% rec.)	Blank limit [4] (ng)	OPR recovery (% rec.)	Surrogate recovery in samples (% rec.)	Blank limit [4] (ng)
$^{13}C_3$-Atrazine									
List 2 Compounds (TCYC)									
Anhydrochlortetracycline (ACTC)	15-200		≤15	9-145		≤15			
Anhydrotetracycline (ATC)	20-160		≤15	25-150		≤15			
Chlortetracycline (CTC)	30-250		≤6	40-180		≤6			
Demeclocycline	35-180		≤15	20-130		≤15			
Doxycycline	35-180		≤6	35-220		≤6			
Epianhydrochlortetracycline (EACTC)	6-130		≤60	5-130		≤60			
Epianhydrotetracycline (EATC)	15-200		≤15	20-160		≤15			
Epichlortetracycline (ECTC)	25-180		≤15	30-200		≤15			
Epioxytetracycline (EOTC)	25-180		≤6	9-145		≤6			
Epitetracycline (ETC)	35-200		≤6	20-250		≤6			
Isochlortetracycline (ICTC)	25-180		≤6	35-140		≤6			
Minocycline	1-250		≤60	9-400		≤60			
Oxytetracycline (OTC)	20-200		≤6	15-150		≤6			
Tetracycline (TC)	20-200		≤6	25-180		≤6			
Surrogate Standard									
d_6-Thiabendazole	25-140	25-140		30-150	25-140				
Recovery Standard									
$^{13}C_3$-Atrazine									

	Aqueous Samples (and POCIS [5])			**Solid/Biosolid[10] Samples**			**Tissue Samples**		
	OPR recovery (% rec.)	**Surrogate recovery in samples (% rec.)**	**Blank limit [4] (ng)**	**OPR recovery (% rec.)**	**Surrogate recovery in samples (% rec.)**	**Blank limit [4] (ng)**	**OPR recovery (% rec.)**	**Surrogate recovery in samples (% rec.)**	**Blank limit [4] (ng)**
List 3 Compounds (ANEG)									
Bisphenol A	70-130		≤500	70-130		≤500	70-150		≤500
Furosemide	70-130		≤40	70-130		≤40	70-130		≤40
Gemfibrozil	70-130		≤1.5	70-130		≤1.5	70-140		≤1.5
Glipizide	70-130		≤6	70-130		≤6	70-140		≤6
Glyburide	70-130		≤3	70-130		≤3	70-150		≤3
Hydrochlorothiazide	70-130		≤20	70-130		≤20	70-130		≤20
2-hydroxy-ibuprofen	70-130		≤80	70-130		≤80	70-130		≤80
Ibuprofen	70-130		≤15	70-130		≤15	70-130		≤15
Naproxen	70-130		≤3	70-135		≤3	70-150		≤3
Triclocarban	70-130		≤3	70-145		≤3	70-130		≤3
Triclosan	70-130		≤60	70-140		≤60	70-150		≤60
Warfarin	70-150		≤1.5	70-140		≤1.5	70-150		≤1.5
Surrogate Standards									
d_6-Bisphenol A	50-150	50-150		50-150	50-150		50-150	50-150	
d_5-Furosemide	50-150	50-150		10-150	10-150		10-150	10-150	
d_6-Gemfibrozil	50-150	50-150		50-150	50-150		50-150	50-150	
d_{11}-Glipizide	50-150	50-150		50-150	50-150		50-150	50-150	
d_3-Glyburide	50-150	50-150		50-150	50-150		50-150	50-150	
$^{13}C_1$-d_2-Hydrochlorothiazide	22-160	22-160		45-150	45-150		35-150	35-150	

	Aqueous Samples (and POCIS [5])			Solid/Biosolid[10] Samples			Tissue Samples		
	OPR recovery (% rec.)	**Surrogate recovery in samples (% rec.)**	**Blank limit [4] (ng)**	**OPR recovery (% rec.)**	**Surrogate recovery in samples (% rec.)**	**Blank limit [4] (ng)**	**OPR recovery (% rec.)**	**Surrogate recovery in samples (% rec.)**	**Blank limit [4] (ng)**
d_6-2-hydroxy-Ibuprofen	39-150	39-150		50-150	50-150		50-150	50-150	
$^{13}C_6$-Ibuprofen	50-150	50-150		28-150	28-150		50-150	50-150	
d_3-Naproxen	50-150	50-150		50-150	50-150		50-150	50-150	
$^{13}C_6$-Triclocarban	35-150	35-150		18-150	18-150		13-150	13-150	
$^{13}C_6$-Triclosan	50-150	50-150		20-150	20-150		40-150	40-150	
d_5-Warfarin	50-150	50-150		50-150	50-150		50-150	50-150	
Recovery Standard									
$^{13}C_3$-Ibuprofen									
$^{13}C_1$-d_3-Naproxen									
List 4 Compounds (BPOS)									
Albuterol	50-160		≤0.3	70-180		≤0.3	60-130		≤0.3
Amphetamine	50-160		≤1.5	70-200		≤1.5	70-130		≤1.5
Atenolol	70-130		≤0.6	70-220		≤0.6	70-130		≤0.6
Atorvastatin	20-130		≤1.5	25-130		≤1.5	70-150		≤1.5
Cimetidine	15-130		≤0.6	70-145		≤0.6	30-130		≤0.6
Clonidine	70-130		≤1.5	70-220		≤1.5	70-130		≤1.5
Codeine	70-130		≤3	70-250		≤3	70-130		≤3
Cotinine	70-130		≤1.5	70-145		≤1.5	70-130		≤1.5
Enalapril	70-130		≤0.3	70-150		≤0.3	70-130		≤0.3
Hydrocodone	70-130		≤1.5	70-220		≤1.5	70-130		≤1.5

	Aqueous Samples (and POCIS [5])			Solid/Biosolid[10] Samples			Tissue Samples		
	OPR recovery (% rec.)	Surrogate recovery in samples (% rec.)	Blank limit [4] (ng)	OPR recovery (% rec.)	Surrogate recovery in samples (% rec.)	Blank limit [4] (ng)	OPR recovery (% rec.)	Surrogate recovery in samples (% rec.)	Blank limit [4] (ng)
Metformin	70-160		≤3	70-200		≤3	70-130		≤3
Oxycodone	65-130		≤0.6	70-180		≤0.6	70-150		≤0.6
Ranitidine	25-140		≤0.6	30-130		≤0.6	NQ-150 [6]		≤0.6
Triamterene	70-140		≤0.3	50-145		≤0.3	70-130		≤0.3
Surrogate Standards									
d$_3$-Albuterol	20-140	20-140		50-150	20-140		20-150	5-150	
d$_5$-Amphetamine	20-130	20-130		40-150	20-130		30-150	5-150	
d$_7$-Atenolol	50-130	50-130		50-150	50-130		30-150	30-300	
d$_5$-Atorvastatin	25-130	20-130		10-130	10-130		40-150	15-150	
d$_3$-Cimetidine	4-130	15-130		30-150	15-130		20-150	NQ-500 [6]	
d$_4$-Clonidine	50-130	50-130		50-160	50-130		30-150	30-300	
d$_6$-Codeine	50-130	50-130		50-150	50-130		10-150	5-150	
d$_3$-Cotinine	50-140	50-140		50-250	50-140		30-150	30-300	
d$_5$-Enalapril	50-130	50-130		50-180	50-130		30-150	10-150	
d$_3$-Hydrocodone	50-130	50-130		40-150	50-130		30-150	20-150	
d$_6$-Metformin	3-130	3-130		10-150	3-130		10-150	5-200	
d$_6$-Oxycodone	50-150	50-150		40-150	50-150		30-150	30-150	
d$_6$-Ranitidine	4-130	10-130		30-130	10-130		10-130	10-130	
d$_5$-Triamterene	50-130	50-130		20-130	50-150		30-150	40-130	
Recovery Standards									

	Aqueous Samples (and POCIS [5])			Solid/Biosolid[10] Samples			Tissue Samples		
	OPR recovery (% rec.)	Surrogate recovery in samples (% rec.)	Blank limit [4] (ng)	OPR recovery (% rec.)	Surrogate recovery in samples (% rec.)	Blank limit [4] (ng)	OPR recovery (% rec.)	Surrogate recovery in samples (% rec.)	Blank limit [4] (ng)
d_9-Albuterol									
d_3-Amitriptyline									
List 5 Compounds (APOSX)									
Alprazolam	70-130		≤0.3	70-130		≤0.15	70-130		≤0.15
Amitriptyline	70-130		≤0.3	70-130		≤0.15	70-130		≤0.15
Amlodipine	60-130		≤1	70-150		≤0.5	70-130		≤0.5
Benzoylecgonine	70-140		≤0.15	70-130		≤0.15	70-130		≤0.15
Benztropine	70-130		≤0.7	70-130		≤0.35	70-150		≤0.35
Betamethasone	70-130		≤1.5	70-130		≤0.75	70-250		≤0.8
Cocaine	70-130		≤0.15	70-130		≤0.15	70-130		≤0.15
DEET	70-160		≤5	70-140		≤3	70-150		≤2
Desmethyldiltiazem	70-130		≤0.15	70-130		≤0.15	60-130		≤0.15
Diazepam	70-130		≤0.5	70-130		≤0.3	70-130		≤0.3
Fluocinonide	70-130		≤2	70-130		≤1	70-140		≤1
Fluticasone propionate	70-130		≤2	70-130		≤1	60-130		≤1
Hydrocortisone	70-130		≤6	70-130		≤3	70-150		≤3
10-hydroxy-amitriptyline	70-130		≤0.15	60-130		≤0.15	40-130		≤0.15
Meprobamate	70-130		≤1.5	70-150		≤1	70-130		≤0.8
Methylprednisolone	70-130		≤4	70-130		≤2	70-130		≤2
Metoprolol	70-130		≤0.5	70-130		≤0.3	70-130		≤0.3

	Aqueous Samples (and POCIS [5])			Solid/Biosolid[10] Samples			Tissue Samples		
	OPR recovery (% rec.)	Surrogate recovery in samples (% rec.)	Blank limit [4] (ng)	OPR recovery (% rec.)	Surrogate recovery in samples (% rec.)	Blank limit [4] (ng)	OPR recovery (% rec.)	Surrogate recovery in samples (% rec.)	Blank limit [4] (ng)
Norfluoxetine	70-130		≤0.5	70-130		≤0.3	70-130		≤0.3
Norverapamil	70-150		≤0.15	70-150		≤0.15	70-160		≤0.15
Paroxetine	70-130		≤1	70-130		≤0.5	60-130		≤0.5
Prednisolone	70-130		≤4	70-130		≤2	60-130		≤2
Prednisone	70-130		≤6	70-145		≤3	70-130		≤3
Promethazine	70-130		≤0.3	70-130		≤0.15	70-130		≤0.15
Propoxyphene	70-130		≤0.3	70-130		≤0.15	70-130		≤0.15
Propranolol	70-130		≤0.3	70-130		≤0.15	70-130		≤0.15
Sertraline	70-130		≤0.3	70-150		≤0.15	60-130		≤0.15
Simvastatin	70-130		≤2	70-130		≤1	70-130		≤1
Theophylline	70-130		≤2	70-130		≤1	70-130		≤1
Trenbolone	70-130		≤2	70-130		≤1	70-130		≤1
Trenbolone acetate	70-130		≤0.3	60-130		≤0.15	70-130		≤0.15
Valsartan	70-130		≤4	70-130		≤2	70-130		≤2
Verapamil	70-130		≤0.15	70-130		≤0.15	70-130		≤0.15
Surrogate Standards									
d_5-Alprazolam	50-150	50-150		50-150	40-150		50-150	30-150	
d_6-Amitriptyline	50-150	50-150		50-150	40-150		50-150	10-150	
d_4-Amlodipine (2-aminoethoxy-d_4)	30-150	30-150		40-150	5-130		50-150	30-150	
d_8-Benzoylecgonine	50-150	30-150		50-150	5-150		50-150	20-150	

	Aqueous Samples (and POCIS [5])			**Solid/Biosolid[10] Samples**			**Tissue Samples**		
	OPR recovery (% rec.)	**Surrogate recovery in samples (% rec.)**	**Blank limit [4] (ng)**	**OPR recovery (% rec.)**	**Surrogate recovery in samples (% rec.)**	**Blank limit [4] (ng)**	**OPR recovery (% rec.)**	**Surrogate recovery in samples (% rec.)**	**Blank limit [4] (ng)**
d_3-Benztropine	40-140	50-150		50-150	5-150		50-150	30-150	
d_5-Betamethasone	50-150	50-150		50-150	40-150		50-150	30-150	
d_3-Cocaine	50-150	50-150		50-150	5-140		50-150	20-150	
d_7-DEET	35-150	30-150		30-150	5-160		30-150	30-150	
d_4-Desmethyldiltiazem	50-150	50-150		50-150	50-150		50-150	20-150	
d_5-Diazepam	50-150	50-150		50-150	20-160		50-150	20-160	
d_5-Fluticasone Propionate	50-150	40-150		20-150	30-150		20-150	20-150	
d_4-Hydrocortisone	40-150	50-150		50-150	30-240		40-150	20-150	
d_3-Methylprednisolone	50-150	50-150		50-150	40-150		50-150	30-150	
d_7-Metoprolol	50-150	40-150		50-150	5-140		50-150	30-150	
d_5-Norfluoxetine	20-130	50-150		50-150	5-130		40-150	5-150	
d_6-Paroxetine	35-130	50-150		50-150	50-150		50-150	20-150	
d_8-Prednisone	40-140	30-150		50-150	20-150		50-150	20-150	
d_4-Promethazine	30-130	40-150		40-150	5-140		40-150	20-150	
d_5-Propoxyphene	50-150	50-150		50-150	5-150		50-150	30-200	
d_7-Propranolol	50-150	50-150		40-150	5-150		50-150	5-150	
d_3-Sertraline	50-150	50-150		50-150	10-150		50-150	20-150	
d_6-Simvastatin	40-140	30-150		40-140	20-150		10-150	10-150	
$^{13}C_1$-$^{15}N_2$-Theophylline	50-150	20-150		30-130	20-200		50-150	10-150	
d_5-Trenbolone	50-150	50-150		20-150	30-150		40-150	20-150	

	Aqueous Samples (and POCIS [5])			Solid/Biosolid[10] Samples			Tissue Samples		
	OPR recovery (% rec.)	Surrogate recovery in samples (% rec.)	Blank limit [4] (ng)	OPR recovery (% rec.)	Surrogate recovery in samples (% rec.)	Blank limit [4] (ng)	OPR recovery (% rec.)	Surrogate recovery in samples (% rec.)	Blank limit [4] (ng)
d_3-Valsartan	50-150	40-150		40-150	20-150		10-150	20-150	
d_7-Verapamil	40-140	50-150		50-150	5-150		50-150	30-150	
Recovery Standards									
$^{13}C_3$-Atrazine									
d_4-Amlodipine (2-chlorophenyl-d_4)									
d_7-Propranolol (1-methylethyl-d_7)									
List 6 Compounds (APOSY) [7]									
Amsacrine	70-130		≤ 0.8	70-130		≤ 1.6	70-130		≤ 0.3
Azathioprine	70-130		≤ 8	70-130		≤ 8	70-130		≤ 1.3
Busulfan	70-130		≤ 8	70-130		≤ 8	70-130		≤ 1.3
Citalopram	70-130		≤ 2	70-130		≤ 1.6	70-130		≤ 0.3
Clotrimazole	70-130		≤ 2	70-130		≤ 1.6	70-130		≤ 0.3
Colchicine	70-130		≤ 2	70-130		≤ 2	70-150		≤ 0.4
Cyclophosphamide	70-130		≤ 1.6	70-130		≤ 8	70-130		≤ 0.3
Daunorubicin	70-130		≤ 8	70-130		≤ 8	70-130		≤ 3
Diatrizoic acid	60-130		≤ 320	70-140		≤ 160	70-130		≤ 26
Doxorubicin	30-130		≤ 80	70-130		≤ 80	50-130		≤ 13
Drospirenone	70-130		≤ 24	70-130		≤ 24	70-130		≤ 4
Etoposide	70-150		≤ 8	70-130		≤ 8	70-130		≤ 3
Iopamidol	70-130		≤ 4	70-130		≤ 4	70-130		≤ 0.8

	Aqueous Samples (and POCIS [5])			**Solid/Biosolid[10] Samples**			**Tissue Samples**		
	OPR recovery (% rec.)	**Surrogate recovery in samples (% rec.)**	**Blank limit [4] (ng)**	**OPR recovery (% rec.)**	**Surrogate recovery in samples (% rec.)**	**Blank limit [4] (ng)**	**OPR recovery (% rec.)**	**Surrogate recovery in samples (% rec.)**	**Blank limit [4] (ng)**
Medroxyprogesterone acetate	60-130		≤ 4	70-130		≤ 4	70-130		≤ 0.8
Melphalan	70-130		≤ 320	70-130		≤ 320	60-130		≤ 52
Metronidazole	70-130		≤ 8	70-130		≤ 8	70-130		≤ 2
Moxifloxacin	70-130		≤ 8	70-130		≤ 8	70-130		≤ 2
Oxazepam	70-130		≤ 4	70-130		≤ 4	70-130		≤ 0.8
Rosuvastatin	70-130		≤ 4	70-130		≤ 20	70-130		≤ 0.8
Tamoxifen	70-130		≤ 1.6	70-130		≤ 1.6	70-130		≤ 0.3
Teniposide	70-130		≤ 4	40-130		≤ 4	70-130		≤ 0.8
Venlafaxine	70-130		≤ 3.2	70-130		≤ 1.6	70-130		≤ 0.3
Zidovudine	70-130		≤ 80	70-130		≤ 80	70-130		≤ 13
Surrogate Standards									
$^{13}C_4$-Azathioprine	50-150	50-150		50-170	50-150		50-150	20-150	
d_8-Busulfan	50-150	50-150		50-150	50-150		50-150	50-150	
d_6-Citalopram	50-150	50-150		50-150	20-150		50-150	20-150	
d_5-Clotrimazole	50-150	50-150		50-150	50-150		50-150	40-150	
d_6-Colchicine	50-150	50-150		50-150	50-150		30-150	40-150	
d_4-Cyclophosphamide	50-150	50-150		50-150	50-150		50-150	40-150	
$^{13}C_1$-d_3-Daunorubicin	40-150	50-150		50-150	30-200		50-150	30-150	
d_6-Diatrizoic acid	5-130	5-150		5-130	5-150		5-150	10-150	
$^{13}C_3$-Drospirenone	50-150	50-150		50-150	50-150		50-150	50-150	

	Aqueous Samples (and POCIS [5])			**Solid/Biosolid[10] Samples**			**Tissue Samples**		
	OPR recovery (% rec.)	**Surrogate recovery in samples (% rec.)**	**Blank limit [4] (ng)**	**OPR recovery (% rec.)**	**Surrogate recovery in samples (% rec.)**	**Blank limit [4] (ng)**	**OPR recovery (% rec.)**	**Surrogate recovery in samples (% rec.)**	**Blank limit [4] (ng)**
d_3-Etoposide	50-150	50-150		50-150	20-150		50-150	20-150	
d_8-Iopamidol	15-150	15-150		10-130	10-150		10-150	40-150	
d_6-Medroxyprogesterone acetate	50-150	50-150		50-150	50-160		50-150	40-150	
d_8-Melphalan	20-150	30-150		10-130	10-150		15-150	10-150	
d_4-Metronidazole	40-150	50-150		50-160	50-150		50-150	40-150	
$^{13}C_1$-d_3-Moxifloxacin	40-150	50-150		50-150	30-160		50-150	50-180	
d_5-Oxazepam	50-150	50-150		50-150	50-160		50-150	50-180	
d_6-Rosuvastatin	50-150	50-150		50-150	50-150		50-150	50-180	
d_5-Tamoxifen	50-150	30-150		50-150	50-150		50-150	40-150	
d_6-Venlafaxine	50-150	50-150		50-150	50-150		50-150	40-150	
d_3-Zidovudine	50-150	50-150		50-160	50-150		50-150	40-150	
Recovery Standards									
$^{13}C_3$-Atrazine									
d_4-Amlodipine (2-chlorophenyl-d_4)									
d_7-Propranolol (1-methylethyl-d_7)									
HM-APOS									
Allyl trenbolone	70-130		≤ 0.8	70-130		≤ 0.8	n.a.		≤ 0.8
Androstenedione	70-130		≤ 2	70-130		≤ 2	70-130		≤ 2
Androsterone	70-130		≤ 20	70-150		≤ 20	70-150		≤ 20

