Evaluation of Per- and Polyfluoroalkyl Substances in the Indiana Portions of the Great Lake Basins

Quality Assurance Project Plan

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Grant #: 00E02913

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QA Staff
Office of Program Support
Date 02 Mar 2021

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List of Acronyms

AIMS	Assessment Information Management System
ALUS	Aquatic Life Use Support
CAS	Chemical Abstract Service
CLP	Contract Laboratory Program
CRQL	Contract-Required Quantification Limit
CWA	Clean Water Act
DFW	Division of Fish and Wildlife
DQA	Data Quality Assessment
DQO	Data Quality Objective
FCA	Fish Consumption Advisory
IAC	Indiana Administrative Code
IDNR	Indiana Department of Natural Resources
ISDH	Indiana State Department of Health
IUPAC	International Union of Pure and Applied Chemistry
OWQ	Office of Water Quality
PCB	Polychlorinated biphenyl
PI	Principal Investigator
QA	Quality Assurance
QA/QC	Quality Assurance and Quality Control
QAPP	Quality Assurance Project Plan
QC	Quality Control
RFP	Request for Proposals
SOP	Standard Operating Procedure
U.S. EPA	United States Environmental Protection Agency
WAPB	Watershed Assessment and Planning Branch
WQMS	Indiana Water Quality Monitoring Strategy
WW	Wet Weight

Definitions

Bioaccumulate	To accumulate a substance, such as a toxic chemical, in various tissues of a living organism.
Co-jurisdictional waters	According to 312 IAC 5-2-47, the Indiana waters of Lake Michigan, the Ohio River, the Wabash River (where it forms the Indiana-Illinois border), and the Great Miami River.
Contaminant	A biological, chemical, physical, or radiological substance which, in sufficient concentration, can adversely affect living organisms through air, water, soil, or food.
Dry weight	The weight of the sample, corrected for the moisture content.
Emerging Contaminant	New compounds or molecules which were not previously known or were just recently appeared in the scientific literature; Contaminants of emerging interest which were known to exist but for which the environmental contamination issues were not fully realized or apprehended; Emerging issues about old (legacy) contaminants (i.e., situations where new information is jostling our understanding of environmental and human health risks related to legacy contaminants) (Sauvé and Desrosiers 2014).
Fillet	The flesh of the fish, which is composed of the skeletal muscles and fat, as opposed to the bones and internal organs.
Composite Sample	A fish tissue sample comprised of two or more individual organisms of the same species collected at a particular site, of similar size (smallest individual within the composite is no less than 75% of the total length of the largest individual), and analyzed as a single sample.
Hydrologic Unit Code (HUC)	A numeric U.S. Geological Survey code which corresponds to a watershed area. Each area also has a text description associated with the

	numeric code and based on the area of land which drains into a hydrologic feature such as a stream, river, or lake.
Legacy Contaminant	Pollutants, often used or produced by industry, which remain in the environment long after they were first introduced (Smith and Young 2009).
Piscivorous	A carnivorous animal which eats primarily fish.
Total Length	A measurement from the anterior-most part of the fish to the longest caudal fin ray when the lobes of the caudal fin are compressed dorsoventrally (U.S. EPA 2000).
Waters of the state	As defined by Indiana Code 14-8-2-307, a lake; reservoir; marsh; waterway; other water under public ownership, jurisdiction, or lease; or has been used by the public with the acquiescence of any or all riparian owners.
Watershed	An area or region drained by a river, river system or other body of water.
Wet weight	The as-is weight, which includes the solid and liquid portion of the sample.

QAPP Rationale

This Quality Assurance Project Plan (QAPP) is being constructed for the monitoring of per- and polyfluoroalkyl substances (PFAS) and other legacy contaminants at 49 sites in the Lake Michigan and Lake Erie basins located in Indiana. Great Lakes Restoration Initiative (GLRI) direct funding grant number 00E02913, will fund the analysis for PFAS in fish tissue samples collected under the IDEM 2020 Fish Tissue Contaminants Monitoring Work Plan (2020 Fish Tissue WP), a hybrid QAPP-work plan. The IDEM annually funded Fish Tissue Monitoring Program collects fish tissue and is documented in a hybrid QAPP-work plan. Fish tissue sample collection for 2020 is supported by the 2020 Fish Tissue WP identification number in GLNPO QA Track, IDEM section B-047-OWQ-WAP-TGM-20-W-R0. Additional funding, GLRI funding is awarded to the Indiana Department of Environmental Management (IDEM).

