Final Report

Per- and Poly-fluoroalkyl Substances (PFAS) in Sediment
Goal 2 - Sediment as a Source of PFAS to the Food Web

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ACRONYMS AND ABBREVIATIONS

AA	atomic absorption
Ag	silver
As	arsenic
Ba	barium
BAF	bioaccumulation factor
BCF	bioconcentration factor
BSAF	biota-sediment accumulation factors
Ca	calcium
Cd	cadmium
CEC	cation exchange capacity
CID	charge injection device
Cr	chromium
u	copper
DHHS	Department of Health and Human Services
DL	detection limit
DO	dissolved oxygen
EGLE	Michigan Department of Environment, Great Lakes, and Energy
EIS	extracted internal standard
ESI	electrospray ionization
FASA	perfluoroalkane sulfomides and derivatives
FCSV	Fish Consumption Screenings Values
Fe	iron
f_{oc}	fraction organic carbon
FTSA	fluorotelomer sulfonic acids
GAC	granular activated carbon
GC	gas chromatography
GLEC	Great Lakes Environmental Center, Inc.
HCI	hydrochloric acid
Hg	mercury
HRGC	high resolution gas chromatography
HRMS	high resolution mass spectrometry
ICP	inductively coupled plasma
IDS	isotope dilution standard
IIS	injection internal standards
K_d	distribution coefficient
K_{oc}	organic carbon partition coefficients
K_p	sediment-water partition coefficients
LC	liquid chromatography
LM	Largemouth
LOQ	limits of quantitation
M	muscle
Mg	magnesium
Mn	manganese
MS	mass spectrometer
ND	not detected
NDIR	non-dispersive infrared detection
Ni	nickel

OES	optical emission spectrometry
PAH	polycyclic aromatic hydrocarbons
Pb	lead
PCB	polychlorinated biphenyls
PEC	probable effect concentrations
PFAA	perfluorinated alkyl acids
PFAS	per- and poly-fluoroalkyl substances
PFCA	perfluoroalkyl carboxylic acids
PFOA	perfluorooctanoic acid
PFOS	perfluorooctanesulfonic acid
PFSA	perfluoroalkane sulfomides
ppb	parts per billion
PPR	predator-prey ratio
QC	quality control
RL	reporting limit
RPD	relative percent difference
SE	Sample event
Se	selenium
SIM	selective ion monitoring
SOP	standard operating procedure
SPE	solid phase extraction
SUR	surrogate
TEC	threshold effect concentration
TOC	total organic carbon
WB	whole body
WWTP	wastewater treatment plant
Zn	zinc

1.0 INTRODUCTION

Per- and poly-fluoroalkyl substances (PFAS) have been detected in surface waters, sediments and aquatic biota of Michigan and are a risk to aquatic life and possibly human health via consumption of contaminated fish. The Michigan Department of Environment, Great Lakes, and Energy (EGLE) has defined maximum surface water concentrations of perfluorooctanoic acid (PFOA) and perfluorooctanesulfonic acid (PFOS) to protect aquatic life and human health. In Michigan, as elsewhere in the Great Lakes region, PFOS has been the dominant PFAS analyte detected in sediment samples (Remucal, 2019).

In order to gain a better understanding of the effect of PFAS contaminated sediment on aquatic organisms and the food web, EGLE directed Great Lakes Environmental Center, Inc. (GLEC) to initiate a two-part study to assess: the toxicity of PFAS-contaminated sediments to sediment-dwelling aquatic organisms (Goal 1A) and sediment as a source of PFAS to the food web (Goal 2, this study). Although PFOS was, overall, the most frequently detected analyte at the greatest concentration in sediments previously collected from Michigan, and it has been shown to be more toxic than other PFAS to aquatic life in water-only and mesocosm exposures (MacDonald et.al. 2004; Marziali et.al. 2019; Simpson et.al. 2021; and Stefani et. al. 2014), the EGLE two-part study was designed to measure a suite of PFAS in collected samples in order to better understand general PFAS chemistry in contaminated sediment and biota. This report summarizes the findings from the Goal 2 study.

In the Goal 2 study, EGLE was interested in determining whether the sediment continues to serve as a source of PFAS to the water column and/or aquatic biota in a location where source control has occurred and PFAS concentrations in fish have declined or plateaued. EGLE has been monitoring PFAS concentrations in sport fish fillet samples from such systems to provide data to the Michigan Department of Health and Human Services (DHHS) for evaluation of fish consumption advisories, and has found that sport fish continue to have elevated fillet PFAS concentrations, even after source control has occurred. It is currently unknown how long it will take for such systems to recover via natural processes. EGLE plans to continue fillet monitoring in these systems.

Kent Lake is an impoundment of the Huron River located downstream of the confluence of Norton Creek and the Huron River. Fish collected from Kent Lake in 2017 had high fillet PFOS concentrations and resulted in a Michigan Department of Health and Human Services (MDHHS) do-not-eat fish consumption advisory. In June 2018, the main source of PFOS to Norton Creek was identified as the City of Wixom wastewater treatment plant (WWTP), which discharges treated effluent to Norton Creek about 5 miles upstream of Kent Lake. The city of Wixom identified a chrome plating facility, which has been in operation since approximately 2000, as the source of high levels of PFOS (28,000 ng/L) to the City of Wixom's sanitary sewer system[1]. A temporary granular activated carbon (GAC) adsorption treatment system was installed in October

[1] https://www.michigan.gov/pfasresponse/investigations/lakes-and-streams/huron-river

2018 to treat the plating facility wastewater to remove PFOS prior to discharge into the city of Wixom's sanitary sewer, and a permanent system was installed in December 2018. Although PFOS concentrations were lower in fish fillets sampled in 2019 and 2020 than in 2017, the concentrations of PFOS in Largemouth Bass fillets still exceeded the threshold for a MDHHS do-not-eat fish consumption advisory.

Exposure of fish to PFAS may occur through bioconcentration (respiratory uptake through the gills) or bioaccumulation (through dietary ingestion as well as uptake). Goal 2 was designed to monitor sediments, sediment-dwelling invertebrates, whole prey fish (i.e., sport fish dietary items), and whole sport/predator fish to determine if sediments may be contributing PFAS to the food web. If sediments are serving as a source of PFAS to the food web, elevated levels of PFAS should be found in benthic organisms and the fish that feed upon them. For example, Lasier et al. (2011) showed that the aquatic oligochaete, *L. variegatus*, accumulated PFOS and other PFAS when exposed to contaminated field sediments for 28-days. Asher et al. (2012) found that bottom-feeding organisms showed elevated PFOS compared to other aquatic organisms which may be due to sediment contamination of PFOS precursors.

2.0 METHODS

EGLE identified Kent Lake as the study location for the Goal 2 study. Proud Lake, another impoundment on the Huron River located about 8 miles upstream of Kent Lake and about 2 miles upstream of the Norton Creek/Huron River confluence, was selected as a reference site (Table 1). Proud Lake is separated from Kent Lake by Moss Lake Dam, a low-head dam small enough to allow fish passage. Water, sediment, sediment-dwelling organisms and fish were sampled and collected from Kent Lake and Proud Lake and analyzed for PFAS and other constituents as described below. The results of this study, presented in Section 3, may be used by EGLE to direct the frequency of future sampling and possibly inform future fish consumption advisory studies.

Table 1. Goal 2 Sampling Locations

Site	Latitude	Longitude	Narrative Description of Sample Collection Area
Kent Lake (study location)	42.519199	-83.659989	Southwest area of the lake, near the outlet
Proud Lake (reference site)	42.567463	-83.518649	Northeast end of the lake, near the inlet

2.1 Sample Collection and Handling

GLEC collected samples of sediment and water during two sampling events (Table 2, Figures 1 and 2) following GLEC standard operating procedures (SOPs). Sample events 1 and 2 (SE1 and SE2) took place on October 1, 2021 and November 24, 2021, respectively. Sampling was conducted from a small craft, and sites were located using a vessel-mounted or handheld GPS, with accuracy within 15 meters.

Table 2. Water and Sediment Sample Collection

Sampling Event and Date	Site	Latitude	Longitude	Sample ID on Maps*	
				Sediment Subsample	Water Sample
SE1 10.01.2021	Kent Lake	42.5192	-83.65999	KLS1.1	KLW1.1
		42.51864	-83.66118	KLS1.2	
		42.52112	-83.65955	KLS1.3	
	Proud Lake	42.56746	-83.51865	PLS1.1	PLW1.1
		42.56732	-83.51811	PLS1.2	
		42.56795	-83.51911	PLS1.3	
SE2 11.24.2021	Kent Lake	42.52447	-83.64691	KLS2.1	
		42.52297	-83.65127	KLS2.2	
		42.53373	-83.6485	KLS2.3	
		42.52009	-83.66041		KLW2.1
	Proud Lake	42.56902	-83.52297	PLS2.1	
		42.56828	-83.52042	PLS2.2	PLW2.1

* Figures 1 and 2

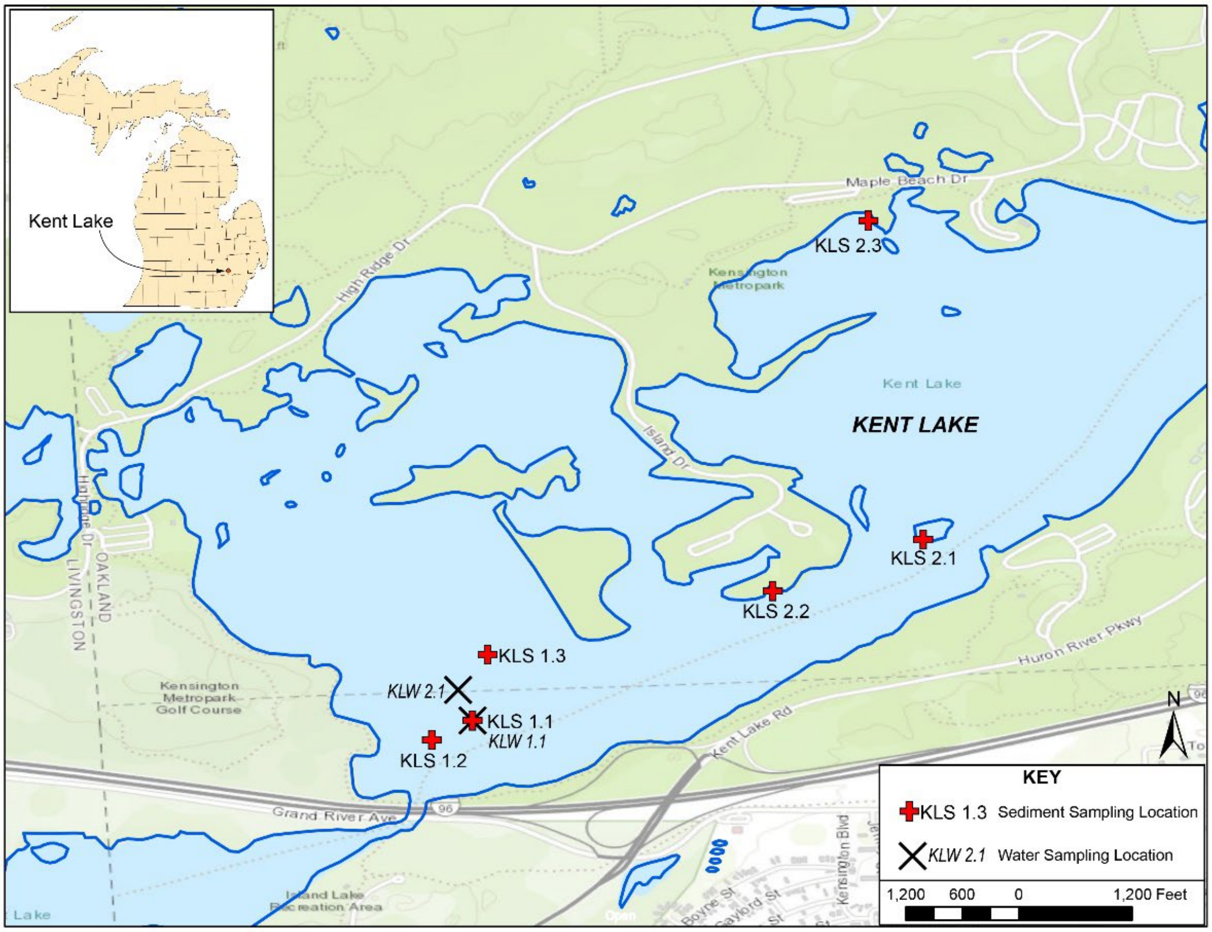

Figure 1. Kent Lake Water and Sediment Sampling Locations

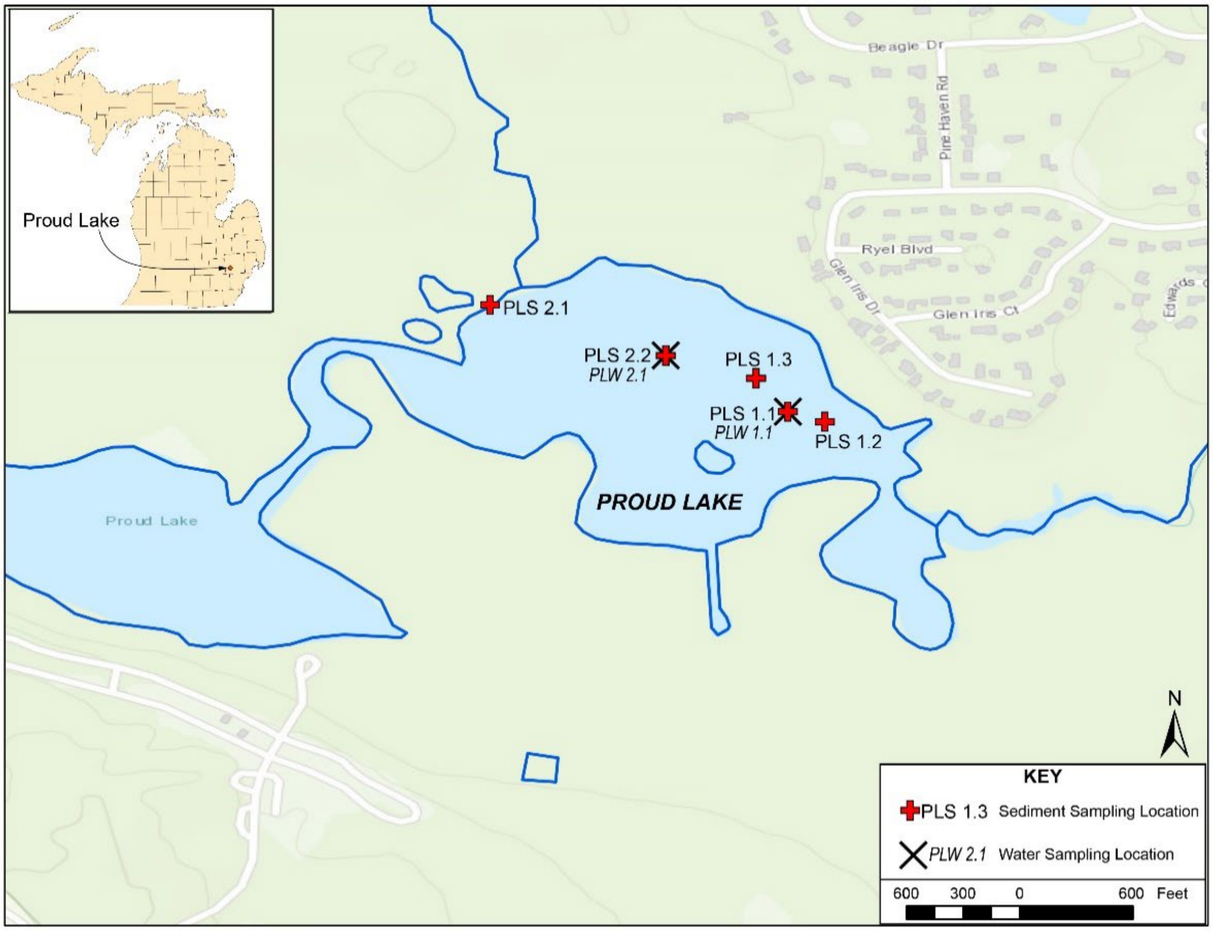

Figure 2. Proud Lake Water and Sediment Sampling Locations

A single surface water sample[2] was collected from each lake during each sampling event, using a depth-integrated sampler. The water samples were deposited into 250 mL HDPE plastic sample bottles, and placed on ice without chemical preservation. Field measurements of water temperature, specific conductivity, pH and dissolved oxygen (DO) were collected, at the same time and place as the water samples, using a YSI® multi-parameter probe which was calibrated each day of use.

Sediment samples were collected using a petite Ponar sampler from three locations in each lake during each sampling event[3]. Sediment samples were combined and mixed in a stainless-steel bowl to form a composite sample, and then distributed to appropriate sample containers using a stainless-steel spoon at each lake. During SE2 at Kent Lake, sediment samples were collected from the eastern portion of the lake to correspond with benthic macroinvertebrate sampling locations. Sample containers were labeled with the location and date, placed on ice in a cooler immediately upon collection, and transported to GLEC for processing.

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[2] No field or equipment blanks, replicates or duplicate were collected in this study.

[3] With the exception of SE2 at Proud Lake, when two sediment samples were collected.

Biota samples were collected by EGLE (Table 3). Fish samples were collected from Kent Lake and Proud Lake on May 11 and 16, 2021, respectively. Two predator and two prey fish samples were collected from each lake, with each sample consisting of multiple individual fish. Fish samples were double ziplock bagged, frozen, and transported to GLEC where they were held frozen. Benthic macroinvertebrate[4] samples were collected by EGLE on November 10 and 27, 2021 as kick-net samples (i.e., sweep net), and transported to GLEC where they were held refrigerated pending sorting. Organisms were sorted into major taxonomic groups (e.g., Odonata and Amphipoda) – the sediment was sieved through a 500-micron sieve, and the organisms were hand-picked, rinsed with deionized water and blotted dry to remove detritus (e.g., see Figure 3); the samples were frozen for shipment to the laboratory for analysis.

Figure 3. Proud Lake Odonate

Table 3. Biota Sample Collection

Waterbody	Biota type	Date collected (mm.dd.yyyy)	Taxonomic descriptor	Number of individuals and/or weight	Sample ID/ Description
Kent Lake	Predator fish	05.11.2021	Largemouth (LM) bass	5	40234558005 Predator fish #1
			Lepomis spp. (sunfish)	5	40237220001 Predator fish #2
	Prey/forage fish	05.11.2021	*Lepomis spp.*	~100 g	40237220002 Forage fish #1
			Micropterus spp. (juvenile bass)	>10, ~100 g	40237220003 Forage fish #2
	Benthic macro-invertebrate	11.10.2021	Odonata (dragonflies/ damselflies)	~3 g	40237220004 Invertebrate #1
		11.27.2021	Odonata	23, 2.44 g	40238788038 Invertebrate #2
			Amphipoda (crustaceans)	5.12 g	40238788039 Invertebrate #3
			Other taxa	6.54 g	40238788040 Invertebrate #4

[4] Also referred to as benthos and as sediment-dwelling organisms.

Waterbody	Biota type	Date collected (mm.dd.yyyy)	Taxonomic descriptor	Number of individuals and/or weight	Sample ID/Description
Proud Lake	Predator fish	05.16.2021	LM bass	5	40234558001 Predator fish #1
			Pumpkinseed	5	40234558002 Predator fish #2
	Prey/forage fish	05.16.2021	*Lepomis spp.*	>10, ~100 g	40234558003 Forage fish #1
			Micropterus spp.	>10, ~100 g	40234558004 Forage fish #2
	Benthic macro-invertebrate	11.10.2021	Odonata	4.15 g	40237220005 Invertebrate #1
		11.27.2021	Odonata	19, 6.04 g	40238788037 Invertebrate #2

2.2 Sample Shipment and Analysis

Samples of all matrices to be analyzed for chemical parameters and physical characteristics (Table 4) were shipped in coolers with wet or dry (fish samples) ice via FedEx overnight delivery to Pace Analytical laboratories for analysis.

Sediment samples were analyzed for PFAS as well as other contaminants known to contribute to the toxicity of sediment to aquatic organisms: elements (trace metals), polycyclic aromatic hydrocarbons (PAHs), and polychlorinated biphenyls (PCBs). These contaminants were analyzed for consistency with EGLE's sediment dredge procedure, WRD-048, and also with Goal 1A of this project. The objective was to assess whether sediment contaminants other than PFAS could be having an impact on the food web at the study location.

Table 4. Number of Goal 2 Samples

Analysis Description	Matrix	Number of Samples
36 PFAS compounds in sediment	Sediment	4
36 PFAS compounds in surface water	Water	4
35 PFAS compounds in biota	Tissue	14
Percent lipids	Tissue	14
pH	Sediment	4
Cation Exchange Capacity (CEC)	Sediment	4
Grain size	Sediment	4
Total Organic Carbon (TOC)	Sediment	4
Mg and Ca	Sediment	4
PCB Congeners (209)	Sediment	2*
PAHs (17 parent PAHs)	Sediment	2*
Elements (13): As, Ba, Cd, Cr, Cu, Fe, Pb, Mn, Hg, Ni, Se, Ag, Zn	Sediment	2*

* Analyzed once at each site.

Field-collected and quality control (QC) samples were analyzed for physical and chemical parameters following Pace SOPs (Table 5). For all methods, reported detection limits (DLs) and reported limits of quantitation (LOQs, or reporting limits (RLs)) were adjusted to account for actual measured sample volume/weight and dilution. Results less than the reported LOQ/RL but greater than the reported DL are reported as estimated concentrations, with a J flag. Undetected results are reported as less than the reported DL.

Table 5. Analytical Methods for Physical and Chemical Parameters

Parameter	Matrix	Pace SOP	Reference Method
36 PFAS compounds	sediment, surface water	Pace ME003NI-04 Determination of Per- and Polyfluoroalkyl Substances (PFAS) by LC/MS/MS (Isotope Dilution)	Lab SOP, PFAS by ID-SPE
35 PFAS compounds	biota	Pace ENV-SOP-MIN4-0178 Determination of Selected 36 Per- and Polyfluoroalkyl Substances (PFAS) by LC/MS/MS (Isotope Dilution)	Lab SOP, PFAS by ID
Tissue processing	biota	Pace ENV-SOP-GBAY-0129 Sample Homogenization, Compositing and Sub-Sampling	Not applicable
Lipids	biota	Pace ENV-SWI-MIN4-0016 Lipid Determination	Lab SOPs
pH	sediment	Pace ENV-SOP-GBAY-0047	EPA Method SW846 9045D
CEC	sediment	Pace ENV-SOP-SHRT-0046	EPA Method 200.7/SW846 9081
Grain Size	sediment	Pace 158 Grain Size Analysis	ASTM D422
TOC	sediment	Pace GBAY-0051	Lloyd Khan Method
PCB Congeners (209)	sediment	Pace ENV-SOP-MIN4-0031 Preparation and Analysis of Samples for the Determination of Chlorinated Biphenyl Congeners by EPA 1668A/C	EPA Method 1668A and 1668C
PAHs (17 parent PAHs)	sediment	Pace ENV-SOP-GBAY-0077- Rev.01 Microwave Extraction for the Determination of PAH, BNA and TPH-DS in a Solid Matrix; ENV-SOP-GBAY-0081 Determination of Semi-Volatile Organics by GC/MS (Selective Ion Monitoring)	EPA Method SW846 3546C (extraction); 8270C SIM (analysis)
Elements As, Ba, Cd, Ca, Cr, Cu, Fe, Pb, Mg, Mn, Ni, Se, Ag, Zn	sediment	Pace ENV-SOP-GBAY-0009 Determination of Metals by Inductively Coupled Plasma (ICP) Spectroscopy by 6010D and 200.7	EPA Methods 6010D/200.7
Mercury (Hg)	sediment	Pace ENV-SOP-GBAY-0013 Determination of Mercury by Cold Vapor Atomic Absorption Spectroscopy - CETAC M-7500 (7470A/7471B_245.1)	EPA Method SW846 7470A/7471B/245.1

2.2.1 PFAS in aqueous samples

A 250 mL water sample was fortified with a solution of surrogate/extracted internal standard/isotope dilution standard (SUR/EIS/IDS) compounds and passed through a Phenomenex Strata-XL-AW 100 µm Polymeric Weak Anion solid phase extraction (SPE) cartridge to extract the method analytes and surrogates. The compounds were eluted from the SPE cartridge with 4 mL of methanol and 4 mL of 0.6 % ammonia in methanol. The extract was then filtered by Phenomenex® Strata PFAS SPE, with a tube rinse of clean methanol. With the filtration tube rinse, the final extract volume was approximately 10 mL. An aliquot of the extract was fortified with injection internal standards (IIS). 10 µL of the fortified aliquot was injected onto an Agilent 1260 liquid chromatography (LC) system equipped with a Phenomenex Gemini® 3µm C18 110Å LC column (50 x 3 mm) coupled to a Sciex tandem mass spectrometer (MS/MS) detector in negative ion electrospray ionization (ESI) mode. The analytes were separated and identified by comparing the acquired mass spectra and retention times to the reference spectra and retention times for calibration standards acquired under identical LC/MS/MS conditions. The concentration of each analyte was determined by using the internal standard isotope dilution technique. DLs and LOQs based on the SOP are listed in Attachment A, Table A.1.

2.2.2 PFAS in sediment samples

Approximately 1 g of solid sample was fortified with SUR/EIS/IDS, mixed with 4 mL of methanol and 4 mL of 0.6 % ammonia in methanol, and then shaken on an orbital shaker, followed by sonication and centrifugation. The extract was then filtered by Phenomenex® Strata PFAS SPE, with a tube rinse of clean methanol. With the filtration tube rinse, the final extract volume was approximately 10 mL. An aliquot of the extract was fortified with IIS. 10 µL of the fortified aliquot was injected onto an Agilent 1260 LC system equipped with a Phenomenex Gemini® 3µm C18 110Å LC column (50 x 3 mm) coupled to a Sciex tandem MS/MS detector in negative ion ESI mode. The analytes were separated and identified by comparing the acquired mass spectra and retention times to the reference spectra and retention times for calibration standards acquired under identical LC/MS/MS conditions. The concentration of each analyte on a dry weight[5] basis was determined using the internal standard isotope dilution technique. DLs and LOQs based on the SOP are listed in Attachment A, Table A.1.