	Aqueous Samples (and POCIS [5])			Solid/Biosolid[10] Samples			Tissue Samples		
	OPR recovery (% rec.)	Surrogate recovery in samples (% rec.)	Blank limit [4] (ng)	OPR recovery (% rec.)	Surrogate recovery in samples (% rec.)	Blank limit [4] (ng)	OPR recovery (% rec.)	Surrogate recovery in samples (% rec.)	Blank limit [4] (ng)
Desogestrel	60-160		≤ 120	70-135		≤ 120	70-130		≤ 120
17beta-Estradiol 3-benzoate	65-150			70-130			n.a.		
Mestranol	70-150		≤ 40	70-135		≤ 40	45-130		≤ 40
Norethindrone	70-130		≤ 4	70-130		≤ 4	70-130		≤ 4
Norgestrel	70-130		≤ 4	65-150		≤ 4	50-150		≤ 4
Progesterone	70-130		≤ 0.8	70-140		≤ 0.8	70-130		≤ 0.8
Testosterone	70-130		≤ 0.8	70-130		≤ 0.8	70-130		≤ 0.8
Surrogate Standards									
$^{13}C_3$-Androstenedione	50-150	50-150		50-150	50-150		50-150	50-150	
d_4-Androsterone	50-150	50-150		50-160	50-160		14-800	14-800	
$^{13}C_2,d_2$-Desogestrel	25-150	25-150		12-150	12-150		3-180	3-180	
d_3-17beta-Estradiol 3-benzoate	35-150	35-150		30-150	30-150		n.a.	n.a.	
d_4-Mestranol	45-150	45-150		50-150	50-150		35-400	35-400	
d_6-Norethindrone	50-150	50-150		50-200	50-200		50-180	50-180	
d_6-Norgestrel	50-150	50-150		50-160	50-160		50-150	50-150	
d_9-Progesterone	50-150	50-150		50-150	50-150		50-150	50-150	
$^{13}C_3$-Testosterone	50-150	50-150		50-170	50-170		50-200	50-200	
Recovery Standards									
$^{13}C_3$-17alpha-Hydroxyprogesterone									

	Aqueous Samples (and POCIS [5])			Solid/Biosolid[10] Samples			Tissue Samples		
	OPR recovery (% rec.)	Surrogate recovery in samples (% rec.)	Blank limit [4] (ng)	OPR recovery (% rec.)	Surrogate recovery in samples (% rec.)	Blank limit [4] (ng)	OPR recovery (% rec.)	Surrogate recovery in samples (% rec.)	Blank limit [4] (ng)
HM-ANEG									
17alpha-Dihydroequilin	70-130		≤ 4	70-130		≤ 4	40-130		≤ 4
Equilenin	70-150		≤ 0.8	70-150		≤ 0.8	70-220		≤ 0.8
Equilin	70-130		≤ 8	70-130		≤ 8	70-130		≤ 8
17alpha-Estradiol	70-140		≤ 4	70-130		≤ 4	70-130		≤ 4
17beta-Estradiol	70-130		≤ 4	70-130		≤ 4	70-130		≤ 4
Estriol	70-140		≤ 48	70-130		≤ 48	70-130		≤ 48
Estrone	70-130		≤ 4	70-130		≤ 4	70-130		≤ 4
17alpha-Ethinylestradiol	70-150		≤ 20	70-130		≤ 20	65-150		≤ 20
Surrogate Standards									
d_3-Equilenin	50-150	50-150		50-150	50-150		45-150	45-150	
d_3-17alpha-Estradiol	50-150	50-150		50-150	50-150		50-150	50-150	
d_4-17beta-Estradiol	50-150	50-150		50-150	50-150		50-150	50-150	
d_3-Estriol	20-150	20-150		30-150	30-150		14-150	14-150	
$^{13}C_3$-Estrone	50-150	50-150		50-150	50-150		50-150	50-150	
$^{13}C_2$-17alpha-Ethinylestradiol	50-150	50-150		50-150	50-150		50-150	50-150	
Recovery Standards									
d_4-Estrone									

Footnotes:

[1] OPR recovery, surrogate recovery and blank limits derived from actual method performance data.

[2] Analysis result is classified as “Information Value” of estimated concentration.

[3] Background level of Erythromycin - H_2O in the associated labeled surrogate may elevate the Erythromycin - H_2O blank value. Sample results may be blank corrected where acceptable by contract.

[4] Higher blank levels are acceptable where sample concentrations exceed 10 times the blank limits.

[5] POCIS samples have the same acceptance limits as aqueous samples, but for POCIS samples the limits are interim guidelines only.

[6] NQ = Not Quantifiable. Low recovery rate may preclude quantification.

[7] The acceptance limits for List 6 compounds are guidelines based on initial estimates; recoveries outside of these limits do not invalidate results.

[8] Recoveries outside limits may be accepted based on application and professional judgment

[9] List 2 compounds have not been validated for tissue matrix.

[10] List 2 compounds are not available for soil/sediment samples

QC Specification Table: Instrumental Acceptance Specifications

QC Parameter	Specification
Instrument Sensitivity	Daily, S:N ≥ 3:1 for all analytes for lowest calibration point.
Initial Calibration (native compounds)	For Lists 1 and 2: Initial, (1/X) weighted linear regression using a minimum of 5 calibration concentrations; repeated as necessary to maintain Cal/Ver results within established acceptance ranges. Calculated concentrations 70-130%, one point per compound may be 60-140%. Internal guideline – coefficient of determination r2 ≥0.985. Calibration curves with lower correlation coefficient values meeting all above criteria may be accepted based on batch specific QC results and professional judgement. For hydrocortisone, an increased frequency of Initial Calibration variance from method acceptance limits has been observed and is attributed to transient instrumental instability of response correctable by instrumental re-analysis. If the results are deemed to be fit for the intended purpose the hydrocortisone data may be flagged and reported with an explanation of the variance, otherwise instrumental re-analysis to correct the QC variance is required. For initial calibration using only 4 calibration concentrations the following requirements must be met: Calculated concentrations of native compounds 70-130%, Correlation coefficient r2 ≥0.985 (this is a requirement, not a guideline). For List 3, 4, 5, 6, Hormones (POS) and Hormones (NEG): Constant RRF calibration. %RSD of RRF ≤ 25%. Calculated concentrations 70-130%, one point per compound may be 60-140%.
Initial Calibration (labeled compounds)	Calculated concentrations 50-150%.
OPENING Calibration Verification	Native compounds: A mid-level calibration solution is analyzed every 12 hours or every 20 samples, whichever occurs first. Determined concentrations within 70-130% of actual. Allowable exception: A maximum of 1 compound per List or 10% of the compounds on a List, whichever is greater, may fall outside 70-130% provided they are in the range 60-140% of actual. Labeled compounds: Calculated concentrations 50-150%.
CLOSING Calibration Verification	Native compounds: 1) Determined concentrations shall be within 70-130% of actual. Results for the greater of 1 compound or 10% of the compounds on a List may fall outside 70-130% provided they are: a) within 60-140% or, b) within 50-150% and the RPD between the CLOSING result and the OPENING result is ≤40%. 2) Closing calibration verification limits do not apply to Furosemide and Hydrochlorothiazide. Labeled compounds: Calculated concentrations 50-150%.
Instrumental Carryover And Instrument Background	Every Initial Calibration, Cal/Ver, or SPM: ≤ 0.3% carryover. Area response of analytes in instrument blank ≤ ½ of response in A-Cal judged following two previous methanol blank injections.
Duplicate Samples or MS/MSD *	If conc. >5 times R.L., RPD ≤ 40% If conc. <5 times R.L., RPD ≤ 40% for 60% of analytes

* Specifications for duplicate samples and MS/MSD are guidelines. Wider limits may be applied based on sample characteristics and professional judgment.

APPENDIX I: LIMITATIONS TO PERFORMANCE

1. SOIL/SEDIMENT SAMPLES

Experience has shown that recoveries of certain compounds from some soil/sediment matrices may be low. Sample data must be evaluated with consideration of the recoveries achieved for each analyte in the matrix spike sample prepared with each analytical batch. Poor performance of specific analytes in the matrix spike may result in those analytes being flagged (NQ) in related project samples.

In particular, the surrogates listed in the table below can show recoveries in soil and sediment samples that do not meet method criteria. The exact reason is not known, as recoveries are in the normal range for other matrices including biosolids samples that undergo identical processing, and for aqueous samples as well. The interaction of dissolved inorganic components of the matrix with the analytes and the material in the Oasis HLB cartridge is the most likely cause for compounds in List 1 and List 5 showing low recovery.

Surrogate	List	Issue
$^{13}C_3$,^{15}N-Ciprofloxacin	List 1	Low Recovery
^{13}C-d_3-Naproxen	List 3	Low Recovery
$^{13}C_3$-Ibuprofen	List 3	Low Recovery
$^{13}C_6$-Triclocarban	List 3	Low Recovery
d_5-Warfarin	List 3	Low Recovery
d_6-Bisphenol A	List 3	Low Recovery
d_6-Gemfibrozil	List 3	Low Recovery
d_4-Desmethyldiltiazem	List 5	Low Recovery
d_6-Paroxetine	List 5	Low Recovery
d_3-Sertraline	List 5	Low Recovery
^{13}C,d_3-Daunorubicin[1]	List 6	Low Recovery
^{13}C,d_3-Moxifloxacin	List 6	Low Recovery

[1] ^{13}C,d_3-Daunorubicin must have a minimum recovery of 30% to report both Doxorubicin and Daunorubicin in sediment samples. ^{13}C,d_3-Daunorubicin shows generally poor recovery in soil/sediments and it is unlikely that this specification will be met.

Analytes and surrogates not meeting method criteria are flagged using protocols detailed in SGS AXYS Document QDO-027 *“Rules for the Application of Non-Quantifiable Flags (NQ) to MLA-075 Results”*.

2. 1,7-DIMETHYLXANTHINE, THEOPHYLLINE AND THEOBROMINE

1,7-Dimethylxanthine is an analyte in List 1, Theophylline or 1,3-dimethylxanthine is an analyte in List 5 of the same method. These analytes are isomers, and hence co-elute in both List 1 and List 5 instrumental runs, leading to a systematic over-reporting of each compound in the Spiked Matrix

(SPM) samples. The recovery criteria for these compounds takes into account the effect of the cross interference on data accuracy. Any positive detection of either analyte is presumed to be a sum of the two analytes. Neither the HPLC, nor the mass spectrometer, can differentiate between the two compounds.

3. ROXITHROMYCIN, CLARITHROMYCIN AND TYLOSIN REQUANTIFICATION

Roxithromycin, Clarithromycin and Tylosin are all quantified against ^{13}C-Sulfamethazine. This surrogate is chemically different from the analytes and can sometimes show low recovery in samples even when the three analytes are not affected. If the recovery of ^{13}C-Sulfamethazine is less than 10%, upon request, Roxithromycin, Clarithromycin and Tylosin are requantified against the recovery standard ^{13}C-Atrazine and flagged as estimated minimum concentrations if detected. The data is evaluated and flagged using procedures outlined in SGS AXYS Document QDO-027 *"Rules for the Application of Non-Quantifiable Flags (NQ) to MLA-075 Results"*.

4. METHYL ESTER INTERFERENCE OF BETA-LACTAM ANTIBIOTICS

Cloxacillin, oxacillin and penicillin G are reported as 'Information Values' of estimated concentration. These compounds are determined by LC-MS/MS using ions from the methanol adduct of the compound ($M+CH_3OH$). There is indication that methyl esters of these compounds can also form in standard solutions over time. Ions from these methyl esters cannot be distinguished from methanol adduct ions formed from the parent compound. The consequence of this reaction could be a slow, but continuous increase of instrument response for these compounds in the calibration solutions. The rate of change in response is different for each compound. This behavior has not yet been observed/documented in client samples. The result of this standard transformation is to confer greater uncertainty on measured concentrations of these three compounds.

5. POTENTIAL AMPHETAMINE INTERFERENCE

The presence of an interfering compound with potential to obscure or cause false positive detection of amphetamine has been observed in some water and solids samples. Use of the secondary transition response, itself prone to interference, is not reliable in overcoming the interference problem. Partial or complete chromatographic resolution of the interfering compound has been observed - i.e. a shift of the native compound peak RT (retention time) relative to that of the d5-amphetamine surrogate is indicative of the interference. Where evidence of this interference is observed, amphetamine results are flagged in reports as "estimated maximum possible values". The typical level of this interference is between the range of the A- and B-Calibration standards.

i. Extracts will not be routinely diluted and reinjected for amelioration of the amphetamine interference alone, as there is no evidence of efficacy.

ii. Where multiple injection data for a sample are available (e.g. a neat and a diluted run), instrument analysts will report amphetamine from the chromatogram producing the most definitive result based on an evaluation of peak shape and peak resolution. The result will be quantified as amphetamine but flagged as an “estimated maximum possible concentration” on reports. The flag must be edited by hand in LIMS; EMPC, K or NDR dependent on client flagging requirements.

iii. For amphetamine with a high peak area response above the SPM, the 1st channel should be confirmed by the 2nd channel. If no peak is present in the 2nd channel, the peak in the 1st channel is possibly not amphetamine and should be removed from the 1st channel.

6. POTENTIAL DEGRADATION OF RANITIDINE IN THE STANDARD SOLUTION

Degradation of ranitidine in the standard solution used to prepare OPR tests has been observed intermittently under the specific conditions of the storage. Where OPR test results indicate the possibility of spiking solution degradation, the ranitidine OPR assigned value is adjusted based on the results of a secondary QC test solution (SAR) prepared from the same ampoule that has been analyzed alongside samples. This problem has been demonstrated to have no impact on sample data accuracy

APPENDIX II: EFFECTS OF ADDING ASCORBIC ACID TO SAMPLES.

Ascorbic acid is added to quench free chlorine in aqueous samples that have been chlorinated. The presence of free chlorine has severe effects on the recovery of analytes and most surrogate compounds. 50 mg/L of ascorbic acid is usually added to samples. The vast majority of analytes and standards are not affected by ascorbic acid addition. It is possible that some analytes may show enhanced recovery. The effects of ascorbic acid on each analyte/standard is shown below.

Analyte	List	Effect	Surrogates	List	Effect
Acetaminophen	List 1	Normal	$^{13}C_2$,^{15}N-Acetaminophen	List 1	Normal
Azithromycin	List 1	Normal	$^{13}C_3$-Caffeine	List 1	Normal
Caffeine	List 1	Normal	$^{13}C_3$,^{15}N-Ciprofloxacin	List 1	Normal
Carbadox	List 1	Normal	$^{13}C_2$-Erythromycin-H_2O	List 1	Normal
Carbamazepine	List 1	Normal	d5-Fluoxetine	List 1	Normal
Cefotaxime	List 1	Normal	$^{13}C_6$-Sulfamethazine	List 1	Normal
Ciprofloxacin	List 1	Normal	$^{13}C_6$-Sulfamethoxazole	List 1	Normal
Clarithromycin	List 1	Normal	d6-Thiabendazole	List 1	Normal
Clinafloxacin	List 1	Normal	$^{13}C_3$-Trimethoprim	List 1	Normal
Cloxacillin	List 1	Normal	d6-Thiabendazole	List 2	Normal
Dehydronifedipine	List 1	Normal	d6-Bisphenol	List 3	Normal
Diphenhydramine	List 1	Marginal low bias	d6-Gemfibrozil	List 3	Normal
Diltiazem	List 1	Marginal low bias	d11-Glipizide	List 3	Normal
Digoxin	List 1	Normal	d3-Glyburide	List 3	Normal
Digoxigenin	List 1	Normal	$^{13}C_3$-Ibuprofen	List 3	High bias
Enrofloxacin	List 1	Normal	^{13}C-d3-Naproxen	List 3	Normal
Erythromycin-H_2O	List 1	Normal	$^{13}C_6$-Triclocarban	List 3	Normal
Flumequine	List 1	Normal	$^{13}C_{12}$-Triclosan	List 3	Normal
Fluoxetine	List 1	Normal	d5-Warfarin	List 4	Normal
Lincomycin	List 1	Normal	d3-Albuterol	List 4	Normal
Lomefloxacin	List 1	Normal	d6-Metformin	List 4	Normal
Miconazole	List 1	Normal	d3-Cotinine	List 4	Normal
Norfloxacin	List 1	Normal	d3-Cimetidine	List 4	Normal
Norgestimate	List 1	Normal	d5-Enalapril	List 4	Normal
Ofloxacin	List 1	Normal	d6-Oxycodone	List 4	Normal
Ormetoprim	List 1	Normal	d4-Clonidine	List 4	Normal
Oxacillin	List 1	Normal	d5-Amphetamine	List 4	Normal
Oxolinic Acid	List 1	Normal	d6-Codeine	List 4	Normal
Penicillin G	List 1	Normal	d3-Hydrocodone	List 4	Normal
Penicillin V	List 1	Normal	d7-Atenolol	List 4	Normal
Roxithromycin	List 1	Normal	d5-Alprazolam	List 5	Normal
Sarafloxacin	List 1	Normal	d6-Amitriptyline	List 5	Normal
Sulfachloropyridazine	List 1	Normal	d8-Benzoylecgonine	List 5	Normal
Sulfadiazine	List 1	Normal	d3-Benztropine	List 5	Normal
Sulfadimethoxine	List 1	Normal	d3-Cocaine	List 5	Normal

Sulfamerazine	List 1	Normal	d7-DEET	List 5	Normal
Sulfamethazine	List 1	Normal	d5-Diazepam	List 5	Normal
Sulfamethizole	List 1	Normal	d3-Methylprednisolone	List 5	Normal
Sulfamethoxazole	List 1	Normal	d7-Metoprolol	List 5	Normal
Sulfanilamide	List 1	Normal	d5-Norfluoxetine	List 5	Normal
Sulfathiazole	List 1	Normal	d6-Paroxetine	List 5	Normal
Thiabendazole	List 1	Normal	d4-Promethazine	List 5	Normal
Trimethoprim	List 1	Normal	d5-propoxyphene	List 5	Normal
Tylosin	List 1	Normal	d7-Propranolol	List 5	Normal
Virginiamycin M1	List 1	Normal	$^{13}C,^{15}N_2$-Theophylline	List 5	Normal
1,7- Dimethylxanthine	List 1	Normal	d4-Hydrocortisone	List 5	Normal
CTC	List 2	Normal	$^{13}C_3$-Androstenedione	HM-APOS	TBD
ECTC	List 2	Normal	d_4-Androsterone	HM-APOS	TBD
ACTC	List 2	Normal	$^{13}C_3$-d_2-Desogestrel	HM-APOS	TBD
EACTC	List 2	Normal	d_3-17alpha-Estradiol	HM-ANEG	TBD
ICTC	List 2	Normal	$^{13}C_2$-17beta-Estradiol	HM-ANEG	TBD
Demeclocycline	List 2	Normal	d_4-17beta-Estradiol	HM-ANEG	Normal
Doxycycline	List 2	Normal	d_3-17beta-Estradiol 3-Benzoate	HM-APOS	TBD
OTC	List 2	Normal	d_3-Estriol	HM-ANEG	TBD
EOTC	List 2	Normal	$^{13}C_3$-Estrone	HM-ANEG	TBD
TC	List 2	Normal	$^{13}C_2$-17alpha-Ethinyl Estradiol	HM-ANEG	TBD
ETC	List 2	Normal	d_3-Equilenin	HM-ANEG	TBD
EATC	List 2	High Bias	d_4-Mestranol	HM-APOS	TBD
ATC	List 2	Normal	d_6-Norethindrone	HM-APOS	Normal
Minocycline (458>441)	List 2	Normal	d_6-Norgestrel	HM-APOS	Normal
Bisphenol A	List 3	Normal	d_9-Progesterone	HM-APOS	Normal
Furosemide	List 3	Normal	$^{13}C_3$-Testosterone	HM-APOS	TBD
Gemfibrozil	List 3	Normal			
Glipizide	List 3	Normal			
Glyburide	List 3	Normal			
Hydrochlorothiazide	List 3	Normal			
2-hydroxy-ibuprofen	List 3	Normal			
Ibuprofen	List 3	Normal			
Naproxen	List 3	Normal			
Triclocarban	List 3	Normal			
Triclosan	List 3	Normal			
Warfarin	List 3	Normal			
Albuterol	List 4	Normal			
Amphetamine	List 4	Normal			
Atenolol	List 4	Normal			
Atorvastatin	List 4	Normal			
Cimetidine	List 4	Normal			
Clonidine	List 4	Normal			
Codeine	List 4	Normal			
Cotinine	List 4	Normal			
Enalapril	List 4	Normal			
Hydrocodone	List 4	Normal			
Metformin	List 4	Normal			
Oxycodone	List 4	Normal			

Ranitidine	List 4	Normal			
Triamterene	List 4	Normal			
Alprazolam	List 5	Normal			
Amitriptyline	List 5	Normal			
Amlodipine	List 5	Normal			
Benzoylecgonine	List 5	Normal			
Benztropine	List 5	Normal			
Betamethasone	List 5	Normal			
Cocaine	List 5	Normal			
DEET	List 5	Normal			
Desmethyldiltiazem	List 5	Normal			
Diazepam	List 5	Normal			
Fluocinonide	List 5	Normal			
Fluticasone Propionate	List 5	Normal			
Hydrocortisone	List 5	Normal			
10-hydroxy-amitriptyline	List 5	Normal			
Meprobamate	List 5	Normal			
Methylprednisolone	List 5	Normal			
Metoprolol	List 5	Normal			
Norfluoxetine	List 5	Normal			
Norverapamil	List 5	Normal			
Paroxetine	List 5	High Bias			
Prednisolone	List 5	Normal			
Prednisone	List 5	Normal			
Promethazine	List 5	Normal			
Propoxyphene	List 5	Normal			
Propranolol	List 5	Normal			
Sertraline	List 5	Normal			
Simvastatin	List 5	Normal			
Theophylline	List 5	Normal			
Trenbolone	List 5	Normal			
Trenbolone acetate	List 5	Normal			
Valsartan	List 5	Normal			
Verapamil	List 5	Normal			
Allyl trenbolone	HM-APOS	Normal			
Androstenedione	HM-APOS	Normal			
Androsterone	HM-APOS	Normal			
Desogestrel	HM-APOS	Normal			
17beta-Estradiol 3-benzoate	HM-APOS	TBD			
Mestranol	HM-APOS	Normal			
Norethindrone	HM-APOS	Normal			
Norgestrel	HM-APOS	Normal			
Progestrone	HM-APOS	Normal			
Testosterone	HM-APOS	Normal			
17alpha-Dihydroequilin	HM-ANEG	Normal			
Equilenin	HM-ANEG	Normal			
Equilin	HM-ANEG	Normal			
17alpha-Estradiol	HM-ANEG	Normal			

17beta-Estradiol	HM-ANEG	Normal			
Estriol	HM-ANEG	Normal			
Estrone	HM-ANEG	Normal			
17alpha-Ethinyl-estradiol	HM-ANEG	Normal			

APPENDIX III: SUMMARY COMPARISON OF USEPA METHOD 1694 AND SGS AXYS METHOD MLA-075.