The analyses will characterize the location and magnitude of PFAs in Lake Michigan, Lake Michigan tributaries, and Lake Erie tributaries. This study will increase spatial coverage of PFAs to better understand concentrations, distributions, and contaminant sources of emerging contaminants of concern in the Great Lakes basins while also supporting the Indiana Fish Consumption Advisory.

About PFAS:

PFAS are a group of man-made chemicals which have been manufactured and used in a variety of industries since the 1940s. This class of chemicals is persistent in the environment and have been shown to accumulate in fish tissue and the human body. There is evidence PFAS exposure is associated with chronic, developmental, and reproductive health effects. Epidemiological literature suggests fish is an important source of exposure to PFAS.

A. Project Management

The elements in this group address the basic area of project management, including the project history, objectives, and roles and responsibilities of the participants. These elements ensure the project has a defined goal, the participants understand the goal and the planned approach, and planning outputs are documented.

A.1 Title and Approval Sheet

See cover page.

A.2 Table of Contents

See table of contents.

A.3 Distribution List

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A.4 Project Organization

Figure 1. Principal lines of project communications

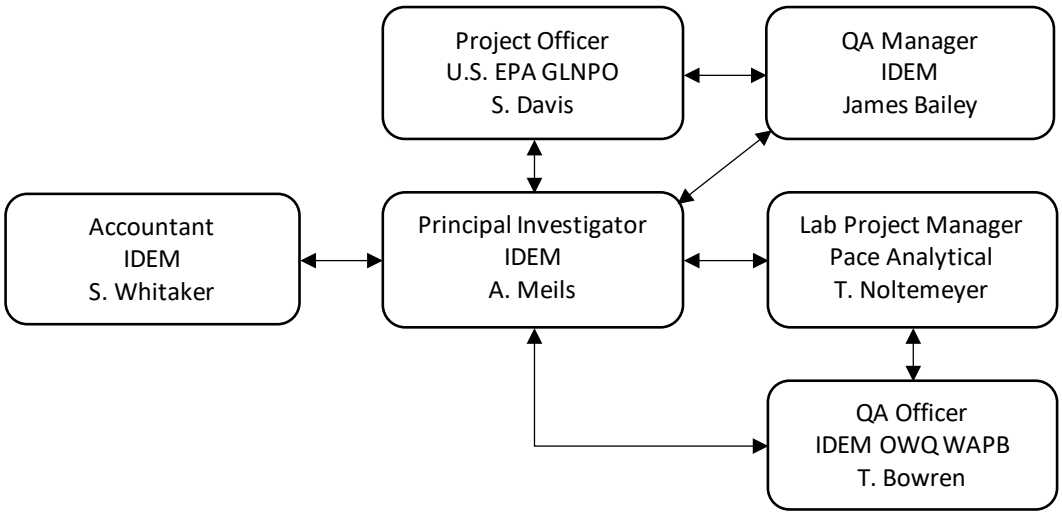


Table 1. Key project partners

Name	Affiliation	Role
Ali Meils	Indiana Department of Environmental Management	Principal Investigator – Overall project oversight, field sampling, U.S. EPA grant reporting, and QAPP maintenance
Stephanie Davis	U.S. Environmental Protection Agency	Project Officer
James Bailey	Indiana Department of Environmental Management	IDEM QA Manager – QAPP review and approval
Tod Noltemeyer	Pace Analytical Services, LLC	Oversight of laboratory analysis

IDEM, as grant recipient, will be responsible for all aspects of project completion. IDEM will ensure subagreements are maintained with required contract laboratories for the length of the project. These subagreements will ensure analysis is conducted in such a manner as to meet the project data quality objectives (DQOs) listed in Section A.7.

Although the key project partners are listed in Table 1, this data would ultimately be made available to decision makers at IDEM and U.S. EPA

Region 5 Great Lakes National Program Office (GLNPO), as well as members of the public.