2.2.3 PFAS in biota samples

Approximately 2 g of wet ground tissue was fortified with SUR/EIS/IDS and extracted with 7 mL of 1% ammonia acetonitrile for 16 hours. The extract was treated with ENVI-Carb™ and filtered prior to Phenomenex® Strata PFAS SPE cleanup. The extract was concentrated to ~0.1 mL with nitrogen gas, spiked with IIS, and then diluted to 1 mL with 96:4% (vol/vol) methanol:water. 3 µL was injected onto an Agilent 1290 LC system equipped with a 30 mm isolator (delay) column followed by a Phenomenex Gemini® 3µm C18 reverse phase LC column (100 x 3 mm) coupled to a Sciex quadropole tandem MS/MS detector in negative ion ESI mode. The concentration of each analyte

[5] Fraction dry weight was determined gravimetrically by weighing a portion of each sediment sample before and after drying it in an oven.

was determined on a wet weight basis using the isotope dilution and internal standard techniques, depending on target analyte. SUR/EIS/IDS was added to all calibration standards, field samples and QC samples to monitor the extraction efficiency of the method analytes. DLs and LOQs based on the SOP are listed in Attachment A, Table A.1.

2.2.4 Biota tissue processing and percent lipids

Biological tissue samples were processed as whole-body composites. Individual fish of the same species and similar size were chopped into cubes and ground in a meat grinder. Multiple benthic macroinvertebrate specimens collected from each location during each sampling event were ground. Ground tissue was homogenized by blending with liquid nitrogen to form a composite sample.

Percent lipids were determined gravimetrically, following the extraction of a subsample of ground tissue with methylene chloride by sonication. For all the Kent Lake benthos samples, as well as Proud Lake Invertebrate #2, an inadequate weight of organisms was obtained to support lipid analysis.

2.2.5 Polychlorinated Biphenyls (PCBs)

Concentrations of 209 polychlorinated biphenyls (PCBs) in sediment were determined by EPA Method 1668, Revisions A and C by high resolution gas chromatography/high resolution mass spectrometry (HRGC/HRMS). Sediment sample extracts were cleaned-up using an acid wash and multi-layer silica prior to analysis. Approximately 125 PCB congeners were sufficiently resolved to be reported as individual congeners, while approximately 70 were reported as mixtures of co-eluting isomers. DLs and LOQs based on the SOP are listed in Attachment A, Table A.2.

2.2.6 Polycyclic Aromatic Hydrocarbons (PAHs)

Concentrations of seventeen polycyclic aromatic hydrocarbons (PAHs) in sediment were determined using gas chromatography/mass spectroscopy (GC/MS) in selective ion monitoring (SIM) mode following documented procedures listed in EPA SW846 Method 8270C SIM for both identification and quantification of analytes. DLs and LOQs based on the SOP are listed in Attachment A, Table A.3.

2.2.7 Elements

Concentrations of 14 trace elements (As, Ba, Cd, Ca, Cr, Cu, Fe, Pb, Mg, Mn, Ni, Se, Ag, Zn) in sediment were determined by ICP by EPA Methods 6010D and 200.7. Samples were digested by heating with appropriate acids and oxidizing agents to solubilize the target elements. Portions of the digestates were pumped into a nebulizer to produce an aerosol. The aerosol was aspirated into the torch of an argon ICP-OES where it was evaporated and decomposed into atoms and ions. The plasma energy caused the target atoms to become excited and, during relaxation, emit characteristic light in the visible and/or ultraviolet emissions. Each element in the sample emits photons at a discrete wavelength(s), which are specific to that element. The light emissions were separated into wavelength and order by passing through a prism and onto an Echelle grating. The signal was then read and quantified by a charge injection device (CID). The intensities of the wavelengths were proportional to the quantity of the target elements, determined through a comparison to known concentrations from a calibration curve. Background correction was required to compensate for spectral interferences. DLs and LOQs based on the SOP are listed in Attachment A, Table A.3.

2.2.8 Mercury

Mercury in sediment was determined by cold vapor atomic absorption (AA) spectroscopy following EPA Methods SW846 7470A, 7471B and 245.1. Cold vapor AA utilizes the volatile property of elemental mercury at a wavelength of 253.7 nm. 0.6 grams of a homogenized sediment sample was digested with oxidizing reagents and acids in a hot block to release mercury from organic complexes. After digestion, the oxidizing reagents were neutralized. Stannous chloride was added to reduce ionic mercury to the ground state. A flow injection analysis system swept the volatile elemental mercury out of the sample and into the cell of an AA spectrophotometer, the absorbance signal of which was proportional to the amount of mercury in the sample. The LOQ for this method was 0.035 µg/kg. DLs and LOQs based on the SOP are listed in Attachment A, Table A.3.

2.2.9 Total organic carbon

Total organic carbon (TOC) in sediment was analyzed by the Lloyd Kahn Method. A weighed sample was acidified with hydrochloric acid (HCl) to remove inorganic forms of carbon (i.e., carbonates and bicarbonates), and then dried in an oven at 75°C to remove excess moisture and HCl. Approximately 1 g of sample was then combusted in a furnace at 1,000°C, producing CO_2 gas. The amount of CO_2 formed was determined by direct non-dispersive infrared detection (NDIR), and was proportional to the carbon in the sample. The LOQ was 100 µg/kg. DLs and LOQs based on the SOP are listed in Attachment A, Table A.3.

2.2.10 pH in sediment

Approximately 20 g of a sediment sample was mixed with reagent water, and the pH of the
resulting aqueous suspension was measured in the same manner as a water sample, electrometrically using a combination pH electrode with temperature compensation.

2.2.11 Cation exchange capacity

Approximately 5 g of air-dried sediment was mixed with an excess of sodium acetate solution, resulting in an exchange of the added sodium cations for the matrix cations. The sample was then washed with isopropyl alcohol to remove sodium not attached to the exchange sites. With the addition of ammonium acetate solution, the adsorbed sodium was replaced with ammonium. The concentration of displaced sodium was then analytically determined by inductively coupled plasma-optical emission spectrometry (ICP-OES).

2.2.12 Grain size

A representative sample of air-dried sediment was weighed to 0.1 g, placed in the sieve shaker stack of a RO-TAP® particle analysis machine, and shaken for 15 minutes. The amount of sieved material in each sieve was weighed to determine the size passing each sieve.

3.0 RESULTS

3.1 Field Measurements

Field measurements of water quality parameters collected *in situ* in Kent Lake and Proud Lake during both sampling events are presented in Table 6. Water quality parameters were generally comparable between the two lakes. Notable changes between the first and second sampling events included significant cooling in both lakes, declines in pH, and increases in DO (especially in Proud Lake).

Table 6. Field Measurements

Site	Sampling Event	Temperature (°C)	pH (SU)	DO (mg/L)	Conductivity (µS/cm)
Kent Lake	SE1	18.7	8.29	11.7	667
	SE2	4.2	7.88	11.8	777
Proud Lake	SE1	18.2	7.81	7.8	710
	SE2	5.1	7.67	11.0	795

3.2 PFAS in Sediment, Water and Biota Samples

PFAS results are summarized in Tables 7 through 10 and Appendix I, and detailed analytical reports are provided in Appendix II. In the analysis and presentation of these data, estimated concentration values reported below the LOQ (i.e., J-flagged results) were included. Note that concentrations are reported in units of µg/kg (part per billion, ppb) dry weight for sediment, µg/kg (ppb) wet weight for biota, and ng/L for surface water (part per trillion, ppt).

3.2.1 PFAS Concentrations in Sediment and Surface Water

Table 7 presents the PFAS results for sediment and surface water samples in Kent Lake and Proud Lake. PFOS and 6:2 FTS were the only PFAS compounds detected in Kent Lake sediment; PFOS made up 81 to 100% of the total concentration of PFAS. In contrast, no PFAS were detected in the Proud Lake sediment samples. The concentration of PFOS was nearly four times higher in the Kent Lake sediment sample collected during SE1 than in SE2. It is not clear whether the same was true for 6:2 FTS because this compound was not detected in the SE1 sediment above the reported DL of 2.0 µg/kg for the sample.

Nine PFAS compounds were detected in Kent Lake surface water, while eight PFAS compounds were detected in water at the Proud Lake area reference site (Table 7). PFOS made up 2 to 4%, and 6:2 FTS made up 42 to 49%, of the total concentration of PFAS in Kent Lake water samples. Neither PFOS nor 6:2 FTS were detected in water from Proud Lake. Comparing the concentrations of other PFAS compounds detected in surface water from both lakes, based on the median concentration(s) in the two sampling events, we found:

- concentrations of three PFAS compounds (PFPeA, PFHxA, and PFHpA) were 5 to 10 times higher in Kent Lake than in Proud Lake; and
- concentrations of four PFAS compounds (PFBA, PFOA, PFBS and PFHxS) were similar in the two lakes.

Notably, PFOS and 6:2 FTS were measured in Kent Lake water as well as sediment, but were not detected in Proud Lake water or sediment. Aside from 6:2 FTS, the highest PFAS concentrations in Kent Lake water were the compounds PFHxA, PFPeA (both biodegradation products of 6:2 FTS; Guelfo et al., 2021) and PFHpA.

Table 7. Kent Lake and Proud Lake Sediment and Surface Water PFAS Results[a]

		Kent Lake				Proud Lake			
	Matrix	Sediment		Surface Water		Sediment		Surface Water	
	Sample Event	SE1	SE2	SE1	SE2	SE1	SE2	SE1	SE2
	Lab Sample #	WJ080 59-003	WL010 91-008	WJ080 59-004	WL010 91-006	WJ080 59-001	WL010 91-007	WJ080 59-002	WL010 91-005
	Units	µg/kg (ppb) dry weight		ng/L (ppt)		µg/kg (ppb) dry weight		ng/L (ppt)	
Analyte		Result							
1	PFBA	< 2.7	< 0.56	**9.1**	**6.2**	< 1.8	< 2.0	**4.6**	**4.9**
2	PFPeA	< 1	< 0.21	**30**	**17**	< 0.68	< 0.78	**3.0 (J)**	**3.3**
3	PFHxA	< 1.2	< 0.25	**19**	**11**	< 0.79	< 0.91	**2.7 (J)**	**3.2 (J)**
4	PFHpA	< 0.91	< 0.19	**14**	**12**	< 0.61	< 0.70	**1.2 (J)**	**1.5 (J)**
5	PFOA	< 1.4	< 0.29	**2.0 (J)**	**2.1 (J)**	< 0.91	< 1.0	**2.6 (J)**	**2.5 (J)**
6	PFNA	< 0.96	< 0.20	< 0.39	< 0.39	< 0.64	< 0.73	**0.48 (J)**	**0.43 (J)**
7	PFDA	< 1	< 0.21	< 0.45	< 0.45	< 0.68	< 0.78	< 0.45	< 0.44
8	PFUdA	< 1.2	< 0.25	< 0.53	< 0.53	< 0.79	< 0.91	< 0.54	< 0.52
9	PFDoA	< 1.1	< 0.24	< 0.40	< 0.40	< 0.75	< 0.86	< 0.41	< 0.39
10	PFTrDA	< 1.1	< 0.23	< 0.45	< 0.45	< 0.74	< 0.85	< 0.46	< 0.44
11	PFTeDA	< 1.2	< 0.25	< 0.51	< 0.51	< 0.81	< 0.93	< 0.52	< 0.50
12	PFHxDA	< 1.4	< 0.30	< 0.69	< 0.70	< 0.96	< 1.1	< 0.70	< 0.68
13	PFODA	< 2.2	< 0.47	< 0.85	< 0.85	< 1.5	< 1.7	< 0.86	< 0.84
14	PFBS	< 0.84	< 0.18	**2.9 (J)**	**2.8 (J)**	< 0.56	< 0.64	**3.0 (J)**	**3.0 (J)**
15	PFPeS	< 1.2	< 0.25	< 0.51	< 0.51	< 0.80	< 0.92	< 0.51	< 0.50
16	PFHxS	< 1.1	< 0.24	**0.92 (J)**	**0.96 (J)**	< 0.76	< 0.87	**1.2 (J)**	**1.0 (J)**
17	PFHpS	< 1.1	< 0.24	< 0.42	< 0.43	< 0.75	< 0.86	< 0.43	< 0.42
18	PFOS	**7.4**	**1.9**	**3.6**	**3.6**	< 1.5	< 1.8	< 1.7	< 1.7
19	PFNS	< 1.4	< 0.30	< 0.61	< 0.61	< 0.94	< 1.1	< 0.61	< 0.60
20	PFDS	< 1.4	< 0.30	< 0.66	< 0.66	< 0.96	< 1.1	< 0.67	< 0.65
21	PFDOS	< 1.7	< 0.35	< 0.89	< 0.89	< 1.1	< 1.3	< 0.90	< 0.87

		Kent Lake				Proud Lake			
	Matrix	Sediment		Surface Water		Sediment		Surface Water	
	Sample Event	SE1	SE2	SE1	SE2	SE1	SE2	SE1	SE2
	Lab Sample #	WJ080 59-003	WL010 91-008	WJ080 59-004	WL010 91-006	WJ080 59-001	WL010 91-007	WJ080 59-002	WL010 91-005
	Units	µg/kg (ppb) dry weight		ng/L (ppt)		µg/kg (ppb) dry weight		ng/L (ppt)	
Analyte		Result							
22	PFOSA	< 1.1	< 0.24	< 0.52	< 0.52	< 0.76	< 0.87	< 0.53	< 0.51
23	EtFOSE	< 1.5	< 0.31	< 0.81	< 0.81	< 0.98	< 1.1	< 0.82	< 0.80
24	MeFOSE	< 2.1	< 0.45	< 1.1	< 1.1	< 1.4	< 1.6	< 1.1	< 1.1
25	EtFOSA	< 2.3	< 0.48	< 1.2	< 1.2	< 1.5	< 1.8	< 1.2	< 1.1
26	MeFOSA	< 2.2	< 0.47	< 1.1	< 1.1	< 1.5	< 1.7	< 1.1	< 1.1
27	EtFOSAA	< 1.9	< 0.39	< 0.64	< 0.64	< 1.2	< 1.4	< 0.65	< 0.63
28	MeFOSAA	< 2.5	< 0.53	< 0.79	< 0.80	< 1.7	< 1.9	< 0.80	< 0.78
29	4:2 FTS	< 1.4	< 0.29	< 0.74	< 0.75	< 0.93	< 1.1	< 0.75	< 0.73
30	6:2 FTS	< 2	**0.44 (J)**	**79**	**40**	< 1.3	< 1.5	< 1.7	< 1.7
31	8:2 FTS	< 1.8	< 0.37	< 1.4	< 1.4	< 1.2	< 1.3	< 1.4	< 1.3
32	10:2 FTS	< 2.4	< 0.50	< 1.0	< 1.0	< 1.6	< 1.8	< 1.0	< 1.0
33	GenX	< 3.7	< 0.78	< 1.8	< 1.8	< 2.5	< 2.9	< 1.8	< 1.7
34	ADONA	< 0.96	< 0.20	< 0.41	< 0.41	< 0.64	< 0.74	< 0.42	< 0.40
35	9Cl-PF3ONS	< 1	< 0.21	< 0.41	< 0.41	< 0.68	< 0.78	< 0.42	< 0.40
36	11Cl-PF3OUdS	< 1.1	< 0.23	< 0.56	< 0.57	<0.73	<0.84	<0.57	<0.55
	Total PFAS[b]	7.4	2.34	160	96	ND	ND	19	20
	Percent Solids (%)	13.3	63.0			19.3	19.4		

[a] Detected concentrations above the reported LOQ appear in bold.

[b] ND = Not detected. Results reported as < [DL] were treated as 0 (zero) concentration in the Total PFAS summation in this table.

J = Estimated concentration below the LOQ but above the DL. See Appendix I for reported DLs and Appendix II for reported LOQs.

Plots display the PFAS concentrations measured in Kent Lake sediment (Figure 4), Kent Lake surface water (Figure 5), and Proud Lake surface water[6] (Figure 6) on a radial log scale. Concentrations of 15 of the most commonly-detected PFAS compounds (PFBA, PFPeA, PFHxA, PFHpA, PFOA, PFNA, PFDA, PFUdA, PFDoA, PFBS, PFOS, PFDS, EtFOSAA, MeFOSAA and 6:2 FTS) are displayed in these radar plots, the patterns of which can be used as a “fingerprint” to illustrate similarities and differences between samples. The data plotted in Figures 4 through 6 illustrates that:

- Within each lake and sample media, PFAS concentrations and their distribution are very similar between SE1 and SE2.
- The concentrations of PFOS and 6:2 FTS in Kent Lake sediment differed between SE1 and SE2. These are considered in Section 4.1.

[6] A radar plot is not presented for PFAS in Proud Lake sediment because no PFAS concentrations were detected.

- Between Kent Lake sediment and water, the distributions of PFAS are different.
- Between Kent Lake and Proud Lake water, the distributions of PFAS are fairly similar, although the concentrations are higher in Kent Lake.

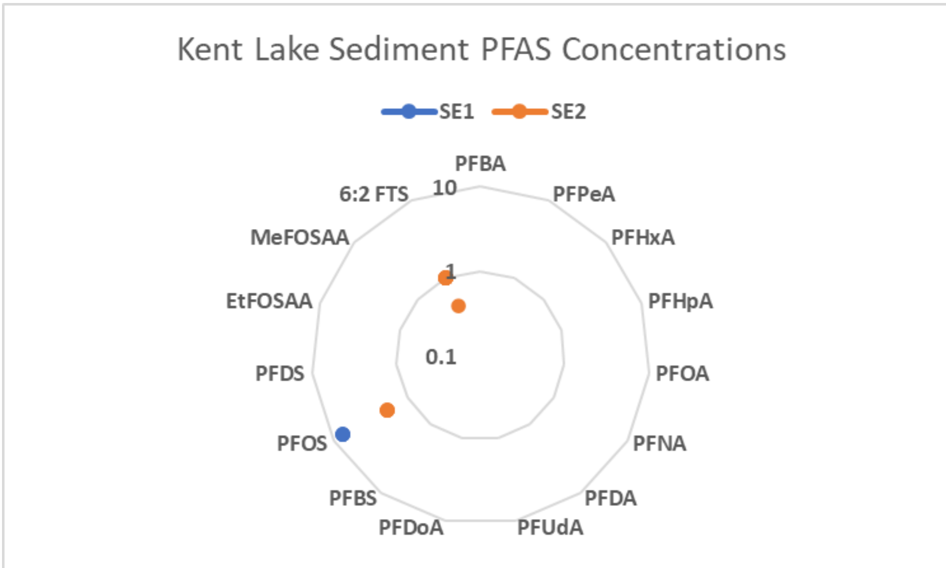


Figure 4. Radar plot of Kent Lake sediment sample PFAS concentrations in µg/kg (ppb) dry weight

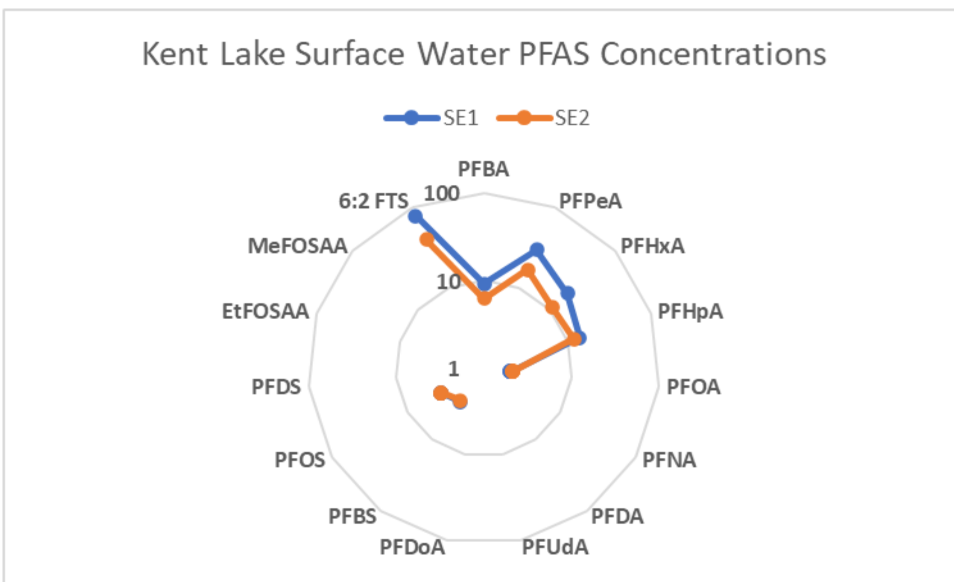


Figure 5. Radar plot of Kent Lake surface water sample PFAS concentrations in ng/L (ppt)

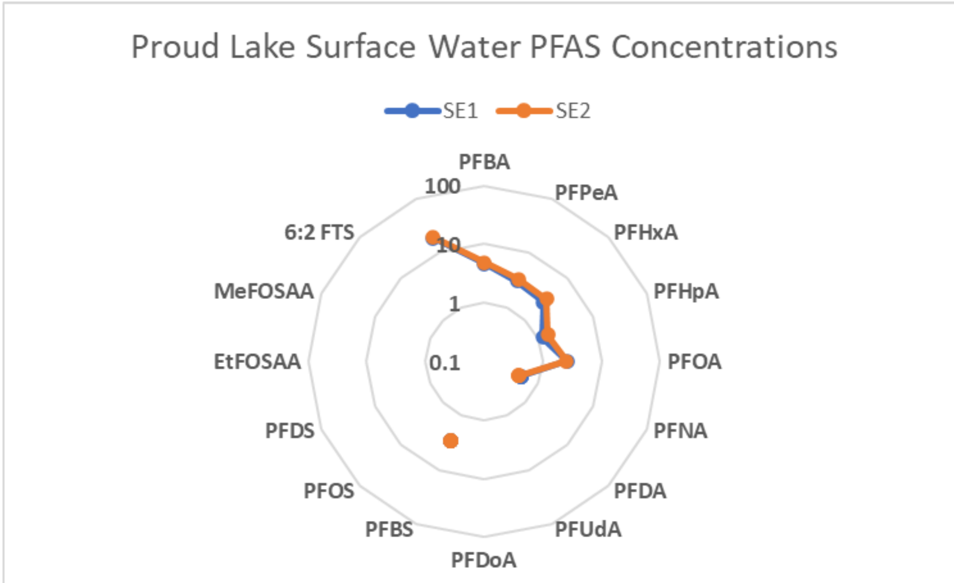


Figure 6. Radar plot of Proud Lake surface water sample PFAS concentrations in ng/L (ppt)

3.2.2 PFAS Concentrations in Kent Lake Biota

Table 8 contains results for PFAS analysis of biota samples (predator and forage fish) in Kent Lake. Eleven to 13 and 12 to 16 PFAS compounds were detected in predator and forage fish in Kent Lake, respectively, with PFOS making up 92 to 96 percent of the total PFAS concentration in the sampled fish. The total PFAS concentration was about 35% higher, on average, in predator fish versus prey fish. The PFOS concentrations measured in Kent Lake predator fish were 630 µg/kg (Largemouth Bass) and 110 µg/kg (sunfish); PFOS concentrations in forage fish were 160 µg/kg (sunfish) and 380 µg/kg (juvenile bass). In comparison, fish sampled throughout the Huron River watershed from 2017 to 2022 had PFOS concentrations that ranged from 0.7 to 2,000 µg/kg[7], with the highest concentration found in Kent Lake in 2017. As another comparison at the National scale, the maximum PFOS concentration measured in fish from EPA's 2013-14 National Rivers and Streams Assessment (NRSA) was 283 µg/kg in a channel catfish collected from the Ohio River (Barbo et al., 2023).

Table 8. Kent Lake Fish PFAS Results[a]

	Lab Sample #	40234558005	40237220001	40237220002	40237220003
	Sample Description[b]	**Predator fish #1**	**Predator fish #2**	**Forage fish #1**	**Forage fish #2**
	Taxonomic Information	**LM bass**	***Lepomis spp.* (sunfish)**	***Lepomis spp.***	***Micropterus spp.* (juvenile bass)**
Analyte		**Result in µg/kg (ppb) wet weight**			
1	PFBA	< 0.090	< 0.089	< 0.090	< 0.086
2	PFPeA	< 0.086	< 0.085	< 0.086	< 0.082
3	PFHxA	**0.43 (I)**	< 0.12	**0.19 (I, J)**	< 0.12
4	PFHpA	< 0.12	< 0.12	< 0.12	< 0.11
5	PFOA	< 0.078	< 0.078	< 0.078	< 0.075
6	PFNA	< 0.084	**0.15 (I, J)**	**0.18 (J)**	< 0.081
7	PFDA	**8.8**	**2.6**	**3.5**	**8.7**
8	PFUdA	**5.7**	**2.4**	**2.5**	**5.2**
9	PFDoA	**2.3**	**0.98**	**1.1**	**1.4**
10	PFTrDA	**0.81**	**0.44**	**0.35**	**0.47**
11	PFTeDA	**0.44**	**0.19 (J)**	**0.23 (J)**	**0.22 (J)**
12	PFHxDA	< 0.078	**0.078 (I, J)**	**0.087 (I, J)**	< 0.075
13	PFODA	< 0.098	< 0.097	< 0.098	< 0.094
14	PFBS	< 0.094	< 0.094	< 0.094	< 0.090
15	PFPeS	< 0.10	< 0.10	< 0.10	< 0.097
16	PFHxS	< 0.094	< 0.093	< 0.094	< 0.090

[7] Michigan.gov/pfasresponse/investigations/lakes-and-streams/huron-river

Lab Sample #		40234558005	40237220001	40237220002	40237220003
Sample Description[b]		Predator fish #1	Predator fish #2	Forage fish #1	Forage fish #2
Taxonomic Information		LM bass	*Lepomis spp.* (sunfish)	*Lepomis spp.*	*Micropterus spp.* (juvenile bass)
Analyte		Result in μg/kg (ppb) wet weight			
17	PFHpS	< 0.086	< 0.086	< 0.086	< 0.082
18	PFOS	**630**	**110**	**160**	**380**
19	PFNS	**0.93**	**0.17 (J)**	**0.21 (J)**	**0.62**
20	PFDS	**5.3**	**1.4**	**1.8**	**4.2**
21	PFDOS	< 0.11	< 0.11	< 0.11	< 0.11
22	PFOSA	**0.14 (J)**	**0.23 (J)**	**0.28**	**0.23 (J)**
23	EtFOSE	< 0.082	**0.088 (J)**	**0.14 (J)**	< 0.079
24	MeFOSE	< 0.095	< 0.094	< 0.095	< 0.091
25	EtFOSA	< 0.087	< 0.087	< 0.087	< 0.083
26	MeFOSA	< 0.067	< 0.067	< 0.067	< 0.064
27	EtFOSAA	< 0.093	< 0.093	**0.097 (J)**	**0.16 (J)**
28	MeFOSAA	**0.14 (I, J)**	< 0.092	**0.11 (J)**	**0.16 (I, J)**
29	4:2 FTS	< 0.078	< 0.078	< 0.078	< 0.075
30	6:2 FTS	< 0.12	**0.25**[c]	**1.3**	**0.13 (I, J)**
31	8:2 FTS	< 0.12	< 0.12	< 0.12	< 0.11
32	10:2 FTS	< 0.093	< 0.093	< 0.093	< 0.089
33	GenX	not analyzed			
34	ADONA	< 0.078	< 0.078	< 0.078	< 0.075
35	9Cl-PF3ONS	< 0.11	< 0.11	< 0.11	< 0.11
36	11Cl-PF3OUdS	< 0.096	< 0.095	< 0.096	< 0.092
Total PFAS[d]		**654.99**	**118.98**	**172.07**	**401.49**
Percent lipids (%)		3.44	1.97	1.75	0.88

[a] Detected concentrations above the reported LOQ appear in bold.