Area	EPA 1694	MLA-075
Applicable Matrices	Aqueous, Solids	Aqueous, Solids, Tissue, POCIS samplers
Analytes Offered	73 compounds, 2 fractions, 4 instrumental runs	158 compounds, 2 fractions, 8 instrumental runs
Sample Containers	Amber glass	Amber glass or HDPE
Chlorine Quenching (water samples)	80 mg sodium thiosulfate per liter, ascorbic acid allowable alternative	50 mg ascorbic acid per liter
Sample Preservation	pH 5-9 if hold time >48hr or freeze	None
Sample Storage Temperature	< 6ºC or frozen (aqueous, solids)	Aqueous: < 4 ºC; Solids: <-20 ºC
Sample Hold Time (guideline only)	Aqueous, 7 days at < 6ºC, undefined for frozen storage Solids, 7 days at <-10 ºC	Aqueous: 7 days for < 4 ºC storage Solids: 7 days for -20 ºC storage
Extract Hold Time	40 days	40 days
Extraction (separate acid, base fractions)	Aqueous: adjust to pH 2 or pH 10, stabilize with EDTA. Solids: adjust to pH 2 or pH 10, stabilize with EDTA, ultrasonic extract into buffered acetonitrile, exchange to water solution.	Aqueous: adjust to pH 2 or pH 10, stabilize with EDTA. Solids: adjust to pH 2 or pH 10, stabilize with EDTA, ultrasonic extract into buffered acetonitrile, exchange to water solution. Tissue: Ultrasonic extract into acetonitrile and pH 2 or pH 10 buffer, exchange to water solution, stabilize with EDTA. POCIS: Dialysis with solvent.
Clean-up (separate acid, base fractions)	SPE (HLB), elute in methanol	SPE (HLB), elute in methanol
Instrumental Acquisition	LC-MS/MS, 3 +ESI runs, 1 -ESI run	UPLC/MS/MS, 4 +ESI runs, 2 -ESI runs LC/MS/MS 2 +ESI runs

Calibration Range for LC-MS/MS, ng/mL in standard	Minimum 5 points, range 0.25- 25000 mg/mL	
Calibration Range for UPLC/MS/MS, ng/mL in standard		Minimum 5 points, range 0.02- 4000 ng/mL
Calibration Model	Multi-level, constant RRF; alternative models allowable	Multi-level, 1/x weighted linear regression for list 1 and 2 compounds, average RRF for list 3, 4 5, 6 and hormones
Initial Calibration Limits	RSD of RRF >20% (isotope dilution) or <35% (internal standard)	Calculated points 70-130% of actual (allowable exception per compound 60-140%)
Calibration Verification Limits	70-130%	Calculated points 70-130% of actual (allowable exception one compound per list or 10% of compounds per list may be 60-140%)
Quantification Type	Isotope dilution or internal standard	Isotope dilution or internal standard
Quantification References	18 isotopically labeled compounds	100 isotopically labeled compounds UPLC : List 3: 14; List 4: 14; List 5: 26; List 6: 20; Hormones: 15; HPLC: List 1: 10; List 2: 1
Initial Precision and Recovery (IPR) Limits, %	range 6-180%	performance based, generally 3-250%
On-Going Precision and Recovery (OPR) Limits, %	range 5-200%	performance based, generally 2-300%
Blank Limits, ng per sample	range 1-500 ng	performance based, generally 0.15–320 ng
Surrogate Recovery Limits, %	range 5-200%	performance based, generally 3-250%
Lower Reporting Limit, ng per sample based on low calibration standard	range 1–500 ng	performance based, generally 0.3–500 ng

Appendix E:

COLLECTION AND HANDLING OF SAMPLES FOR ANALYSIS OF PFOA, PFOS AND OTHER FLUORINATED COMPOUNDS

COLLECTION AND HANDLING OF SAMPLES FOR ANALYSIS OF PFOA, PFOS AND OTHER FLUORINATED COMPOUNDS

Avoid all contact of samples with Teflon, sticky labels and adhesive tape. Sampling personnel should avoid wearing jackets and other outer clothing that is new or that has not been washed a few times.

Water Samples:
Collect the water in the required bottles (500mL/1L/50mL). These should have screw cap lids. The bottles should be made of high-density polyethylene (HDPE). Teflon bottles should not be used. (Axys can supply appropriately proofed bottles.) Once filled, each bottle of water should be sealed individually in a Ziploc or whirlpak type bag and stored in a cooler at 4 ºC. Water samples (and any field blanks) should be shipped to the laboratory in a cooler on packaged wet ice or with sufficient blue ice packs to maintain the cool temperature.

Sediment/soil Samples:
Collect an ample amount of sample in 125mL or 250mL HDPE jars making sure not to use any Teflon material. Do not fill the jars all the way to the top especially if there is significant moisture in the samples. Once filled, each jar of sample should be sealed individually in a Ziploc or whirlpak type bag and frozen or stored in a cooler at 4 ºC. Sediment or soil samples should be shipped to the laboratory in a cooler on packaged wet ice or with sufficient blue ice packs to maintain the cool temperature.

Tissue Samples:
Tissue samples, if small enough, can be put into 125mL or 250mL HDPE jars. If larger, wrap in clean foil first. Each sample should be sealed individually in a Ziploc or whirlpak type bag and frozen or stored in a cooler at 4 ºC, if a freezer is not available. The samples should be shipped to the laboratory in a cooler on packaged wet ice or with sufficient blue ice packs to maintain the cool temperature.

GENERAL PFAS SAMPLING GUIDANCE

This document contains an introduction to PFAS, biosecurity recommendations, and general recommendations to decrease the possibility of cross-contamination.

Revised 10/16/2018

Michigan
Department of
Environmental
Quality

Michigan Department of Environmental Quality

GENERAL PFAS SAMPLING

Guidance

CONTENTS

Acronyms

Acronyms used throughout the **General PFAS Sampling Guidance** document and/or each sampling guidance are as follows:

AFFF – Aqueous film forming foam

CAS Number – Chemical abstracts service number

COC – Chain of Custody

DEPA – Danish Environmental Protection Agency (Denmark)

EINECS – European List of Notified Chemical Substances (European Union)

ENCS – Existing and New Chemical Substances Inventory (Japan)

ETFE – Ethylene-tetrafluoroethylene

FCMP – Fish Contaminant monitoring program

FCSV – Fish consumption screening values

FDA – Food and Drug Administration (United States of America)

FEP – Fluorinated ethylene propylene

HASP – Health and Safety Plan

HDPE – High-density polyethylene

IECSC – Inventory of Existing Chemical Substances Produced or Imported in China

ITRC – Interstate Technology & Regulatory Council

KECI – Korea Existing Chemicals Inventory (South Korea)

KEMI – Swedish Chemical Agency (Sweden)

LDPE – Low-density polyethylene

LHA – Lifetime Health Advisory (United States Environmental Protection Agency)

MDEQ – Michigan Department of Environmental Quality

MDHHS – Michigan Department of Health and Human Services

MPART – Michigan PFAS Action Response Team

MSDS – Material Safety Data Sheet (former reference)

ng/L – Nanograms per liter

NZIoC – New Zealand Inventory of Chemicals (New Zealand)

PCPs – Personal care products

PID – Photoionization detector

PFAA – Perfluoroalkyl acids

PFAS – Per- and Polyfluoroalkyl Substances

PFC – Polyfluorocarbons

PFCA – Perfluoroalkyl carboxylic acids

PFOA – Perfluorooctanoic acid

PFOS – Perfluorooctanesulfonic acid

PFPE – Perfluoropolyethers

PFSA – Perfluoroalkyl sulfonic acids

PICCS – Philippine Inventory of Chemicals and Chemical Substances (Philippines)

ppb – Parts per billion

PPE – Personal protection equipment

ppt – Parts per trillion

PTFE – Polytetrafluoroethylene

PVC – Polyvinyl chloride

PVDF – Polyvinylidene fluoride

PVF – Polyvinyl fluoride

QA/QC – Quality assurance/quality control

QAPP – Quality Assurance Project Plan

OECD – Organization for Economic Cooperation and Development

SDS – Safety Data Sheet

SWAS – Surface Water Assessment Section (MDEQ)

TSCA – Toxic Substances Control Act (United States of America)

USEPA – United States Environmental Protection Agency

UV – Ultraviolet

VOC – Volatile organic compounds

WRD – Water resources division (MDEQ)

Disclaimer

The Michigan Department of Environmental Quality (MDEQ) intends to update the information contained within this PFAS Sampling Guidance document as new information becomes available. The user of this PFAS Sampling Guidance is encouraged to visit the Michigan PFAS Action Response Team webpage (www.michigan.gov/PFASresponse) to access the current version of this document.

1. Introduction

Per- and polyfluoroalkyl Substances (PFAS) are a class of **emerging contaminants** composed of more than 3,000 human-made, fluorinated, organic chemicals (Buck et al., 2011, Wang et al., 2017). The actual number of compounds is continuously changing, as some PFAS are no longer produced due to regulatory and voluntary actions, while new ones are created as alternatives. The carbon-fluorine bond that exists in PFAS is one of the strongest bonds in nature, they are tough to break and are resistant to thermal, chemical, and biological degradation.

NOTE: Emerging Contaminants are chemicals and materials in the environment and present real or potential human health or environmental risks, and either…

- Do not have peer-reviewed human health standards

  or:
- Standards/regulations are evolving due to new science, new laboratory analytical capabilities, and new knowledge about the chemicals.

Due to their unique chemical properties, various PFAS can lower surface tension (act as surfactants), are oil-repelling (oleophobic), and are water-repelling (hydrophobic), yet are also relatively water soluble. They have been used extensively in many industries worldwide for a wide variety of applications. PFAS were first invented in the late 1930's and commercially used from the 1940's as non-stick coatings. PFAS continued to be used in many industries and various products as more PFAS were developed with unique chemical properties. Some of the documented PFAS uses are in hydraulic fluids, biocides, construction products, fire-fighting foams, household products, wetting and mist suppressing agents, surfactants for oil and natural gas recovery enhancement, polymerization agents, low-friction bearings and seals, insulators, cables, wires, protective coatings for a wide variety of materials, nonstick coatings, surgical patches, cardiovascular grafts, implants, oil and water repellent coatings for a wide range of materials such as paper and cardboard packaging products, carpets, leather products, and textiles (OECD, 2013). The presence of PFAS in these materials is a potential source of environmental concern and cross-contamination.

The probability of false positives is relatively high during PFAS sample collection due to the potential for many sources of cross-contamination, combined with low laboratory detection limits (nanograms per liter (ng/L) or parts per trillion (ppt)). There are many products that could be found in the sampling environment, that have not been documented to either contain or not contain PFAS, and may come into contact with the samples, introducing causing cross-contamination.

The United States Environmental Protection Agency (USEPA) has established a Lifetime Health Advisory (LHA) for Perfluorooctanoic acid (PFOA) and Perfluorooctanesulfonic acid (PFOS), separately or combined, of 70 ppt. The MDEQ cleanup criteria protective of groundwater used for drinking water purposes is also 70 ppt for PFOS and PFOA, individually or combined. The MDEQ has also promulgated a standard under Rule 57 for PFOS of 11 ppt for surface water that is used as a drinking water source and 12 ppt for surface water that is not used as a drinking water source.

2. Purpose and Objectives

The purpose of this document is to provide guidance and information to staff who will:

- Collect or handle PFAS environmental samples.
- Perform subsurface activities such as soil borings and/or well installation or well abandonment at PFAS sites.

This document is intended to supplement the MDEQ media-specific PFAS sampling guidance documents and is a resource for PFAS sampling.

The objectives of this document are as follows:

- Provide guidance on avoiding PFAS cross-contamination during sampling.
- Improve sampling consistency and data quality.
- Provide guidance to MDEQ staff and contractors.

Because PFAS are emerging contaminants and information about their use in various materials is still not available; the MDEQ will update this document as new information becomes available.

NOTE: This guidance does not include specific information for sampling environmental media and should not be used to replace specific sampling guidance documents required for use by MDEQ staff.

3. Farm Biosecurity

In the event PFAS sampling occurs on or near a farm, staff need to follow the requirements in this document when conducting sample collection, to reduce the likelihood of transporting animal diseases.

3.1 Scheduling

To avoid cross-contamination from previous sampling locations, it is preferable that staff visit only one farm in a day.

3.2 Before Sampling

Staff should review **Section 4.2.4. Field Clothing and Personal Protective Equipment (PPE)** before going into the field.

Staff must have a clean vehicle, clean clothing, and clean boots to visit the sampling location. Before arriving at the farm, staff should call the owner of the farm to indicate they will be arriving shortly and ask if there are any additional biosecurity requirements for their farm. Once at the farm, staff should park away from any animals and barns; preferably in a designated visitor area or on concrete.

Immediately before exiting the vehicle, place disposable PFAS-free boot covers over boots. (*NOTE: Disposable boot covers can be slippery, especially in icy/snowy conditions.*)

3.3 While Sampling

Staff should not approach animal areas unless necessary for testing. If access to an animal area is needed, staff should always be accompanied by farm personnel.

3.4 After Sampling

Dispose of used disposable boot covers at the facility if possible; otherwise, place in a plastic bag, seal and place in the vehicle trunk to dispose of properly later.

4. General PFAS Sampling

The following sections discuss technical issues such as the need to use PFAS-free water; information about PFAS-free clothing and PPE; and laboratory issues that should be considered when sampling for PFAS.

4.1 Sampling Objectives

Before conducting any PFAS sampling, it is recommended that a project-specific Quality Assurance Project Plan (QAPP) should be developed. The QAPP must meet MDEQ policy and should include the analyte list, method of analysis, environmental matrices, and reporting limits, which are based on the project objectives. All of these considerations will be discussed in more detail in this guidance document.

4.2 PFAS Cross-Contamination Potential Sources

Potential sources of PFAS cross-contamination in the typical sampling environment include water used during drilling or decontamination, materials used within the sampling environment, sampling equipment, field clothing and personal protective equipment (PPE), sun and biological protection products, personal hygiene and personal care products (PCPs), food packaging, and the environment itself.

The materials associated with sampling that have the potential for PFAS cross-contamination have been divided into three major groups:

- Prohibited (●) identifies items and materials that should not be used when sampling. It is well documented that they contain PFAS or that PFAS are used in their manufacture.
- Allowable (■) identifies items and materials that have been proven not to be sources of PFAS cross contamination and are considered allowable for sampling.
- Needs Screening (▲) identifies items and materials that have the potential for PFAS cross-contamination due to a lack of scientific data or statements from manufacturers to prove otherwise. These items and materials are further sub-divided into two categories:
    - **Category 1:** Items and materials that will come in direct contact with the sample. These should not be used when sampling unless they are known to be PFAS-free, by collecting an equipment blank sample prior to use.
    - **Category 2:** Items and materials that will not come in direct contact with the sample. These should be avoided, if possible, unless they are known to be PFAS-free by collecting an equipment blank sample prior to use.

All of the materials or items discussed in each of the MDEQ's PFAS Sampling Guidance Documents will be divided into ● Prohibited ■ Allowable, or ▲ Needs Screening. Several examples of prohibited and allowable materials and materials that need screening are listed in the **MDEQ PFAS Sampling Quick Reference Field Guide** at the end of this document. Also, materials and items that are specific to a particular environmental media or sampling method will be thoroughly explained in that media's sampling guidance document (such as peristaltic pumps for groundwater sampling).

NOTE: If recommended PPE will be used during sampling, **Category 2** materials are not expected to be a source of cross-contamination as long as they do not come into contact with the samples.

Please note that at this time no published research is available that documents the use of various materials and their effect on sample results. Therefore, a conservative approach is recommended in this guidance based on the evaluation of multiple environmental samples at various PFAS sites. Field sampling occurring during extreme weather (e.g., rainfall, snowfall, or extreme heat) should be conducted while wearing the appropriate clothing that will not pose a risk for cross-contamination but will also ensure the safety of the field personnel.

4.2.1 PFAS-Free Water

The term PFAS-free water is defined here as water that does not contain significant concentrations of any compound in a specific PFAS analyte list that is being analyzed at a project-defined level. The significant concentrations depend on project data quality objectives and could, for instance, be less than the laboratory reporting limit, <1/2 the limit of quantitation, or other defined criteria for the specific PFAS compound of interest (ITRC, 2017).

NOTE: The confirmation of PFAS-free water should always be performed prior to the commencement of work. Site or public water supplies have been identified in many instances to contain detectable levels of PFAS.

One important consideration for each project is to identify a PFAS-free water source to use for decontamination of sampling and drilling equipment when applicable. The decontamination of sampling tools or small equipment parts can be performed using laboratory-supplied verified PFAS-free water. Other water can only be used for decontamination purposes if it has been analyzed and shown to be PFAS-free as defined for the project.

4.2.2 Materials Screening

Materials screening should be performed during the Health and Safety Plan (HASP) and QAPP development or the planning phase of sampling programs. The screening should be performed on all of the items and materials that are expected to come into contact with the samples and defined as **Category 1**.

Material screening should include a review of Safety Data Sheets (SDSs; formerly Material SDS [MSDSs]). Make sure the review uses current SDSs, because the actual composition of a particular item or material may have changed over time without changing the actual item or material name. All products from the United States or abroad should be screened. Text fragments such as “perfluoro,” “fluoro,” or “fluorosurfactant” may identify the use of PFAS in specific items or materials.

NOTE: Manufacturers can change the chemical composition of any product. As a result, equipment blank samples should be collected for all materials that will come into direct contact with the sample media, regardless of what category they might be in, to confirm they are “PFAS-free”, i.e. will not contaminate samples at detectable levels. **There is no guarantee that materials in the ‘Allowable category will always be PFAS-free.**

Some countries have official national lists of industrial chemicals defined by regulations, such as:

- Toxic Substances Control Act (TSCA) in the United States.
- European List of Notified Chemical Substances (EINECS), as well as substances pre-registered under the Registration, Evaluation, Authorization, and restriction of Chemicals (REACH) in the European Union.
- Swedish Chemical Agency (KEMI) in Sweden.

- Domestic Substances List (DSL) in Canada.
- Inventory of Existing New Chemical Substances Produced or Imported in China (IECSC)
- Existing and New Chemical Substances Inventory (ENCS) in Japan.
- Korea Existing Chemicals Inventory (KECI) in South Korea.
- New Zealand Inventory of Chemicals (NZIoC) in New Zealand.
- Philippine Inventory of Chemicals and Chemical Substances (PICCS) in the Philippines.

The information available on these lists includes the chemical names and various identity numbers, which is usually the Chemical Abstracts Service number (CAS Number) (KEMI, 2015). The lists may not contain a substantial amount of information because of laws in regards to proprietary information, which gives the suppliers the right to not name newly developed chemicals. The information is not always sufficient to identify if the items or materials contain PFAS, as many of the PFAS do not have an assigned CAS Number at this time (KEMI, 2015). The most recent summary conducted by the Organization for Economic Co-operation and Development (OECD) identified 4,730 PFAS-related CAS numbers (OECD, 2018).

Sometimes manufacturers provide information about their products online or upon request, which may indicate if PFAS were used in the manufacturing of a particular item or material.