A.4.1. IDEM QA Staff Responsibility

James Bailey will serve as the IDEM QA Manager and will be responsible for review and approval of this QAPP throughout the project period.

A.4.2. IDEM Principal Investigator Responsibility

Ali Meils, IDEM's Contaminants Monitoring Program manager, will serve as the principal investigator (PI) and project manager. Oversee and track all aspects of the project and be responsible for administering the grant. Specific tasks include:

- Oversight of IDEM OWQ sampling
- Oversight of contract laboratory work
- Budgetary oversight
- Project QA, including QAPP development
- Submittal of project progress and final reports to U.S. EPA.

A.4.3 WAPB QA Responsibilities

The WAPB quality assurance manager (WAPB QAM), Tim Bowren, is responsible for coordinating all QA activities. The quality assurance officers (QAOs), assigned to projects by the WAPB QAM, coordinate and audit QA/QC activities, prepare and review program QAPPs, act as liaison to external laboratories, and report to management on the QA aspects of a project. QA staff perform data validation review, data assessment, data qualification, and internal performance and system audits for projects assigned under the direction of the WAPB QAM. Assigned QA personnel review all work plans and SOPs for compliance with the WAPB 2017 Indiana Surface Water Programs (Surface Water) QAPP (IDEM 2017a). In short, this involves all sample collection conducted for this project.

Each laboratory performing data analysis for the IDEM OWQ is responsible for data validation of results before reporting to IDEM OWQ WAPB or the IDEM PI. IDEM Requests for Proposal (RFPs) require each OWQ contract analytical laboratory, in this case Pace Analytical Services (Pace), have a QAO and a written QA plan. Data from these laboratories is reviewed and QA/QC'd by IDEM's OWQ.

A.4.4. WAPB Field Responsibilities

The PI is responsible for sampling and data collection efforts, and assigning participating staff to data collection duties for this project. The PI ensures participating staff follow this QAPP, WAPB SOPs, and other QA documents in the course of sampling and data collection activities. Any nonconformities are reported to the PI who will collaborate with the QAO and technical staff concerning documenting and addressing the

nonconformity through the corrective action process. Although supervisory responsibility rests with the Targeted Monitoring Section chief, the IDEM PI is ultimately responsible for ensuring all work performed complies with this QAPP.

A.4.5 WAPB Laboratory and Analytical Laboratories Responsibilities

IDEM RFPs set forth requirements and technical specifications for OWQ contract laboratory analysis of water samples for various contaminants or pollutants.

In short, OWQ contract laboratories must have and maintain a documented QA/QC program, capable of demonstrating data have a specified degree of precision and reliability. Laboratories must be able to validate each method used and each analysis performed by the method using the QA/QC program.

QA/QC measures must be documented. All documentation must be maintained and made available for the use by IDEM OWQ for five years after the expiration date of the contract. QA/QC documentation must be submitted as required.

Laboratories must maintain and document continual evaluation of the accuracy and precision of an analytical procedure and the ability of individual analysts to meet laboratory performance for a procedure.

All fish tissue samples will be analyzed by Pace laboratory, which maintains a QA and compliance program (Table 6, Table 7, and Table 8). All OWQ contract laboratories are responsible for complying with the IDEM RFP 16-088 (State of Indiana 2016).

A.4.6 Other Laboratory Monitoring and Oversight

The project grant work plan, this QAPP, the RFPs, and contracts with selected vendors serve a valuable oversight function for this project. The laboratories conducting contaminant analyses must have and maintain a documented QA/QC program sufficient to satisfy the QA/QC requirements set forth in this document. Demonstrations of qualifications to perform the work are part of the RFP process. In addition, QA/QC measures must be documented and provided to IDEM upon request.

The IDEM PI will track project progress through receipt of project progress reports, data reports, and raw data deliverables (Section A.9). In addition, trained and experienced staff at IDEM's WAPB will analyze and validate the data.

A.5 Problem Definition and Background

A.5.1 The Study

Other than select samples from Lake Michigan proper, Indiana has not analyzed fish tissue for PFAS chemicals in the Lake Michigan or Lake Erie basins due to the additional analytical costs. The data collected from this project will be used to support FCA improvements in the basins and inform program areas of potential hotspots in the area. Table 2 contains decision rules concerning total PCBs, total mercury, and PFOS.