[b] See Table 3 for sampling dates and other information.

[c] This result should be considered estimated due to anomalously high recovery for corresponding SUR/EIS/IDS compound $^{13}C_2$_6:2 FTS (see Section 3.2.4 and Attachment B Table B.2).

[d] Results < [DL] were treated as 0 (zero) µg/kg in the Total PFAS summation in this table.

J = Estimated concentration below the LOQ but above the DL. See Appendix I for reported DLs and Appendix II for reported LOQs.

I = Interference present as evidenced by incorrect isotope ratios.

Table 9 contains results for PFAS analysis of benthos samples in Kent Lake. Nine to 16 PFAS compounds were detected in benthos sampled in Kent Lake. PFOS made up 38 to 60 percent of the total PFAS concentration in the benthos. The total PFAS concentration was, on average, 19 times higher in prey fish compared to benthos, due primarily to the relatively high concentrations of PFOS and PFDS measured in prey fish.

Table 9. Kent Lake Benthos PFAS Results[a]

Lab Sample #		40237220004	40238788038	40238788039	40238788040
Sample Description[b]		Invertebrate #1	Invertebrate #2	Invertebrate #3	Invertebrate #4
Taxonomic Information		Odonata (dragonflies/ damselflies)	Odonata	Amphipod (crustaceans)	Other taxa
Analyte		Result in µg/kg (ppb) wet weight			
1	PFBA	< 0.36	< 0.076	**0.11 (J)**	< 0.079
2	PFPeA	< 0.34	< 0.073	< 0.084	< 0.075
3	PFHxA	< 0.48	< 0.10	**0.23 (J)**	< 0.11
4	PFHpA	< 0.47	< 0.099	**0.15 (I, J)**	< 0.10
5	PFOA	< 0.31	**0.083 (J)**	**0.67**	**0.17 (J)**
6	PFNA	**0.42 (J)**	**0.26**	**1.4**	**0.48**
7	PFDA	**1.1**	**0.49**	**1.3**	**0.72**
8	PFUdA	**0.84 (J)**	**0.40**	**1.0**	**0.60**
9	PFDoA	**0.41 (J)**	**0.15 (J)**	**0.56**	**0.31**
10	PFTrDA	< 0.38	< 0.080	**0.19 (J)**	**0.097 (J)**
11	PFTeDA	< 0.42	< 0.089	< 0.10	< 0.091
12	PFHxDA	< 0.31	< 0.066	< 0.077	< 0.068
13	PFODA	< 0.39	< 0.083	< 0.096	< 0.086
14	PFBS	< 0.38	< 0.080	< 0.093	< 0.083
15	PFPeS	< 0.41	< 0.086	< 0.100	< 0.089
16	PFHxS	< 0.38	< 0.080	**0.18 (J)**	< 0.082
17	PFHpS	< 0.35	< 0.073	< 0.085	< 0.075
18	PFOS	**11**	**2.8**	**10**	**5.3**
19	PFNS	< 0.42	< 0.088	< 0.10	< 0.091
20	PFDS	**0.44 (J)**	**0.14 (J)**	**0.26**	**0.17 (J)**
21	PFDOS	< 0.45	< 0.095	< 0.11	< 0.098
22	PFOSA	< 0.42	**0.21**	**0.12 (J)**	< 0.091
23	EtFOSE	< 0.33	< 0.070	< 0.081	< 0.072
24	MeFOSE	**0.54 (J)**	< 0.080	< 0.093	< 0.083
25	EtFOSA	< 0.35	< 0.074	< 0.085	< 0.076
26	MeFOSA	< 0.27	< 0.057	< 0.066	< 0.059
27	EtFOSAA	**0.41 (I, J)**	**0.22 (I)**	**0.19 (J)**	**0.10 (J)**
28	MeFOSAA	< 0.37	**0.16 (J)**	**0.25**	**0.14 (J)**
29	4:2 FTS	< 0.31	< 0.066	< 0.077	< 0.068
30	6:2 FTS	**3.2 (I)[c]**	**1.6**	**10**	**2.4**

Lab Sample #		40237220004	40238788038	40238788039	40238788040
Sample Description[b]		Invertebrate #1	Invertebrate #2	Invertebrate #3	Invertebrate #4
Taxonomic Information		Odonata (dragonflies/ damselflies)	Odonata	Amphipod (crustaceans)	Other taxa
Analyte		Result in µg/kg (ppb) wet weight			
31	8:2 FTS	< 0.47	< 0.100	< 0.12	< 0.10
32	10:2 FTS	< 0.37	< 0.079	< 0.092	< 0.082
33	GenX	not analyzed			
34	ADONA	< 0.31	< 0.066	< 0.077	< 0.068
35	9Cl-PF3ONS	< 0.45	< 0.095	< 0.11	< 0.097
36	11Cl-PF3OUdS	< 0.38	< 0.081	< 0.094	< 0.084
Total PFAS[d]		**18.36**	**6.51**	**26.61**	**10.49**
Percent lipids (%)		NA[e]	NA[e]	NA[e]	NA[e]

[a] Detected concentrations above the reported LOQ appear in bold.

[b] See Table 3 for sampling dates and other information.

[c] This result should be considered estimated due to anomalously high recovery for corresponding SUR/EIS/IDS compound $^{13}C_2$_6:2 FTS (see Section 3.2.4 and Attachment B Table B.2).

[d] Results < [DL] were treated as 0 (zero) µg/kg in the Total PFAS summation in this table.

[e] Not available. Insufficient tissue available for lipid analysis.

J = Estimated concentration below the LOQ but above the DL. See Appendix I for reported DLs and Appendix II for reported LOQs.

I = Interference present as evidenced by incorrect isotope ratios.

Table 9 contains results for PFAS analysis of benthos samples in Kent Lake. Nine to 16 PFAS compounds were detected in benthos sampled in Kent Lake. PFOS made up 38 to 60 percent of the total PFAS concentration in the benthos. The total PFAS concentration was, on average, 19 times higher in prey fish compared to benthos, due primarily to the relatively high concentrations of PFOS and PFDS measured in prey fish.

Table 10. Kent Lake Benthos PFAS Results[a]

	Lab Sample #	40237220004	40238788038	40238788039	40238788040
	Sample Description[b]	Invertebrate #1	Invertebrate #2	Invertebrate #3	Invertebrate #4
	Taxonomic Information	Odonata (dragonflies/ damselflies)	Odonata	Amphipod (crustaceans)	Other taxa
Analyte		Result in µg/kg (ppb) wet weight			
1	PFBA	< 0.36	< 0.076	**0.11 (J)**	< 0.079
2	PFPeA	< 0.34	< 0.073	< 0.084	< 0.075
3	PFHxA	< 0.48	< 0.10	**0.23 (J)**	< 0.11
4	PFHpA	< 0.47	< 0.099	**0.15 (I, J)**	< 0.10
5	PFOA	< 0.31	**0.083 (J)**	**0.67**	**0.17 (J)**
6	PFNA	**0.42 (J)**	**0.26**	**1.4**	**0.48**
7	PFDA	**1.1**	**0.49**	**1.3**	**0.72**
8	PFUdA	**0.84 (J)**	**0.40**	**1.0**	**0.60**
9	PFDoA	**0.41 (J)**	**0.15 (J)**	**0.56**	**0.31**
10	PFTrDA	< 0.38	< 0.080	**0.19 (J)**	**0.097 (J)**
11	PFTeDA	< 0.42	< 0.089	< 0.10	< 0.091
12	PFHxDA	< 0.31	< 0.066	< 0.077	< 0.068
13	PFODA	< 0.39	< 0.083	< 0.096	< 0.086
14	PFBS	< 0.38	< 0.080	< 0.093	< 0.083
15	PFPeS	< 0.41	< 0.086	< 0.100	< 0.089
16	PFHxS	< 0.38	< 0.080	**0.18 (J)**	< 0.082
17	PFHpS	< 0.35	< 0.073	< 0.085	< 0.075
18	PFOS	**11**	**2.8**	**10**	**5.3**
19	PFNS	< 0.42	< 0.088	< 0.10	< 0.091
20	PFDS	**0.44 (J)**	**0.14 (J)**	**0.26**	**0.17 (J)**
21	PFDOS	< 0.45	< 0.095	< 0.11	< 0.098
22	PFOSA	< 0.42	**0.21**	**0.12 (J)**	< 0.091

	Lab Sample #	40237220004	40238788038	40238788039	40238788040
	Sample Description[b]	Invertebrate #1	Invertebrate #2	Invertebrate #3	Invertebrate #4
	Taxonomic Information	Odonata (dragonflies/ damselflies)	Odonata	Amphipod (crustaceans)	Other taxa
	Analyte	Result in µg/kg (ppb) wet weight			
23	EtFOSE	< 0.33	< 0.070	< 0.081	< 0.072
24	MeFOSE	**0.54 (J)**	< 0.080	< 0.093	< 0.083
25	EtFOSA	< 0.35	< 0.074	< 0.085	< 0.076
26	MeFOSA	< 0.27	< 0.057	< 0.066	< 0.059
27	EtFOSAA	**0.41 (I, J)**	**0.22 (I)**	**0.19 (J)**	**0.10 (J)**
28	MeFOSAA	< 0.37	**0.16 (J)**	**0.25**	**0.14 (J)**
29	4:2 FTS	< 0.31	< 0.066	< 0.077	< 0.068
30	6:2 FTS	**3.2 (I)[c]**	**1.6**	**10**	**2.4**
31	8:2 FTS	< 0.47	< 0.100	< 0.12	< 0.10
32	10:2 FTS	< 0.37	< 0.079	< 0.092	< 0.082
33	GenX	not analyzed			
34	ADONA	< 0.31	< 0.066	< 0.077	< 0.068
35	9Cl-PF3ONS	< 0.45	< 0.095	< 0.11	< 0.097
36	11Cl-PF3OUdS	< 0.38	< 0.081	< 0.094	< 0.084
	Total PFAS[d]	**18.36**	**6.51**	**26.61**	**10.49**
	Percent lipids (%)	NA[e]	NA[e]	NA[e]	NA[e]

[a] Detected concentrations above the reported LOQ appear in bold.

[b] See Table 3 for sampling dates and other information.

[c] This result should be considered estimated due to anomalously high recovery for corresponding SUR/EIS/IDS compound $^{13}C_2$_6:2 FTS (see Section 3.2.4 and Attachment B Table B.2).

[d] Results < [DL] were treated as 0 (zero) µg/kg in the Total PFAS summation in this table.

[e] Not available. Insufficient tissue available for lipid analysis.

J = Estimated concentration below the LOQ but above the DL. See Appendix I for reported DLs and Appendix II for reported LOQs.

I = Interference present as evidenced by incorrect isotope ratios.

Radar plots display the PFAS concentrations measured in Kent Lake fish (Figure 7) and Kent Lake invertebrates (Figure 8). The data plotted in these figures illustrates that:

- Within each biota type, PFAS concentrations and their distribution are very similar.
- Between Kent Lake fish and invertebrates, there are some similarities in the distribution of PFAS, although there are also differences. The differences may be due to detectability of some compounds in the invertebrate samples; low sample weight caused analytical reporting limits to be elevated, resulting in more non-detects.

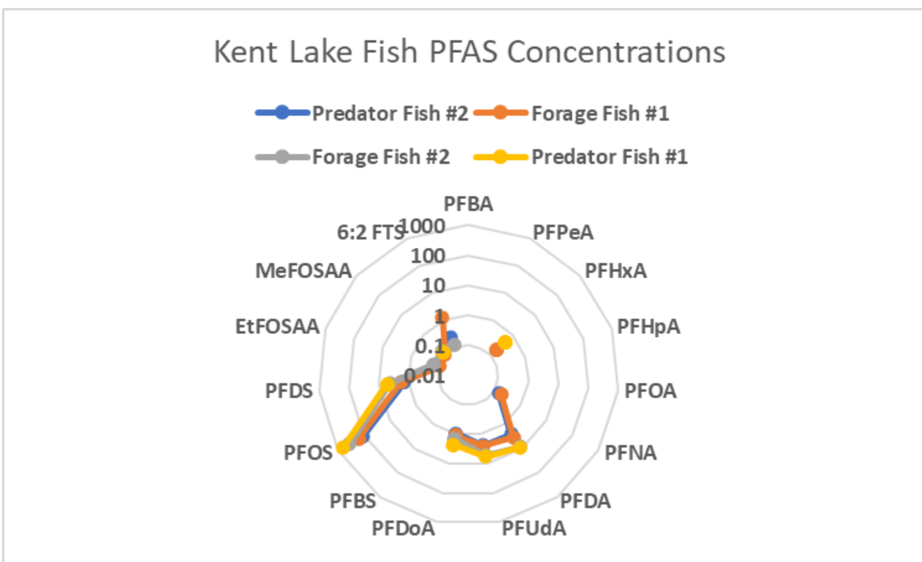


Figure 7. Radar plot of PFAS concentrations in µg/kg (ppb) wet weight in Kent Lake fish samples

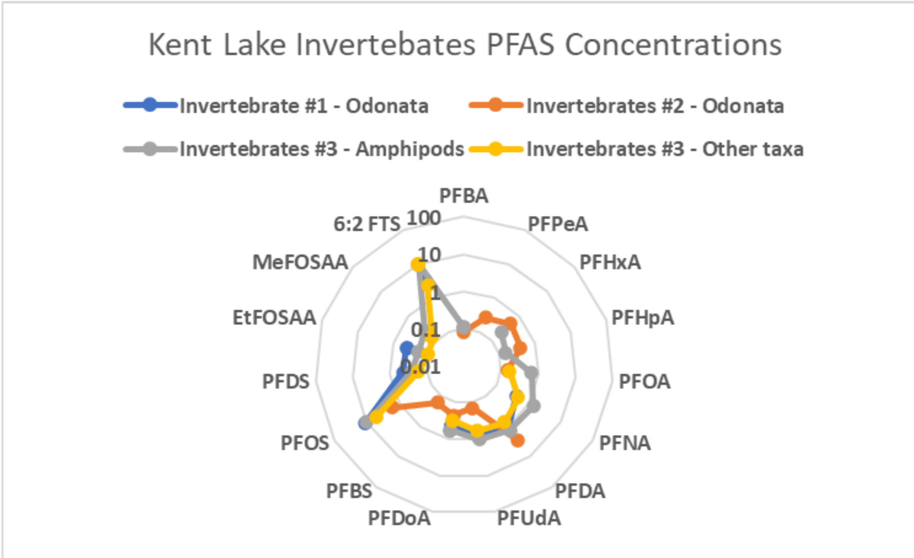


Figure 8. Radar plot of PFAS concentrations in µg/kg (ppb) wet weight in Kent Lake invertebrate samples

3.2.3 PFAS Concentrations in Proud Lake Biota

Table 10 summarizes the PFAS results for Proud Lake biota samples (predator fish, forage fish and benthos). Ten PFAS compounds were detected in predator and forage fish, and 5 were detected in both invertebrate samples. PFOS made up 50 to 67 percent of the total PFAS concentration in Proud Lake predator and forage fish, and 28 to 73 percent in benthos. Total PFAS was only slightly higher, on average, in predator fish versus prey fish, while total PFAS was higher in fish compared to invertebrates.

Table 11. Proud Lake Biota PFAS Results[a]

Lab Sample #		**40234558-001**	**40234558-002**	**40234558-003**	**40234558-004**	**40237220-005**	**40238788-037**
Sample Description[b]		**Predator fish #1**	**Predator fish #2**	**Forage fish #1**	**Forage fish #2**	**Inverte-brate #1**	**Inverte-brate #2**
Taxonomic Information		**LM bass**	**Pumpkinseed**	***Lepomis spp.***	***Micropterus spp.***	**Odonata**	**Odonata**
Analyte		**Result in µg/kg (ppb) wet weight**					
1	PFBA	< 0.089	< 0.085	< 0.087	< 0.086	< 0.35	< 0.090
2	PFPeA	< 0.085	< 0.081	< 0.083	< 0.082	< 0.33	< 0.086
3	PFHxA	**0.21 (I, J)**	**0.39 (I)**	**0.55 (I)**	**0.27 (I)**	< 0.47	< 0.12
4	PFHpA	< 0.12	< 0.11	< 0.11	< 0.11	< 0.45	< 0.12
5	PFOA	< 0.077	< 0.074	**0.08**	< 0.075	< 0.30	< 0.078
6	PFNA	**0.1 (J)**	0.28	**0.35**	**0.27**	**0.37 (J)**	**0.18 (J)**
7	PFDA	**8.7**	**2.5**	**4.1**	**8.6**	**1.2**	**0.35**
8	PFUdA	**6.7**	**1.8**	**2.7**	**5.0**	**0.73 (J)**	**0.34**
9	PFDoA	**4.2**	**1.1**	**1.7**	**2.7**	**0.50 (J)**	**0.27**
10	PFTrDA	**1.7**	**0.44**	**0.65**	**0.81**	< 0.36	< 0.094
11	PFTeDA	**1.5**	**0.46**	**0.63**	**0.64**	< 0.41	< 0.10
12	PFHxDA	**0.085 (J)**	< 0.074	< 0.076	< 0.075	< 0.30	< 0.078
13	PFODA	< 0.097	< 0.092	< 0.095	< 0.094	< 0.38	< 0.098
14	PFBS	< 0.093	< 0.089	< 0.092	< 0.090	< 0.37	< 0.095
15	PFPeS	< 0.10	< 0.096	< 0.098	< 0.097	< 0.40	< 0.10
16	PFHxS	< 0.093	< 0.088	< 0.091	< 0.090	< 0.36	< 0.094
17	PFHpS	< 0.085	< 0.081	< 0.084	< 0.083	< 0.34	< 0.086
18	PFOS	**48**	**11**	**11**	**25**	**7.4**	**0.44**
19	PFNS	< 0.10	< 0.098	< 0.10	< 0.099	< 0.40	< 0.10
20	PFDS	**0.73**	**0.23**	**0.15**	**0.27**	< 0.35	< 0.090
21	PFDOS	< 0.11	< 0.11	< 0.11	< 0.11	< 0.44	< 0.11
22	PFOSA	< 0.10	**0.11 (J)**	< 0.10	**0.1 (J)**	< 0.41	< 0.10
23	EtFOSE	< 0.081	< 0.077	0.091	< 0.079	< 0.32	< 0.082

Lab Sample #		**40234558-001**	**40234558-002**	**40234558-003**	**40234558-004**	**40237220-005**	**40238788-037**
Sample Description[b]		**Predator fish #1**	**Predator fish #2**	**Forage fish #1**	**Forage fish #2**	**Inverte-brate #1**	**Inverte-brate #2**
Taxonomic Information		**LM bass**	**Pumpkinseed**	***Lepomis spp.***	***Micropterus spp.***	**Odonata**	**Odonata**
Analyte		**Result in µg/kg (ppb) wet weight**					
24	MeFOSE	< 0.094	< 0.089	< 0.092	< 0.091	< 0.37	< 0.095
25	EtFOSA	< 0.086	< 0.082	< 0.084	< 0.083	< 0.34	< 0.087
26	MeFOSA	< 0.066	< 0.063	< 0.065	< 0.064	< 0.26	< 0.067
27	EtFOSAA	< 0.092	< 0.088	< 0.090	< 0.089	< 0.36	< 0.093
28	MeFOSAA	< 0.092	< 0.087	< 0.090	< 0.089	< 0.36	< 0.093
29	4:2 FTS	< 0.077	< 0.074	< 0.076	< 0.075	< 0.30	< 0.078
30	6:2 FTS	< 0.12	< 0.11	< 0.12	< 0.11	< 0.46	< 0.12
31	8:2 FTS	< 0.12	< 0.11	< 0.11	< 0.11	< 0.46	< 0.12
32	10:2 FTS	< 0.092	< 0.088	< 0.090	< 0.089	< 0.36	< 0.093
33	GenX	not analyzed-					
34	ADONA	< 0.077	< 0.074	< 0.076	< 0.075	< 0.30	< 0.078
35	9Cl-PF3ONS	< 0.11	< 0.11	< 0.11	< 0.11	< 0.43	< 0.11
36	11Cl-PF3OUdS	< 0.095	< 0.090	< 0.093	< 0.092	< 0.37	< 0.096
Total PFAS[c]		**71.93**	**18.31**	**22.00**	**43.66**	**10.20**	**1.58**
Percent lipids (%)		1.44	1.84	1.62	1.26	0.78	NA[d]

[a] Detected concentrations above the reported LOQ appear in bold.

[b] See Table 3 for sample collection dates and other information.

[c] Results < [DL] were treated as 0 (zero) µg/kg in the Total PFAS summation in this table.

[d] Not available. Insufficient tissue available for lipid analysis.

J = Estimated concentration below the LOQ but above the DL. See Appendix I for reported DLs and Appendix II for reported LOQs.

D = Result obtained from analysis of diluted sample.

I = Interference present as evidenced by incorrect isotope ratios.

Radar plots display the PFAS concentrations measured in Proud Lake fish (Figure 9), and Proud Lake invertebrates (Figure 10). The data plotted in these figures illustrates that:

- Within each biota type, PFAS concentrations and their distribution are very similar.
- Between Proud Lake fish and invertebrates, there are some similarities in the distribution of PFAS compounds, although there are also differences. Like Kent Lake, the differences may be due to detectability of some compounds in the invertebrate samples; low sample weight caused analytical reporting limits to be elevated, resulting in more non-detects.

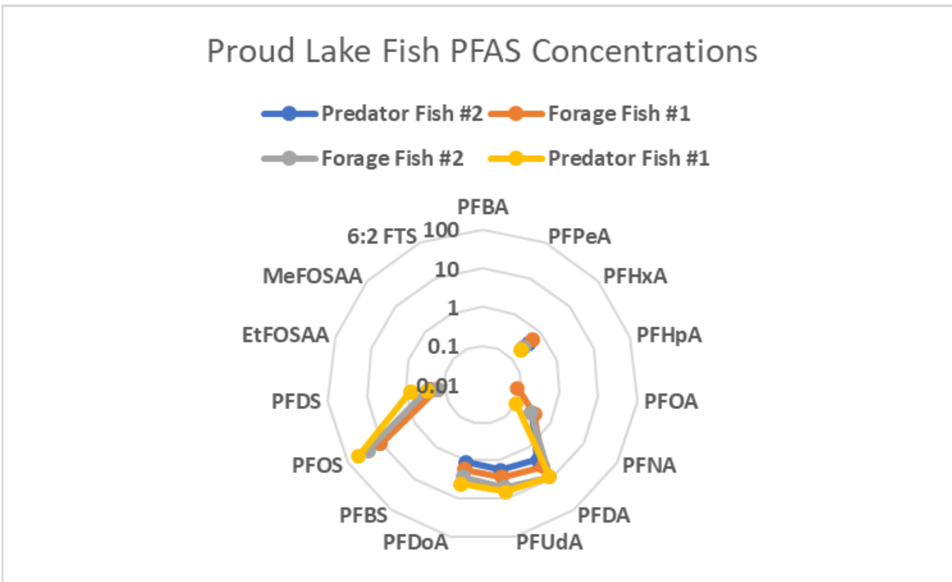


Figure 9. Radar plot of PFAS concentrations in µg/kg (ppb) wet weight in Proud Lake fish samples

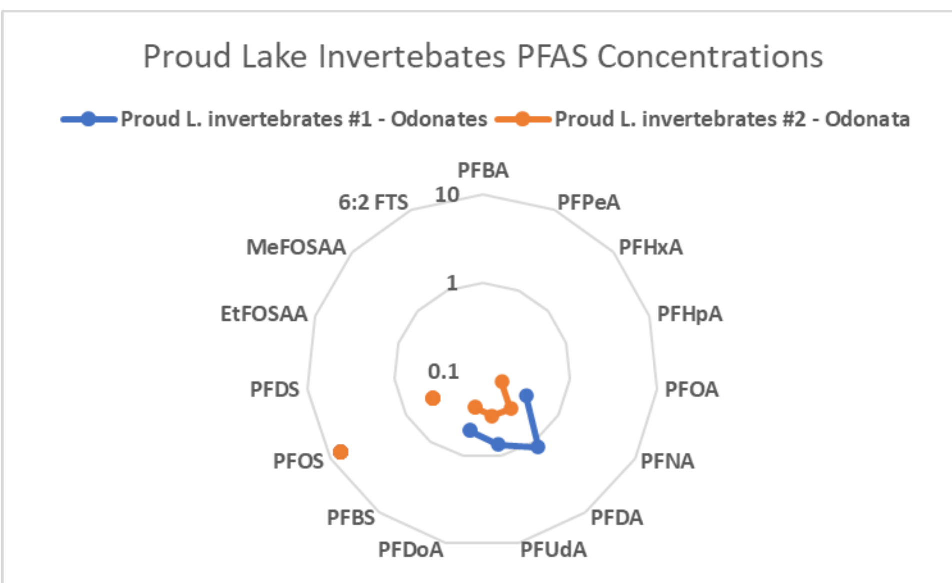


Figure 10. Radar plot of sample PFAS concentrations in µg/kg (ppb) wet weight in Proud Lake invertebrate samples

A comparison of PFAS concentrations in biota from Kent and Proud lakes is summarized below.