4.2.3 Sampling Equipment

The actual list of PFAS-containing materials potentially encountered onsite will change based on the specific sampled media and site-specific sampling conditions. Do not use any equipment that contains any known fluoropolymers. Consider all of the following:

- ● Do not use polytetrafluoroethylene (PTFE) that includes the trademark Teflon® and Hostaflon®, which can be found in many items, including but not limited to the lining of some hoses and tubing, some wiring, certain kinds of gears, and some objects that require the sliding action of parts.
- ● Do not use Polyvinylidene fluoride (PVDF) that includes the trademark Kynar®, which can be found in many items, including but not limited to tubing, films/coatings on aluminum, galvanized or aluminized steel, wire insulators, and lithium-ion batteries.
- ● Do not use Polychlorotrifluoroethylene (PCTFE) that includes the trademark Neoflon®, which can be found in many items, including but not limited to valves, seals, gaskets, and food packaging.
- ● Do not use Ethylene-tetrafluoroethylene (ETFE) that includes the trademark Tefzel®, which can be found in many items, including but not limited to the wire and cable insulation and covers, films for roofing and siding, liners in pipes, and some cable tie wraps.
- ● Do not use Fluorinated ethylene propylene (FEP) that includes the trademarks Teflon® FEP and Hostaflon® FEP, and may also include Neoflon®, which can be found in many items, including but not limited to the wire and cable insulation and covers, pipe linings, and some labware.
- ● Do not use low-density polyethylene (LDPE) for any items that will come into direct contact with the sample media. LDPE can be found in many items, including but not limited to containers and bottles, plastic bags, and tubing.
    - ▲ **However**, LDPE may be used if an equipment blank has confirmed it to be PFAS-free. LDPE does not contain PFAS in the raw material but may contain PFAS cross-contamination from the manufacturing process.

●- Prohibited 🟩- Allowable ▲- Needs Screening

- ■ LDPE bags (e.g., Ziploc®) that do not come into direct contact with the sample media and do not introduce cross-contamination with samples may be used.
- ■ Use materials that are either made of high-density polyethylene (HDPE), polypropylene, silicone, or acetate.
- ■ Glass bottles or containers may be used if they are known to be PFAS-free, however, PFAS have been found to adsorb to glass, especially when the sample is in contact with the glass for a long period of time (e.g. being stored in a glass container). If the sample comes into direct contact with the glass for a short period of time (e.g. using a glass container to collect the sample, then transferring the sample to a non-glass sample bottle), the adsorption is minimal.
- ■ Powderless nitrile gloves (which can be found at some hardware and major retail outlets).
- ▲ Latex gloves should be screened before use.
- ▲ Some sampling guidance documents allow the use of aluminum foil provided the shiny side is placed away from the sample (e.g., fish tissue sampling guidance). As a precaution, MDEQ recommends that aluminum foil not is used unless equipment blank samples confirm it is PFAS-free.

4.2.4 Field Clothing and Personal Protective Equipment (PPE)

Any field planning and mobilization effort should address the physical, chemical, and biological hazards associated with each PFAS site. The mitigation of potential risks may be documented in a site-specific HASP or a QAPP. Due to the extensive use of PFAS in many industries and products, PPE may contain PFAS. During PFAS investigation, PPE containing PFAS should be avoided to prevent cross-contamination. The development of the HASP or QAPP should consider these factors before mobilization in the field. All HASPs or QAPPs need to address the concern of potential exposure of staff to PFAS through PPE.

Personal safety is paramount. The safety of staff should not be compromised by fear of PFAS containing materials without any scientific basis. Any deviation from this guidance, including those necessary to ensure the health and safety of sampling personnel, must be recorded in field notes and discussed in the final report.

Globally, protective coatings for textiles are estimated to be about 50 percent of the total use of PFAS (DEPA, 2015). Due to its unique properties of water and oil repellency, PFAS has been used to coat various clothing (i.e., pants, jackets, and t-shirts) and leather products (i.e., boots, shoes, and jackets). Many of these types of clothing and PPE have the potential to be used in the sampling environment.

NOTE: The Danish Ministry of the Environment identified alternative polymer technology as being PFAS-free. Products treated with this technology are water- resistant, but not oil and dirt repellent to the same extent as products treated with PFAS- based agents (DEPA, 2015).

While preparing for sampling, particular focus should be made on clothing that has been advertised as having waterproof, water-repellant, or dirt and/or stain resistant characteristics. These types of clothing are most likely to have had PFAS used in their manufacturing.

Field Clothing and PPE that should be avoided (●) in the immediate sampling environment include the following:

●- Prohibited ■- Allowable ▲- Needs Screening

● Do not use clothing that has been washed with fabric softener which may contain PFAS.

● Do not use clothing that has been made with or washed with water, dirt, and/or stain resistant chemicals.

● Do not use clothing chemically treated for insect resistance and ultraviolet protection (See **Section 4.2.5** on biological hazards).

● Do not use clothing or PPE items that have any of the brand or product names that have been found to contain PFAS by the Danish Ministry of the Environment and presented in **Table 1** below (DEPA, 2015).

Field Clothing and PPE that are allowable (■) to wear within the immediate sampling environment include the following:

■ Powderless nitrile gloves.

■ Polyvinyl chloride (PVC) or wax-coated fabrics.

■ Neoprene.

■ Any boots made of polyurethane and/or PVC. If the HASP requires 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.

NOTE: There could be many PPE materials used during various sampling events, including hard hats and safety glasses. All clothing and PPE should be evaluated prior to sampling.

Field Clothing and PPE that must be evaluated (▲) before wearing within the immediate sampling environment include the following:

▲ Latex gloves.

▲ Water resistant or stain-treated clothing and PPE.

▲ Tyvek suits and clothing that contain Tyvek® (USEPA PFAS sampling guidance from USEPA Region 2 prohibits the use of Tyvek; available product information suggests Tyvek® may be used if required. Coated Tyvek® requires further evaluation; therefore, MDEQ recommends the collection of an Equipment Blank before Tyvek® use).

Table 1 below provides a list of prohibited field clothing (DEPA, 2015). However, the manufacturer and/or vendor for the field clothing and/or PPE should be contacted to confirm that these brand or product names still contain PFAS. There have been instances where manufacturers have kept the same brand and/or product name but have changed the chemicals used during the manufacturing of a particular item.

Table 1. Prohibited Field Clothing and PPE Brand and Product Names

● Prohibited Materials[1] (DEPA, 2015)	
Advanced Dual Action Teflon® fabric protector.	Release Teflon®
Repel Teflon® fabric protector	High-Performance Release Teflon®
High performance Repel Teflon® fabric protector	Ultra Release Teflon®
NK Guard S series	GreenShield®
Tri-Effects Teflon® fabric protector	Lurotex Protector RL ECO®
Oleophobol CP®	Repellan KFC®
Rucostar® EEE6	UnidyneTM
Bionic Finish®	RUCO-GUARD®
RUCOSTAR®	RUCO-COAT®
RUCO-PROTECT®	RUCOTEC®
RUCO®	Resist Spills™
Resists Spills and Releases Stains™	Scotchgard™ Fabric Protector

[1] This list is not considered to be a complete listing of prohibited materials. All materials should be evaluated before use during sampling.

4.2.5 Sun and Biological Protection

Because biological hazards (sunburn, mosquitos, ticks, etc.) may be encountered during sampling, the elimination of specific clothing materials or PPE (sunscreens and insect repellants) could pose a health and safety hazard to staff.

The safety of staff should not be compromised by fear of PFAS containing materials without any scientific basis. Personal safety is paramount. Any deviation from this guidance, including those necessary to ensure the health and safety of MDEQ staff, should be recorded in field notes and discussed in the final report.

Prolonged sun exposure will require sunscreens, which may have included PFAS in their manufacture. Protection against insects may require the use of insect repellant. **Table 2** contains a detailed list of sunscreens and insect repellants that have been analyzed and found to be PFAS-free as of the date of this document. Note that this is not a comprehensive list of allowable insect repellants or sunscreens; other products may meet the requirements for use. Listing or omission of any product does not imply endorsement or disapproval. Also, there is no guarantee that these products will always remain PFAS free.

NOTE: Sunscreens and insect repellants must be evaluated on a case-by-case basis. Refer to **Section 4.6 Quality Control Samples** for details on collecting equipment blanks.

The MDEQ recommends that additional sunscreens and insect repellents be treated as (▲) Needs Screening and should be evaluated before use.

- ● Sunscreens and insect repellants should not be applied near the sample collection area.
- ■ Hands should be well washed after application or handling of these products, and afterwards, powderless nitrile gloves should be worn.

●- Prohibited ■- Allowable ▲- Needs Screening

Table 2. Sunscreen and Insect Repellents[1]

	🟩 Allowable Insect Repellants
Photos	**Insect Repellent Spray**
	• OFF Deep Woods • Sawyer Permethrin
	🟩 Allowable Sunscreens
Photos	**Sunscreens**
  	• Banana Boat Sport Performance Sunscreen Lotion Broad Spectrum SPF 30. • Meijer Sunscreen Lotion Broad Spectrum SPF 30. • Neutrogena Ultra-Sheer Dry-Touch Sunscreen Broad Spectrum SPF 30.

🟩 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 repellant, Herbal Armor, California Baby Natural Bug Spray, Baby Ganics.
Sunscreen and Insect Repellent: Avon Skin So Soft Bug Guard Plus – SPF 30 Lotion.

[1] This table is not considered to be a complete listing of allowable materials and materials that require screening. All materials should be evaluated before use during sampling. Some of the sunscreen and insect repellent testing has been performed using a PFAS screening Method known as Particle Induce Gamma-Ray Emission (PIGE). The use of approved gloves should always be used, and the sample should never come into contact with any of the sunscreen or insect repellent products. An Equipment Blank sample could also be collected to verify the product as PFAS-free.

●- Prohibited 🟩- Allowable ▲- Needs Screening

If an insect repellant has not been approved and staff needs protection against biting insects:

NOTE: The words "Natural" and/or "Organic" in the product name or to describe it does not mean that it is PFAS-free.

- Tuck pant legs into socks and/or boots to seal the gap between the boots and the pants to reduce the risk of being bitten by ticks.
- Wear well-washed, light-colored clothing to easily see ticks during field activities.
- Light-colored clothing, long sleeves, and large-brimmed hats also prevent sunburn.
- Equipment Blank samples should be collected to verify that the preferred insect repellant or sunscreen is PFAS-free by using the testing procedures identified in **Section 4.6 Quality Control Samples**.

4.2.6 Personnel Hygiene and Personal Care Products (PCPs)

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 (OECD, 2002, Fujii, 2013, Borg and Ivarsson, 2017). However, if the MDEQ's sampling SOPs are followed, these items should not come into contact with the sampling equipment or the sample being collected. As of the date of this sampling guidance, cross-contamination of samples due to the use of PCPs has not been documented during the collection of thousands of samples. However, field personnel should be aware of the potential of cross-contamination if the sampling equipment or actual samples would come into contact with these products. The following precautions should be taken when dealing with personal hygiene or PCPs before sampling:

● 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.

4.2.7 Food Packaging

PFAS has been used by the paper industry as a special protective coating against grease, oil, and water for paper and paperboards, including food packaging since the late 1950s (Trier et al., 2018). PFAS application for food packaging includes paper products that come into contact with food such as paper plates, food containers, bags, and wraps (OECD, 2002). In January 2016, the Food and Drug Administration (FDA) banned the use of PFAS which has eight carbon atoms (such as PFOA and PFOS) or more, in food packaging materials. However, PFOA and PFOS or other eight or more carbon chain PFAS may still be detected in food packaging because of the use of recycled paper which may contain PFAS. Various studies have found up to 57percent detection frequency in food contact materials such as paper (Trier et al., 2011; Rosenmai et al., 2013; Schaider et al., 2017).

NOTE: Short-chain PFAS have not been banned for use in the manufacturing of contact food materials in the United States.

PFAS has been used by the paper industry as a special protective coating against grease, oil, and water for paper and paperboards, including food packaging since the late 1950s (Trier et al., 2018). PFAS application for food packaging includes paper products that come into contact with food such as paper plates, food containers, bags, and wraps (OECD, 2002). Pre-wrapped food or snacks (such as candy bars, microwave popcorn, etc.) must not be in the sampling and staging areas during sampling due to PFAS contamination of the packaging. When staff requires a break to eat or drink, they should remove their gloves, coveralls, and any other PPE, if worn, in the staging area and move to the designated area for food and beverage consumption. When finished, staff should wash their hands and put on a fresh pair of powderless nitrile gloves at the staging area, before returning to the sampling area.

● Do not handle, consume, or otherwise interact with pre-wrapped food or snacks, carry-out food, fast food, or other food items while on-site during sampling.

■ Move to the staging area and remove PPE prior to leaving the sampling and staging areas if consuming food on site becomes necessary.

4.3 PFAS Sampling Procedures

4.3.1 Sample Containers, Handling, and Collection

All bottles used for PFAS sampling should come from the laboratory that will also be performing the PFAS analysis. Commercial laboratories that have demonstrated awareness and elimination of possible PFAS cross-contamination from sample containers and laboratory supplies should be used. Recommended sampling containers will be discussed for each environmental media. Any sampling containers provided by the laboratory should be verified as PFAS-free.

Before sampling, staff may come into contact with textiles and fabrics treated with PFAS, such as carpets and car interiors. Staff should be aware that these materials, and any other surfaces that repel water and are stain resistant, have the potential of being treated with PFAS. However, these are considered **Category 2** materials and the field personnel should be aware of the possible PFAS use. Sample containers and equipment that will be used for sampling should not be stored on or come into contact with materials suspected to contain PFAS.

For all environmental media, hands should be well washed before sampling. Clean powderless nitrile gloves must be put on before sample collection, handling of sample containers, and handling sampling equipment. The sample container must be kept sealed at all times and only open during the sample collection. The sampling container cap or lid should never be placed on any surface unless it is PFAS-free. The sampling container cap or lid must never be placed directly on the ground. A list of various materials used in sampling and handling can be found in the **MDEQ Quick Reference Field Guide** located at the end of this document.

In the absence of formal USEPA guidance for PFAS sample storage, the documentation in EPA Method 537 Rev. 1.1 should be used as a guide for thermal preservation (holding temperature), and holding times for other environmental media samples (with the exception of biota – in order to limit microbial growth, biota samples such as fish and vegetation are recommended to be kept frozen until the sample is prepared).

If published analytical reference methods, other than EPA Method 537 Rev. 1.1 are used, follow the guidelines or requirements in those methods for sample storage, preservation, and hold times. Otherwise EPA Method 537 Rev. 1.1 requries that samples must be chilled during storage and shipment, and must not exceed 50°F (10°C) during the first 48 hours after collection.

4.3.2 Sample Shipment

In general, for all environmental media sampled for PFAS, samples must be kept on ice from the time of sample collection to the arrival at the laboratory. The following procedures should be used for sample shipment:

- ■ Regular ice should be used to cool and maintain the sample at or below the proper temperature.
    - ▲ Chemical or blue ice may be used if it is known to be PFAS-free and it is absolutely certain that the sample is cooled and maintained at or below the proper temperature during collection and through transit to the laboratory.
- ■ Refresh with regular ice, if needed, double bagged in LDPE resealable storage bags if needed.
- ■ Fish and other wildlife samples should be placed on dry ice and frozen before the shipment to the lab. If fish is frozen, shipping the samples overnight on ice should be acceptable.
- ■ The samples, ice, and chain of custody (COC) should always be bagged in polyethylene (i.e., Ziploc®) bags.
- ■ Chain of Custody and other forms should be single bagged in LDPE resealable storage bags and taped to the inside of the cooler lid.
- ■ The cooler should be taped closed with a custody seal and shipped by overnight courier.
- ■ Samples should be shipped as soon as possible (e.g. overnight) to ensure the samples arrive within the analytical holding time specified by the lab.

4.3.3 Preferential Sampling Sequence

A preferred sampling sequence should be established before any sampling event to reduce the risk of cross-contamination. In general, the sampling sequence should be such that sampling starts in areas where it is expected or known to be least contaminated, to areas anticipated or identified to be most contaminated. If analytical results from past sampling events are available, the sampling sequence can be readily determined.

For many PFAS investigation sites, no PFAS sampling has been conducted. In these cases, all site information on possible PFAS uses and potential PFAS migration patterns (e.g., upgradient, downgradient) from PFAS sources at the site should be reviewed before the sampling event to help establish the sampling sequence.

If multiple samples (i.e., monitoring wells) will be collected for an area where a particular or potential PFAS release in the environment might have been documented, samples that are known to be upgradient from the impacted area should be sampled first, followed by those that are furthest downgradient from the suspected source. The remaining wells should be progressively sampled from the most distant downgradient to those closer to the known PFAS source.

If no information is available about the site, samples are to be collected in the following order:

1) drinking water (e.g., residential wells).
2) surface water.
3) groundwater.

4.4 Decontamination Procedures

It is customary with sampling that equipment is decontaminated at the conclusion of the sampling event. If the previous user of the equipment is not known, and it is unclear how the equipment was handled, especially rental equipment, the equipment should be decontaminated.

Disposable **Category 1** sampling equipment should be used, especially for sample bottles and other materials that are used where the sample may be in contact with the sampling equipment for an extended time period.

●- Prohibited ■- Allowable ▲- Needs Screening

Non-disposable sampling equipment used at multiple sites or sampling locations can become highly contaminated with PFAS. Decontamination procedures must be implemented to prevent cross-contamination, especially between individual sample locations. It is customary to decontaminate sampling equipment at the end of the sampling event, whether the event is a single sampling location or several sites that conclude at the end of the workday.

Throughout the sampling guidance documents, information will be provided about any media-specific decontamination procedures. For non-dedicated **Category 1** sampling equipment, there are many decontamination methods, two of which are listed below.

Decontamination Method 1:

- ● Do not use Decon 90®.
- ● Do not put equipment away without decontaminating it.
- 🟩 Laboratory supplied PFAS-free deionized water is preferred for decontamination.
- 🟩 Alconox®, Liquinox®, and Citranox® can be used for equipment decontamination.
- 🟩 Sampling equipment can be scrubbed using polyethylene or polyvinylchloride (PVC) brush to remove particulates.
- 🟩 Decontamination procedures should include triple rinsing with PFAS-free water.
- 🟩 Do decontaminate sampling equipment after sampling at each location, or at the end of the workday.
- 🟩 Commercially available deionized water in an HDPE container may be used for decontamination if the water is verified to be PFAS-free as defined in **Section 4.2.1** of this document.
- ▲ Municipal drinking water may be used for decontamination purposes if it is known to be PFAS-free.

Decontamination Method 2:

1. In a PFAS-free bucket, wash the equipment with a mixture of PFAS-free water and PFAS-free soap (bucket #1)
2. In a second PFAS-free bucket (bucket #2), rinse the equipment with PFAS-free water
3. A second rinse should be done with PFAS-free water using either a third bucket (bucket #3) or, if washed and rinsed, the second bucket (bucket #2).
4. For decontamination of additional equipment, change the decontamination water between cleanings.

4.5 Laboratory Considerations

The PFAS analytical list is available on the MPART website (www.michigan.gov/PFASresponse) under Testing and Treatment. This list includes the 14 analytes required to be analyzed for drinking water samples when using USEPA Method 537 Rev. 1.1, and the 24 analytes the MDEQ recommends be analyzed for all other environmental media. The MPART website should be visited to download the most recent document. Laboratories should be able to analyze and report PFAS results that will meet the project-specific data quality objectives identified in the QAPP.

●- Prohibited 🟩- Allowable ▲- Needs Screening

Drinking Water Samples

USEPA Method 537 Revision 1.1 must be used for testing finished drinking water samples. Other methods are available for non-drinking water samples. Many laboratories refer to the isotope dilution method as 'modified Method 537,' however, the USEPA does not recognize isotope dilution as an acceptable modification of USEPA Method 537 Rev. 1.1 for drinking water analysis. USEPA drinking water methods are generally prescriptive, and only limited modifications are allowed because the finished treated drinking water is assumed to be free of significant interferences.

NOTE: USEPA Method 537 Rev. 1.1 was developed to be used only for finished drinking water samples, and contains specific requirements for sample preservation, shipping storage, and holding times.

USEPA Method 537 Rev. 1.1 was designed for finished drinking water and chemical preservation using Trizma® to buffer the sample and remove free chlorine. Non-chlorinated finished drinking water may also be analyzed using USEPA Method 537 Rev. 1.1.

Other Environmental Media Samples

There are currently no published USEPA methods using isotope dilution for determining PFAS in non-drinking water matrices or other sample media. There are USEPA methods for analyzing PFAS in additional matrices going through the development and validation process and may be available as early as fall of 2018. Some commercial laboratories have developed isotope dilution methods based on existing published methods, however, there may be significant differences between SOPs from different commercial laboratories regarding the details of the preparation and analysis of PFAS samples. A review of the laboratory's procedure and certifications should be done to ensure that the laboratory is capable of providing data that meet the data quality objectives of the project. MDEQ is implementing a laboratory SOP review process. Staff should refer to the MDEQ internal shared drive to see whether SOPs have been reviewed for the lab they are considering.

The following non-USEPA analytical methods have been published for use in determining PFAS in various media:

- ISO (International Organization for Standardization) Method 25101 (ISO, 2009) - Water quality Determination of PFOA and PFOS - Method for unfiltered samples of drinking water, groundwater, and surface water, using solid phase extraction and liquid chromatography/mass spectrometry (HPLC/MS/MS.)

- ASTM D7979 (ASTM, 2017) - Standard Test Method for Determination of Per- and Polyfluoroalkyl Substances in Water, Sludge, Influent, Effluent and Wastewater by Liquid Chromatography-Tandem Mass Spectrometry (LC/MS/MS). This method has been investigated for use with surface water, sludge, and wastewater for selected PFAS. This method has not been evaluated on drinking water matrices. Some commercial laboratories have modified this method and are using isotope dilution.