- Fish concentrations were higher in Kent Lake for: PFOS, PFNS, PFDS, PFOSA, MeFOSAA and 6:2 FTS.
- Fish concentrations were similar (based on overlapping concentration ranges in the two lakes) for PFHxA, PFNA, PFDA, PFUdA, PFDoA and PFTrDA.
- Fish concentrations of PFTeDA were higher in Proud Lake.
- Invertebrate concentrations were higher in Kent Lake for: PFOA, PFDS, EtFOSAA, MeFoSAA and 6:2 FTS.
- Invertebrate concentrations were similar for PFNA, PFDA, PFUdA, PFDoA and PFOS.
- PFDA, PFUdA and PFDoA were detected in fish and inverebrates although they were not detected in water or sediment.

In evaluating these findings, it is important to recognize that movement of fish between lakes is possible.

3.2.4 Quality Control Summary for PFAS

QC results from the analysis of PFAS in water and sediment samples, and biota samples, are summarized in Attachment B Tables B.1 and B.2, respectively, and the details are included in Appendix II. Laboratory method blank samples were free of the analytes at the RLs, indicating that sample processing procedures did not contribute contamination. The recovery of analytes in laboratory control samples, prepared by fortifying clean water or reference matrix material, passed the criteria indicating that sample extraction performed as expected. All other QC results for PFAS analyses were within acceptance limits, with the following exceptions:

- In water samples: surrogate recovery for $^{13}C_2$_6:2 FTS in a lab duplicate sample, surrogate recovery for $^{13}C_2$_4:2 FTS and $^{13}C_2$_6:2 FTS in a matrix spike sample, and relative percent difference (RPD) for analyte PFPeA in a lab duplicate sample were not within acceptance limits. These QC failures did not affect the reported results for the investigative samples.
- Percent recoveries for several target analytes were elevated or diminished in the matrix spike samples analyzed in two of the three biota sample batches. These deviations may be due to the presence of the affected analytes in the sample material, and/or sample inhomogeneity. For instance, zero percent recovery of PFOS in 40234558002-MSD and 40237220003-MS can be attributed to the presence of the analyte in the unspiked samples at concentrations orders of magnitude higher than the fortified concentration.
- With the exception of $^{13}C_2$_PFDA in biota sample #40237220001, the four injection internal standards passed the criteria. Acceptable recovery of injection internal standards provides verification that the instrument detector was working as expected. The laboratory was confident that the instrument detector was working as expected during the analysis of all samples for PFAS.
- There was elevated/diminished recovery of some SUR/EIS/IDS compounds in the biota samples. The use of the isotope dilution method generally precludes any adverse impact on those individual native compounds that have a directly

associated standard. However, in several cases, percent recoveries of labelled FTS SUR/EIS/IDS compounds were anomalously high, and were adversely impacted by matrix. In these cases, the results for the associated native compounds should be considered estimated, as footnoted in Tables 9 and 10.

3.2.5 PFAS Concentrations in Kent Lake and Proud Lake Ecosystems (graphical analysis)

Median concentrations of PFAS detected in water, sediment and biota in Kent Lake and Proud Lake are displayed as composite radar plots in Figures 11 (for Kent Lake) and 12 (Proud Lake). The median concentrations of each PFAS compound were calculated for each lake and sample matrix (sediment, water, fish or invertebrates). Non-detect concentrations were replaced with ½ the reported DL to calculate medians.

Although PFOS was the PFAS compound present at the highest concentrations in sediment and biota in Kent Lake (Figure 11), a number of other PFAS compounds were also detected in multiple sediment and/or biota samples, including PFHxA, PFHpA, PFDA, PFUdA, PFDoA, PFDS and 6:2 FTS. No PFAS compounds were detected in Proud Lake sediments, although PFOS and a number of other PFAS compounds (including PFHxA, PFNA, PFDA, PFUdA and PFDoA) were detected in multiple Proud Lake biota samples (Figure 12). The patterns of PFAS distribution in sediments and biota appear similar in Kent Lake, suggesting that PFAS in the sediment may act as a source of contamination to biota. On the other hand, the patterns of PFAS distribution in water in both lakes are different than the patterns of PFAS distribution in biota.

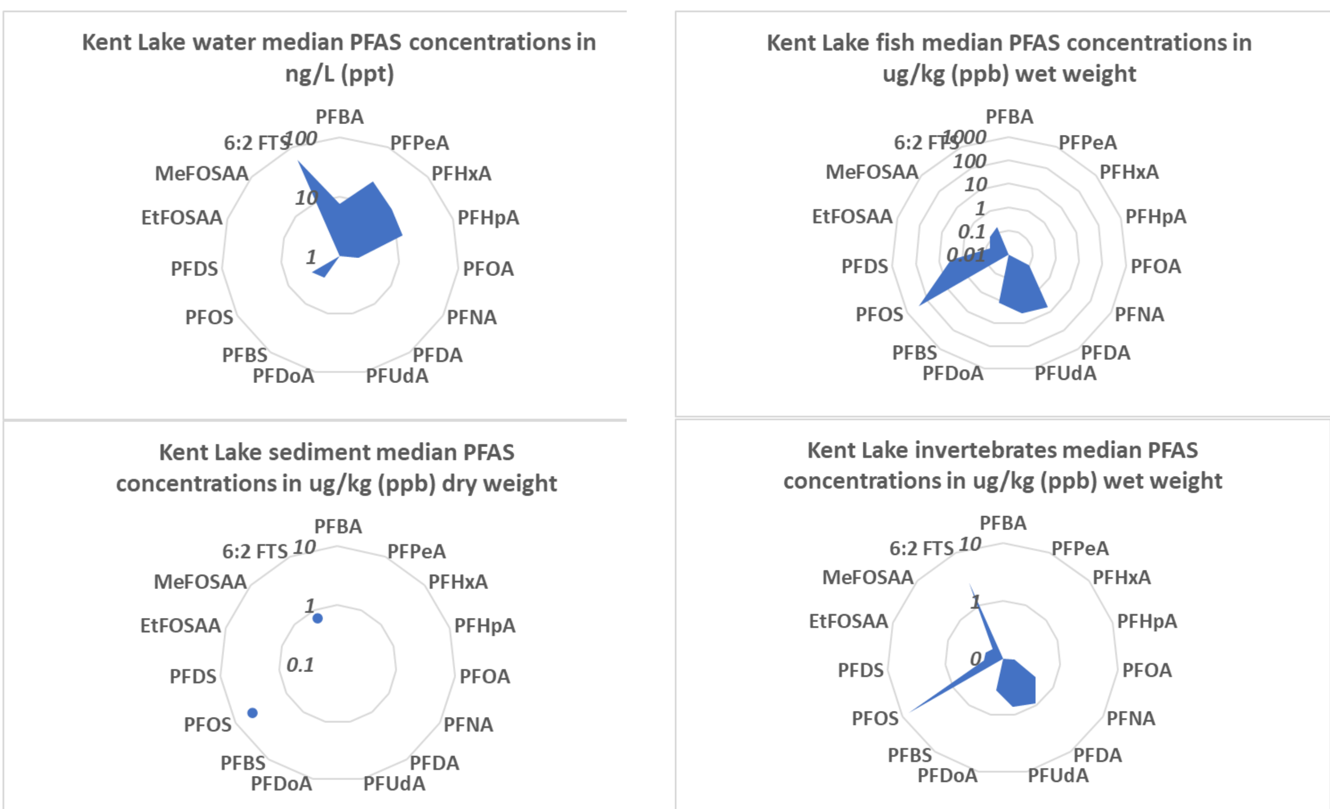
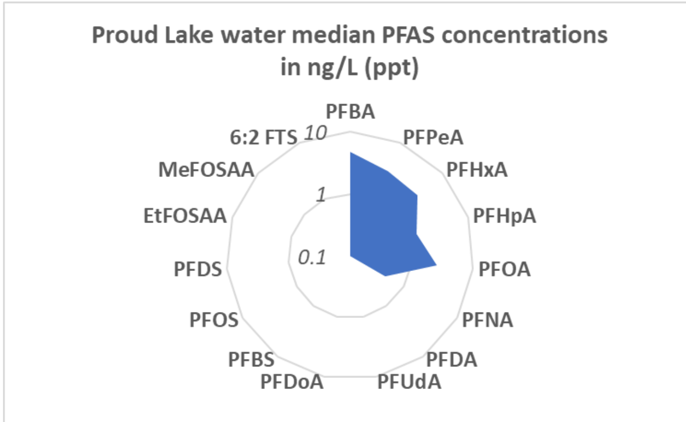
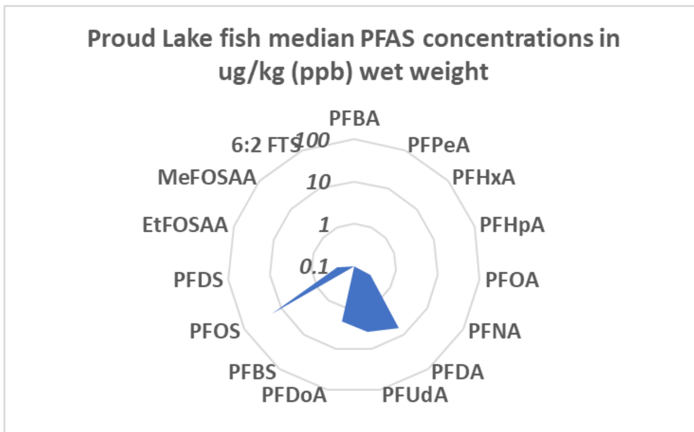


Figure 11. Median concentrations of PFAS compounds detected in water, sediment, fish, and invertebrates collected from Kent Lake.





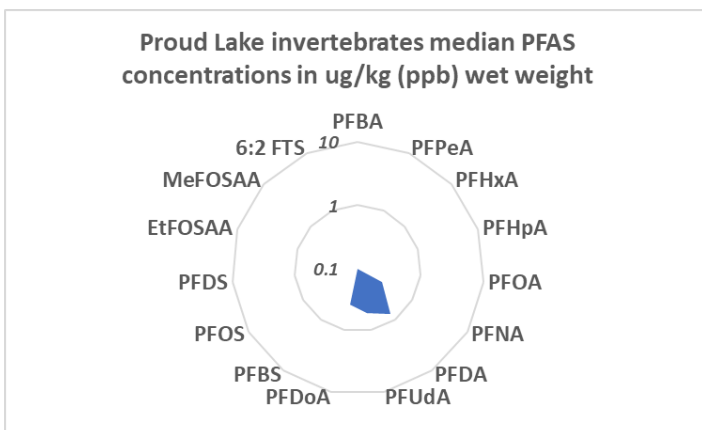


Figure 12. Median concentrations of PFAS compounds detected in water, fish and invertebrates collected from Proud Lake (Note: PFAS were not detected in Proud Lake sediment samples.)

Median concentrations of PFAS detected in water, sediment and/or biota in Kent Lake and Proud Lake are also plotted as bar graphs in Figures 13a, 13b and 14[8]. The median concentrations of each PFAS compound were calculated for each lake and sample matrix (sediment, water, fish and invertebrates). Non-detect concentrations were replaced with ½ the reported DL to calculate medians. The median PFAS concentrations in Kent Lake are presented in two graphs (Figures 13a and 13b) due to the number of PFAS compounds (n=21) detected: a) perfluoroalkyl carboxylic acids (PFCAs) in Figure 13a, and b) perfluoroalkane sulfonic acids (PFSAs), perfluoroalkane sulfomides and derivatives (FASAs), and fluorotelomer sulfonic acids (FTSAs) in Figure 13b. Although PFOS was the PFAS compound detected at the highest concentrations in sediment and biota in Kent Lake, a number of other PFAS compounds were also detected in multiple sediment and/or biota samples, including PFHxA, PFHpA, PFDA, PFUdA, PFDoA, PFDS and 6:2 FTS. No PFAS were detected in Proud Lake sediments, although PFOS and a number of other PFAS, including PFHxA, PFNA, PFDA, PFUdA and PFDoA, were detected in multiple Proud Lake biota samples (Figure 14).

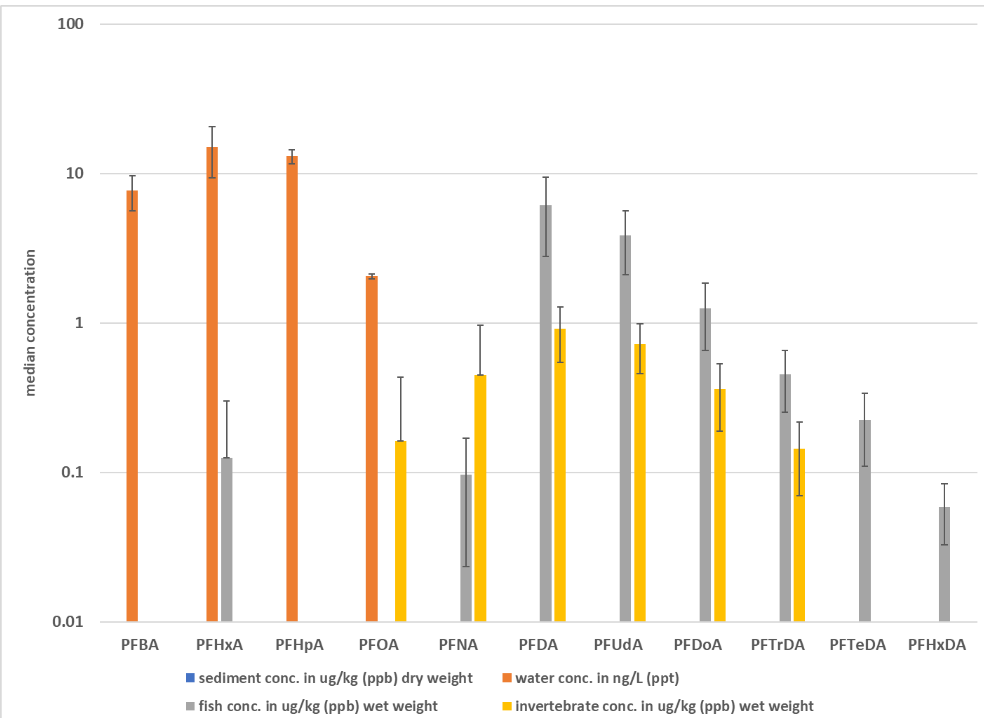


Figure 13(a). Median concentrations of PFCA compounds detected in water, sediment, fish and invertebrates collected from Kent Lake

[8] For clarity in these figures, concentrations of PFAS compounds detected in water only (for example, PFPeA, PFODA and PFBS) were omitted. In addition, the median (± 1 standard deviation) concentration for a compound was plotted only if it was detected in at least half the samples of a particular matrix (sediment, water, fish or invertebrates).

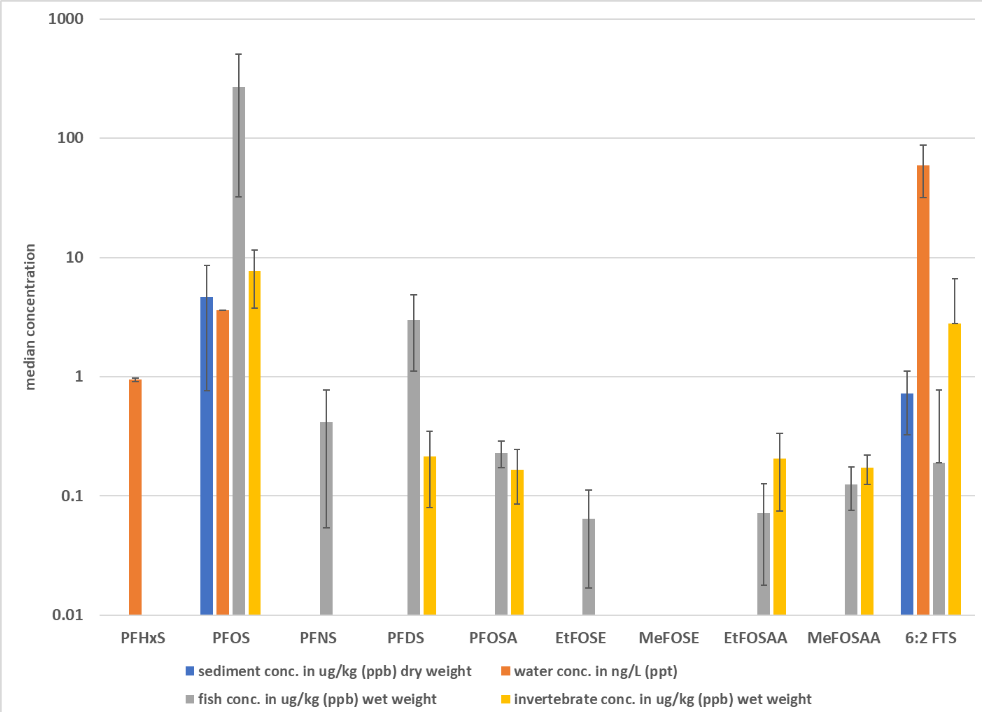


Figure 13(b) Median concentrations of PFSA, FASA and FTSA compounds detected in water, sediment fish and invertebrates in Kent Lake

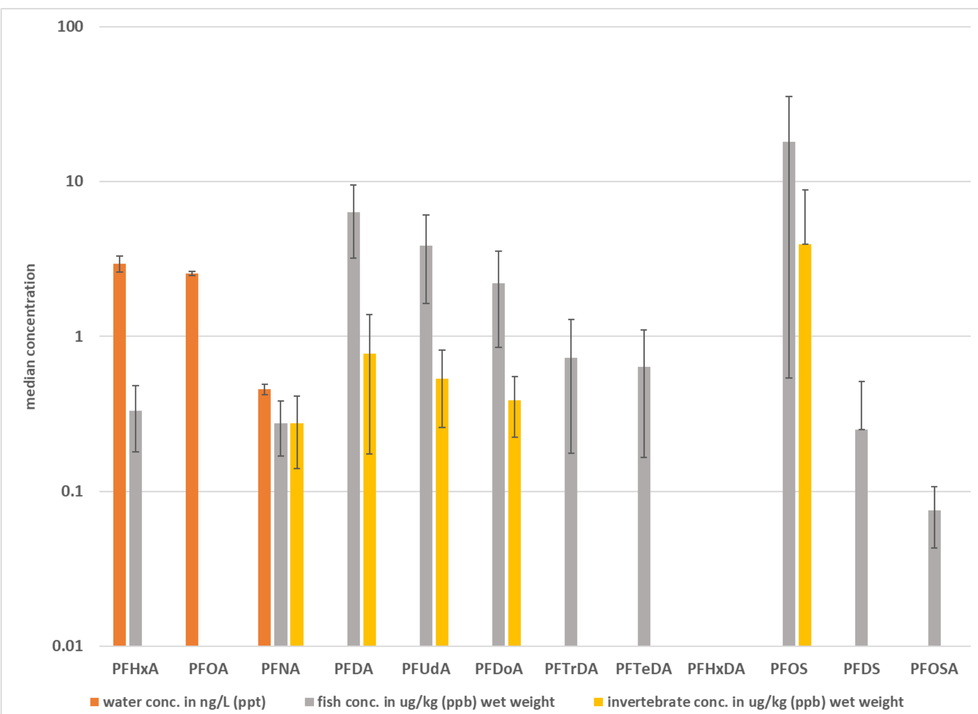


Figure 14. Median concentrations of PFAS compounds detected in water, fish and invertebrates collected from Proud Lake (Note: PFAS were not detected in Proud Lake sediment samples)

3.3 Other Parameters in Sediment Samples

Results for elements, PAHs and physical/aggregate parameters measured in Proud Lake and Kent Lake sediment samples are summarized in Table 11 and in Appendix I. Detailed analytical reports, including QC results, are provided in Appendix II.

All of the elements were detected in Proud Lake and Kent Lake sediments, except for cadmium, selenium, silver and (in Proud Lake) mercury. For the elements that were detected in samples from both lakes, sediment concentrations were within a factor of two except for chromium and nickel, both of which were 2.4 to 2.8 times higher in Kent Lake sediment.

Seven PAHs were detected in Proud Lake sediment, but none were detected in sediment from Kent Lake.

The Kent Lake sediment collected during the first sampling event was predominantly silt and fine sand (USDA/NRCS soil texture classification[9]: sandy loam) with a fraction organic carbon (f_{oc})[10] of 13 percent and cation exchange capacity (CEC) of 110 meq/100 g. In comparison, the Kent Lake sediment collected during the second sampling event was both coarser (predominantly fine sand; USDA/NRCS soil texture classification: sand) and much lower in organic carbon (f_{oc} of 1%) and CEC (4.8 meq/100 g). The differences between these properties of the two Kent Lake sediment samples appears to be correlated with the differences in PFOS concentrations in these samples; this is discussed further in Section 4.1. Proud Lake sediment samples were predominantly silt and fine sand (USDA/NRCS soil texture classification: silt loam (SE1) and loam (SE2)), with a f_{oc} of 6 to 9 percent and CEC of 26 to 48 meq/100 g.

Table 12. Kent Lake and Proud Lake Sediment Results for Elements, PAHs and Physical/Aggregate Properties

		Kent Lake		Proud Lake	
		SE1	SE1	SE1	SE2
		40234540004	40234540002	40234540002	40237648002
Parameter	CAS #	Result			
Elements in mg/kg (ppm) dry weight					
Arsenic	7440-38-2	16.8 (J)		14.7	
Barium	7440-39-3	152		238	
Cadmium	7440-43-9	<1.1		<0.71	

[9] https://stormwater.pca.state.mn.us/index.php?title=File:Soil_texture_triangle.jpg

[10] f_{oc} was calculated as [TOC (mg/kg)] ÷ (10^6 mg/kg)

		Kent Lake		Proud Lake	
		SE1	SE1	SE1	SE2
		40234540004	40234540002	40234540002	40237648002
Parameter	CAS #	Result			
Calcium	7440-70-2	76,500	28,100	232,000	180,000
Chromium	7440-47-3	14.4		5.9	
Copper	7440-50-8	27.4		20.9	
Iron	7439-89-6	17,900		11,500	
Lead	7439-92-1	29.4		21.8	
Magnesium	7439-95-4	3,880	2,150	5,220	2,910
Manganese	7439-96-5	687		1,040	
Mercury	7439-97-6	0.13 (J)		<0.053	
Nickel	7440-02-0	14.3		5.2 (J)	
Selenium	7782-49-2	<10.3		<7.0	
Silver	7440-22-4	<2.4		<1.6	
Zinc	7440-66-6	59.9		56.9	
PAHs in µg/kg (ppb) dry weight					
2-Methylnaphthalene	91-57-6	<60.9		48.2 (J)	
Acenaphthene	83-32-9	<54.0		<35.3	

		Kent Lake		Proud Lake	
		SE1	SE1	SE1	SE2
		40234540004	40234540002	40234540002	40237648002
Parameter	CAS #	Result			
Acenaphthylene	208-96-8	<52.5		<34.3	
Anthracene	120-12-7	<51.7		<33.8	
Benzo(a)anthracene	56-55-3	<53.8		<35.2	
Benzo(a)pyrene	50-32-8	<47.3		<30.9	
Benzo(b)fluoranthene	205-99-2	<57.8		65.6 (J)	
Benzo(g,h,i)perylene	191-24-2	<73.1		<47.8	
Benzo(k)fluoranthene	207-08-9	<53.2		36.4 (J)	
Chrysene	218-01-9	<78.5		81.1 (J)	
Dibenz(a,h)anthracene	53-70-3	<57.6		<37.7	
Fluoranthene	206-44-0	<49.3		110 (J)	
Fluorene	86-73-7	<49.9		<32.6	
Indeno(1,2,3-cd)pyrene	193-39-5	<86.7		<56.7	
Naphthalene	91-20-3	<40.6		<26.5	
Phenanthrene	85-01-8	<47.7		42.1 (J)	
Pyrene	129-00-0	<61.2		79.9 (J)	
Physical and Aggregate Properties					
Percent Solids (%)		12.1	61	18.4	15.1
pH at 25°C (S.U.)		7.4	7.4	7.5	6.9
Total Organic Carbon (mg/kg dry weight)	7440-44-0	128,000	9,870	87,300	60,900
Cation Exchange Capacity (meq/100g)		110	4.8	48.1	26.4
Grain Size Fractional Components (%)					
+3"		0.0	0.0	0.0	0.0

		Kent Lake		Proud Lake	
		SE1	SE1	SE1	SE2
		40234540004	40234540002	40234540002	40237648002
Parameter	CAS #	Result			
gravel - coarse		0.0	0.0	0.0	0.0
gravel - fine		0.0	3.0	0.0	0.1
sand - coarse		0.0	4.4	0.0	0.4
sand - medium		14.2	15.4	4.4	5.0
sand - fine		36.0	69.1	28.7	35.2
fines - silt		43.0	7.7	57.4	48.4
fines - clay		6.8	0.4	9.5	10.9

J = Estimated concentration below the reported LOQ but above the reported DL.
Results reported as < are non-detect above the reported DL.

3.4 PCBs in Sediment

Results from PCB analysis of Proud Lake and Kent Lake sediment samples are summarized in Table 12; congener-specific results are tabulated in Attachment C Table C.1. QC results are summarized in a bulleted list in Attachment B. The total PCB concentration in the Kent Lake sediment sample was 22 times higher than in the Proud Lake sediment sample. A total of 131 congeners and coeluting congeners were detected in the Kent Lake sediment sample, while 90 were detected in the Proud Lake sediment sample. As noted in Table 12, the total PCB concentration in Kent Lake (269 µg/kg) exceeds the Threshold Effect Concentrations (TECs) of 60 µg/kg (MacDonald et al. 2000) and 34 µg/kg (Wisconsin DNR. 2003). This is discussed further in Section 4.10.

Table 13. Kent Lake and Proud Lake Sediment PCB Results in µg/kg (ppb) dry weight

Sample Location	Kent Lake	Proud Lake
Lab Sample #	40234540004	40234540002
Number of PCB congeners/co-eluting congeners detected	90	131
Total PCB concentration	269*	12.4

* Exceeds the TEC of 60 µg/kg (ppb) (MacDonald et al., 2000) and 34 µg/kg (ppb) (Wisconsin DNR, 2003).

4.0 DISCUSSION

4.1 PFOS in Kent Lake and Proud Lake Sediment

The PFOS concentrations measured in sediment samples from Kent Lake were approximately four times higher in the first sampling event than in the second (Table 7). This difference is likely related to the differences in physical properties such as the proportion of fine-grained sediment, as well as TOC content, calcium and magnesium concentrations, and CEC. These sediment properties, which have been suggested in the literature to correlate positively with concentrations of PFOS in sediment, were all higher in the SE1 sediment sample. Researchers have noted that sorption of PFOS and other perfluorochemical surfactants (e.g., perfluorocarboxylates, perfluorosulfonates, and perfluorooctyl sulfonamide acetic acids) is influenced by both sediment-specific and solution-specific parameters (Higgins et al. 2006; Wang et al. 2013). Sediment TOC was the dominant sediment parameter affecting sorption, indicating the importance of hydrophobic interactions (Higgins et al. 2006). However, sorption also increased with increasing calcium in solution and decreasing pH, suggesting that electrostatic interactions play a role. Wang et al. (2013) investigated the distribution of PFOS in water and sediment samples from the Yellow River Estuary (China) and found that the distribution coefficient (K_d) of PFOS was significantly and positively correlated to the TOC and clay content of the sediment. The differences in these sediment properties between the Kent Lake sediment samples from SE1 and SE2 appear to explain the significant difference in measured PFOS concentrations. Other factors, such as differences between the spatial locations of the SE1 and SE2 sediment sample collection should also be considered.

4.2 PFOS and 6:2 FTS Partition Coefficients

Sediment-water partition coefficients (K_p) and organic carbon partition coefficients (K_{oc}) were calculated for the two PFAS compounds measured in Kent Lake sediment, PFOS and 6:2 FTS, using median water concentrations and concentrations measured in individual sediment samples, and log-transformed[11] (Table 13). K_p and K_{oc} were approximately two orders of magnitude (i.e., 2 log units) higher for PFOS than for 6:2 FTS[12]. Table 13 also includes K_p and K_{oc} values reported by Szabo et al. (2022) for PFOS measured in an urban lake in Melbourne, Australia. The median partition coefficients for PFOS in Kent Lake were at least an order of magnitude (1.1 – 1.7 log units) greater than the partition coefficients measured by Szabo et al (2022). No K_p or K_{oc} values could be found in the literature for 6:2 FTS.

[11] It is customary to log-transform contaminant ratios that vary widely in environmental matrices. This includes the contaminant ratios K_p, K_{oc}, and BAF. This convention is followed in this report.

[12] Note that the concentration of 6:2 FTS measured in the sediment collected during SE1 was < reported DL, and ½ reported DL was used as a replacement value to calculate the median concentration. Therefore, K_p and K_{oc} are presented as less than (“<”) values.

Table 14. Log-transformed sediment-water partition coefficients (K_p) and organic carbon partition coefficients (K_{oc}) for PFOS and 6:2 FTS measured in water and sediment samples collected from Kent Lake

PFAS	log K_p (L/kg)			log K_{oc} (L/kg)			Szabo et al. (2022)	
	SE1	SE2	Median	SE1	SE2	Median	log K_p	log K_{oc}
PFOS	3.31	2.72	3.02	4.21	4.73	4.47	1.97	2.75
6:2 FTS	<1.53	0.87	0.98	<2.42	2.87	2.43	-	-

The K_p and K_{oc} measured in Kent Lake for PFOS was compared to values measured by GLEC in surface water and contaminated sediments collected from six sites throughout Michigan in the fall of 2021 for Goal 1A of this study (Table 14). PFOS partition coefficients in Kent Lake (Table 13) were higher than the median values for the six Michigan sites (Table 14), although there was some overlap in the partition coefficients between individual sites. The median log K_p measured in Kent Lake was 0.4 log units greater than the median values for the six Michigan sites, and the median log K_{oc} measured in Kent Lake was 1.1 log units greater than the median values for the six sites. Regardless of the differences between sites, the partition coefficients calculated from our data show that both 6:2 FTS and especially PFOS have a strong tendency to partition from the water column into sediment.

Table 15. Log-transformed K_p and K_{oc} for PFOS measured in surface water and contaminated sediments collected from six sites in Michigan for Goal 1A

Partition coefficient	Fort Gratiot	Clark's Marsh	Huron Norton Cr.	Rogue R.	Beaver Dam Pond	Pigeon R.	Median of 6 sites
log K_p	1.6	2.5	3.3	2.6	2.2	3.2	2.6
log K_{oc}	2.9	2.8	4.0	3.6	3.1	4.2	3.4

4.3 PFAS Bioaccumulation Factors

Bioaccumulation factors (BAFs) were calculated for PFAS compounds in both lakes, using median water concentrations and concentrations measured in individual biota samples. In Kent Lake, BAFs were calculated for seven PFAS compounds (Table 15 and Figure 15). BAFs for PFOS in Kent Lake biota were one to four orders of magnitude higher compared to other PFAS, especially in fish. BAFs for PFOS were higher in forage and predator fish compared to benthos, suggesting a tendency for this compound to biomagnify (see also Section 4.7). The opposite trend was observed for 6:2 FTS (i.e., higher concentrations in invertebrates compared to fish), the only other PFAS for which BAFs could be calculated in different trophic level organisms sampled from Kent Lake. 6:2 FTS has been reported to bioaccumulate to a lesser extent than PFOS in both terrestrial and aquatic ecosystems (Ali et al., 2021; Semerád et al., 2022; Zhi et al., 2022).

Table 16. Log BAFs Calculated for Biota Samples Collected from Kent Lake

Sample Description	Predator fish #1	Predator fish #2	Forage fish #1	Forage fish #2	Inverte-brate #1	Inverte-brate #2	Inverte-brate #3	Inverte-brate #4
Taxon. Info.	LM bass	*Lepomis spp.* (sunfish)	*Lepomis spp.*	*Micropterus spp.* (juvenile bass)	Odonata (dragonflies/ damselflies)	Odonata	Amphipoda (crustaceans)	Other taxa
PFAS	**log BAF (L/kg)**							
PFBA							1.16	
PFHxA	1.46		1.10				1.19	
PFHpA							1.06	
PFOA						1.61	2.51	1.92
PFHxS							2.28	
PFOS	5.24	4.49	4.65	5.02	3.49	2.89	3.44	3.17
6:2 FTS		0.62	1.34	0.34	1.73	1.43	2.23	1.61

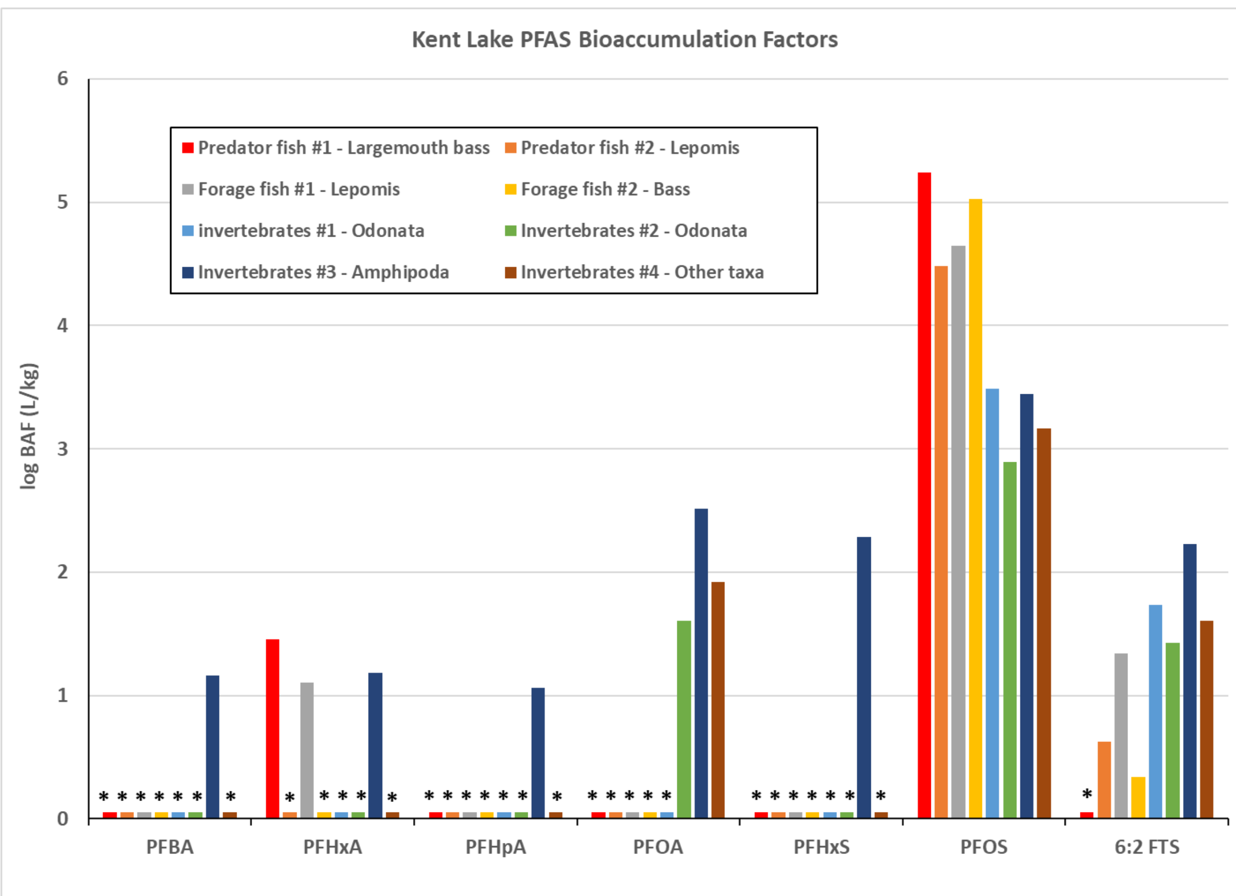


Figure 15. BAFs for PFAS detected in water and biota collected from Kent Lake (Note: asterisk above bar indicates no BAF could be calculated for that compound/organism)

The bioaccumulation factors presented above and the partition coefficients presented in the previous section indicate that PFOS behaves as a persistent, bioaccumulative substance in this aquatic system. The elevated concentration ratios for PFOS (including K_p, K_{oc} and BAF) in Kent Lake may reflect the relatively slower rate of decline in sediment and biota concentrations compared to the concentration in lake water. This is discussed further in Section 4.9.

In Proud Lake, BAFs were calculated for two PFAS compounds, PFHxA and PFNA (Table 16 and Figure 16). BAFs for PFOS could not be calculated for biota collected from Proud Lake because the water concentrations were below the reported DL. However, using a PFOS water concentration of ½ the reported DL results in estimated log BAFs of 4.11 to 4.75 L/kg in predator fish, 4.11 to 4.47 L/kg in forage fish, and 2.71 to 3.94 L/kg in benthos. The agreement between these BAF estimates for PFOS in Proud Lake and those measured in Kent Lake is reasonable considering the assumption applied to estimate the PFOS water concentration in Proud Lake (i.e., non-detect results were estimated as ½ the reported DL).

Table 17. BAFs Calculated for Biota Sample Collected from Proud Lake

Sample Description	Predator fish #1	Predator fish #2	Forage fish #1	Forage fish #2	Inverte-brate #1	Inverte-brate #2
Taxonomic Information	**LM bass**	**Pumpkin-seed**	***Lepomis spp.***	***Micropterus spp.***	**Odonata**	**Odonata**
PFAS	**Log BAF (L/kg)**					
PFHxA	1.85	2.12	2.27	1.96		
PFNA	2.34	2.79	2.89	2.77	2.91	2.60

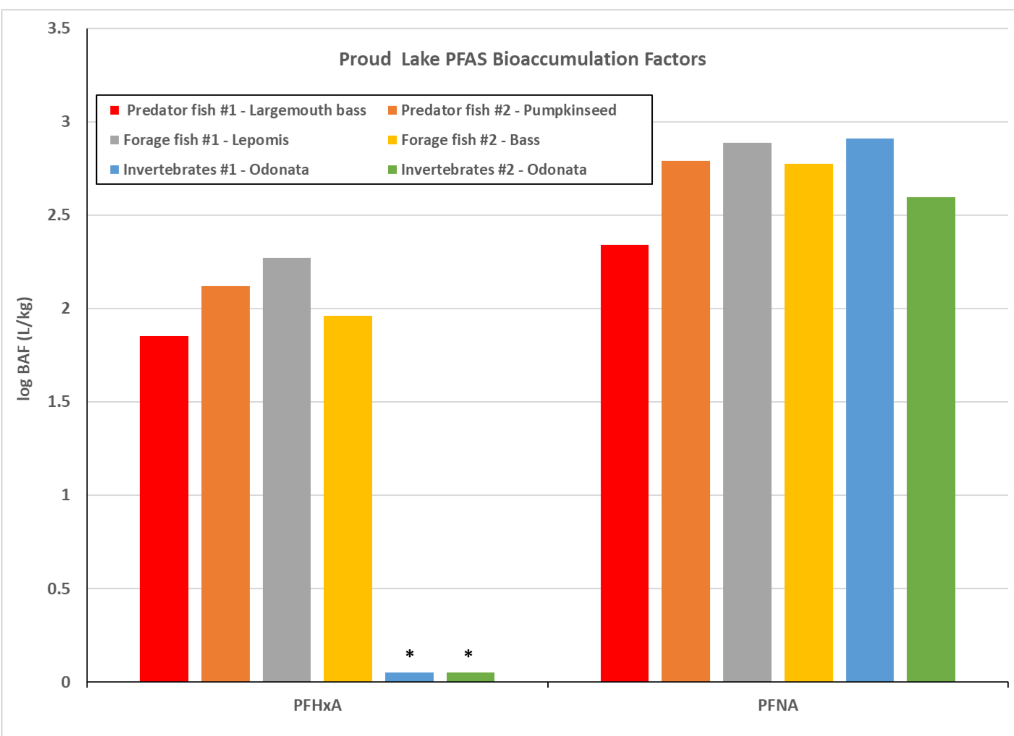


Figure 16. BAFs for PFAS detected in water and biota collected from Proud Lake (Note: asterisk above bar indicates no BAF could be calculated for that compound/organism)

4.4 PFAS BAFs Compared to Data in Literature

The BAFs calculated from PFAS concentrations measured in Kent and Proud Lakes were compared to BAFs compiled from the published literature and summarized by Burkhard (2021). The comparison, summarized in Table 17, shows that the average log BAFs measured in Kent and Proud Lake benthos for PFHxA, PFHpA, PFOA, PFNA, PFHxS and PFOS fall within one standard deviation of the whole-body log BAFs for Malacostraca[13] calculated by Burkhard (2021) for those compounds. For the same compounds, except PFOS, average log BAFs measured in Kent and Proud Lake fish also fall within one standard deviation of the whole-body log BAFs for freshwater ray-finned fish[14] calculated by Burkhard (2021). For fish in Kent Lake, the average log BAF for PFOS was 1.2 log unit (16 times) higher than Burkhard's corresponding average for freshwater ray-finned fish. This exceeded Burkhard's corresponding average by more than one standard deviation and is greater than the 90th percentile of the BAFs compiled by that author for PFOS in freshwater ray-finned fish. This places the Kent Lake fish average PFOS BAF among the high outlying BAFs, according to Burkhard. As was noted above, this may be related to the relatively slower rate of decline in sediment and biota PFOS concentrations compared to the concentration in lake water. For 6:2 FTS, the median log BAF for fish in Kent Lake (0.62 L/kg) was lower than Burkhard's median log BCF for freshwater ray-finned fish (1.54 L/kg) but higher than the lowest log BCF value (0.48 L/kg).

Table 18. Comparison of log BAFs (L/kg wet weight) measured in Kent and Proud Lakes to values summarized by Burkhard (2021)

	This Study			Burkhard (2021) Summary log BAF: average (SD, n)	
PFAS	Lake	Biota type	log BAF: average (SD, n)	Freshwater Malacostraca[a]	Freshwater Teleostei[b]
PFBA	Kent	Benthos	1.16 (NA, 1)		
PFHxA	Kent	Fish	1.28 (0.25, 2)	1.84 (1.02, 6)	1.10 (1.53, 11)
	Proud		2.05 (0.18, 4)		
	Kent	Benthos	1.19 (NA, 1)		
PFHpA	Kent	Benthos	1.06 (NA, 1)	1.61 (0.62, 5)	1.69 (1.54, 6)
PFOA	Kent	Benthos	2.01 (0.46, 3)	1.93 (0.95, 13)	2.20 (0.86, 39)
PFNA	Proud	Fish	2.70 (0.24, 4)	3.17 (0.54, 10)	2.89 (1.23, 36)
		Benthos	2.75 (0.22,2)		
PFHxS	Kent	Benthos	2.28 (NA, 1)	1.76 (0.48, 7)	2.10 (0.77, 20)

[13] At least one of the invertebrate samples collected from Kent Lake was identified as amphipods, which are members of the class Malacostraca.

[14] All fish collected in Kent and Proud Lakes for analysis of PFAS for this study were freshwater ray-finned fishes.

PFOS	Kent	Fish	4.85 (0.35, 4)	3.32 (0.66, 22)	3.65 (0.85, 70)
		Benthos	3.25 (0.28, 4)		
6:2 FTS	Kent	Fish	0.77 (0.52, 3)		1.54 (0.62, 3)[c]
		Benthos	1.75 (0.34, 4)		

[a] The class Malacostraca includes marine, freshwater, and terrestrial crustaceans. Familiar members of the Malacostraca are the stomatopods (mantis shrimp) and euphausiids (krill), as well as the amphipods.

[b] Ray-finned fishes.

[c] Burkhard (2021) found only bioconcentration factors (BCFs) for 6:2 FTS.

4.5 PFAS BAFs Compared to Laboratory Exposure Data

The BAFs calculated from PFAS concentrations measured in amphipods collected from Kent Lake were compared to those measured in *Lumbriculus* that had been exposed for 28-days to contaminated sediments from six sites throughout Michigan for Goal 1A of this study. The *Lumbriculus* BAFs were calculated using PFAS concentrations measured in pore water (Table 18). While there is general agreement between the Kent Lake amphipod BAFs and *Lumbriculus* BAFs from the laboratory tests for a number of the PFAS compounds, PFOA and PFOS log BAFs for Kent Lake amphipods are substantially higher (0.8-1.0 log units) than median log BAFs for the *Lumbriculus*.

Table 19. Comparison of log BAFs (L/kg wet weight) measured in Kent Lake amphipods Goal 2) to log BAFs measured in *Lumbriculus* from 28-day toxicity tests conducted with contaminated sediments from six Michigan sites (Goal 1A)

	Goal 2	Goal 1A						
	Kent Lake	Fort Gratiot	Clark's Marsh	Huron Norton Creek	Rogue River	Beaver Dam Pond	Pigeon River	Median (6 sites)
	log BAFs							
PFBA	1.16	1.4		1.1		1.1		1.1
PFHxA	1.19	0.9				0.77		0.835
PFHpA	1.06	1.3		1.5	1.4	1.3		1.35
PFOA	2.51	1.5	1.8		1.2	1.3	1.6	1.5
PFHxS	2.28		2.2		1.7	1.5		1.7
PFOS	3.44	2.6	2.5	2.8	2.5	2.6	2.7	2.6

4.6 PFAS Biota-sediment Accumulation Factors

Biota-sediment accumulation factors (BSAFs) were calculated for the two PFAS compounds detected in Kent Lake sediment, PFOS and 6:2 FTS, using median sediment sample concentrations (on a dry weight basis) and median concentrations (on a wet weight basis) for all fish and all benthos samples (Table 19). For PFOS, median BSAFs in fish were 36 times higher than for benthos, while for 6:2 FTS the trend was reversed, with median BSAFs in benthos 11 times higher than in fish.

Table 20. Median BSAFs for Fish and Invertebrates Collected from Kent Lake

PFAS	Fish	Benthos
	BSAF (SD, n)	
PFOS	58 (51, 4)	1.6 (0.84, 4)
6:2 FTS	0.35 (0.89, 3)	3.9 (5.4, 4)

Relatively few BSAF data were found in the literature for PFAS. Langberg et al. (2020) reported the following BSAF values for PFOS in muscle tissue sampled from different areas in a large and deep Scandinavian lake that was contaminated by a former industrial discharge: 1.4 – 41.1 (perch), 0.4 – 13.0 (pike), 0.2 – 2.2 (crayfish), 0.4 (char), and 0.5 – 0.6 (trout). The median of the PFOS BSAF values calculated for Kent Lake fish (58) exceeds any of the values reported by Langberg et al., while the range of PFOS BSAFs for benthos in Kent Lake (0.60 – 13.89) is somewhat higher than Langberg's BSAF range for crayfish. No BSAFs could be found in the literature for 6:2 FTS.

BSAFs calculated from PFAS concentrations measured in Kent Lake amphipods were also compared to BSAFs calculated in *Lumbriculus* from the Goal 1A toxicity tests (Table 20). While the Kent Lake amphipod BSAF for PFOS fell within the range of values measured for *Lumbriculus* from the six laboratory tests, the Kent Lake value (2.15) was higher than the median of the six *Lumbriculus* BSAFs (0.87). For 6:2 FTS, the Kent Lake BSAF was about twice the BSAF value measured in the test from the one sediment (Huron Norton Creek) that produced data to calculate a BSAF.

Table 21. Comparison of BSAFs (kg dry sediment/kg wet weight) measured in Kent Lake amphipods (Goal 2) to BSAFs measured in *Lumbriculus* from 28-day toxicity tests conducted with contaminated sediments from six Michigan sites (Goal 1A)

	Goal 2	Goal 1A					
PFAS	Kent Lake	Fort Gratiot	Clark's Marsh	Huron Norton Cr.	Rogue River	Beaver Dam Pond	Pigeon River
PFOS	2.15	6.3	0.74	4.0	1.0	0.17	0.58
6:2 FTS	13.9			7.2			

4.7 PFAS Predator-prey Ratios

Predator-prey ratios (PPRs) offer a means of examining biomagnification of contaminants. Assuming trophic transfer from benthos→ forage fish → predator fish, PPRs were calculated for forage fish/benthos and predator fish/forage fish for all PFAS measured in both types of biota. The results are shown in Figure 17. For many of the PFAS, the predator fish/forage fish PPRs are fairly close to unity (1), implying little or no biomagnification. For a number of the PFAS, however, the forage fish/benthos PPRs are elevated, indicating biomagnification. These include PFOS (PPR=35) and PFDS (PPR=14) in Kent Lake, and also PFDA (PPR=7 to 8), PFUdA (PPR=5 to 7) and PFDoA (PPR=3 to 6) in both Kent and Proud lakes. Interestingly, these latter PFAS compounds were not detected in water or sediment in either lake.

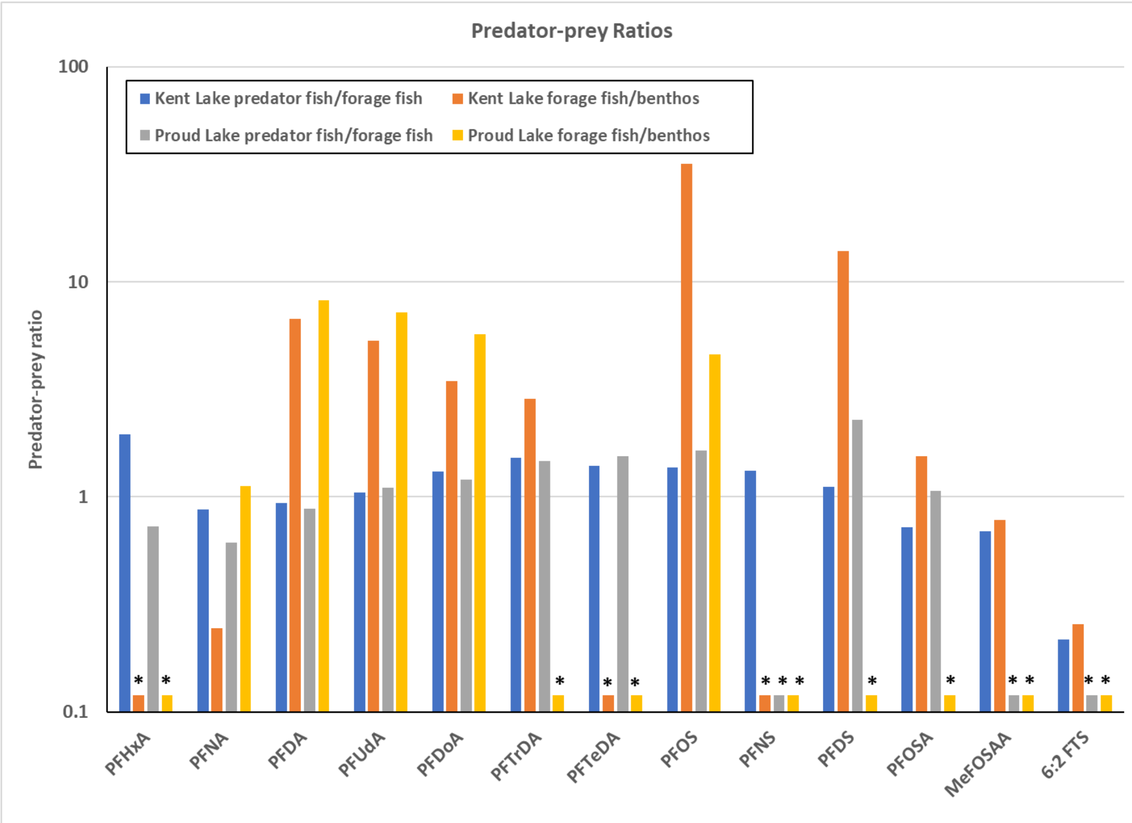


Figure 17. Predator-prey ratios for PFAS compounds in Kent Lake and Proud Lake (Note: asterisk above bar indicates no PPR could be calculated for that compound)

4.8 Comparison of PFAS Detected in Kent Lake and Proud Lake with Wixom WWTP Discharge

As presented above in Section 3.2.1, 9 PFAS compounds were detected in Kent Lake surface water. In comparison, 10 PFAS compounds were measured in final effluent from the Wixom WWTP in 2018 (2 years prior to lake sampling for this study), including the 9 PFAS compounds detected in Kent Lake water (Table 21; AECOM, 2021). The concentrations of these PFAS compounds in WWTP effluent and Kent Lake water are also significantly correlated (Figure 18). This correlation strongly suggests that the WWTP discharge was a source of PFOS and other PFAS compounds in Kent Lake[15]. On the other hand, 8 of the PFAS compounds measured in the Wixom WWTP effluent were also detected in Proud Lake water (albeit at much lower concentrations), but the concentrations of those PFAS compounds in effluent and Proud Lake water were not significantly correlated (not shown). PFAS compounds in Proud Lake, which does not receive discharge of Wixom WWTP effluent via flow from Norton Creek, most likely originate from other sources.