- ASTM D7968 (ASTM, 2017) - Standard Test Method for Determination of Polyfluorinated Compounds in Soil by Liquid Chromatography-Tandem Mass Spectrometry (LC/MS/MS). This procedure utilizes a quick extraction and is not intended to generate an exhaustive accounting of the content of PFAS in difficult soil matrices.

4.6 Quality Control Samples

4.6.1 Laboratory Quality Control Samples

The QAPP should describe what batch quality control (QC) samples – such as method blank (MB), laboratory control sample (LCS), laboratory control sample duplicate (LCSD), field duplicate (FD), matrix spike (MS), and matrix spike duplicate (MSD) – are prepared for each media type. In some cases, depending on the project, additional QC samples may be required. For samples with high concentrations of PFAS, an FD may be warranted. The QAPP should also reference the laboratory SOP.

4.6.2 Field Quality Control Samples

Field QC samples can be used to evaluate the field equipment and supplies as well as assess the possibility of cross-contamination during sampling, transport, and storage of samples. For samples such as equipment rinse blanks (EB), field blanks (FB), and trip blanks (TB) the following is required:

- EB should be collected by passing laboratory verified PFAS-free water over or through decontaminated field sampling equipment before the collection of samples to assess the adequacy of the decontamination process and/or to evaluate potential contamination from the equipment used during sampling. The recommended frequency should be in the QAPP.

- FB are prepared in the laboratory by placing an aliquot of PFAS-free water reagent water in a sample container and treating it as a sample in all respects, including shipment to the sampling site, exposure to sampling site conditions, storage, preservation, and all analytical procedures. The purpose of the FB is to determine if method analytes or other interferences are present in the field environment. The recommended frequency should be in the QAPP.

- TB are a bottle of PFAS-free water that should be prepared in the laboratory, should then travel from the laboratory to the site, and then get transported back to the laboratory without having been exposed to any sampling procedures. Typically, a TB is used for volatile compounds, but it may be recommended for PFAS sampling to assess cross-contamination introduced from the laboratory and during shipping procedures. The recommended frequency should be in the QAPP

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Michigan Department of Environmental Quality

MDEQ PFAS SAMPLING QUICK REFERENCE FIELD GUIDE[1]

All Items Used During Sampling Event

● Prohibited
• Items or materials that contain fluoropolymers such as ○ Polytetrafluoroethylene (PTFE), that includes the trademarks Teflon® and Hostaflon® ○ Polyvinylidene fluoride (PVDF), that includes the trademark Kynar® ○ Polycholotrifluoroethylene (PCTFE), that includes the trademark Neoflon ® ○ Ethylene-tetrafluoro-ethylene (ETFE), that includes the trademark Tefzel® ○ Fluorinated ethylene propylene (FEP), that includes the trademarks Teflon® FEP and Hostaflon® FEP • Items or materials that contain any other fluoropolymer

Pumps, Tubing, and Sampling Equipment

● Prohibited	🟩 Allowable	▲ Needs Screening[2]
• Items or materials containing any fluoropolymer (potential items include tubing, valves, or pipe thread seal tape)	• High-density polyethylene (HDPE) • Low-density polyethylene (LDPE) tubing • Polypropylene • Silicone • Stainless-steel • Any items used to secure sampling bottles made from: ○ Natural rubber ○ Nylon (cable ties) ○ Uncoated metal springs ○ Polyethylene	• Any items or materials that will come into direct contact with the sample that have **not** been verified to be PFAS-free ○ Do not assume that any sampling items or materials are PFAS-free based on composition alone

Sample Storage and Preservation

● Prohibited	🟩 Allowable	▲ Needs Screening[2]
• Polytetrafluoroethylene (PTFE): Teflon® lined bottles or caps	• Glass jars[4] • Laboratory-provided PFAS-Free bottles: ○ HDPE or polypropylene • Regular wet ice • Thin HDPE sheeting • LDPE resealable storage bags (i.e. Ziploc®) that will not contact the sample media[6]	• Aluminium foil[4] • Chemical or blue ice[5] • Plastic storage bags other than those listed as 🟩 Allowable • Low-density polyethylene (LDPE) bottles

Field Documentation

● Prohibited	🟩 Allowable	▲ Needs Screening[2]
• Clipboards coated with PFAS • Notebooks made with PFAS treated paper • PFAS treated loose paper • PFAS treated adhesive paper products	• Loose paper (non-waterproof, non-recycled) • Rite in the Rain® notebooks • Aluminium, polypropylene, or Masonite field clipboards • Ballpoint pens, pencils, and Fine or Ultra-Fine Point Sharpie® markers	• Plastic clipboards, binders, or spiral hard cover notebooks • All markers not listed as 🟩 Allowable • Post-It® Notes or other adhesive paper products • Waterproof field books

Decontamination

● Prohibited	🟩 Allowable	▲ Needs Screening[2]
• Decon 90® • PFAS treated paper towel	• Alconox®, Liquinox®, or Citranox® • Triple rinse with PFAS-free deionized water • Cotton cloth or untreated paper towel	• Municipal water • Recycled paper towels or chemically treated paper towels

www.michigan.gov/pfasresponse 800-662-9278 Revised 10/17/2018

Clothing, Boots, Rain Gear, and PPE

🔴 Prohibited	🟩 Allowable	▲ Needs Screening[2]
• New or unwashed clothing • Anything made of or with: ○ Gore-Tex™ or other water-resistant synthetics • Anything applied with or recently washed with: ○ Fabric softeners ○ Fabric protectors, including UV protection ○ Insect resistant chemicals ○ Water, dirt, and/or stain resistant chemicals	• Powderless nitrile gloves • Well-laundered synthetic or 100% cotton clothing, with most recent launderings not using fabric softeners • Made of or with: ○ Polyurethane ○ Polyvinyl chloride (PVC) ○ Wax coated fabrics ○ Rubber / Neoprene ○ Uncoated Tyvek®	• Latex gloves • Water and/or dirt resistant leather gloves • Any special gloves required by a HASP • Tyvek® suits, clothing that contains Tyvek®, or coated Tyvek®

Food and Beverages

🔴 Prohibited	🟩 Allowable
• No food should be consumed in the staging or sampling areas, including pre-packaged food or snacks. 🟩 If consuming food on-site becomes necessary, move to the staging area and remove PPE. After eating, wash hands thoroughly and put on new PPE.	• Brought and consumed only outside the vicinity of the sampling area: ○ Bottled water ○ Hydration drinks (i.e. Gatorade®, Powerade®)

Personal Care Products (PCPs) - for day of sample collection[6]

🔴 Prohibited	🟩 Allowable	▲ Needs Screening[2]
• Any PCPs[6], sunscreen, and insect repellent applied in the sampling area.	PCPs[6], sunscreens, and insect repellents applied in the staging area, away from sampling bottles and equipment followed by thoroughly washing hands: **PCPs[6]:** • Cosmetics, deodorants/antiperspirants, moisturizers, hand creams, and other PCPs[6] **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 50 • Meijer® Sunscreen Continuous Spray Broad Spectrum SPF 30 • 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+ • Neutrogena® UltraSheer Dry-Touch Sunscreen Broad Spectrum SPF 30 **Insect Repellents:** • OFF® Deep Woods • Sawyer® Permethrin	• Products other than those listed as 🟩Allowable

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[1] This table is not considered to be a complete listing of prohibited or allowable materials. All materials should be evaluated prior to use during sampling. The manufacturers of various products should be contacted in order to determine if PFAS was used in the production of any particular product.

[2] Equipment blank samples should be taken to verify these products are PFAS-free prior to use during sampling.

[3] **For surface water foam samples:** LDPE storage bags may be used in the sampling of foam on surface waters. In this instance, it is allowable for the LDPE bag to come into direct contact with the sample media.

[4] **For fish and other wildlife samples:** Depending on the project objectives, glass jars and aluminum foil might be used for PFAS sampling. PFAS has been found to bind to glass and if the sample is stored in a glass jar, a rinse of the jar is required during the sample analysis. PFAS are sometimes used as a protective layer for some aluminum foils. An equipment blank sample should be collected prior to any aluminum foil use.

[5] Regular ice is recommended as there are concerns that chemical and blue ice may not cool and maintain the sample at or below 42.8˚F (6°C) (as determined by EPA 40 CFR 136 – NPDES) during collection and through transit to the laboratory.

[6] Based on evidence, avoidance of PCPs is considered to be precautionary because none have been documented as having cross-contaminated samples due to their use. However, if used, application of PCPs must be done at the staging area and away from sampling bottles and equipment, and hands must be thoroughly washed after the use of any PCPs prior to sampling.

🔴- Prohibited 🟩- Allowable ▲- Needs Screening

Appendix 3: PFAS Analyte Groups and Names

Appendix 3 PFAS Analyte Groups and Full Names

Analyte group	Compound Abbreviation	Compound Name
Ether carboxylates	ADONA	Decafluoro-3H-4,8-dioxanonoate
Ether carboxylates	HFPO-DA	2,3,3,3-Tetrafluoro-2-(1,1,2,2,3,3,3-heptafluoropropoxy)propionic acid
Ether carboxylates	NFDHA	Perfluoro-3,6-dioxaheptanoate
Ether carboxylates	PFMBA	Perfluoro-4-methoxybutanoate
Ether carboxylates	PFMPA	Perfluoro-3-methoxypropanoate
Ether sulfonates	11Cl-PF3OUdS	11-chloroeicosafluoro-3-oxaundecane-1-sulfonic acid
Ether sulfonates	9Cl-PF3ONS	9-chlorohexadecafluoro-3-oxanonane-1-sulfonic acid
Ether sulfonates	PFEESA	Ether carboxylates
Fluorotelomer carboxylates	3:3 FTCA	2H, 2H, 3H, 3H-perfluorohexanoic acid
Fluorotelomer carboxylates	5:3 FTCA	2H, 2H, 3H, 3H-perfluorooctanoic acid
Fluorotelomer carboxylates	7:3 FTCA	2H, 2H, 3H, 3H-perfluorodecanoic acid
Fluorotelomer sulfonates	4:2 FTS	1H, 1H, 2H, 2H-perfluorohexane sulfonate (4:2 FTS)
Fluorotelomer sulfonates	6:2 FTS	1H, 1H, 2H, 2H-perfluorooctane sulfonate (6:2 FTS)
Fluorotelomer sulfonates	8:2 FTS	1H, 1H, 2H, 2H-perfluorodecane sulfonate (8:2 FTS)
Perfluoroalkyl carboxylates	PFBA	Perfluorobutanoate (PFBA)
Perfluoroalkyl carboxylates	PFDA	Perfluorodecanoate (PFDA)
Perfluoroalkyl carboxylates	PFDoA	Perfluorododecanoate (PFDoA)
Perfluoroalkyl carboxylates	PFHpA	Perfluoroheptanoate (PFHpA)
Perfluoroalkyl carboxylates	PFHxA	Perfluorohexanoate (PFHxA)
Perfluoroalkyl carboxylates	PFNA	Perfluorononanoate (PFNA)
Perfluoroalkyl carboxylates	PFOA	Perfluorooctanoate (PFOA)
Perfluoroalkyl carboxylates	PFPeA	Perfluoropentanoate (PFPeA)
Perfluoroalkyl carboxylates	PFTeDA	Perfluorotetradecanoate (PFTeDA)
Perfluoroalkyl carboxylates	PFTrDA	Perfluorotridecanoate (PFTrDA)
Perfluoroalkyl carboxylates	PFUnA	Perfluoroundecanoate (PFUnA)
Perfluoroalkyl sulfonates	PFBS	Perfluorobutanesulfonate (PFBS)
Perfluoroalkyl sulfonates	PFDoS	Perfluorododecanesulfonate (PFDoS)
Perfluoroalkyl sulfonates	PFDS	Perfluorodecanesulfonate (PFDS)
Perfluoroalkyl sulfonates	PFHpS	Perfluoroheptanesulfonate (PFHpS)
Perfluoroalkyl sulfonates	PFHxS	Perfluorohexanesulfonate (PFHxS)
Perfluoroalkyl sulfonates	PFNS	Perfluorononanesulfonate (PFNS)
Perfluoroalkyl sulfonates	PFOS	Perfluorooctanesulfonate (PFOS)
Perfluoroalkyl sulfonates	PFPeS	Perfluoropentanesulfonate (PFPeS)

Analyte group	Compound Abbreviation	Compound Name
Perfluorooctane sulfonamide ethanols	N-EtFOSE	N-Ethylperfluoro-1-octanesulfonamidoethanol (N-EtFOSE)
Perfluorooctane sulfonamide ethanols	N-MeFOSE	N-Methylperfluoro-1-octanesulfonamidoethanol (N-MeFOSE)
Perfluorooctane sulfonamideacetic acids	EtFOSAA	N-Ethylperfluoro-1-octanesulfonamidoacetic acid (N-EtFOSAA)
Perfluorooctane sulfonamideacetic acids	MeFOSAA	N-Methylperfluoro-1-octanesulfonamidoacetic acid (N-MeFOSAA)
Perfluorooctane sulfonamides	N-EtFOSA	N-Ethylperfluorooctanesulfonamide (N-EtFOSA)
Perfluorooctane sulfonamides	N-MeFOSA	N-Methylperfluorooctanesulfonamide (N-MeFOSA)
Perfluorooctane sulfonamides	PFOSA	Perfluorooctanesulfonamide (PFOSA) 2

Appendix 4: PFAS Detection Limits

Appendix 4: Detection Limits for Surface Water, Sediment, and Fish Tissue

Surface Water Units ng/L	**MLA-060 Phase I Original 13**		**MLA-110 Rev 01 30 Compounds**		**MLA-110 Rev 02 40 Compounds**	
Compound	**Min**	**Max**	**Min**	**Max**	**Min**	**Max**
11Cl-PF3OUdS	na	na	na	na	1.12	12.9
3:3 FTCA	na	na	na	na	1.12	12.9
4:2 FTS	na	na	1.5	1.61	1.12	12.9
5:3 FTCA	na	na	na	na	6.98	80.8
6:2 FTS	na	na	1.35	1.45	1.01	11.7
7:3 FTCA	na	na	na	na	6.98	80.8
8:2 FTS	na	na	1.5	1.61	1.12	12.9
9Cl-PF3ONS	na	na	na	na	1.12	13
ADONA	na	na	na	na	1.12	12.9
EtFOSAA	na	na	0.748	0.806	0.279	3.23
HFPO-DA	na	na	0.355	0.383	1.06	12.3
MeFOSAA	na	na	0.374	0.403	0.279	3.23
N-EtFOSA	na	na	0.935	1.01	0.698	8.08
N-EtFOSE	na	na	2.86	3.08	2.09	24.2
NFDHA	na	na	na	na	0.558	1.8
N-MeFOSA	na	na	0.43	0.464	0.321	3.72
N-MeFOSE	na	na	3.82	4.11	2.79	32.3
PFBA	1.0	2.0	1.53	1.65	1.12	12.9
PFBS	1.0	2.0	0.383	0.413	0.279	3.23
PFDA	1.0	2.0	0.374	0.403	0.279	3.23
PFDoA	1.0	2.0	0.374	0.403	0.279	3.23
PFDoS	na	na	0.355	0.383	0.279	3.23
PFDS	na	na	0.383	0.413	0.279	3.23
PFEESA	na	na	na	na	0.279	3.23
PFHpA	1.0	2.0	0.383	0.413	0.279	3.23
PFHpS	na	na	0.355	0.383	0.279	3.23
PFHxA	1.0	2.0	0.383	0.413	0.279	4.43
PFHxS	1.0	2.0	0.383	0.413	0.279	3.23
PFMBA	na	na	na	na	0.279	3.23
PFMPA	na	na	na	na	0.558	6.46
PFNA	1.0	2.0	0.374	0.403	0.279	3.23
PFNS	na	na	0.355	0.383	0.279	3.23
PFOA	1.0	2.0	0.383	0.413	0.279	4.01
PFOS	1.0	2.0	0.383	0.413	0.279	3.23

Surface Water Units ng/L	MLA-060 Phase I Original 13		MLA-110 Rev 01 30 Compounds		MLA-110 Rev 02 40 Compounds	
Compound	**Min**	**Max**	**Min**	**Max**	**Min**	**Max**
PFOSA	1.0	2.0	0.383	0.413	0.279	3.23
PFPeA	1.0	2.0	0.767	0.826	0.558	6.46
PFPeS	na	na	0.355	0.383	0.28	3.25
PFTeDA	na	na	0.374	0.403	0.353	7.43
PFTrDA	na	na	0.374	0.403	0.279	3.23
PFUnA	1.0	2.0	0.374	0.403	0.279	3.23

Sediment Units ng/g	MLA-060 Phase I Original 13		MLA-110 Rev 01 30 Compounds		MLA-110 Rev 02 40 Compounds	
Compound	**Min**	**Max**	**Min**	**Max**	**Min**	**Max**
11Cl-PF3OUdS	na	na	na	na	0.0736	0.737
3:3 FTCA	na	na	na	na	0.119	0.736
4:2 FTS	na	na	0.145	0.824	0.0736	0.736
5:3 FTCA	na	na	na	na	0.743	4.6
6:2 FTS	na	na	0.131	0.742	0.0663	0.85
7:3 FTCA	na	na	na	na	0.743	4.6
8:2 FTS	na	na	0.145	0.824	0.0736	0.626
9Cl-PF3ONS	na	na	na	na	0.0736	0.738
ADONA	na	na	na	na	0.0736	0.736
EtFOSAA	na	na	0.0726	0.412	0.0184	0.236
HFPO-DA	na	na	0.0345	0.0664	0.0736	0.736
MeFOSAA	na	na	0.0363	0.206	0.0184	0.184
N-EtFOSA	na	na	0.0907	0.515	0.046	0.515
N-EtFOSE	na	na	0.278	1.58	0.138	1.84
NFDHA	na	na	na	na	0.0594	0.368
N-MeFOSA	na	na	0.0417	0.237	0.0212	0.184
N-MeFOSE	na	na	0.37	2.1	0.184	1.84
PFBA	0.1	0.2	0.149	0.844	0.119	0.912
PFBS	0.1	0.2	0.0372	0.211	0.0184	0.184
PFDA	0.1	0.2	0.0363	0.206	0.0184	0.184
PFDoA	0.1	0.2	0.0363	0.206	0.0184	0.147
PFDoS	na	na	0.0345	0.196	0.0233	0.184
PFDS	na	na	0.0372	0.211	0.0269	0.184
PFEESA	na	na	na	na	0.0297	0.184

Sediment Units ng/g	**MLA-060 Phase I Original 13**		**MLA-110 Rev 01 30 Compounds**		**MLA-110 Rev 02 40 Compounds**	
Compound	**Min**	**Max**	**Min**	**Max**	**Min**	**Max**
PFHpA	0.1	0.2	0.0372	0.211	0.0184	0.237
PFHpS	na	na	0.0345	0.196	0.0184	0.184
PFHxA	0.1	0.2	0.0372	0.211	0.0184	0.311
PFHxS	0.1	0.2	0.0372	0.211	0.0184	0.184
PFMBA	na	na	na	na	0.0297	0.184
PFMPA	na	na	na	na	0.0594	0.368
PFNA	0.1	0.2	0.0363	0.206	0.0184	0.184
PFNS	na	na	0.0345	0.196	0.0184	0.184
PFOA	0.1	0.2	0.0372	0.211	0.0184	0.184
PFOS	0.1	0.2	0.0372	0.211	0.0184	0.184
PFOSA	0.1	0.2	0.0372	0.211	0.0184	0.184
PFPeA	0.1	0.2	0.0744	0.422	0.0368	0.368
PFPeS	na	na	0.0345	0.196	0.0184	0.185
PFTeDA	na	na	0.0363	0.206	0.0184	0.184
PFTrDA	na	na	0.0363	0.206	0.0184	0.184
PFUnA	0.1	0.2	0.0363	0.206	0.0184	0.184

Tissue Units ng/g	**MLA-060 Phase I Original 13**		**MLA-110 Rev 02 40 Compounds**	
Compound	**Min**	**Max**	**Min**	**Max**
11Cl-PF3OUdS	na	na	0.341	1.11
3:3 FTCA	na	na	0.34	1.11
4:2 FTS	na	na	0.34	1.11
5:3 FTCA	na	na	2.13	6.94
6:2 FTS	na	na	0.306	1
7:3 FTCA	na	na	2.13	6.94
8:2 FTS	na	na	0.289	0.944
9Cl-PF3ONS	na	na	0.341	1.11
ADONA	na	na	0.34	1.11
EtFOSAA	na	na	0.0851	0.278
HFPO-DA	na	na	0.34	1.11
MeFOSAA	na	na	0.0851	0.278
N-EtFOSA	na	na	0.238	0.778
N-EtFOSE	na	na	0.851	2.78

Tissue Units ng/g	**MLA-060 Phase I Original 13**		**MLA-110 Rev 02 40 Compounds**	
Compound	**Min**	**Max**	**Min**	**Max**
NFDHA	na	na	0.17	1.33
N-MeFOSA	na	na	0.0851	0.278
N-MeFOSE	na	na	0.851	2.78
PFBA	0.5	1.0	0.34	1.11
PFBS	0.5	1.0	0.0851	0.278
PFDA	0.5	1.0	0.0851	0.278
PFDoA	0.5	1.0	0.0681	0.222
PFDoS	na	na	0.0851	0.278
PFDS	na	na	0.0851	0.278
PFEESA	na	na	0.0851	0.278
PFHpA	0.5	1.0	0.0851	0.278
PFHpS	na	na	0.0851	0.278
PFHxA	0.5	1.0	0.0851	0.278
PFHxS	0.5	1.0	0.0851	0.278
PFMBA	na	na	0.0851	0.278
PFMPA	na	na	0.17	0.556
PFNA	0.5	1.0	0.0851	0.278
PFNS	na	na	0.0851	0.278
PFOA	0.5	1.0	0.0851	0.278
PFOS	0.5	1.0	0.0851	0.495
PFOSA	0.5	1.0	0.0851	0.278
PFPeA	0.5	1.0	0.17	0.556
PFPeS	na	na	0.0855	0.279
PFTeDA	na	na	0.0851	0.303
PFTrDA	na	na	0.0851	0.278
PFUnA	0.5	1.0	0.0851	0.278

Appendix 5: PFAS Data Surface Water

PFAS in Surface Water (ng/L): *Note: Empty cells denote levels below detection limit. Cells that contain “na” denote analytes not quantified in the method used for that sample.*

Site	Sample ID	11Cl-PF3OUdS	3:3 FTCA	4:2 FTS	5:3 FTCA	6:2 FTS	7:3 FTCA	8:2 FTS	9Cl-PF3ONS	ADONA	EtFOSAA
1	LL1A	na	na		na		na		na	na	
1	LL1B	na	na		na		na		na	na	
1	LL1C	na	na		na		na		na	na	
1	LL1D	na	na		na		na		na	na	
2	LL2D0721211038										
2	LL2A										
2	LL2F										
2	LL2G										1.13
2	LL4										
2	LL2B										
2	LL2C										0.627
2	LL2D0721211042										
2	LL2E										
2	LL2H6in										
2	LL2H6indup										
2	LL2HSUR										12.9
3	LL3A										
3	LL3B										
3	LL3CPOND										
3	LL3CSW										
4	04-FF1B-070821										
4	04-FFIA-070821					11.6					
5	5-FF2A										
5	5FF2B										
6	CW 1	na	na		na		na		na	na	
6	CW 2	na	na		na		na		na	na	
6	CW 3	na	na		na		na		na	na	
6	CW 3.1	na	na		na	6.58	na		na	na	
6	CW 4	na	na		na		na		na	na	
7	7SFFdwn071019		na		na		na				
8	FF6up100719		na		na		na				
8	FF 5 dwn		na		na		na				
9	9AGPdwn071019		na		na		na				
9	9AGPup071019		na		na		na				
10	10-CW5-071821										
10	10-CW6-070821										
10	10-CW7-070821										

Appendix *5* PFAS in Surface Water
Note: Empty cells denote levels below detection limit.
Cells that contain “na” denote analytes not quantified in the method used for that sample.