[15] The departure of the PFOS concentration in Kent Lake water from the linear regression line plotted in Figure 18 may be related to the addition of pretreatment to remove this compound from the wastewater, as will be discussed subsequently.

Table 22. PFAS compound concentrations in Kent Lake water, Proud Lake water, and Wixom WWTP effluent

PFAS Compound	Kent L. water[a]	Proud L. water[a]	Wixom WWTP effluent[b]
	Concentration in ng/L (ppt)		
PFBA	7.7	4.8	90
PFPeA	24	3.2 (J)	790
PFHxA	15	3.0 (J)	440
PFHpA	13	1.4 (J)	330
PFOA	2.1 (J)	2.6 (J)	9.9
PFNA	ND	0.46 (J)	3.4
PFBS	2.9 (J)	3.0 (J)	13
PFHxS	0.94 (J)	1.1 (J)	2.8
PFOS	3.6	ND	270
6:2 FTS	50	ND	3,000

[a] Median concentration measured in samples collected for this study.

[b] AECOM. 2021.

ND = Not detected at a concentration > reported DL.

J = Estimated concentration below the reported LOQ but above the reported DL.

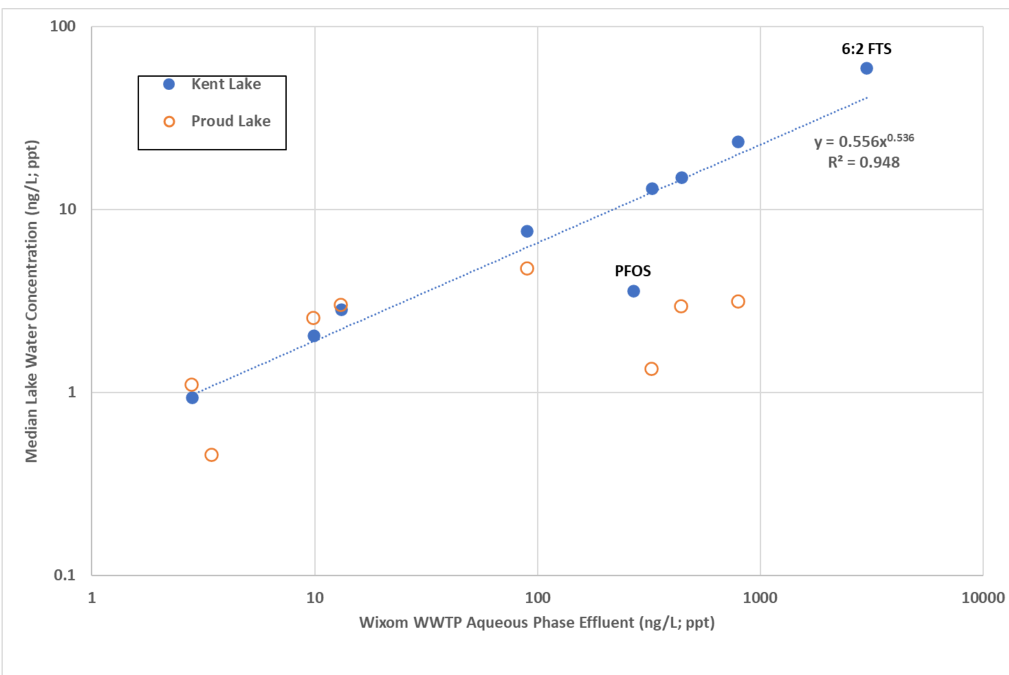
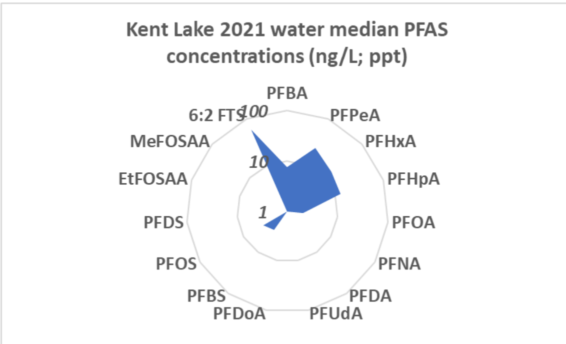


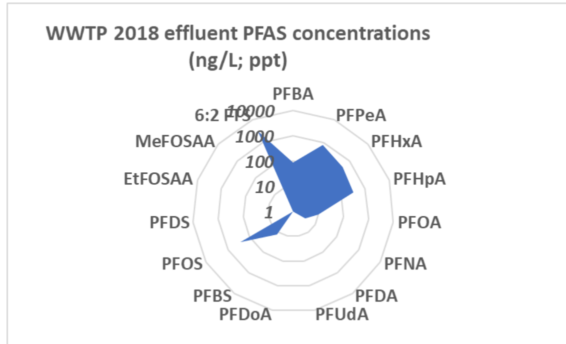
Figure 18. Scatterplot of PFAS compound concentrations measured in Wixom WWTP effluent vs. median concentrations in Kent Lake and Proud Lake water[16]

The strong similarity in PFAS concentration distributions measured in Kent Lake water and Wixom WWTP effluent is also evident in radar plots of these data (Figure 19).

[16] Regression line, equation, and labels for PFOS and 6:2 FTS are only presented for Kent Lake:Wixom WWTP data.

Figure 19 also includes a radar plot for PFAS concentrations measured in WWTP biosolids, which was applied to farm fields as fertilizer in the early 2010s[17]. 6:2 FTS and its degradation products are predominant PFAS in lake water; these compounds are also predominant in the treatment plant influent, effluent, and sludge (biosolids).





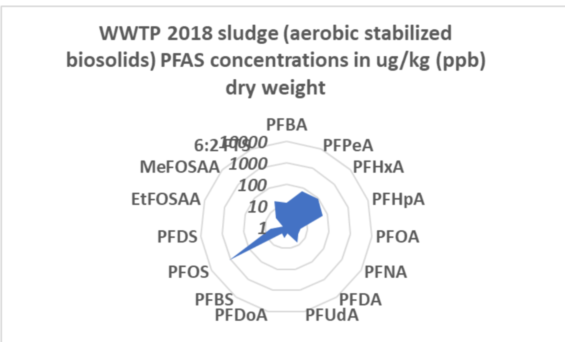
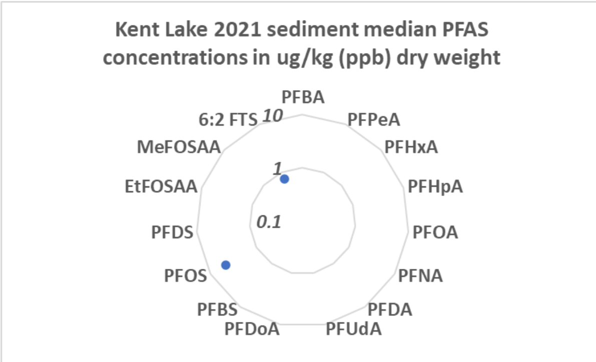


Figure 19. Median concentrations of PFAS compounds detected in Kent Lake water, Wixom WWTP effluent and Wixom WWTP biosolids

The radar plot in Figure 20 compares PFAS concentrations measured in 2021 in Kent Lake sediment (this study) to PFAS measured in sediment collected from the Wixom WWTP outfall pond in 2022 (EGLE, 2022). The agreement in PFAS concentration distributions between these samples, especially the prominence of PFOS and 6:2 FTS, is notable. These data show that a significant inventory of PFOS remains in place in sediments near the WWTP discharge, even 4 years following source control.

[17] MLive, *Michigan farmer sues auto supplier after PFAS taints cattle herd*. Published August 26, 2022.



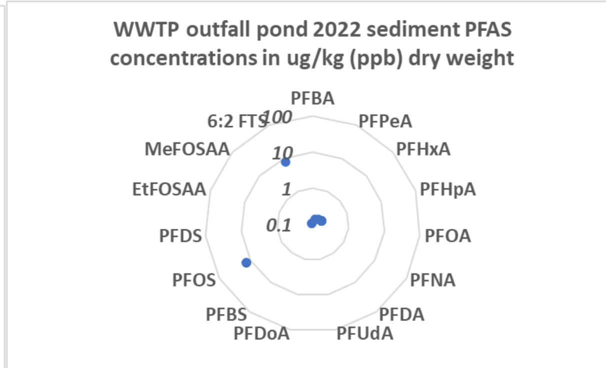


Figure 20. Median concentrations of PFAS compounds detected in Kent Lake sediment and sediment collected from the Wixom WWTP outfall pond in 2022

4.9 Understanding PFOS and 6:2 FTS Contamination in Kent Lake

As presented in Section 3.2.1, PFOS and 6:2 FTS were the only PFAS compounds detected in Kent Lake sediment. PFOS contamination in Kent Lake is well-documented and related to usage and discharge by a plating facility (EGLE 2019). During electrochemical plating, chromic acid mists, which pose a health risk to workers, are produced. Surfactants are added to the plating bath to suppress mist formation and decrease surface wettability (Kim et al. 2021). PFOS was traditionally used as a popular mist suppressant in functional chromium plating and in plastic etching. The usage and sale of PFOS as a fume suppressant added to chromium plating tanks was ended in 2015 by EPA's final chromium electroplating NESHAP rule[18]. 6:2 FTS has been widely used as a non-PFOS mist suppressant in the chromate plating process dating from 2012 to 2015 (NASF 2019). While 6:2 FTS is reported to be less toxic and bioaccumlative then PFOS, high levels of 6:2 FTS have been detected in environmental media at sites associated with point-sources of contamination such as fluorochemical manufacturing facilities or fire fighter training sites where PFAS-containing aqueous film forming foam (AFFF) has been used (NASF 2019). 6:2 FTS was measured at a concentration of 3,000 ng/L in final effluent from the Wixom WWTP in 2018 (AECOM, 2021), and at 1,100 ng/L in the Norton Creek receiving water in November of 2021 (GLEC, 2022). These data indicate that PFOS discharge from the WWTP, presumably from industrial discharges originating from the plating facility, continued well after the 2015 chromium electroplating NESHAP rule. As mentioned above, WWTP discharge was also a likely source of other PFAS compounds in Kent Lake, and this includes 6:2 FTS.

To put the results of this study in context, it is useful to review information gathered by EGLE regarding the time course of PFAS contamination in Kent Lake (EGLE, 2019). The Wixom, MI WWTP began discharge monitoring for PFAS in August 2018. Elevated PFOS concentrations (as high as 4,800 ng/L) were measured and traced to wastewater discharged from the metal plating facility. That facility added GAC treatment in October 2018 as a pretreatment process to remove PFAS from their sewer discharge. A 95% and 99% drop in PFOS concentrations at the WWTP discharge was measured in 69 days and in one year, respectively. An 81% drop in total PFAS concentrations was also

[18] National Emission Standards for Hazardous Air Pollutants: Hard and Decorative Chromium Electroplating and Chromium Anodizing Tanks (40 CFR Part 63, Subpart N).

measured in 69 days. As noted previously, the WWTP discharged to Norton Creek, a small tributary that flows to the Huron River upstream of Kent Lake.

Reductions in PFOS concentrations in Kent Lake were also measured over the same period. Between October 2018 and August 2020, PFOS concentrations measured in Kent Lake water dropped 82%; between August 2020 to October 2021[19], PFOS concentrations dropped another 7%. PFOS in small Kent Lake fish dropped 90% between 2017 and 2019, and decreased an additional 10% between 2019 and 2021[20]. PFOS in large Kent Lake fish dropped 78% between 2017 and 2019, and decreased another 62% between 2019 and 2021[10]. Figure 21 is a timeseries plot of PFOS concentrations in Wixom WWTP plant effluent, Kent Lake water, and small and large fish in Kent Lake. In each of these cases, PFOS concentrations in the lake initially dropped quite rapidly after the contamination source was addressed, but after two years, further reductions appeared generally less dramatic (most notably in water and small fish, based on limited data). Sampling for this study took place in May and November of 2021, during the period of comparatively slow changes in PFOS (and presumably other PFAS compound) concentrations.

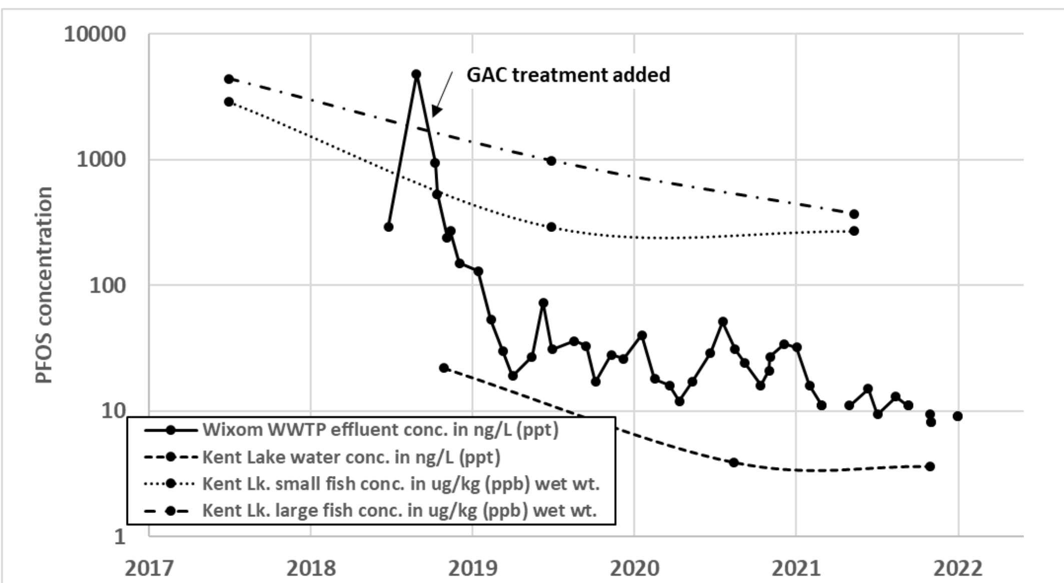


Figure 21. PFOS concentrations in Wixom WWTP plant effluent, Kent Lake water, and small and large fish in Kent Lake.

PFOS and other PFAS have accumulated in the sediments and food web of Kent Lake and, to a lesser degree, in Proud Lake. Although PFOS concentrations in Kent Lake have declined 84% in water and 92% in large fish since a major contamination source

[19] PFOS concentrations reported in this study.

[20] Comparison of PFOS concentrations in fish sampled in 2021 vs. earlier years is complicated by the fact that in 2021 whole fish were analyzed while in 2017 and 2019 the analysis was conducted on fish fillets. To overcome this difference, we determined the ratio of PFOS concentrations between muscle (fillets) and whole fish. Based on data from the literature, PFOS muscle:whole fish ratios were determined for six fish species and ranged from 0.245 to 0.535, with an average of 0.396. The 0.396 ratio was then used to estimate whole body PFOS concentrations from the 2019 filet data.

was addressed by treatment, the changes have been far slower than the 4-day hydraulic residence time[21] of the lake. In the absence of other significant sources, it is logical to conclude that sediment is acting as a reservoir for PFOS in Kent Lake. Furthermore, the relatively slow changes in PFOS concentrations in Kent Lake are likely due to the "natural recovery" from PFOS in the lake sediments, through sediment scouring and burial, diffusion from pore water, and possibly biotic or abiotic transformation. PFOS may continue to be available to the aquatic ecosystem via bioaccumulation by benthic invertebrates and trophic transfer to fish, as well as possibly physical-chemical transport (scour and diffusion; Kong et al. 2018) of the contaminant from the sediment back into the lake water. It is also possible that the slow rate of PFOS decline observed in large fish may reflect the elimination of this compound from body burdens that accumulated in the past, when aqueous and dietary exposures may have also been higher, although this is not supported by laboratory studies or bioaccumulation modeling[22].

PFOS has continued to accumulate in the aquatic food chain of Kent Lake for a number of years after sources were controlled and water concentrations declined. As natural recovery occurs, concentrations of PFOS and other PFAS may be expected to decline in sediment, sediment-dwelling organisms and fish that feed upon these organisms. The results of this study, together with results from comparable studies conducted over time, may help inform risk assessors regarding when to expect a decline in fish tissue concentrations and/or whether natural recovery is the appropriate management decision for the waterbody, or if other actions like sediment capping or dredging should be explored. Based on the reductions in PFOS concentrations observed in Kent Lake, fish tissue concentrations declined by about one order-of-magnitude over four years (2017 to 2021) by natural recovery. Further studies would be required to extrapolate this result to other aquatic ecosystems and/or longer time periods. There are still unanswered questions about PFAS contamination in the Huron River waterway that includes Proud and Kent Lakes, including: What are the sources of PFAS other than PFOS and 6:2 FTS in both lakes? What is the role of chemical transformation on the distribution of PFAS throughout this aquatic ecosystem?

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[21] Hydraulic residence time was calculated as the ratio of lake volume (1,200-acre surface area·6-foot average depth= 5.2×10^7 ft^2) to outflow rate (141 ft^3/s; USGS 20-year average flow rate of the Huron River at Milford, MI).

[22] Research suggests that fish eliminate PFOS and other PFAS fairly rapidly (adult rainbow trout elimination half-lives for PFOS were 8-20 days depending upon tissue type; Falk et al., 2015), apparently due to substantial renal elimination (Sun et al., 2022).

4.11 Non-PFAS Chemical Parameters in Comparison to Sediment Quality Criteria

Concentrations of other (non-PFAS) chemical parameters measured in Kent and Proud Lake sediments were compared to threshold and probable effect concentrations (TEC and PEC, respectively) to assess the toxicity of these chemicals to sediment-dwelling organisms. Effect concentrations for metals, PAHs and total PCBs were obtained from EPA (2002), MacDonald et al. (2000) and the Wisconsin Department of Natural Resources (2003). The only non-PFAS parameter measured at concentrations exceeding these effect concentrations was total PCBs in Kent Lake sediment. The total PCB concentration in Kent Lake (269 µg/kg) falls below the PEC of 676 µg/kg (MacDonald et al., 2000) and 277 µg/kg (Wisconsin DNR. 2003), but exceeds the TEC of 60 µg/kg (MacDonald et al., 2000) and 34 µg/kg (Wisconsin DNR. 2003). The total PCB concentration in Proud Lake (12.4 µg/kg) falls below these effect concentrations.

No impacts to the food web can be attributed to non-PFAS sediment contaminants, including PCBs, with any certainty. Because benthic invertebrates were scarce in both the Kent Lake and Proud Lake sediments during the sampling events, the scarcity of organisms in the Kent Lake sediments cannot reasonably be attributed to the toxic effects of PCBs. Furthermore, toxic effects of PCBs at 269 µg/kg to benthos are uncertain. Finkelstein et al. (2017) calculated 23.7% benthic injury (sublethal reproduction effects) due to chronic exposure to Aroclor 1254 (a PCB mixture) of 1,000 µg/kg. Another factor potentially explaining the scarcity of benthic invertebrates is the time of year that samples were collected (November).

5.0 SUMMARY AND CONCLUSIONS

PFOS and 6:2 FTS were the only PFAS compounds detected in Kent Lake sediment; PFOS made up 81 to 100% of the total concentration of PFAS. In contrast, no PFAS compounds were detected in the Proud Lake (reference site) sediment samples.

Nine PFAS compounds were detected in Kent Lake surface water, while eight PFAS compounds were detected in water at the Proud Lake area reference site. PFOS made up 2 to 4%, and 6:2 FTS made up 42 to 49%, of the total concentration of PFAS in Kent Lake water samples. The concentrations of nine PFAS compounds measured in Kent Lake water in this study were significantly correlated to concentrations of the same PFAS compounds measured in 2018 in the effluent from the Wixom WWTP. An industry discharging to this WWTP was known to be a major source of PFOS contamination prior to the installation of a treatment system. Neither PFOS nor 6:2 FTS were detected in water from Proud Lake.

A dozen or more PFAS compounds were detected in predator and forage fish in Kent Lake, with PFOS making up 92 to 96 percent of the total PFAS concentration in the sampled fish. The total PFAS concentration was about 35% higher, on average, in predator fish than in prey fish. Ten to 17 PFAS compounds were detected in benthic invertebrates sampled in Kent Lake. PFOS made up 38 to 60 percent of the total PFAS concentration in the Kent Lake invertebrates. The total PFAS concentration was, on average, 19 times higher in prey fish compared to benthos, due primarily to the relatively high concentrations of PFOS and PFDS measured in prey fish.

The PFOS concentrations measured in Kent Lake predator fish were 630 µg/kg

(Largemouth Bass) and 110 µg/kg (sunfish), while PFOS concentrations in forage fish were 160 µg/kg (sunfish) and 380 µg/kg (juvenile bass). In comparison, fish sampled throughout the Huron River watershed from 2017 to 2022 had PFOS concentrations that ranged from 0.7 to 2,000 µg/kg[23], and the maximum PFOS concentration measured in fish from EPA's 2013-14 NRSA was 283 µg/kg (Barbo et al., 2023).

The primary objective of this study was to determine whether sediment acts as a source of PFAS to the aquatic food web, following source control to curtail active loading. The following line of evidence was developed in this study that supports this hypothesis:

- The patterns of PFAS distribution in the sediments and biota of Kent Lake appear similar, as illustrated by radar plots (Figures 11), suggesting that PFAS in the sediment may act as a source of contamination to invertebrates, forage fish and predator fish.

For PFOS and 6:2 FTS, this study provides additional lines of evidence:

- A major source of PFOS contamination to the Huron River watershed and Kent Lake was identified and controlled in 2018, yet biota concentrations remain elevated.
- The presumptive food chain (benthic invertebrates→ forage fish → predator fish) in Kent Lake provides a trophic transfer mechanism that could explain how sediment acts as a source of PFAS to the aquatic food web.
- The partition coefficients calculated from the data for Kent Lake, as well as other PFAS-impacted water bodies in Michigan, show that PFOS and 6:2 FTS have a strong tendency to partition from the water column into sediment.
- The tendency for PFOS and 6:2 FTS to partition from the water column into the sediment probably led to significant accumulation of PFOS in the sediments of Kent Lake, as well as upstream of Kent Lake, during the years of active PFOS discharge from the Wixom WWTP. In other words, sediments probably acted as a sink for PFOS during this time.
- Once the PFOS source was controlled, the initial decline in water concentrations was rapid (Figure 21). Sediment PFOS concentrations would be expected to decline more slowly, continuing to contaminate the food chain during "active recovery" of the sediments. Thus, sediments probably went from being a <u>sink</u> to a <u>source</u> of PFOS in Kent Lake.
- Physical-chemical transport (scour and diffusion) could also reintroduce PFOS from the sediment back into the water column, where additional bioaccumulation could take place.

However, the scope of this study also has limitations that should be considered:

- The sources of "other" PFAS to the study and reference lakes are not fully understood.
- It is not clear to what extent the sources of these "other" PFAS have been controlled.
- Although the "other" PFAS were detected and measured in water, benthic

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[23] Michigan.gov/pfasresponse/investigations/lakes-and-streams/huron-river

invertebrates and fish in Kent Lake as well as Proud Lake, they were not detected in the sediments of either lake.

- For all of the media that were sampled (sediment, water, fish and invertebrates), the sample sizes were quite small ($2 \le n \le 4$) which raises the issue of whether the median PFAS concentrations were truly representative of the lake ecosystems.
- Two of the four invertebrate samples from Kent Lake were Odonates (dragonflies and damselflies) that may not typically serve as prey for forage fish.
- The PFOS and PFAS concentrations in the two sediment samples from Kent Lake differed by a factor of 4, most likely due to differences in the sorption properties of the sediments.
- Collection and PFAS analysis of additional sediment samples from Kent Lake would be useful to confirm the median concentrations.
- Sampling and analysis of sediments from the same locations over time (perhaps annually) would be useful to determine whether PFOS and other PFAS concentrations are declining, and at what rate.

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Attachment A. Analytes, Limits of Quantitation and Detection Limits[24]

Table A.1. Minimum LOQs and DLs for PFAS Compounds Analyzed in Goal 2 Sediment, Surface Water and Biota Samples

				PFAS in sediment		PFAS in surface water		PFAS in biota	
			Method						
			SOP Reference	ME003NI-04		ME003NI-04		MIN4-0178	
	Analyte	Abbreviation	CAS#	LOQ	DL	LOQ	DL	LOQ	DL
	Perfluoroalkyl carboxylic acid	***PFCA***		µg/kg		ng/L		µg/kg	
1	Perfluorobutanoic acid	PFBA	375-22-4	1	0.2	4	0.60	0.250	0.046
2	Perfluoropentanoic acid	PFPeA	2706-90-3	1	0.2	4	0.54	0.250	0.049
3	Perfluorohexanoic acid	PFHxA	307-24-4	1	0.2	4	0.69	0.250	0.069
4	Perfluoroheptanoic acid	PFHpA	375-85-9	1	0.2	4	0.45	0.250	0.091
5	Perfluorooctanoic acid - br/lin	PFOA	335-67-1	1	0.2	4	0.83	0.250	0.058
6	Perfluorononoic acid	PFNA	375-95-1	1	0.2	4	0.46	0.250	0.029
7	Perfluorodecanoic acid	PFDA	335-76-2	1	0.2	4	0.52	0.250	0.106
8	Perfluoroundecanoic acid	PFUdA	2058-94-8	1	0.2	4	0.63	0.250	0.064
9	Perfluorododecanoic acid	PFDoA	307-55-1	1	0.2	4	0.47	0.250	0.074
10	Perfluorotridecanoic acid	PFTrDA	72629-94-8	1	0.2	4	0.53	0.250	0.050

[24] LOQs and DLs presented here are minimum limits. Reported limits were adjusted to account for actual measured sample volume/weight (adjusted for percent solid in sediment samples) and dilution.

Table A.1. Minimum LOQs and DLs for PFAS Compounds Analyzed in Goal 2 Sediment, Surface Water and Biota Samples

			Method	PFAS in sediment		PFAS in surface water		PFAS in biota	
			SOP Reference	ME003NI-04		ME003NI-04		MIN4-0178	
	Analyte	Abbreviation	CAS#	LOQ	DL	LOQ	DL	LOQ	DL
11	Perfluorotetradecanoic acid	PFTeDA	376-06-7	1	0.2	4	0.60	0.250	0.071
12	Perfluorohexadecanoic acid	PFHxDA	67905-19-5	2	0.5	8	0.82	0.250	0.032
13	Perfluorooctandecanoic acid	PFODA	16517-11-6	1	0.2	8	1.00	0.250	0.054
	Perfluoroalkane sulfonic acid	***PFSA***							
14	Perfluorobutanesulfonic acid	PFBS	375-73-5	1	0.2	4	0.41	0.221	0.043
15	Perfluoropentanesulfonic acid	PFPeS	2706-91-4	1	0.2	4	0.59	0.235	0.058
16	Perfluorohexanesulfonic acid - br/lin	PFHxS	355-46-4	1	0.2	4	0.55	0.228	0.044
17	Perfluoroheptanesulfonic acid	PFHpS	375-92-8	1	0.2	4	0.50	0.238	0.034
18	Perfluorooctanesulfonic acid - br/lin	PFOS	1763-23-1	1	0.2	4	2.00	0.231	0.034
19	Perfluorononesulfonic acid	PFNS	68259-12-1	1	0.2	4	0.71	0.240	0.048
20	Perfluorodecanesulfonic acid	PFDS	335-77-3	1	0.2	4	0.78	0.241	0.040
21	Perfluorododecanesulfonic acid	PFDoS	79780-39-5	1	0.2	8	1.00	0.243	0.029
	Perfluoroalkane sulfomides and derivatives	***FASA***							

Table A.1. Minimum LOQs and DLs for PFAS Compounds Analyzed in Goal 2 Sediment, Surface Water and Biota Samples

			Method	PFAS in sediment		PFAS in surface water		PFAS in biota	
			SOP Reference	ME003NI-04		ME003NI-04		MIN4-0178	
	Analyte	Abbreviation	CAS#	LOQ	DL	LOQ	DL	LOQ	DL
22	Perfluorooctanesulfomide	PFOSA	754-91-6	1	0.2	4	0.61	0.250	0.034
23	N-ethyl perfluorooctane sulfomidoethanol	EtFOSE	1691-99-2	2	0.5	8	0.95	0.250	0.048
24	N-methyl perfluorooctane sulfomidoethanol	MeFOSE	24448-09-7	2	0.5	8	1.30	0.250	0.050
25	N-ethyl perfluorooctane sulfomide	EtFOSA	4151-50-2	2	0.5	8	1.40	0.250	0.034
26	N-methyl perfluorooctane sulfomide	MeFOSA	31506-32-8	2	0.5	16	1.30	0.250	0.038
27	N-ethyl perfluorooctanesulfomidoacetic acid - br/lin	EtFOSAA	2991-50-6	2	0.5	8	0.75	0.250	0.083
28	N-methyl perfluorooctanesulfomidoacetic acid - br/lin	MeFOSAA	2355-31-9	2	0.5	8	0.93	0.250	0.025
	Fluorotelomer sulfonic acid	***FTSA***							
29	4:2 Fluorotelomer sulfonic acid	4:2 FTS	757124-72-4	2	0.5	8	0.87	0.234	0.042
30	6:2 Fluorotelomer sulfonic acid	6:2 FTS	27619-97-2	2	0.5	8	2.00	0.238	0.113
31	8:2 Fluorotelomer sulfonic acid	8:2 FTS	39108-34-4	2	0.5	8	1.60	0.241	0.091
32	10:2 Fluorotelomer sulfonic acid	10:2 FTS	120226-60-0	2	0.5	8	1.20	0.241	0.041
	Perfluoroalkyl ether carboxylic acid	***PFECA***							

Table A.1. Minimum LOQs and DLs for PFAS Compounds Analyzed in Goal 2 Sediment, Surface Water and Biota Samples

			Method	PFAS in sediment		PFAS in surface water		PFAS in biota	
			SOP Reference	ME003NI-04		ME003NI-04		MIN4-0178	
	Analyte	Abbreviation	CAS#	LOQ	DL	LOQ	DL	LOQ	DL
33	Hexafluoropropylene oxide dimer acid	HFPO-DA or GenX	13252-13-6	4	1.0	8	2.10	--	--
34	4,8-dioxa-3H-perfluorononoic acid	ADONA	919005-14-4	2	0.5	8	0.48	0.236	0.065
	Polyfluoroalkyl ether sulfonic acid	***PFESA***							
35	9-Chlorohexadecafluoro-3-oxanone-1-sulfonic acid	9Cl-PF3ONS	756426-58-1	2	0.5	8	0.48	0.233	0.031
36	11-chloroeicosafluoro-3-oxaundecane-1-sulfonic acid	11Cl-PF3OUdS	763051-92-9	2	0.5	8	0.66	0.235	0.036

Table A.2. Minimum DLs and LOQs for PCB Congeners in Goal 2 Sediment Samples

PCB Congener	IUPAC#	DL (ng/kg)	LOQ (ng/kg)
2-Chlorobiphenyl	PCB-1	3.07	25
3-Chlorobiphenyl	PCB-2	3.54	25
4-Chlorobiphenyl	PCB-3	5.50	25
2,2'-Dichlorobiphenyl	PCB-4	7.11	25
2,6-Dichlorobiphenyl	PCB-10	2.68	25
2,5-Dichlorobiphenyl	PCB-9	3.90	25
2,4-Dichlorobiphenyl	PCB-7	4.40	25
2,3'-Dichlorobiphenyl	PCB-6	3.86	25
2,3-Dichlorobiphenyl	PCB-5	3.36	25
2,4'-Dichlorobiphenyl	PCB-8	7.31	25
3,5-Dichlorobiphenyl	PCB-14	2.75	25
3,3'-Dichlorobiphenyl	PCB-11	117.0	174
PCB-(13/12)	PCB-(13/12)	7.2	50
4,4'-Dichlorobiphenyl	PCB-15	6.58	50
2,2',6-Trichlorobiphenyl	PCB-19	3.98	50
PCB-(30/18)	PCB-(30/18)	12.4	50
2,2',4-Trichlorobiphenyl	PCB-17	6.96	25
2,3',6-Trichlorobiphenyl	PCB-27	2.81	25
2,3,6-Trichlorobiphenyl	PCB-24	3.19	25
2,2',3-Trichlorobiphenyl	PCB-16	8.49	25
2,4',6-Trichlorobiphenyl	PCB-32	5.99	25
2',3,5-Trichlorobiphenyl	PCB-34	4.46	25
2,3,5-Trichlorobiphenyl	PCB-23	5.35	25
PCB-(26/29)	PCB-(26/29)	8.7	100
2,3',4-Trichlorobiphenyl	PCB-25	3.68	25
2,4',5-Trichlorobiphenyl	PCB-31	38.1	130
PCB-(28/20)	PCB-(28/20)	34.3	130
PCB-(21/33)	PCB-(21/33)	33.3	270
2,3,4'-Trichlorobiphenyl	PCB-22	21.1	190
3,3',5-Trichlorbiphenyl	PCB-36	4.07	25
3,4',5-Trichlorobiphenyl	PCB-39	4.92	25
3,4,5-Trichlorobiphenyl	PCB-38	5.33	25
3,3',4-Trichlorobiphenyl	PCB-35	4.06	25
3,4,4'-Trichlorobiphenyl	PCB-37	13.0	53
2,2',6,6'-Tetrachlorbiphenyl	PCB-54	2.73	50
PCB-(50/53)	PCB-(50/53)	11.9	100
PCB-(45/51)	PCB-(45/51)	13.7	100
2,2',3,6'-Tetrachlorobiphenyl	PCB-46	4.78	50
2,2',5,5'-Tetrachlorobiphenyl	PCB-52	29.8	123
2,3',5',6-Tetrachlorobiphenyl	PCB-(73/43)	9.7	100
PCB-(69/49)	PCB-(69/49)	14.6	100
2,2',4,5-Tetrachlorobiphenyl	PCB-48	5.82	100

Table A.2. Minimum DLs and LOQs for PCB Congeners in Goal 2 Sediment Samples

PCB Congener	IUPAC#	DL (ng/kg)	LOQ (ng/kg)
PCB-(44/47/65)	PCB-(44/47/65)	39.6	300
PCB-(59/62/75)	PCB-(59/62/75)	13.7	150
2,2',3,4'-Tetrachlorobiphenyl	PCB-42	8.49	100
PCB-(41/40/71)	PCB-(41/40/71)	16.8	150
2,3,4',6-Tetrachlorobiphenyl	PCB-64	14.90	50
2,3',5,5'-Tetrachlorobiphenyl	PCB-72	4.53	50
2,3',4,5'-Tetrachlorobiphenyl	PCB-68	4.98	50
2,3,3',5-Tetrachlorobiphenyl	PCB-57	4.21	50
2,3,3',5'-Tetrachlorobiphenyl	PCB-58	4.44	50
2,3',4,5-Tetrachlorobiphenyl	PCB-67	3.57	50
2,3,4',5-Tetrachlorobiphenyl	PCB-63	4.47	50
PCB-(61/70/74/76)	PCB-(61/70/74/76)	40.1	200
2,3',4,4'-Tetrachlorobiphenyl	PCB-66	25.4	84
2,3,3',4-Tetrachlorobiphenyl	PCB-55	4.63	50
2,3,3',4'-Tetrachlorobiphenyl	PCB-56	14.10	50
2,3,4,4'-Tetrachlorobiphenyl	PCB-60	10.00	50
3,3',5,5'-Tetrachlorobiphenyl	PCB-80	3.78	50
3,3',4,5'-Tetrachlorobiphenyl	PCB-79	3.16	50
3,3',4,5-Tetrachlorobiphenyl	PCB-78	4.23	50
3,4,4',5-Tetrachlorobiphenyl	PCB-81	5.15	50
3,3',4,4'-Tetrachlorobiphenyl	PCB-77	4.88	50
2,2',4,6,6'-Pentachlorobiphenyl	PCB-104	5.78	50
2,2',3,6,6'-Pentachlorobiphenyl	PCB-96	4.31	50
2,2',4,5',6-Pentachlorobiphenyl	PCB-103	4.76	50
2,2',3,5,6'-Pentachlorobiphenyl	PCB-94	4.94	50
2,2',3,5',6-Pentachlorobiphenyl	PCB-95	20.0	95
PCB-(100/93/102/98)	PCB-(100/93/102/98)	16.8	200
PCB-(88/91)	PCB-(88/91)	7.3	100
2,2',3,3',6-Pentachlorobiphenyl	PCB-84	7.60	50
2,2',3,4,6'-Pentachlorobiphenyl	PCB-89	5.52	50
2,3',4,5',6-Pentachlorobiphenyl	PCB-121	4.24	50
2,2',3,5,5'-Pentachlorobiphenyl	PCB-92	3.81	50
PCB-(113/90/101)	PCB-(113/90/101)	16.7	300
2,2',3,3',5-Pentachlorobiphenyl	PCB-83	4.12	50
2,2',4,4',5-Pentachlorobiphenyl	PCB-99	8.83	100
2,3,3',5,6-Pentachlorobiphenyl	PCB-112	4.76	50
PCB-(108/119/86/97/125/87)	PCB-(108/119/86/97/125/87)	19.4	300
PCB-(117/116/85)	PCB-(117/116/85)	9.6	150
PCB-(110/115)	PCB-(110/115)	27.1	200
2,2',3,3',4-Pentachlorobiphenyl	PCB-82	5.56	50
2,3,3',5,5'-Pentachlorobiphenyl	PCB-111	4.65	50

Table A.2. Minimum DLs and LOQs for PCB Congeners in Goal 2 Sediment Samples			
PCB Congener	**IUPAC#**	**DL (ng/kg)**	**LOQ (ng/kg)**
2,3',4,5,5'-Pentachlorobiphenyl	PCB-120	4.18	50
PCB-(107/124)	PCB-(107/124)	6.62	100
2,3,3',4,6-Pentachlorobiphenyl	PCB-109	3.30	50
2,3',4,4',5'-Pentachlorobiphenyl	PCB-123	6.02	50
2,3,3',4,5-Pentachlorobiphenyl	PCB-106	2.51	50
2,3',4,4',5-Pentachlorobiphenyl	PCB-118	18.40	50
2,3,3',4',5'-Pentachlorobiphenyl	PCB-122	3.65	50
2,3,4,4',5-Pentachlorobiphenyl	PCB-114	4.65	50
2,3,3',4,4'-Pentachlorobiphenyl	PCB-105	10.20	100
3,3',4,5,5'-Pentachlorobiphenyl	PCB-127	3.60	50
3,3',4,4',5-Pentachlorobiphenyl	PCB-126	6.40	50
2,2',4,4',6,6'-Hexachlorobiphenyl	PCB-155	4.84	50
2,2',3,5,6,6'-Hexachlorobiphenyl	PCB-152	4.97	50
2,2',3,4',6,6'-Hexachlorobiphenyl	PCB-150	3.60	50
2,2',3,3',6,6'-Hexachlorobiphenyl	PCB-136	3.69	50
2,2',3,4,6,6'-Hexachlorobiphenyl	PCB-145	4.65	50
2,2',3,4',5,6'-Hexachlorobiphenyl	PCB-148	4.86	50
PCB-(151/135)	PCB-(151/135)	6.4	100
2,2'4,4',5,6'-Hexachlorobiphenyl	PCB-154	3.26	50
2,2',3,4,5',6-Hexachlorobiphenyl	PCB-144	3.07	50
PCB-(147/149)	PCB-(147/149)	15.50	200
PCB-(134/143)	PCB-(134/143)	10.1	100
PCB-(139/140)	PCB-(139/140)	10.0	100
2,2'3,3',4,6-Hexachlorobiphenyl	PCB-131	4.39	50
2,2',3,4,5,6-Hexachlorobiphenyl	PCB-142	5.16	50
2,2',3,3',4,6'-Hexachlorobiphenyl	PCB-132	7.77	100
2,2',3,3',5,5'-Hexachlorobiphenyl	PCB-133	4.67	50
2,3,3',5,5',6-Hexachlorobiphenyl	PCB-165	5.05	50
2,2',3,4',5,5'-Hexachlorobiphenyl	PCB-146	3.26	50
2,3,3',4,5',6-Hexachlorobiphenyl	PCB-161	4.01	50
PCB-(153/168)	PCB-(153/168)	15.2	200
2,2',3,4,5,5'-Hexachlorobiphenyl	PCB-141	6.24	50
2,2',3,3',4,5'-Hexachlorobiphenyl	PCB-130	5.44	50
2,2',3,4,4',5-Hexachlorobiphenyl	PCB-137	5.63	50
2,3,3',4',5',6-Hexachlorobiphenyl	PCB-164	4.11	50
PCB-(138/163/129)	PCB-(138/163/129)	23.8	300
2,3,3',4,5,6-Hexachlorobiphenyl	PCB-160	4.51	50
2,3,3',4,4',6-Hexachlorobiphenyl	PCB-158	2.84	50
PCB-(128/166)	PCB-(128/166)	9.2	100
2,3,3',4,5,5'-Hexachlorobiphenyl	PCB-159	4.40	50
2,3,3',4',5,5'-Hexachlorobiphenyl	PCB-162	3.51	50
2,3',4,4',5,5'-Hexachlorobiphenyl	PCB-167	4.51	50

Table A.2. Minimum DLs and LOQs for PCB Congeners in Goal 2 Sediment Samples

PCB Congener	IUPAC#	DL (ng/kg)	LOQ (ng/kg)
PCB-(156/157)	PCB-(156/157)	9.4	100
3,3',4,4',5,5'-Hexachlorobiphenyl	PCB-169	4.70	50
2,2',3,4',5,6,6'-Heptachlorobiphenyl	PCB-188	4.66	50
2,2',3,3',5,6,6'-Heptachlorobiphenyl	PCB-179	3.18	50
2,2',3,4,4',6,6'-Heptachlorobiphenyl	PCB-184	3.79	50
2,2',3,3',4,6,6'-Heptachlorobiphenyl	PCB-176	3.70	50
2,2',3,4,5,6,6'-Heptachlorobiphenyl	PCB-178	5.37	50
2,2',3,3',5,5',6-Heptachlorobiphenyl	PCB-175	3.91	50
2,2',3,3',4,5',6-Heptachlorobiphenyl	PCB-187	5.50	50
2,2',3,4',5,5',6-Heptachlorobiphenyl	PCB-182	2.24	50
2,2',3,4,4',5,6'-Heptachlorobiphenyl	PCB-186	4.24	50
PCB-(183/185)	PCB-(183/185)	6.2	100
2,2',3,3',4,5,6'-Heptachlorobiphenyl	PCB-174	4.08	50
2,2'3,3',4,5',6'-Heptachlorobiphenyl	PCB-177	3.05	50
2,2',3,4,4',5,6-Heptachlorbiphenyl	PCB-181	4.81	50
PCB-(171/173)	PCB-(171/173)	5.74	100
2,2'3,3',4,5,5'-Heptachlorobiphenyl	PCB-172	3.69	50
2,3,3',4,5,5',6-Heptachlorobiphenyl	PCB-192	3.76	50
PCB-(180/193)	PCB-(180/193)	8.0	100
2,3,3',4,4',5',6-Heptachlorobiphenyl	PCB-191	4.36	50
2,2',3,3',4,4',5-Heptachlorobiphenyl	PCB-170	4.30	50
2,3,3',4,4',5,6-Heptachlorobiphenyl	PCB-190	4.38	50
2,3,3',4,4',5,5'-Heptachlorobiphenyl	PCB-189	5.91	50
2,2',3,3',5,5',6,6'-Octachlorobiphenyl	PCB-202	4.54	75
2,2',3,3',4,5',6,6'-Octachlorobiphenyl	PCB-201	3.59	75
2,2',3,4,4',5,6,6'-Octachlorobiphenyl	PCB-204	4.31	75
PCB-(197/200)	PCB-(197/200)	6.0	150
PCB-(198/199)	PCB-(198/199)	8.0	150

Table A.2. Minimum DLs and LOQs for PCB Congeners in Goal 2 Sediment Samples

PCB Congener	IUPAC#	DL (ng/kg)	LOQ (ng/kg)
2,2',3,3',4,4',5,6'-Octachlorobiphenyl	PCB-196	3.76	75
2,2',3,4,4',5,5',6-Octachlorobiphenyl	PCB-203	2.28	75
2,2',3,3',4,4',5,6-Octachlorobiphenyl	PCB-195	3.93	75
2,2',3,3',4,4',5,5'-Octachlorobiphenyl	PCB-194	3.75	75
2,3,3',4,4',5,5',6-Octachlorobiphenyl	PCB-205	5.20	75
2,2'3,3',4,4',5,5',6,6'-Nonachlorobiphenyl	PCB-208	4.63	75
2,2'3,3',4,4',5,6,6'-Nonachlorobiphenyl	PCB-207	4.03	75
2,2',3,3',4,4',5,5',6-Nonachlorobiphenyl	PCB-206	6.10	75
Decachlorobiphenyl	PCB-209	12.60	75

Table A.3. Minimum DLs and LOQs for Other Parameters Analyzed in Goal 2 Sediment Samples				
Reference Method/Pace SOP	**Analyte**	**CAS Number**	**DL (µg/kg)**	**LOQ (µg/kg)**
PAHs in Sediment by EPA Methods SW846 3546C/8270C Pace SOPs ENV-GBAY-0077/GBAY-0081	2-Methylnaphthalene	91-57-6	2.44	16.7
	Acenaphthene	83-32-9	2.17	16.7
	Acenaphthylene	208-96-8	2.10	16.7
	Anthracene	120-12-7	2.07	16.7
	Benzo(a)anthracene	56-55-3	2.16	16.7
	Benzo(a)pyrene	50-32-8	1.90	16.7
	Benzo(b)fluoranthene	205-99-2	2.32	16.7
	Benzo(e)pyrene	192-97-2	1.95	16.7
	Benzo(g,h,i)perylene	191-24-2	2.93	16.7
	Benzo(k)fluoranthene	207-08-9	2.31	16.7
	Chrysene	218-01-9	3.15	16.7
	Dibenz(a,h)anthracene	53-70-3	2.31	16.7
	Fluoranthene	206-44-0	1.98	16.7
	Fluorene	86-73-7	2.00	16.7
	Indeno(1,2,3-cd)pyrene	193-39-5	3.48	16.7
	Naphthalene	91-20-3	1.63	16.7
	Phenanthrene	85-01-8	1.9	16.7
	Pyrene	129-00-0	2.45	16.7
Metals in Sediment by EPA Methods 6010D/200.7 Pace SOP ENV-GBAY-0009	Arsenic (As)	7440-38-2	1.47	2.50
	Barium (Ba)	7440-39-3	0.15	0.50
	Cadmium (Cd)	7440-43-9	0.13	0.50
	Calcium (Ca)	7440-70-2	14.33	50.00
	Chromium (Cr)	7440-47-3	0.28	1.00
	Copper (Cu)	7440-50-8	0.28	1.00
	Iron (Fe)	7439-89-6	3.16	10.00
	Lead (Pb)	7439-92-1	0.60	2.00
	Magnesium (Mg)	7439-95-4	18.42	100.00
	Manganese (Mn)	7439-96-5	0.19	0.50
	Nickel (Ni)	7440-02-0	0.27	1.00
	Selenium (Se)	7782-49-2	1.31	4.00
	Silver (Ag)	7440-22-4	0.31	1.00
	Zinc (Zn)	7440-66-6	1.20	4.00
Mercury in Sediment EPA Method SW846 7470A/7471B/245.1 Pace SOP ENV-GBAY-0013	Mercury (Hg)	7439-97-6	0.010	0.035

Table A.3. Minimum DLs and LOQs for Other Parameters Analyzed in Goal 2 Sediment Samples

Reference Method/Pace SOP	Analyte	CAS Number	DL (µg/kg)	LOQ (µg/kg)
Total Organic Carbon in Sediment Lloyd Khan Method Pace SOP ENV-GBAY-0051	TOC	7440-44-0	50.55	100.0

Attachment B. Summary of QC Results

Table B.1. Summary of QC results for PFAS in water and sediment samples

QC result	Batch WJ08059 (SE1)		Batch WL01091 (SE2)	
	Water	Sediment	Water	Sediment
Field sample % surrogate recoveries (surr rec)	Passed the criteria	Passed the criteria	Passed the criteria	Passed the criteria
Method blank sample analyte results, % surr rec	Passed the criteria	Passed the criteria	Passed the criteria	Passed the criteria
Lab control sample % analyte rec, % surr rec	Passed the criteria	Passed the criteria	Passed the criteria	Passed the criteria
Lab duplicate sample analyte RPDs, % surr rec	analyte RPDs passed the criteria; all % surr rec passed the criteria except $^{13}C_2$_6:2 FTS		analyte RPDs passed the criteria except PFPeA[a]; all % surr rec passed the criteria	
Matrix spike sample % analyte rec, % surr rec	% analyte rec – acceptance limit exceeded for 6:2 FTS[b]; % surr rec – $^{13}C_2$_4:2 FTS and $^{13}C_2$_6:2 FTS outside acceptance limits		% analyte rec passed the criteria; % surr rec passed the criteria	

[a] Lab duplicate sample was not a split of a Goal 2 field sample.

[b] The Kent Lake sample was fortified to prepare the matrix spike sample.

ND = Not detected at a concentration greater than the DL.

RPD = relative percent difference.

Table B.2. Summary of QC results for PFAS in biota samples			
QC result	**Batch 40234558**	**Batch 40237220**	**Batch 40238788**
Lab method blank sample analyte results	Passed the criteria	Passed the criteria	Passed the criteria
Lab control sample % recovery (% rec) of analytes	Passed the criteria	Passed the criteria	Passed the criteria except PFODA, which was not detected in any Goal 2 samples analyzed in the batch
Matrix spike sample % rec of analytes	% rec of several analytes was elevated	% rec of several analytes was elevated or diminished. However, the RPDs between the matrix spike and matrix spike duplicate samples passed the criteria	Not reported because matrix spike sample was prepared using a sample from a different project
Injection internal standard (IIS) % rec	Passed the criteria	Passed the criteria with the exception of $^{13}C_2$_PFDA in sample #40237220001	Passed the criteria in the Goal 2 samples analyzed in this batch
Surrogate/extracted internal standard/isotope dilution standard (SUR/EIS/IDS) % rec	Elevated % rec for multiple SUR/EIS/IDS compounds in all samples, including anomalously high recoveries for $^{13}C_2$_4:2 FTS, $^{13}C_2$_6:2 FTS, $^{13}C_2$_8:2 FTS. The results for these native compounds should be considered estimated.	Elevated/diminished % rec for multiple SUR/EIS/IDS compounds in all samples. The result for 6:2 FTS in sample #40237220001 should be considered estimated due to anomalously high % rec of $^{13}C_2$_6:2 FTS.	Diminished rec for one SUR/EIS/IDS compound in each of two Goal 2 samples analyzed in this batch

A summary of the QC results for analysis of PCBs in sediment samples follows:

- Recoveries of the isotopically-labeled PCB internal standards in the sample extracts ranged from 50 to 144%, passing the method criteria. Since the quantification of the native congeners was based on isotope dilution and internal standard methodology, the data were corrected for recovery to obtain accurate results.
- Incorrect isotope ratios were obtained for selected PCB congeners. The affected congeners were flagged "I" on the results table.
- Results for selected PCB congeners were derived from the analysis of diluted sample extracts due to retention time shift in the primary run, and the affected values were flagged "DN2" on the results tables.
- A laboratory method blank was prepared and analyzed with the sample batch as part of the lab's routine quality control procedures. The results show the blank to contain a trace level of congener #66. The sample extracts contained this congener at levels over ten times higher than seen in the method blank. This indicates that the sample processing procedures did not significantly contribute to the PCB content determined for the sample material.
- Laboratory control samples were prepared using reference material that was fortified with native standards. The spiked native compounds were recovered at 93 to 104%, with relative percent differences of 0.0 to 5.9%, passing the criteria.
- A matrix spike sample was not prepared and analyzed using a Goal 2 sample.