PFAS in Surface Water (ng/L)

Site	Sample ID	11Cl-PF3OUdS	3:3 FTCA	4:2 FTS	5:3 FTCA	6:2 FTS	7:3 FTCA	8:2 FTS	9Cl-PF3ONS	ADONA	EtFOSAA
11	POTW 11 dwn		na		na		na				
11	POTW 11 up		na		na		na				
12	POTW 8		na		na	6.6	na				
13	13-WHIP					1.93					
14	POTW 14 dwn		na		na		na				
15	POTW 6		na		na		na				
15	POTW6-15-DS										
15	POTW6-15-UPS										
16	FTM020										
16	HORICON 081623										
16	FSW1		na		na	4.08	na				
16	FSW2		na		na	3.57	na				
16	FSW3		na		na		na				
17	ATLMST (4)										
17	ATLPC1 (1)		5.33								
17	ATLPC2 (2)										
17	ATLSBA6					6.78					
17	ATLSU3 (4b)										
18	18RNG2										
18	18RNG4										
18	18RNG6 FR					2.74					
18	18RNG9CNT (3)										
19	FTM022										
20	FTM028										
21	TSB080321										
22	BUTTERWORTH POND	na	na		na		na		na	na	
22	CEDAR POND	na	na		na		na		na	na	
22	ECHO LAKE POND #1	na	na		na		na		na	na	
22	ECHO LAKE POND #2	na	na		na		na		na	na	
22	LOCUSTWOOD POND	na	na		na		na		na	na	

Appendix *5* PFAS in Surface Water
Note: Empty cells denote levels below detection limit.
Cells that contain “na” denote analytes not quantified in the method used for that sample.

PFAS in Surface Water (ng/L)

Site	Sample ID	HFPO-DA	MeFOSAA	N-EtFOSA	N-EtFOSE	NFDHA	N-MeFOSA	N-MeFOSE	PFBA	PFBS	PFDA
1	LL1A							na	7.96	81.4	
1	LL1B							na	3.8	1.26	0.658
1	LL1C							na		0.811	
1	LL1D							na		5.86	
2	LL2D0721211038								3.61	0.821	0.48
2	LL2A								4	1.19	0.639
2	LL2F								5.48	1.32	0.453
2	LL2G		0.744						6.25	4.9	2.4
2	LL4								3.39	0.622	0.673
2	LL2B								3.76	1.88	0.564
2	LL2C								5.07	2.36	1
2	LL2D0721211042								3.75	0.857	0.471
2	LL2E								6.34	1.47	0.453
2	LL2H6in								6.06	3.2	0.965
2	LL2H6indup								5.39	2.5	0.544
2	LL2HSUR										38.9
3	LL3A								22.9	39	0.372
3	LL3B								1.56		0.678
3	LL3CPOND								46.8	115	3.99
3	LL3CSW								49.1	109	7.14
4	04-FF1B-070821								2.19	1.48	
4	04-FFIA-070821								32.1	2.92	2.55
5	5-FF2A								56.1	31.9	6.73
5	5FF2B								19.3	6.94	6.3
6	CW 1							na		1.37	0.666
6	CW 2							na	3.32	3.42	
6	CW 3							na	1.87	1.41	
6	CW 3.1							na		1.21	0.865
6	CW 4							na		0.456	
7	7SFFdwn071019							na	7.83	2.46	
8	FF6up100719							na	4.16	3.16	
8	FF 5 dwn							na	3.92	2.6	
9	9AGPdwn071019							na		0.753	
9	9AGPup071019							na		0.728	
10	10-CW5-071821									1.07	0.679
10	10-CW6-070821								4.47	5.58	0.755
10	10-CW7-070821								3.84	4.82	0.559
11	POTW 11 dwn							na		2.26	
11	POTW 11 up							na	4.55	4.15	
12	POTW 8							na	8.96	4.32	1.03
13	13-WHIP								6.3	2.39	0.624
14	POTW 14 dwn							na	5.79	3.62	
15	POTW 6							na	5.38	3.11	
15	POTW6-15-DS								2.88	2.25	0.497
15	POTW6-15-UPS								3.85	2.21	0.631
16	FTM020								1.71	1.06	
16	HORICON 081623									0.74	
16	FSW1					na					
16	FSW2					na				2.03	
16	FSW3					na					
17	ATLMST (4)								2.61	4.76	
17	ATLPC1 (1)									3.08	1.32
17	ATLPC2 (2)									0.483	

Appendix *5* PFAS in Surface Water

Note: Empty cells denote levels below detection limit.

Cells that contain “na” denote analytes not quantified in the method used for that sample.

PFAS in Surface Water (ng/L)

Site	Sample ID	HFPO-DA	MeFOSAA	N-EtFOSA	N-EtFOSE	NFDHA	N-MeFOSA	N-MeFOSE	PFBA	PFBS	PFDA
17	ATLSBA6								5.76	5.39	0.488
17	ATLSU3 (4b)										
18	18RNG2								2.29		
18	18RNG4								1.48	0.546	
18	18RNG6 FR								1.91	0.811	
18	18RNG9CNT (3)								4.7	2.47	0.468
19	FTM022								3.56	6.02	0.51
20	FTM028								5.95	3.73	0.963
21	TSB080321								1.8	0.88	
22	BUTTERWORTH POND	0.705				na			4.54		
22	CEDAR POND					na			2.75		0.493
22	ECHO LAKE POND #1					na					
22	ECHO LAKE POND #2					na					
22	LOCUSTWOOD POND	0.583				na			4.26	0.792	1.9

Appendix *5* PFAS in Surface Water
Note: Empty cells denote levels below detection limit.
Cells that contain “na” denote analytes not quantified in the method used for that sample.

PFAS in Surface Water (ng/L)

Site	Sample ID	PFDoA	PFDoS	PFDS	PFEESA	PFHpA	PFHpS	PFHxA	PFHxS	PFMBA	PFMPA
1	LL1A				na	25.4	0.991	32.3	18.6	na	na
1	LL1B				na	2.75	0.421	2.58	1.78	na	na
1	LL1C				na	0.556		0.893	0.967	na	na
1	LL1D				na	2.55	0.458	2.41	2.57	na	na
2	LL2D0721211038					2.11		2.91	0.706		
2	LL2A					2.72		4.62	3.59		
2	LL2F					2.87		6.51	1.77		
2	LL2G					5.56	0.49	17.9	4.3		
2	LL4					2.1		3.29	1.05		
2	LL2B					2.78		4.31	2.04		
2	LL2C					3.26		8.77	1.67		
2	LL2D0721211042					2.47		3.98	0.731		
2	LL2E					1.78		3.65	1.14		
2	LL2H6in					4.05		5.9	2.79		
2	LL2H6indup					3.63		6.04	2.72		
2	LL2HSUR	5.01				4.8		6.45			
3	LL3A					31.9	0.499	57.8	6.84		
3	LL3B					1.05		1.53			
3	LL3CPOND					73.5	1.32	113	12.6		
3	LL3CSW	0.758		0.552		71.8	1.58	143	13.6		
4	04-FF1B-070821					1.44		1.03	0.58		
4	04-FFIA-070821					50.5		78.4	7		
5	5-FF2A					27.4		39.3	26.4		
5	5FF2B					28.3	14.9	72.4	215		
6	CW 1				na	12.2	1.09	6.45	24.6	na	na
6	CW 2				na	1.06		1.61	6.33	na	na
6	CW 3				na	1.57		2.96	7.32	na	na
6	CW 3.1				na	7.06	1.43	6.95	31.6	na	na
6	CW 4				na	0.91		0.84	0.854	na	na
7	7SFFdwn071019				na	3.96		5.03	4.53	na	na
8	FF6up100719				na	3.67		5.84	3.23	na	na
8	FF 5 dwn				na	4.18		5.36	4.23	na	na
9	9AGPdwn071019				na	0.991		1.13	1.87	na	na
9	9AGPup071019				na	0.814			2.02	na	na
10	10-CW5-071821					2.34		2.73	5.51		
10	10-CW6-070821					3.07		3.32	1.48		
10	10-CW7-070821					2.91		3.02	1.42		
11	POTW 11 dwn				na	2.58		2.56	4.48	na	na
11	POTW 11 up				na	3.21		4.17	5.68	na	na
12	POTW 8				na	4.05		11.5	4.53	na	na
13	13-WHIP					4.14		7.09	3.62		
14	POTW 14 dwn				na	3.54		12.7	4.81	na	na
15	POTW 6				na	2.88		5.45	3.61	na	na
15	POTW6-15-DS					2.83		4.58	2.49		
15	POTW6-15-UPS					2.59		4.86	2.38		
16	FTM020					1.65		1.63	4.73		
16	HORICON 081623					1.03		1.7	4.03		
16	FSW1				na	3.09		2.77	10.7	na	na
16	FSW2				na	2.6	0.985	4.96	13.1	na	na
16	FSW3				na	0.846		0.752	0.887	na	na
17	ATLMST (4)					5.57	1.26	7.95	25.9		
17	ATLPC1 (1)					4.12		5.13	1.56		
17	ATLPC2 (2)					0.752		0.922	1.08		

Appendix *5* PFAS in Surface Water
Note: Empty cells denote levels below detection limit.
Cells that contain “na” denote analytes not quantified in the method used for that sample.

Site	Sample ID	PFDoA	PFDoS	PFDS	PFEESA	PFHpA	PFHpS	PFHxA	PFHxS	PFMBA	PFMPA
17	ATLSBA6					8.13	2.61	18.6	55.1		
17	ATLSU3 (4b)										
18	18RNG2					0.536		0.463			
18	18RNG4					0.578		0.646	1.32		
18	18RNG6 FR					0.717		1.46	0.909		
18	18RNG9CNT (3)					1.59		2.99	1.71		
19	FTM022					3.32		4.02	1.73		
20	FTM028					3.04		3.68	2.85		
21	TSB080321					1.51		1.69	1.52		
22	BUTTERWORTH POND				na	2.18		1.73		na	na
22	CEDAR POND				na	1.46		1.34		na	na
22	ECHO LAKE POND #1				na					na	na
22	ECHO LAKE POND #2				na					na	na
22	LOCUSTWOOD POND				na	3.34		2.68	0.42	na	na

Appendix *5* PFAS in Surface Water
Note: Empty cells denote levels below detection limit.
Cells that contain “na” denote analytes not quantified in the method used for that sample.

PFAS in Surface Water (ng/L)

Site	Sample ID	PFNA	PFNS	PFOA	PFOS	PFOSA	PFPeA	PFPeS	PFTeDA	PFTrDA	PFUnA
1	LL1A	1.87		79.3	9.45		21.7	2.91			
1	LL1B	2.4		8.36	9.91	0.743	2.72				0.505
1	LL1C	0.655		1.34	1.79	0.407					
1	LL1D	1.09		12.3	4.41	0.493	1.58				
2	LL2D0721211038	0.69		3.94	2.86		3.85				
2	LL2A	1.79		4.94	16.4		4.17	0.472			
2	LL2F	3.04		5.08	4.58		6.11	0.41			
2	LL2G	3.47		15.8	31.9	0.727	18.5	0.969			
2	LL4	1.95		3.43	1.34		3.69				0.375
2	LL2B	2.16		6.53	7.39		4.91	0.463			
2	LL2C	2.97		7.7	9.66		8.23	0.376			
2	LL2D0721211042	0.856		3.83	3.37		2.88				
2	LL2E	0.805		3.52	5.61		2.41				
2	LL2H6in	2.74		6.88	10.3		7.36	0.535			0.668
2	LL2H6indup	2.57		7.11	9.46		6.34	0.612			
2	LL2HSUR	7.15		8.71	92.9	4.54	6.95				93.7
3	LL3A	0.868		74.3	1.39		44.4	0.758			
3	LL3B	1.15		1.61	0.61		1.64				0.373
3	LL3CPOND	17		136	23.6		93.4	1.5			0.537
3	LL3CSW	21.4		135	29.5		90.3	1.45	0.436	0.373	1.72
4	04-FF1B-070821	0.758		2.27	1.63		2.31				
4	04-FFIA-070821	15.4		22.7	18.1		99.4	0.852			
5	5-FF2A	11.6		66.7	72.9		41.7	4.17			
5	5FF2B	18		45.5	537		40	14.2			3.93
6	CW 1	2.68		9.83	37.8	0.464	3.82	1.96			
6	CW 2	0.941		4.26	6.39	0.629	0.845				
6	CW 3	0.866		5.15	3.28		2.28	0.767			
6	CW 3.1	2.59		9.99	51.2	0.441	3.97	2.25			0.389
6	CW 4	0.813		2.2	1.19	0.81					
7	7SFFdwn071019	2.55		19.8	13		6.29				
8	FF6up100719	1.75		15.1	6.31		5.41				
8	FF 5 dwn	1.51	na	16.4	11.4	0.844	4.67				
9	9AGPdwn071019	1.45		3.51	2.76		1.69				
9	9AGPup071019			2.52	2.14						
10	10-CW5-071821	1.6		5.18	20		2.43	0.647			0.444
10	10-CW6-070821	1.22		5.98	4.96		3.35				
10	10-CW7-070821	1.26		6.02	4.54		4.29				
11	POTW 11 dwn	1.07	na	15.6	6.69		1.95				
11	POTW 11 up	0.954	na	15.7	6.48		3.57	0.814			
12	POTW 8	1.98	na	12.3	11.2		15.8	0.809			
13	13-WHIP	1.67		9.39	9.04		11.6	0.848			
14	POTW 14 dwn		na	21.2	4.18		26.7				
15	POTW 6	1.82	na	10	9.23		5.43				
15	POTW6-15-DS	1.36		7.06	7.15		4.87	0.435			
15	POTW6-15-UPS	1.42		7.77	7.38		4.88	0.4			
16	FTM020	1.06		2.86	5.71		1.76	0.766			
16	HORICON 081623	0.902		2.51	7.14		1.43	0.592			
16	FSW1	1.97		6.28	28.6		2.13				
16	FSW2	1.71		8.87	78.7		3.09	1.83			
16	FSW3	1.5		4.78	3.42	0.852					
17	ATLMST (4)	4.84		19.7	39.4	0.622	5.29	3.05			
17	ATLPC1 (1)	6.45		14.8	15.1		5.25				
17	ATLPC2 (2)	0.495		2.08	2.3		0.835				

Appendix *5* PFAS in Surface Water

Note: Empty cells denote levels below detection limit.

Cells that contain “na” denote analytes not quantified in the method used for that sample.

Site	Sample ID	PFNA	PFNS	PFOA	PFOS	PFOSA	PFPeA	PFPeS	PFTeDA	PFTrDA	PFUnA
17	ATLSBA6	6.64		35.2	112	0.633	15	6.45			
17	ATLSU3 (4b)			0.476							
18	18RNG2			2.53	2.88						
18	18RNG4			2.85	4.59	0.493					
18	18RNG6 FR	0.42		2.56	4.29	0.372	1.67				
18	18RNG9CNT (3)	1.32		9.47	8.08		3.51				
19	FTM022	1.63		14	5.29		5.23				
20	FTM028	1.32		9.28	8.27		6.25				
21	TSB080321	1.9		3.81	2.75		1.78				
22	BUTTERWORTH POND	1.58		2.95	1.38		1.33				
22	CEDAR POND	2.46		2.11	1.72		1.5				1.24
22	ECHO LAKE POND #1				1.82						
22	ECHO LAKE POND #2	0.416		0.689	0.868						
22	LOCUSTWOOD POND	10		6.86	3.76	0.475	2.71				1.81

Appendix *5* PFAS in Surface Water
Note: Empty cells denote levels below detection limit.
Cells that contain “na” denote analytes not quantified in the method used for that sample.

Appendix 6: PFAS Data Sediment

PFAS in Sediment (ng/g, dry weight basis): *Note: Empty cells denote levels below detection limit. Cells that contain “na” denote analytes not quantified in the method used for that sample.*

Site ID	Sample ID	11Cl-PF3OUdS	3:3 FTCA	4:2 FTS	5:3 FTCA	6:2 FTS	7:3 FTCA	8:2 FTS	9Cl-PF3ONS	ADONA	EtFOSAA
1	LL1A	na	na		na		na		na	na	
1	LL1B	na	na		na		na		na	na	
1	LL1C	na	na		na		na		na	na	
1	LL1D	na	na		na		na		na	na	
2	LL2A										
2	LL2F										
2	LL4										
2	LL2B										0.061
2	LL2C										0.513
2	LL2D										0.343
2	LL2E										0.238
2	LL2G										0.374
2	LL2H										
3	LL3B										
3	LL3C SW										
3	LL3A										
4	04-FF1A-070821										0.529
4	04-FF1B-070821										
5	FF5dwn100719		na		na		na				
5	FF6up100719		na		na		na				
5	5FF2A										
5	5FF2B				22	1.11	5.37	9.01			0.49
6	CW 1	na	na		na		na		na	na	0.125
6	CW 2	na	na		na		na		na	na	
6	CW 3	na	na		na		na		na	na	
6	CW 3.1	na	na		na		na		na	na	
6	CW 4	na	na		na		na		na	na	
7	7SFFdwn071019		na		na		na				
7	7SFFup071019		na		na		na				
9	9AGPdwn071019		na		na		na				
9	9AGPup071019		na		na		na				
10	10-CW6-070821										0.068
10	10-CW7-070821										0.093
11	POTW 11 #1 up		na		na		na				
11	POTW 11 #2 dwn		na		na		na				
12	POTW8100719		na		na		na				

PFAS in Sediment (ng/g, dry weight basis)

Site ID	Sample ID	11Cl-PF3OUdS	3:3 FTCA	4:2 FTS	5:3 FTCA	6:2 FTS	7:3 FTCA	8:2 FTS	9Cl-PF3ONS	ADONA	EtFOSAA
13	13-WHIP										0.189
14	POTW 14 #2 dwn		na		na		na				0.15
15	POTW6100719		na		na	1.81	na				
16	FSW1		na		na		na				
16	FSW2		na		na		na				
16	FSW3		na		na		na				
16	FTM020										0.067
19	FTM022										
20	FTM028										
Pond	BUTTERWORTH POND	na	na		na		na		na	na	
Pond	CEDAR POND	na	na		na		na		na	na	
Pond	ECHO LAKE POND #1	na	na		na		na		na	na	
Pond	ECHO LAKE POND #2	na	na		na		na		na	na	
Pond	LOCUSTWOOD POND	na	na		na		na		na	na	

Note: Empty cells denote levels below detection limit.
Cells that contain “na” denote analytes not quantified in the method used for that sample.