Attachment C. Results of PCB congener analysis of sediments

Table C.1. Proud Lake and Kent Lake Sediment PCB Congener Results				
		Sample Location	Proud Lake	Kent Lake
		Lab Sample #	40234540002	40234540004
PCB Congener	**IUPAC #**	**CAS#**	**Result (µg/kg dry weight)**	
2-Chlorobiphenyl	PCB-1	2051-60-7	ND	0.0661
3-Chlorobiphenyl	PCB-2	2051-61-8	0.0250 (J)	0.0605 (J)
4-Chlorobiphenyl	PCB-3	2051-62-9	0.0153 (J)	0.208
2,2'-Dichlorobiphenyl	PCB-4	13029-08-8	ND (DN2)	0.0773 EMPC (I, J, DN2)
2,3-Dichlorobiphenyl	PCB-5	16605-91-7	ND (DN2)	ND (DN2)
2,3'-Dichlorobiphenyl	PCB-6	25569-80-6	ND (DN2)	0.300 (DN2)
2,4-Dichlorobiphenyl	PCB-7	33284-50-3	ND (DN2)	0.0899 (J, DN2)
2,4'-Dichlorobiphenyl	PCB-8	34883-43-7	0.0507 (J, DN2)	2.510 (DN2)
2,5-Dichlorobiphenyl	PCB-9	34883-39-1	ND (DN2)	0.0694 (J, DN2)
2,6-Dichlorobiphenyl	PCB-10	33146-45-1	ND (DN2)	ND (DN2)
3,3'-Dichlorobiphenyl	PCB-11	2050-67-1	ND (DN2)	ND (DN2)
3,4'-; 3,4-Dichlorobiphenyl	PCB-13/12	2974-90-5 2974-92-7	ND (DN2)	0.574 (DN2)
3,5-Dichlorobiphenyl	PCB-14	34883-41-5	ND (DN2)	ND (DN2)
4,4'-Dichlorobiphenyl	PCB-15	2050-68-2	0.110 (DN2)	1.010 (DN2)
2,2',3-Trichlorobiphenyl	PCB-16	38444-78-9	0.0376 (J, DN2)	0.343
2,2',4-Trichlorobiphenyl	PCB-17	37680-66-3	0.0429 (J, DN2)	0.717
2,4,6-; 2,2',5-Trichlorobiphenyl	PCB-30/18	35693-92-6 37680-65-2	ND (DN2)	0.150
2,2',6-Trichlorobiphenyl	PCB-19	38444-73-4	ND (DN2)	0.0553

Table C.1. Proud Lake and Kent Lake Sediment PCB Congener Results				
		Sample Location	Proud Lake	Kent Lake
		Lab Sample #	40234540002	40234540004
PCB Congener	IUPAC #	CAS#	Result (µg/kg dry weight)	
2,4,4'-; 2,3,3'-Trichlorobiphenyl	PCB-28/20	7012-37-5 38444-84-7	0.379 (DN2)	11.300
2,3,4-; 2,3',4'-Trichlorobiphenyl	PCB-21/33	55702-46-0 38444-86-9	0.159 (J, DN2)	8.540
2,3,4'-Trichlorobiphenyl	PCB-22	38444-85-8	ND (DN2)	1.69
2,3,5-Trichlorobiphenyl	PCB-23	55720-44-0	ND (DN2)	ND
2,3,6-Trichlorobiphenyl	PCB-24	55702-45-9	ND (DN2)	0.0204 (J)
2,3',4-Trichlorobiphenyl	PCB-25	55712-37-3	0.0193 (J, DN2)	0.537
2,3',5-; 2,4,5-Trichlorobiphenyl	PCB-26/29	38444-81-4 15862-07-4	ND (DN2)	0.660
2,3',6-Trichlorobiphenyl	PCB-27	38444-76-7	ND (DN2)	0.0860
2,4',5-Trichlorobiphenyl	PCB-31	16606-02-3	0.193 (DN2)	4.13
2,4',6-Trichlorobiphenyl	PCB-32	38444-77-8	ND (DN2)	0.395
2',3,5-Trichlorobiphenyl	PCB-34	37680-68-5	ND (DN2)	0.182
3,3',4-Trichlorobiphenyl	PCB-35	37680-69-6	ND (DN2)	0.0483 (J)
3,3',5-Trichlorbiphenyl	PCB-36	38444-87-0	ND (DN2)	ND
3,4,4'-Trichlorobiphenyl	PCB-37	38444-90-5	0.227 (DN2)	4.06
3,4,5-Trichlorobiphenyl	PCB-38	53555-66-1	ND (DN2)	ND
3,4',5-Trichlorobiphenyl	PCB-39	38444-88-1	ND (DN2)	0.0441 (J)

Table C.1. Proud Lake and Kent Lake Sediment PCB Congener Results				
		Sample Location	Proud Lake	Kent Lake
		Lab Sample #	40234540002	40234540004
PCB Congener	IUPAC #	CAS#	Result (µg/kg dry weight)	
2,2',3,4-; 2,2',3,3'-; 2,3',4',6-Tetrachlorobiphenyl	PCB-41/40/71	52663-59-9 38444-93-8 41464-46-4	0.124 (J)	4.93
2,2',3,4'-Tetrachlorobiphenyl	PCB-42	36559-22-5	0.0767	2.99
2,2',3,5-Tetrachlorobiphenyl	PCB-43	70362-46-8	ND	0.292
2,2',3,5'-; 2,2',4,4'-; 2,3,5,6-Tetrachlorobiphenyl	PCB-44/47/65	41464-39-5 2437-79-8 33284-54-7	0.317	11.1
2,2',3,6-; 2,2',4,6'-Tetrachlorobiphenyl	PCB-45/51	70362-45-7 68194-04-7	0.0436 (J)	0.994
2,2',3,6'-Tetrachlorobiphenyl	PCB-46	41464-47-5	0.0136 (J)	0.329
2,2',4,5-Tetrachlorobiphenyl	PCB-48	70362-47-9	0.0559	1.94
2,3',4,6-; 2,2',4,5'-Tetrachlorobiphenyl	PCB-69/49	60233-24-1 41464-40-8	0.196	7.99
2,2',4,6-; 2,2',5,6'-Tetrachlorobiphenyl	PCB-50/53	62796-65-0 41464-41-9	0.0309 (J)	0.872
2,2',5,5'-Tetrachlorobiphenyl	PCB-52	35693-99-3	0.389	12.8
2,2',6,6'-Tetrachlorbiphenyl	PCB-54	15968-05-5	ND	ND

Table C.1. Proud Lake and Kent Lake Sediment PCB Congener Results				
		Sample Location	Proud Lake	Kent Lake
		Lab Sample #	40234540002	40234540004
PCB Congener	IUPAC #	CAS#	Result (µg/kg dry weight)	
2,3,3',4-Tetrachlorobiphenyl	PCB-55	74338-24-2	ND	ND
2,3,3',4'-Tetrachlorobiphenyl	PCB-56	41464-43-1	0.215	8.11
2,3,3',5-Tetrachlorobiphenyl	PCB-57	70424-67-8	ND	0.0309 (J)
2,3,3',5'-Tetrachlorobiphenyl	PCB-58	41464-49-7	ND	0.0895
2,3,3',6-; 2,3,4,6-; 2,4,4',6-Tetrachlorobiphenyl	PCB-59/62/75	74472-33-6 54230-22-7 32598-12-2	0.0335 (J)	0.950
2,3,4,4'-Tetrachlorobiphenyl	PCB-60	33025-41-1	0.0898	2.88
2,3,4,5-; 2,3',4',5-; 2,4,4',5-; 2,3',4',5'-Tetrachlorobiphenyl	PCB-61/70/74/76	33284-53-6 32598-11-1 32690-93-0 70362-48-0	0.325	18.0
2,3,4',5-Tetrachlorobiphenyl	PCB-63	74472-34-7	ND	0.670
2,3,4',6-Tetrachlorobiphenyl	PCB-64	52663-58-8	0.112	5.38
2,3',4,4'-Tetrachlorobiphenyl	PCB-66	32598-10-0	0.408	21.4
2,3',4,5-Tetrachlorobiphenyl	PCB-67	73575-53-8	ND	0.184

Table C.1. Proud Lake and Kent Lake Sediment PCB Congener Results				
		Sample Location	Proud Lake	Kent Lake
		Lab Sample #	40234540002	40234540004
PCB Congener	IUPAC #	CAS#	Result (µg/kg dry weight)	
2,3',4,5'-Tetrachlorobiphenyl	PCB-68	73575-52-7	ND	0.136
2,3',5,5'-Tetrachlorobiphenyl	PCB-72	41464-42-0	ND	0.0824
2,3',5',6-Tetrachlorobiphenyl	PCB-73	74338-23-1	ND	ND
3,3',4,4'-Tetrachlorobiphenyl	PCB-77	32598-13-3	0.0883	1.54
3,3',4,5-Tetrachlorobiphenyl	PCB-78	70362-49-1	ND	ND
3,3',4,5'-Tetrachlorobiphenyl	PCB-79	41464-48-6	0.00783 (J)	0.145
3,3',5,5'-Tetrachlorobiphenyl	PCB-80	33284-52-5	ND	ND
3,4,4',5-Tetrachlorobiphenyl	PCB-81	70362-50-4	ND	0.0395 (J)
2,2',3,3',4-Pentachlorobiphenyl	PCB-82	52663-62-4	0.0179 (J)	0.528
2,2',3,3',5-Pentachlorobiphenyl	PCB-83	60145-20-2	0.0358 (J)	0.763
2,2',3,3',6-Pentachlorobiphenyl	PCB-84	52663-60-2	0.0617	2.43
2,3,4',5,6-; 2,3,4,5,6-; 2,2',3,4,4'-Pentachlorobiphenyl	PCB-117/116/85	68194-11-6 18259-05-7 65510-45-4	0.137	3.51

Table C.1. Proud Lake and Kent Lake Sediment PCB Congener Results				
		Sample Location	Proud Lake	Kent Lake
		Lab Sample #	40234540002	40234540004
PCB Congener	IUPAC #	CAS#	Result (µg/kg dry weight)	
2,3,3',4,5'-; 2,3',4,4',6-; 2,2',3,4,5-; 2,2',3,4',5'-; 2,3',4',5',6-; 2,2',3,4,5'-Pentachlorobiphenyl	PCB-108/119/86/97/125/87	70362-41-3 56558-17-9 55312-69-1 41464-51-1 74472-39-2 38380-02-8	0.345	8.07
2,2',3,4,6-; 2,2',3,4',6-Pentachlorobiphenyl	PCB-88/91	55215-17-3 68194-05-8	0.0343 (J)	1.80
2,2',3,4,6'-Pentachlorobiphenyl	PCB-89	73575-57-2	ND	0.234
2,3,3',5',6-; 2,2',3,4',5-; 2,2',4,5,5'-Pentachlorobiphenyl	PCB-113/90/101	68194-10-5 68194-07-0 37680-73-2	0.475	11.6
2,2',3,5,5'-Pentachlorobiphenyl	PCB-92	52663-61-3	0.115	2.40
2,2',4,4',6-; 2,2',3,5,6-; 2,2',4,5,6'-; 2,2',3,4',6'-Pentachlorobiphenyl	PCB-100/93/102/98	39485-83-1 73575-56-1 68194-06-9 60233-25-2	ND	0.565
2,2',3,5,6'-Pentachlorobiphenyl	PCB-94	73575-55-0	ND	0.0548 (J)
2,2',3,5',6-Pentachlorobiphenyl	PCB-95	38379-99-6	0.141	6.38

Table C.1. Proud Lake and Kent Lake Sediment PCB Congener Results				
		Sample Location	Proud Lake	Kent Lake
		Lab Sample #	40234540002	40234540004
PCB Congener	IUPAC #	CAS#	Result (µg/kg dry weight)	
2,2',3,6,6'-Pentachlorobiphenyl	PCB-96	73575-54-9	ND	0.104
2,2',4,4',5-Pentachlorobiphenyl	PCB-99	38380-01-7	0.255	7.91
2,2',4,5',6-Pentachlorobiphenyl	PCB-103	60145-21-3	ND	0.122
2,2',4,6,6'-Pentachlorobiphenyl	PCB-104	56558-16-8	ND	ND
2,3,3',4,4'-Pentachlorobiphenyl	PCB-105	32598-14-4	0.254	5.18
2,3,3',4,5-Pentachlorobiphenyl	PCB-106	70424-69-0	ND	ND
2,3,3',4',5-; 2,3',4',5,5'-Pentachlorobiphenyl	PCB-107/124	70424-68-9 70424-70-3	0.0231 (J)	0.248
2,3,3',4,6-Pentachlorobiphenyl	PCB-109	74472-35-8	0.0452	0.897
2,3,3',4',6-; 2,3,4,4',6-Pentachlorobiphenyl	PCB-110/115	38380-03-9 74472-38-1	0.611	14.8
2,3,3',5,5'-Pentachlorobiphenyl	PCB-111	39635-32-0	ND	ND
2,3,3',5,6-Pentachlorobiphenyl	PCB-112	74472-36-9	ND	0.0232 (J)
2,3,4,4',5-Pentachlorobiphenyl	PCB-114	74472-37-0	0.0102 (J)	0.207
2,3',4,4',5-Pentachlorobiphenyl	PCB-118	31508-00-6	0.536	10.5

Table C.1. Proud Lake and Kent Lake Sediment PCB Congener Results				
		Sample Location	Proud Lake	Kent Lake
		Lab Sample #	40234540002	40234540004
PCB Congener	IUPAC #	CAS#	Result (µg/kg dry weight)	
2,3',4,5,5'-Pentachlorobiphenyl	PCB-120	68194-12-7	ND	0.0644 (J)
2,3',4,5',6-Pentachlorobiphenyl	PCB-121	56558-18-0	ND	ND
2,3,3',4',5'-Pentachlorobiphenyl	PCB-122	76842-07-4	0.0112 (J)	0.200
2,3',4,4',5'-Pentachlorobiphenyl	PCB-123	65510-44-3	0.0138 (J)	0.265
3,3',4,4',5-Pentachlorobiphenyl	PCB-126	57465-28-8	ND	0.0459 (J)
3,3',4,5,5'-Pentachlorobiphenyl	PCB-127	39635-33-1	ND	0.0145 (J)
2,2',3,3',4,4'-; 2,3,4,4',5,6-Hexachlorobiphenyl	PCB-128/166	38380-07-3 41411-63-6	0.119	1.31
2,2',3,4,4',5'-; 2,3,3',4',5,6-; 2,2',3,3',4,5-Hexachlorobiphenyl	PCB-138/163/129	35065-28-2 74472-44-9 55215-18-4	0.769	7.60
2,2',3,3',4,5'-Hexachlorobiphenyl	PCB-130	52663-66-8	0.0561	0.626
2,2'3,3',4,6-Hexachlorobiphenyl	PCB-131	61798-70-7	ND	0.0593 (J)
2,2',3,3',4,6'-Hexachlorobiphenyl	PCB-132	38380-05-1	0.175	2.15
2,2',3,3',5,5'-Hexachlorobiphenyl	PCB-133	35694-04-3	0.0158 (J)	0.140

Table C.1. Proud Lake and Kent Lake Sediment PCB Congener Results				
		Sample Location	Proud Lake	Kent Lake
		Lab Sample #	40234540002	40234540004
PCB Congener	IUPAC #	CAS#	Result (µg/kg dry weight)	
2,2',3,3',5,6-; 2,2',3,4,5,6'-Hexachlorobiphenyl	PCB-134/143	52704-70-8 68194-15-0	0.0286 (J)	0.378
2,2',3,5,5',6-; 2,2',3,3',5,6'-Hexachlorobiphenyl	PCB-151/135	52663-63-5 52744-13-5	0.203	2.01
2,2',3,3',6,6'-Hexachlorobiphenyl	PCB-136	38411-22-2	0.0557	0.683
2,2',3,4,4',5-Hexachlorobiphenyl	PCB-137	35694-06-5	0.0245 (J)	0.334
2,2',3,4,4',6-; 2,2',3,4,4',6'-Hexachlorobiphenyl	PCB-139/140	56030-56-9 59291-64-4	ND	0.149
2,2',3,4,5,5'-Hexachlorobiphenyl	PCB-141	52712-04-6	0.115	0.967
2,2',3,4,5,6-Hexachlorobiphenyl	PCB-142	41411-61-4	ND	ND
2,2',3,4,5',6-Hexachlorobiphenyl	PCB-144	68194-14-9	0.0257 (J)	0.235
2,2',3,4,6,6'-Hexachlorobiphenyl	PCB-145	74472-40-5	ND	ND
2,2',3,4',5,5'-Hexachlorobiphenyl	PCB-146	51908-16-8	0.125	1.33
2,2',3,4',5,6-; 2,2',3,4',5',6-Hexachlorobiphenyl	PCB-147/149	68194-13-8 38380-04-0	0.436	4.81
2,2',3,4',5,6'-Hexachlorobiphenyl	PCB-148	74472-41-6	ND	ND

Table C.1. Proud Lake and Kent Lake Sediment PCB Congener Results				
		Sample Location	Proud Lake	Kent Lake
		Lab Sample #	40234540002	40234540004
PCB Congener	IUPAC #	CAS#	Result (µg/kg dry weight)	
2,2',3,4',6,6'-Hexachlorobiphenyl	PCB-150	68194-08-1	ND	ND
2,2',3,5,6,6'-Hexachlorobiphenyl	PCB-152	68194-09-2	ND	ND
2,2',4,4',5,5'-; 2,3',4,4',5',6-Hexachlorobiphenyl	PCB-153/168	35065-27-1 59291-65-5	0.629	6.37
2,2'4,4',5,6'-Hexachlorobiphenyl	PCB-154	60145-22-4	0.00968 (J)	0.149
2,2',4,4',6,6'-Hexachlorobiphenyl	PCB-155	33979-03-2	ND	ND
2,3,3',4,4',5-; 2,3,3',4,4',5'-Hexachlorobiphenyl	PCB-156/157	38380-08-4 69782-90-7	0.0826 (J)	0.982
2,3,3',4,4',6-Hexachlorobiphenyl	PCB-158	74472-42-7	0.0576	0.469
2,3,3',4,5,5'-Hexachlorobiphenyl	PCB-159	39635-35-3	ND	0.0362 (J)
2,3,3',4,5,6-Hexachlorobiphenyl	PCB-160	41411-62-5	ND	ND
2,3,3',4,5',6-Hexachlorobiphenyl	PCB-161	74472-43-8	ND	ND
2,3,3',4',5,5'-Hexachlorobiphenyl	PCB-162	39635-34-2	ND	0.0419 (J)
2,3,3',4',5',6-Hexachlorobiphenyl	PCB-164	74472-45-0	0.0537	0.552
2,3,3',5,5',6-Hexachlorobiphenyl	PCB-165	74472-46-1	ND	ND

Table C.1. Proud Lake and Kent Lake Sediment PCB Congener Results				
		Sample Location	Proud Lake	Kent Lake
		Lab Sample #	40234540002	40234540004
PCB Congener	IUPAC #	CAS#	Result (µg/kg dry weight)	
2,3',4,4',5,5'-Hexachlorobiphenyl	PCB-167	52663-72-6	0.0343 (J)	0.274
3,3',4,4',5,5'-Hexachlorobiphenyl	PCB-169	32774-16-6	ND	ND
2,2',3,3',4,4',5-Heptachlorobiphenyl	PCB-170	35065-30-6	0.208	1.80
2,2',3,3',4,4',6-; 2,2',3,3',4,5,6-Heptachlorobiphenyl	PCB-171/173	52663-71-5 68194-16-1	0.0597 (J)	0.492
2,2'3,3',4,5,5'-Heptachlorobiphenyl	PCB-172	52663-74-8	0.0437	0.331
2,2',3,3',4,5,6'-Heptachlorobiphenyl	PCB-174	38411-25-5	0.188	1.35
2,2',3,3',4,5',6-Heptachlorobiphenyl	PCB-175	40186-70-7	0.00855 (J)	0.0625 (J)
2,2',3,3',4,6,6'-Heptachlorobiphenyl	PCB-176	52663-65-7	0.0197 (J)	0.155
2,2'3,3',4,5',6'-Heptachlorobiphenyl	PCB-177	52663-70-4	0.126	1.01
2,2',3,3',5,5',6-Heptachlorobiphenyl	PCB-178	52663-67-9	0.0504	0.355
2,2',3,3',5,6,6'-Heptachlorobiphenyl	PCB-179	52663-64-6	0.0676	0.469
2,2',3,4,4',5,5'-; 2,3,3',4',5,5',6-Heptachlorobiphenyl	PCB-180/193	35065-29-3 69782-91-8	0.456	3.31
2,2',3,4,4',5,6-Heptachlorbiphenyl	PCB-181	74472-47-2	ND	ND

Table C.1. Proud Lake and Kent Lake Sediment PCB Congener Results				
		Sample Location	Proud Lake	Kent Lake
		Lab Sample #	40234540002	40234540004
PCB Congener	IUPAC #	CAS#	Result (µg/kg dry weight)	
2,2',3,4,4',5,6'-Heptachlorobiphenyl	PCB-182	60145-23-5	ND	0.0283 (J)
2,2',3,4,4',5',6-; 2,2',3,4,5,5',6-Heptachlorobiphenyl	PCB-183/185	52663-69-1 52712-05-7	0.128	0.956
2,2',3,4,4',6,6'-Heptachlorobiphenyl	PCB-184	74472-48-3	ND	ND
2,2',3,4,5,6,6'-Heptachlorobiphenyl	PCB-186	74472-49-4	ND	ND
2,2',3,4',5,5',6-Heptachlorobiphenyl	PCB-187	52663-68-0	0.281	1.80
2,2',3,4',5,6,6'-Heptachlorobiphenyl	PCB-188	74487-85-7	ND	ND
2,3,3',4,4',5,5'-Heptachlorobiphenyl	PCB-189	39635-31-9	ND	0.0893
2,3,3',4,4',5,6-Heptachlorobiphenyl	PCB-190	41411-64-7	0.0410 (J)	0.334
2,3,3',4,4',5',6-Heptachlorobiphenyl	PCB-191	74472-50-7	ND	0.0494 (J)
2,3,3',4,5,5',6-Heptachlorobiphenyl	PCB-192	74472-51-8	ND	ND
2,2',3,3',4,4',5,5'-Octachlorobiphenyl	PCB-194	35694-08-7	0.120	0.837
2,2',3,3',4,4',5,6-Octachlorobiphenyl	PCB-195	52663-78-2	0.0592	0.394
2,2',3,3',4,4',5,6'-Octachlorobiphenyl	PCB-196	42740-50-1	0.0692	0.425
2,2',3,3',4,4',6,6'-; 2,2',3,3',4,5,6,6'-Octachlorobiphenyl	PCB-197/200	33091-17-7 52663-73-7	0.0233 (J)	0.123 (J)

Table C.1. Proud Lake and Kent Lake Sediment PCB Congener Results				
		Sample Location	**Proud Lake**	**Kent Lake**
		Lab Sample #	**40234540002**	**40234540004**
PCB Congener	**IUPAC #**	**CAS#**	**Result (µg/kg dry weight)**	
2,2',3,3',4,5,5',6-; 2,2',3,3',4,5,5',6'-Octachlorobiphenyl	PCB-198/199	68194-17-2 52663-75-9	0.165	0.910
2,2',3,3',4,5',6,6'-Octachlorobiphenyl	PCB-201	40186-71-8	0.174 (J)	0.0837
2,2',3,3',5,5',6,6'-Octachlorobiphenyl	PCB-202	2136-99-4	0.0329 (J)	0.149
2,2',3,4,4',5,5',6-Octachlorobiphenyl	PCB-203	52663-76-0	0.0969	0.512
2,2',3,4,4',5,6,6'-Octachlorobiphenyl	PCB-204	74472-52-9	ND	ND
2,3,3',4,4',5,5',6-Octachlorobiphenyl	PCB-205	74472-53-0	ND	0.0427 (J)
2,2',3,3',4,4',5,5',6-Nonachlorobiphenyl	PCB-206	40186-72-9	0.085	0.280
2,2'3,3',4,4',5,6,6'-Nonachlorobiphenyl	PCB-207	52663-79-3	0.0122 (J)	0.0385 EMPC (I, J)
2,2'3,3',4,4',5,5',6,6'-Nonachlorobiphenyl	PCB-208	52663-77-1	0.0274 (J)	0.0819
Decachlorobiphenyl	PCB-209	2051-24-3	0.0711	0.143
		Total PCBs (EMPC=0)	12.4	269
		Total PCBs (EMPC=Result)	12.4	269

DN2 = Values obtained from analyses of a diluted sample extract. Results were taken from secondary analyses due to retention time shift in the primary run.
EMPC = Estimated maximum possible concentration.
I = Interference present, evidenced by incorrect isotope ratios.
J = Estimated concentration below the LOQ but above the DL See Appendix I for reported LOQs and Appendix II for reported DLs.
ND = Not detected at a concentration greater than the DL. ND results were treated as 0 (zero) concentration in the Total PCB summation.

Attachment D. Muscle to whole body ratios

A muscle to whole body (M/WB) ratio for PFOS was calculated for freshwater fish. Paired muscle (M) and whole body (WB) tissue PFOS concentrations were available for six freshwater fish species from three studies (Lescord et al. 2015, Shi et al. 2015, Valsecchi et al. 2021). Muscle and whole PFOS concentrations (ng/g wet weight) were reported as site level averages in all studies. For each species, a M/WB ratio was calculated at each site, and then the average of those site-level ratios was calculated to represent the species-level M/WB ratio. A final freshwater fish M/WB ratio was calculated as the average of the six species-level M/WB ratios (Table D.1). Across species, PFOS M/WB ratios ranged from 0.245 to 0.535, with an average ratio of 0.396 (Table D.1).

Table D.1. Muscle to whole body (M/WB) conversion factors in freshwater fish species

Species	Site	M PFOS (ng/g)	WB PFOS (ng/g)	M/WB	Average M/WB	Reference
Char (*Salvelinus alpinus*)	Meretta Lake	77	181	0.425	0.245	Lescord et al. 2015
	Resolute Lake	27	224	0.121		
	Char Lake	0.54	1.5	0.360		
	Small Lake	0.06	0.80	0.075		
Crucian carp (*Carassius carassius*)	Tangxum Lake	180[a]	477[a]	0.378	0.467	Shi et al. 2015
	Xiaoqing River	3.29[a]	5.91[a]	0.556		
Burbot (*Lota lota*)	Lake Geneva	8.88	16.58	0.535	0.535	Valsecchi et al. 2021
Roach (*Rutilus rutilus*)	Lake Geneva	10.78	20.59	0.513	0.513	Valsecchi et al. 2021
Brown trout (*Salmo trutta*)	Lake Iseo	0.71	2.43	0.293	0.293	Valsecchi et al. 2021
Shad (*Alosa agone*)	Lake Maggiore	4.07	15.56	0.286	0.324	Valsecchi et al. 2021
	Lake Como	1.87	6.05	0.297		
	Lake Iseo	3.47	10.35	0.370		
	Lake Garda	6.05	17.26	0.342		
Six fish species average					0.396	

[a] Calculated from author-reported water concentration and tissue-specific BAF.

APPENDICES

APPENDIX I

Goal 2 Compiled Results
(sent as a separate Excel files)

APPENDIX II

Pace Analytical Reports of Analysis by Batch Number
(sent as a separate pdf files)