PFAS in Sediment (ng/g, dry weight basis)

Site ID	Sample ID	HFPO-DA	MeFOSAA	N-EtFOSA	N-EtFOSE	NFDHA	N-MeFOSA	N-MeFOSE	PFBA	PFBS	PFDA
1	LL1A					na					
1	LL1B					na					0.438
1	LL1C					na					
1	LL1D					na					
2	LL2A										0.09
2	LL2F										
2	LL4										
2	LL2B										0.047
2	LL2C		0.186								0.199
2	LL2D										0.262
2	LL2E		0.061								0.174
2	LL2G		0.308								0.13
2	LL2H								0.396		0.708
3	LL3B										0.178
3	LL3C SW									0.116	0.303
3	LL3A									0.18	0.238
4	04-FF1A-070821		0.095								0.477
4	04-FF1B-070821										
5	FF5dwn100719					na					
5	FF6up100719					na					
5	5FF2A										0.135
5	5FF2B		0.205						0.615	0.14	10.6
6	CW 1		0.205			na					0.115
6	CW 2					na					
6	CW 3					na					
6	CW 3.1		0.045			na					0.718
6	CW 4					na					
7	7SFFdwn071019					na					
7	7SFFup071019					na					
9	9AGPdwn071019					na					
9	9AGPup071019					na					
10	10-CW6-070821										0.046
10	10-CW7-070821										0.145
11	POTW 11 #1 up					na					
11	POTW 11 #2 dwn					na					
12	POTW8100719					na					
13	13-WHIP		0.047							0.064	0.119
14	POTW 14 #2 dwn		0.125			na			3.74		0.153
15	POTW6100719					na					
16	FSW1					na					

Appendix 6 PFAS in Sediment

Note: Empty cells denote levels below detection limit.

Cells that contain “na” denote analytes not quantified in the method used for that sample.

PFAS in Sediment (ng/g, dry weight basis)

Site ID	Sample ID	HFPO-DA	MeFOSAA	N-EtFOSA	N-EtFOSE	NFDHA	N-MeFOSA	N-MeFOSE	PFBA	PFBS	PFDA
16	FSW2					na					
16	FSW3					na					0.043
16	FTM020										
19	FTM022										0.056
20	FTM028										
Pond	BUTTERWORTH POND	na				na					0.198
Pond	CEDAR POND	na	0.189			na					0.287
Pond	ECHO LAKE POND #1	na				na					0.434
Pond	ECHO LAKE POND #2	na				na					0.506
Pond	LOCUSTWOOD POND	na				na					0.684

Appendix 6 PFAS in Sediment
Note: Empty cells denote levels below detection limit.
Cells that contain “na” denote analytes not quantified in the method used for that sample.

PFAS in Sediment (ng/g, dry weight basis)

Site ID	Sample ID	PFDoA	PFDoS	PFDS	PFEESA	PFHpA	PFHpS	PFHxA	PFHxS	PFMBA	PFMPA
1	LL1A				na					na	na
1	LL1B	0.952		0.586	na					na	na
1	LL1C			0.062	na					na	na
1	LL1D			0.074	na					na	na
2	LL2A	0.226									
2	LL2F	0.064									
2	LL4										
2	LL2B	0.077									
2	LL2C	0.121		0.078							
2	LL2D	0.769		0.289		0.123					
2	LL2E	0.585		0.167							
2	LL2G	0.109		0.098							
2	LL2H	0.285		0.044		0.183		0.301			
3	LL3B	0.455									
3	LL3C SW	0.352		0.361		0.065		0.058			
3	LL3A	0.537		0.551		0.221		0.301	0.109		
4	04-FF1A-070821	0.302		0.358		0.271		0.183			
4	04-FF1B-070821	0.171									
5	FF5dwn100719	0.085			na					na	na
5	FF6up100719	0.231		0.12	na					na	na
5	5FF2A	0.154							0.202		
5	5FF2B	8.02	1.03	3.47		0.797	1.63	1.61	9.87		
6	CW 1	1.32		0.087	na			0.062	0.116	na	na
6	CW 2				na					na	na
6	CW 3				na					na	na
6	CW 3.1	1.64		0.321	na	0.057		0.106	0.328	na	na
6	CW 4				na					na	na
7	7SFFdwn071019				na					na	na
7	7SFFup071019				na					na	na
9	9AGPdwn071019				na					na	na
9	9AGPup071019	0.083			na					na	na
10	10-CW6-070821	0.142		0.042							
10	10-CW7-070821	0.317		0.11							
11	POTW 11 #1 up				na					na	na
11	POTW 11 #2 dwn				na					na	na
12	POTW8100719	0.162			na					na	na
13	13-WHIP	0.29		0.188				0.094	0.517		
14	POTW 14 #2 dwn	0.199		0.126	na					na	na
15	POTW6100719	0.089			na					na	na
16	FSW1				na					na	na

Appendix 6 PFAS in Sediment
Note: Empty cells denote levels below detection limit.
Cells that contain “na” denote analytes not quantified in the method used for that sample.

PFAS in Sediment (ng/g, dry weight basis)

Site ID	Sample ID	PFDoA	PFDoS	PFDS	PFEESA	PFHpA	PFHpS	PFHxA	PFHxS	PFMBA	PFMPA
16	FSW2				na					na	na
16	FSW3	0.025			na	0.043		0.03		na	na
16	FTM020	0.07		0.085					0.048		
19	FTM022	0.059									
20	FTM028								0.055		
Pond	BUTTERWORTH POND	0.074			na					na	na
Pond	CEDAR POND				na	0.253		0.272		na	na
Pond	ECHO LAKE POND #1	0.325			na	0.242		0.139		na	na
Pond	ECHO LAKE POND #2	0.645			na					na	na
Pond	LOCUSTWOOD POND	0.263			na	0.122		0.079		na	na

Appendix 6 PFAS in Sediment
Note: Empty cells denote levels below detection limit.
Cells that contain “na” denote analytes not quantified in the method used for that sample.

PFAS in Sediment (ng/g, dry weight basis)

Site ID	Sample ID	PFNA	PFNS	PFOA	PFOS	PFOSA	PFPeA	PFPeS	PFTeDA	PFTrDA	PFUnA
1	LL1A				0.052						
1	LL1B	0.17		0.094	2.58				0.474	0.93	1.75
1	LL1C				0.089						0.063
1	LL1D				0.103					0.04	0.081
2	LL2A	0.084		0.075	0.815				0.153	0.256	0.43
2	LL2F				0.138					0.07	0.153
2	LL4										0.065
2	LL2B	0.039			0.265				0.04	0.081	0.131
2	LL2C	0.039		0.067	1.69	0.199	0.115		0.064	0.1	0.162
2	LL2D	0.156		0.226	1	0.095	0.411		0.567	0.815	0.609
2	LL2E				1.01	0.087	0.5		0.394	0.443	0.311
2	LL2G	0.041		0.048	0.814	0.111					0.09
2	LL2H	0.248		0.312	1.22		0.588		0.046	0.109	2.14
3	LL3B				0.12				0.354	0.504	0.708
3	LL3C SW	0.113		0.236	0.62				0.142	0.214	0.43
3	LL3A	0.071		1.14	0.374		0.428		0.183	0.353	0.86
4	04-FF1A-070821	0.34		0.22	2.02	0.146	0.858		0.199	0.247	0.293
4	04-FF1B-070821	0.306			0.848				0.253	0.416	0.268
5	FF5dwn100719			0.155	0.324	0.212					0.098
5	FF6up100719	0.128		0.15	0.547				0.2	0.56	0.35
5	5FF2A	0.12		0.227	3.12		0.088		0.086	0.235	0.449
5	5FF2B	4.96	2.7	3.43	283	8.22	0.78	0.314	2.09	12	30.6
6	CW 1				1.75	0.262			0.547	0.404	0.153
6	CW 2				0.103						
6	CW 3				0.06						
6	CW 3.1	0.132	0.046	0.11	2.92	0.187	0.139		0.859	0.348	0.266
6	CW 4				0.12						0.071
7	7SFFdwn071019				0.252						
7	7SFFup071019				0.346						0.102
9	9AGPdwn071019	0.107		0.117	0.464						0.173
9	9AGPup071019				0.217					0.086	0.081
10	10-CW6-070821	0.049			0.238		0.144		0.1	0.132	0.177
10	10-CW7-070821	0.059		0.099	0.582		0.248		0.257	0.227	0.217
11	POTW 11 #1 up										
11	POTW 11 #2 dwn				0.086						
12	POTW8100719				0.485				0.104	0.198	0.231
13	13-WHIP	0.059		0.051	0.987	0.041	0.119		0.181	0.18	0.268
14	POTW 14 #2 dwn			0.245	1.27	0.178			0.254	0.173	
15	POTW6100719				0.406					0.171	0.131
16	FSW1				0.288						

Appendix 6 PFAS in Sediment
Note: Empty cells denote levels below detection limit.
Cells that contain “na” denote analytes not quantified in the method used for that sample.

PFAS in Sediment (ng/g, dry weight basis)

Site ID	Sample ID	PFNA	PFNS	PFOA	PFOS	PFOSA	PFPeA	PFPeS	PFTeDA	PFTrDA	PFUnA
16	FSW2				0.561						
16	FSW3	0.08		0.108	0.305					0.035	0.089
16	FTM020				0.209				0.041	0.094	0.096
19	FTM022	0.042		0.068	0.177					0.054	0.087
20	FTM028										
Pond	BUTTERWORTH POND	0.329		0.256	0.364					0.261	0.374
Pond	CEDAR POND	0.682		0.698	0.587						0.334
Pond	ECHO LAKE POND #1	0.453		1.04	1.2		0.149		0.186	0.36	0.679
Pond	ECHO LAKE POND #2	0.369		0.409	1.09				0.541	1.04	1.05
Pond	LOCUSTWOOD POND	1.7		0.355	0.969		0.094		0.124	0.716	2.96

Note: Empty cells denote levels below detection limit.
Cells that contain “na” denote analytes not quantified in the method used for that sample.

Appendix 7: PFAS Data Fish Tissue

PFAS in Fish Tissue (ng/g): Note: Empty cells denote levels below the detection limit.

Site	Sample ID	PFDA	PFDoA	PFDoS	PFDS	PFEESA	PFHpA	PFHpS
2	FTM073LMB0121	2.57	1.97		0.549			
2	FTM073BBH0121	0.276	0.396					
2	FTM073BBH0221	0.191	0.258					
2	FTM073BBH0321	0.16	0.412					
2	FTM073BC0121	1.76	0.941		0.327			
2	FTM073BC0221	1.69	1.13		0.468			
2	FTM073BC0321	1.29	0.722		0.276			
2	FTM073LMB0221	1.42	1.69		0.573			
2	FTM073LMB0321	2.09	1.66		0.457			
2	FTM073PS0121	0.6	0.435		0.116			
2	FTM073PS0221	1.1	0.985		0.283			
2	FTM073PS0321	1.27	1.53		0.389			
2	FTM073YP0121	2.13	1.58		0.354			
2	FTM073YP0221	1.93	1.11		0.246			
2	FTM073YP0321	0.994	0.594					
6	CW2BBH0121	0.277	2.64		0.313			
6	CW2BBH0221	0.349	3.07		0.334			
6	CW2BBH0321	0.316	2.85		0.311			
6	CW2BG0221	1.09	3.84		0.79			0.142
6	CW2BG0321	1.03	3.63		0.845			
6	CW2CC0121	1.62	6.34	0.118	1.05			0.317
6	CW2CC0221	1.09	3.6		0.701			0.17
6	CW2CC0321	1.17	4.47		1.18		0.133	0.362
6	CW2BG0121	1.56	6.67		1.34			0.115
6	CW2LMB0121	3.4	12.5	0.227	2.7			0.328
6	CW2LMB0221	1.33	6.51		1.11			0.137
6	CW2LMB0321	1.55	6.04		1.04			0.209
8	FF5BG0121	0.702	1.53		0.351			
8	FF5BG0221	1.03	1.97		0.348			
8	FF5BG0321	0.882	1.19		0.274			
8	FF5CC0121	0.314	0.793		0.126		0.132	
8	FF5CC0221	0.346	1.08		0.187		0.215	
8	FF5CC0321	0.355	1.1		0.137			
8	FF5LMB0121	1.55	3.5		0.713			
8	FF5LMB0221	1.6	2.35		0.439			
8	FF5LMB0321	1.23	1.65		0.462			
12	POTW8BG0121	1.83	1.87		0.582			
12	POTW8BG0221	1.4	2.56		1.23			
12	POTW8BG0321	2.2	3.73		1.6			

Site	Sample ID	PFDA	PFDoA	PFDoS	PFDS	PFEESA	PFHpA	PFHpS
12	POTW8CC0121	1.39	1.74		0.278			
12	POTW8CC0221	1.69	1.97		0.929			0.105
12	POTW8CC0321	1.11	1.62		0.56			
12	POTW8LMB0121	1.1	2.1		0.773			
12	POTW8LMB0221	1.01	1.78		0.74			
12	POTW8LMB0321	2.52	2.16		1.01			
12	POTW8YBH0121	0.172	0.85		0.098			
12	POTW8YBH0221	0.209	0.641					
12	POTW8YBH0321	0.115	0.761					
12	POTW8YP0121	0.603	1.21		0.155			
12	POTW8YP0221	0.948	1.32		0.311			
12	POTW8YP0321	1.11	1.57		0.322			
16	FSW1BG0121	1.03	0.711					
16	FSW1BG0221	0.8	0.434					
16	FSW1BG0321	1.06	0.49					
16	FSW1CP0121	0.997	0.977					0.152
16	FSW1CP0221	1.86	1.1		0.121			0.192
16	FSW1CP0321	1.09	0.713					0.176
16	FTM145AE0121	0.331	0.868		0.246			0.363
16	FTM145AE0221	0.49	1.14		0.383			0.794
16	FTM145AE0321	0.511	1.09		0.344			1.06
16	FTM145BBH0121	0.145	0.571		0.284			
16	FTM145BBH0321	0.157	0.416		0.346			
19	FTM022PS0121	0.502	0.798		0.22			
19	FTM022PS0221	0.792	0.807		0.303			
19	FTM022PS0321	1.19	1.31		0.639			
19	FTM022SB0121	0.298	0.32		0.133			
19	FTM022SB0221	0.336	0.453		0.135			
19	FTM022WCF0221	0.983		0.106				
19	FTM022WCF0321	0.228	0.691					
19	FTM022YP0121	0.675	1.14		0.487			
19	FTM022YP0221	1.17	1.56		0.666			
19	FTM022YP0321	1.08	1.35		0.288			
20	FTM028BBH0121	0.177	1.23		0.23			
20	FTM028BBH0221	0.155	0.665		0.177			
20	FTM028BG0121	0.52	1.36		0.578			
20	FTM028BG0221	0.594	1.04		0.419			
20	FTM028BG0321	0.671	1.22		0.367			
20	FTM028CC0121	0.57	1.17		0.573			
20	FTM028CC0221	0.492	1.95		0.71			

Note: Empty cells denote levels below detection limit.

Site	Sample ID	PFDA	PFDoA	PFDoS	PFDS	PFEESA	PFHpA	PFHpS
20	FTM028CC0321	0.397	1.65		0.533			
20	FTM028LMB0121	0.679	1.61		0.887			
20	FTM028LMB0221	0.689	1.63		1.04			
20	FTM028LMB0321	1.16	2.15		0.933			
20	FTM028WP0121	0.648	1.46		0.744			
20	FTM028WP0221	0.458	1.15		0.561			
21	FTM030BG0121	0.578	0.245					
21	FTM030BG0221	0.465	0.438					
21	FTM030BG0321	1.17	0.676		0.096			
21	FTM030LMB0121	1.75	0.882		0.118			
21	FTM030LMB0221	1.74	1.09		0.174			
21	FTM030LMB0321	1.09	0.896		0.109			

Site	Sample ID	PFHxA	PFHxS	PFMBA	PFMPA	PFNA	PFNS	PFOA
2	FTM073LMB0121					0.229		
2	FTM073BBH0121							
2	FTM073BBH0221							
2	FTM073BBH0321							
2	FTM073BC0121		0.266			0.858		
2	FTM073BC0221		0.218			0.854		
2	FTM073BC0321		0.194			0.552		
2	FTM073LMB0221							
2	FTM073LMB0321					0.184		
2	FTM073PS0121					0.218		
2	FTM073PS0221					0.245		
2	FTM073PS0321					0.259		
2	FTM073YP0121					0.375		
2	FTM073YP0221					0.719		
2	FTM073YP0321					0.266		
6	CW2BBH0121		0.201					
6	CW2BBH0221		0.251					
6	CW2BBH0321		0.426					
6	CW2BG0221		0.361				0.113	
6	CW2BG0321		0.269				0.131	
6	CW2CC0121		2.49			0.212	0.139	0.112
6	CW2CC0221		1.33			0.149		
6	CW2CC0321		3.19			0.205	0.107	
6	CW2BG0121		0.406				0.233	
6	CW2LMB0121		0.809			0.147	0.469	
6	CW2LMB0221		1.07				0.162	
6	CW2LMB0321		0.957			0.188	0.163	
8	FF5BG0121					0.119		
8	FF5BG0221					0.159		
8	FF5BG0321					0.146		
8	FF5CC0121	0.13						
8	FF5CC0221	0.112				0.133		
8	FF5CC0321					0.098		
8	FF5LMB0121					0.124		
8	FF5LMB0221					0.206		
8	FF5LMB0321							
12	POTW8BG0121					0.165		
12	POTW8BG0221					0.106		
12	POTW8BG0321					0.106		
12	POTW8CC0121		0.193			0.289		

Note: Empty cells denote levels below detection limit.

Site	Sample ID	PFHxA	PFHxS	PFMBA	PFMPA	PFNA	PFNS	PFOA
12	POTW8CC0221		0.256			0.279		
12	POTW8CC0321		0.131			0.202		
12	POTW8LMB0121							
12	POTW8LMB0221					0.107		
12	POTW8LMB0321					0.277		
12	POTW8YBH0121							
12	POTW8YBH0221							
12	POTW8YBH0321					0.133		
12	POTW8YP0121		0.113			0.207		
12	POTW8YP0221		0.165			0.311		
12	POTW8YP0321					0.312		
16	FSW1BG0121							
16	FSW1BG0221							
16	FSW1BG0321					0.206		
16	FSW1CP0121		0.419			0.29		
16	FSW1CP0221		0.204			0.139	0.127	
16	FSW1CP0321		0.22			0.112		
16	FTM145AE0121		2.11			0.351	0.131	0.389
16	FTM145AE0221		5.87			0.792	0.188	0.425
16	FTM145AE0321	0.149	9.05			0.81	0.2	0.542
16	FTM145BBH0121		1.2					
16	FTM145BBH0321		1.22			0.114	0.117	
19	FTM022PS0121					0.183		
19	FTM022PS0221					0.298		0.403
19	FTM022PS0321					0.498		0.357
19	FTM022SB0121					0.125		
19	FTM022SB0221					0.124		
19	FTM022WCF0221		0.115		1.52			0.657
19	FTM022WCF0321							
19	FTM022YP0121					0.184		
19	FTM022YP0221					0.326		
19	FTM022YP0321					0.753		0.347
20	FTM028BBH0121							
20	FTM028BBH0221							
20	FTM028BG0121					0.115		
20	FTM028BG0221							
20	FTM028BG0321		0.108			0.19		
20	FTM028CC0121					0.134		
20	FTM028CC0221					0.11		
20	FTM028CC0321							

Note: Empty cells denote levels below detection limit.

Site	Sample ID	PFHxA	PFHxS	PFMBA	PFMPA	PFNA	PFNS	PFOA
20	FTM028LMB0121							
20	FTM028LMB0221							
20	FTM028LMB0321							
20	FTM028WP0121					0.194		
20	FTM028WP0221							
21	FTM030BG0121					0.212		
21	FTM030BG0221					0.11		
21	FTM030BG0321					0.469		
21	FTM030LMB0121					0.136		
21	FTM030LMB0221					0.127		
21	FTM030LMB0321					0.158		

Site	Sample ID	PFOS	PFOSA	PFPeA	PFPeS	PFTeDA	PFTrDA	PFUnA
2	FTM073LMB0121	24.3				0.803	1.8	6.03
2	FTM073BBH0121	2.06				0.193	0.405	0.901
2	FTM073BBH0221	0.891				0.19	0.444	0.494
2	FTM073BBH0321	0.707				0.321	0.492	
2	FTM073BC0121	29				0.286	0.824	3.16
2	FTM073BC0221	25.9				0.547	1.17	3.52
2	FTM073BC0321	20.1				0.314	0.599	2.51
2	FTM073LMB0221	18				0.873	1.77	4.7
2	FTM073LMB0321	18.5				0.783	1.86	4.75
2	FTM073PS0121	4.89	0.149			0.159	0.448	1.58
2	FTM073PS0221	8.51	0.125			0.38	0.911	3.04
2	FTM073PS0321	11.2	0.172			1.03	2.06	3.26
2	FTM073YP0121	19.8	0.358			0.605	1.57	5.1
2	FTM073YP0221	16.4	0.15			0.441	1.08	4.12
2	FTM073YP0321	7.7				0.307	0.802	2.24
6	CW2BBH0121	6.63	0.914			4.56	1.38	0.503
6	CW2BBH0221	10.8	1.21			3.62	1.6	0.854
6	CW2BBH0321	8.04	1.54			2.65	1.31	0.699
6	CW2BG0221	68.9	0.759			2.14	1.34	2.17
6	CW2BG0321	94	0.405			2.04	1.46	1.61
6	CW2CC0121	58.8	0.642			4.15	2.36	1.54
6	CW2CC0221	43.8	0.726			2.12	1.51	1.43
6	CW2CC0321	47.6	1.05			4.22	2.38	1.54
6	CW2BG0121	142	0.624			4	3.17	3.15
6	CW2LMB0121	249	0.253			6.16	4.35	4.34
6	CW2LMB0221	76.2	0.212			3.64	2.9	2.09
6	CW2LMB0321	87.3	0.185			3.65	2.37	2.1
8	FF5BG0121	13.1				1.43	1.82	1.35
8	FF5BG0221	18.5				1.39	1.87	1.84
8	FF5BG0321	13.6				0.886	1.03	1.16
8	FF5CC0121	1.97				0.885	1.14	0.49
8	FF5CC0221	3.2				1.26	1.38	0.611
8	FF5CC0321	2.39				1.02	1.54	0.706
8	FF5LMB0121	17.8				2.78	4.27	3.32
8	FF5LMB0221	14.9				1.62	1.8	2.64
8	FF5LMB0321	16.4				1.25	1.43	1.77
12	POTW8BG0121	27.7	0.129			1.25	1.9	3.3
12	POTW8BG0221	19.4	0.104			2.44	2.73	2.19
12	POTW8BG0321	34.9	0.141			3.03	4.48	3.65
12	POTW8CC0121	12.7				1.11	2.01	3.26

Note: Empty cells denote levels below detection limit.

Site	Sample ID	PFOS	PFOSA	PFPeA	PFPeS	PFTeDA	PFTrDA	PFUnA
12	POTW8CC0221	27.8	0.21			1.65	2.2	2.59
12	POTW8CC0321	9.83	0.153			1.22	1.7	1.76
12	POTW8LMB0121	15.6				2.17	2.77	2.26
12	POTW8LMB0221	17.6				1.48	1.97	1.87
12	POTW8LMB0321	35.4	0.125			1.48	2.27	2.99
12	POTW8YBH0121	0.926				0.87	1.16	0.788
12	POTW8YBH0221	1.21	0.114			0.6	0.819	0.667
12	POTW8YBH0321	0.589				1.27	1.42	0.514
12	POTW8YP0121	8.05	0.331			1.06	1.52	1.92
12	POTW8YP0221	13.7	0.22			1.26	1.87	2.38
12	POTW8YP0321	15.1	0.196			1.42	2.22	
16	FSW1BG0121	52.7				0.645	1.28	2.11
16	FSW1BG0221	45.4				0.327	0.616	1.83
16	FSW1BG0321	46.2				0.29	0.629	1.62
16	FSW1CP0121	67.1	0.198			0.46	1.21	2.12
16	FSW1CP0221	119	0.25			0.416	0.937	3.68
16	FSW1CP0321	84.3	0.298			0.391	0.752	1.98
16	FTM145AE0121	103	0.193		0.142	0.914	2.82	0.865
16	FTM145AE0221	127	0.136		0.165	1.33	2.41	1.34
16	FTM145AE0321	138	0.318		0.319	0.996	1.93	1.18
16	FTM145BBH0121	20.2	4.48			1.03	1.49	0.623
16	FTM145BBH0321	30.1	9.62			0.728	0.966	0.539
19	FTM022PS0121	5.23	0.115			0.77	0.975	0.653
19	FTM022PS0221	7.42	0.124			0.476	0.881	1.41
19	FTM022PS0321	13.9	0.116			1.13	1.85	2.37
19	FTM022SB0121	3.79	0.631			0.312	0.326	0.436
19	FTM022SB0221	4.85	0.968			0.322	0.419	0.568
19	FTM022WCF0221	1.19	0.782					
19	FTM022WCF0321	1.3				0.556	0.942	0.616
19	FTM022YP0121	12	0.281			1.26	2.44	1.43
19	FTM022YP0221	16.5	0.375			1.36	2.89	2.31
19	FTM022YP0321	12.6	0.346			1.04	1.62	1.61
20	FTM028BBH0121	0.949	0.206			1.31	1.15	0.579
20	FTM028BBH0221	0.888	0.198			0.398	0.527	0.4
20	FTM028BG0121	8.83	0.174			0.562	0.939	0.963
20	FTM028BG0221	10.6	0.155			0.461	0.748	0.927
20	FTM028BG0321	6.79	0.153			0.622	0.911	0.96
20	FTM028CC0121	4.25				0.857	0.863	0.901
20	FTM028CC0221	4.76	0.135			1.43	1.59	1.12
20	FTM028CC0321	4.55	0.115			1.77	1.58	0.888

Note: Empty cells denote levels below detection limit.

PFAS in Fish Tissue (ng/g)

Site	Sample ID	PFOS	PFOSA	PFPeA	PFPeS	PFTeDA	PFTrDA	PFUnA
20	FTM028LMB0121	12.7	0.115			0.894	1.18	1.23
20	FTM028LMB0221	12.7				0.99	1.38	1.15
20	FTM028LMB0321	16.1	0.133			0.895	1.38	1.45
20	FTM028WP0121	8.79	0.25			1.05	1.16	0.983
20	FTM028WP0221	8.83	0.735			0.58	0.787	0.772
21	FTM030BG0121	7.94					0.216	1.05
21	FTM030BG0221	8.36				0.384	0.522	1.81
21	FTM030BG0321	17.2				0.278	0.6	3.17
21	FTM030LMB0121	18.6				0.227	0.708	3.61
21	FTM030LMB0221	19.5				0.444	0.905	4.99
21	FTM030LMB0321	12				0.396	0.809	3.21

Note: Empty cells denote levels below detection limit.

Appendix 8: Detection Limits for CECs

PBDE IUPAC and Detection Limits in Fish Tissue

PBDE Congener Name	IUPAC #	Min DL (ppt)	Max DL (ppt)
2,2',3,3',4,4',5,5',6,6'-DeBDE	209	4.04	8.36
2,2',3,3',4,4',5,5',6-NoBDE	206	0.554	1.07
2,2',3,3',4,4',5,6,6'-NoBDE	207	0.742	1.43
2,2',3,3',4,4'-HxBDE	128	0.444	1.07
2,2',3,3',4,5,5',6,6'-NoBDE	208	0.83	1.6
2,2',3,4,4',5,5',6-OcBDE	203	0.19	0.651
2,2',3,4,4',5,6-HpBDE	181	0.226	0.535
2,2',3,4,4',5',6-HpBDE	183	0.163	0.385
2,2',3,4,4',5'-HxBDE	138	0.261	0.578
2,2',3,4,4',6'-HxBDE	140	0.185	0.409
2,2',3,4,4'-PeBDE	85	0.479	0.919
2,2',4,4',5,5'-HxBDE	153	0.238	0.515
2,2',4,4',5,6'-HxBDE	154	0.146	0.335
2,2',4,4',5-PeBDE	99	0.373	0.709
2,2',4,4',6,6'-HxBDE	155	0.152	0.337
2,2',4,4',6-PeBDE	100	0.268	0.533
2,2',4,4'-TeBDE	47	0.0993	0.137
2,2',4,5'-TeBDE	49	0.11	0.176
2,2',4,6'-TeBDE	51	0.0993	0.143
2,2',4-TriBDE	17	0.239	1.71
2,3,3',4,4',5,6-HpBDE	190	0.388	0.918
2,3,3',4,4'-PeBDE	105	0.611	1.17
2,3,4,4',5,6-HxBDE	166		
2,3',4,4',6-PeBDE	119	0.615	1.18
2,3',4,4'-TeBDE	66	0.135	0.216
2,3',4,5,5'-PeBDE	120		
2,3,4,5,6-PeBDE	116	0.866	1.66
2,3',4',6-TeBDE	71	0.123	0.197
2,3',4-TriBDE	25		
2',3,4-TriBDE	33		
2,4,4',6-TeBDE	75	0.0993	0.154
2,4,4'-TriBDE	28	0.226	1.61
2,4,6-TriBDE	30	0.273	1.95
2,4',6-TriBDE	32	0.212	1.52
2,4'-DiBDE	8	0.177	0.276
2,4-DiBDE	7	0.229	0.358
2,6-DiBDE	10	0.283	0.443
3,3',4,4',5-PeBDE	126	0.33	0.656
3,3',4,4'-TeBDE	77	0.0987	0.103

Appendix 8: Detection Limits of Four CECs

PBDE Congener Name	IUPAC #	Min DL (ppt)	Max DL (ppt)
3,3',4,5'-TeBDE	79	0.0987	0.125
3,3',4-TriBDE	35	0.154	1.1
3,3'-DiBDE	11		
3,4,4'-TriBDE	37	0.14	0.999
3,4'-DiBDE	13		
3,4-DiBDE	12	0.15	0.235
4,4'-DiBDE	15	0.135	0.211

Detection Limit of Chlorinated Paraffins in three media

	Fish Tissue		Sediment		Surface Water	
Compound	Min DL (ng/g)	Max DL (ng/g)	Min DL (ng/g)	Max DL (ng/g)	Min DL (ng/L)	Max DL (ng/L)
C10Cl5	4.91	5.01	4.25	6.25	58.5	70.7
C10Cl6	1.47	1.5	1.27	1.88	17.6	21.2
C10Cl7	4.91	5.01	4.25	6.25	58.5	70.7
C11Cl5	4.91	5.01	4.25	6.25	58.5	70.7
C11Cl6	4.91	5.01	4.25	6.25	58.5	70.7
C11Cl7	4.91	5.01	4.25	6.25	58.5	70.7
C11Cl8	2.94	3.01	2.55	3.75	35.1	42.4
C11Cl9	2.94	3.01	2.55	3.75	35.1	42.4
C12Cl10	0.981	1	0.85	1.25	11.7	14.1
C12Cl5	4.91	5.01	4.25	6.25	58.5	70.7
C12Cl6	4.91	5.01	4.25	6.25	58.5	70.7
C12Cl7	2.94	3.01	2.55	3.75	35.1	42.4
C12Cl8	2.94	3.01	2.55	3.75	35.1	42.4
C12Cl9	1.96	2	1.7	2.5	23.4	28.3
C13Cl10	1.96	2	1.7	2.5	23.4	28.3
C13Cl5	2.45	2.51	2.12	3.13	29.3	35.4
C13Cl6	1.96	2	1.7	2.5	23.4	28.3
C13Cl7	2.45	2.51	2.12	3.13	29.3	35.4
C13Cl8	4.91	5.01	4.25	6.25	58.5	70.7
C13Cl9	1.96	2	1.7	2.5	23.4	28.3
C14Cl10	0.981	1	0.85	1.25	11.7	14.1
C14Cl11	0.981	1	0.85	1.25	11.7	14.1
C14Cl5	0.491	0.501	0.425	0.625	5.85	7.07
C14Cl6	1.47	1.5	1.27	1.88	17.6	21.2
C14Cl7	2.94	3.01	2.55	3.75	35.1	42.4
C14Cl8	2.94	3.01	2.55	3.75	35.1	42.4
C14Cl9	4.91	5.01	4.25	6.25	58.5	70.7
C15Cl10	1.47	1.5	1.27	1.88	17.6	21.2

Appendix 8: Detection Limits of Four CECs

	Fish Tissue		Sediment		Surface Water	
Compound	**Min DL (ng/g)**	**Max DL (ng/g)**	**Min DL (ng/g)**	**Max DL (ng/g)**	**Min DL (ng/L)**	**Max DL (ng/L)**
C15Cl11	1.47	1.5	1.27	1.88	17.6	21.2
C15Cl5	0.981	1	0.85	1.25	11.7	14.1
C15Cl6	2.94	3.01	2.55	3.75	35.1	42.4
C15Cl7	3.43	3.51	2.97	4.38	41	49.5
C15Cl8	3.43	3.51	2.97	4.38	41	49.5
C15Cl9	1.47	1.5	1.27	1.88	17.6	21.2
C16Cl10	1.96	2	1.7	2.5	23.4	28.3
C16Cl11	0.491	0.501	0.425	0.625	5.85	7.07
C16Cl5	2.94	3.01	2.55	3.75	35.1	42.4
C16Cl6	9.81	10	8.5	12.5	117	141
C16Cl7	9.81	10	8.5	12.5	117	141
C16Cl8	2.94	3.01	2.55	3.75	35.1	42.4
C16Cl9	9.81	10	8.5	12.5	117	141
C17Cl10	0.981	1	0.85	1.25	11.7	14.1
C17Cl11	0.491	0.501	0.425	0.625	5.85	7.07
C17Cl5	0.981	1	0.85	1.25	11.7	14.1
C17Cl6	0.981	1	0.85	1.25	11.7	14.1
C17Cl7	1.96	2	1.7	2.5	23.4	28.3
C17Cl8	1.96	2	1.7	2.5	23.4	28.3
C17Cl9	4.91	5.01	4.25	6.25	58.5	70.7
C18Cl10	4.91	5.01	4.25	6.25	58.5	70.7
C18Cl5	1.96	2	1.7	2.5	23.4	28.3
C18Cl6	63.8	65.1	55.2	81.3	761	919
C18Cl7	12.8	13	11	16.3	152	184
C18Cl8	9.81	10	8.5	12.5	117	141
C18Cl9	4.91	5.01	4.25	6.25	58.5	70.7
C19Cl10	2.94	3.01	2.55	3.75	35.1	42.4
C19Cl6	2.94	3.01	2.55	3.75	35.1	42.4
C19Cl7	4.91	5.01	4.25	6.25	58.5	70.7
C19Cl8	3.93	4.01	3.4	5	46.8	56.6
C19Cl9	1.96	2	1.7	2.5	23.4	28.3
C20Cl6	4.91	5.01	4.25	6.25	58.5	70.7
C20Cl7	2.94	3.01	2.55	3.75	35.1	42.4
C20Cl8	1.96	2	1.7	2.5	23.4	28.3
C20Cl9	1.96	2	1.7	2.5	23.4	28.3

Appendix 8: Detection Limits of Four CECs

PPCPs (APOS) Detection Limits

	Tissue (ng/g)		**Sediment (ng/g)**		**Surface Water (ng/L)**	
PPCP	**Min**	**Max**	**Min**	**Max**	**Min**	**Max**
10-hydroxy-amitriptyline	0.0577	0.06	0.108	0.153	0.137	0.15
Alprazolam	0.115	0.12	0.216	0.306	0.273	0.3
Amitriptyline	0.115	0.12	0.216	0.306	0.273	0.3
Amlodipine	0.387	0.402	0.726	1.03	0.915	1.01
Benzoylecgonine	0.0577	0.06	0.108	0.153	0.137	0.15
Benztropine	0.269	0.28	0.505	0.714	0.637	0.7
Betamethasone	0.577	0.6	1.08	1.53	1.37	1.5
Cocaine	0.0577	0.06	0.108	0.153	0.137	0.15
DEET	0.115	0.12	0.216	0.306	0.273	0.3
Desmethyldiltiazem	0.0404	0.042	0.108	0.153	0.137	0.15
Diazepam	0.193	0.201	0.362	0.512	0.457	0.502
Fluocinonide	0.773	0.804	1.45	2.05	1.83	2.01
Fluticasone propionate	0.773	0.804	1.45	2.05	1.83	2.01
Hydrocortisone	2.31	2.4	4.33	6.12	5.46	6
Meprobamate	0.577	0.6	1.08	1.53	1.37	1.5
Methylprednisolone	1.54	2.19	2.88	4.08	3.64	4
Metoprolol	0.193	0.201	0.362	0.512	0.457	0.502
Norfluoxetine	0.193	0.201	0.362	0.512	0.457	0.502
Norverapamil	0.0577	0.06	0.108	0.153	0.137	0.15
Paroxetine	0.387	0.402	0.726	1.03	0.915	1.01
Prednisolone	1.54	1.6	2.88	4.08	3.64	4
Prednisone	2.31	2.4	4.33	6.12	5.46	6
Promethazine	0.115	0.12	0.216	0.306	0.273	0.3
Propoxyphene	0.115	0.12	0.216	0.306	0.273	0.3
Propranolol	0.115	0.12	0.216	0.306	0.273	0.3
Sertraline	0.115	0.12	0.216	0.306	0.273	0.3
Simvastatin	0.773	0.804	1.45	2.05	1.83	2.01
Theophylline	2.31	5.72	4.33	6.12	5.46	6
Trenbolone	0.773	0.804	1.45	2.05	1.83	2.01
Trenbolone acetate	0.115	0.12	0.216	0.306	0.273	0.3
Valsartan	1.54	1.6	2.88	4.08	3.64	4
Verapamil	0.0577	0.06	0.108	0.153	0.137	0.15

PPCPs (ANEG) Detection Limits

	Tissue (ng/g)		Sediment (ng/g)		Surface Water (ng/L)	
	Min	**Max**	**Min**	**Max**	**Min**	**Max**
2-Hydroxy-ibuprofen	1.54	1.54	2.88	4.08	3.64	3.72
Bisphenol A	2.31	2.31	4.33	6.12	5.46	5.58
Furosemide	1.54	1.54	2.88	4.08	3.64	3.72
Gemfibrozil	0.308	0.308	0.577	0.816	0.728	0.744
Glipizide	0.308	0.308	0.577	0.816	0.728	0.744
Glyburide	0.308	0.308	0.577	0.816	0.728	0.744
Hydrochlorothiazide	3.38	3.38	2.88	4.08	3.64	3.72
Ibuprofen	1.54	1.54	2.88	4.08	3.64	3.72
Naproxen	0.769	0.769	1.44	2.04	1.82	1.86
Triclocarban	0.154	0.154	0.288	0.408	0.364	0.372
Triclosan	2.31	2.31	4.33	6.12	5.46	5.58
Warfarin	0.154	0.154	0.288	0.408	0.364	0.372

Pesticide Detection Limits

	Fish Tissue (ng/g)		Surface Water (ng/L)		Sediment (ng/g)	
Compound	**Min**	**Max**	**Min**	**Max**	**Min**	**Max**
2,4'-DDD	0.001	0.0096	0.01	0.0112	0.0009	0.0152
2,4'-DDE	0.001	0.0046	0.01	0.0112	0.001	0.0066
2,4'-DDT	0.001	0.0182	0.01	0.0112	0.0009	0.0522
4,4'-DDD	0.001	3.24	0.01	0.0112	0.0009	0.046
4,4'-DDE	0.001	3.24	0.01	0.0112	0.001	0.0101
4,4'-DDT	0.001	3.24	0.01	0.0112	0.0009	0.0402
Aldrin	0.001	0.0046	0.01	0.0115	0.001	0.0193
alpha-Endosulphan	0.001	3.24	0.01	0.0112	0.001	0.015
beta-Endosulphan	0.001	3.24	0.01	0.0112	0.001	0.0182
Chlordane, alpha (cis)	0.001	3.24	0.01	0.0112	0.001	0.0131
Chlordane, gamma (trans)	0.001	3.24	0.01	0.0112	0.001	0.0107
Chlordane, oxy-	0.001	3.24	0.01	0.0112	0.001	0.0046
Dieldrin	0.001	3.24	0.01	0.0112	0.001	0.0058
Endosulphan Sulphate	0.001	0.0038	0.01	0.0112	0.001	0.0103
Endrin	0.001	3.24	0.0105	0.0138	0.0017	0.068
Endrin Aldehyde	0.001	0.0038	0.0108	0.016	0.0032	0.275
Endrin Ketone	0.001	0.0038	0.01	0.0112	0.001	0.0527
HCH, alpha	0.001	3.24	0.01	0.0112	0.001	0.0065
HCH, beta	0.001	3.24	0.01	0.0112	0.001	0.0127
HCH, delta	0.001	0.0039	0.01	0.0112	0.001	0.0082

	Fish Tissue (ng/g)		Surface Water (ng/L)		Sediment (ng/g)	
Compound	**Min**	**Max**	**Min**	**Max**	**Min**	**Max**
HCH, gamma	0.001	3.24	0.01	0.0112	0.001	0.0063
Heptachlor	0.001	3.24	0.01	0.0112	0.001	0.0131
Heptachlor Epoxide	0.001	3.24	0.01	0.0112	0.001	0.0163
Hexachlorobenzene	0.001	3.24	0.01	0.0112	0.0009	0.0031
Methoxychlor	0.001	0.0038	0.01	0.0112	0.001	0.0177
Mirex	0.001	3.24	0.01	0.0112	0.001	0.0123
Nonachlor, cis-	0.001	10.1	0.0101	0.014	0.001	0.0296
Nonachlor, trans-	0.001	8.31	0.01	0.0112	0.001	0.0087