A.5.2 The Goal

Understanding emerging contaminants in Indiana wild fish is an objective of fish tissue monitoring. To further support this objective the GLRI funding is targeted for exploring the Great Lakes basins PFAS concentrations in fish tissue. The State of Indiana will utilize the data collected during this project to better understand PFAS patterns and distributions in the region, as well as, use the data in the Indiana Fish Consumption Advisory to inform Indiana wild caught fish consumers (Table 2).

A.5.3. Introduction

Fish tissue and sediment contaminants monitoring in Indiana has been conducted regularly since the early 1970s. First by the Indiana State Board of Health and then by IDEM. Fish tissue contaminant monitoring was first incorporated into the rotating basin methodology in 1997. The program currently follows a five-year rotating basin schedule, as described in the 2017-2021 WQMS (IDEM 2017b). Twenty-three fixed station program sites form the original fish tissue sampling network which began operating in the late 1970s in cooperation with the U.S. EPA. Prior to 1997, fish tissue samples were collected from these sites on a biennial basis. Post 1997, sampling has been conducted at the original fish tissue sampling sites once every five years in accordance with the WQMS rotating basin methodology.

In addition to the fish tissue sampling network, other sample sites are targeted based on historical environmental problems, water body access, use for fishing, date of last sampling event, potential contaminant sources, and monitoring recommendations by other agencies and entities. Sampling targets approximately 35 to 45 sites annually, including any sites from the original sampling network in the target basin (Table 4). An average of 3 to 5 composite or individual fish tissue samples are collected per site. In addition to these sites, samples from other agencies are accepted which were collected, prepared, and preserved using the same WAPB techniques. Sometimes, fish tissue samples are collected by other offices or agencies for analysis under IDEM's laboratory services contract (IDOA 2019). Results support specific projects such as mercury fate and transport; trends and status; and natural resource damage assessments. Samples are prepared using the whole fish or from the edible portion (skin-on or skin-off fillets) of fish. Whether whole fish or fillets are used

depends upon the site location, and size and species of fish retrieved. Typically, whole fish are only used when processing noncarp minnow species, or fish less than or equal to ten centimeters (four inches) in length.

Previous assessments in the Great Lakes basins include determination of the aquatic life use impairments for fish consumption based on concentrations of total PCBs and mercury for the 305(b) and 303(d) Integrated Report on water quality in the state of Indiana. The data collected also supports the ISDH issuance, modification, and removal of FCA's on waters of the state.

The FCA provides fish consumers with information about the risks associated with consuming potentially contaminated fish caught in Indiana. The FCA helps consumers make informed decisions regarding the size and species of fish, and how often to eat sport caught or commercially bought fish. After annual analytical results are received, the FCA workgroup meets to discuss the findings of recent fish monitoring data and develop the updated statewide FCA. Members of the Indiana Interagency FCA work group are ISDH, IDEM, and IDNR. Indiana's FCAs are issued by ISDH. IDEM collects and manages the majority of the data used to make FCA decisions for the state. IDNR has been instrumental in the collection of fish tissue samples from Lake Michigan and a number of inland lakes, where special studies are conducted.

Table 2. Criteria for Decision Making Using Fish Tissue

Parameter	IDEM Derived Criteria values for 303(d) ALUS* Determination (µg/kg** WW)	Indiana FCA decision levels (µg/kg wet weight)
Total PCB	>20 for any single sample	>50 – 1900 limited consumption >1900 No consumption
Total mercury	>300 (Fish tissue trophic level consumption weighted arithmetic mean concentration in edible portions for a sampling event.)	>50 – 950 limited consumption for sensitive populations @ >950 No consumption @ >160 limited consumption for the general population
PFOS	NA	>10-200 limited consumption >200 No consumption

* ALUS=Aquatic Life Use Support
**micrograms per kilogram
@Sensitive populations include women under age 50; women who are pregnant, breastfeeding, or planning to become pregnant; people with compromised immune systems; and children under the age of 15.

Indiana's sport fish consumption advisories are currently based on concentrations of mercury, PCBs, and PFOS found in the edible portions

of fish tissue. During the last three decades, more than 5,900 fish tissue samples have been analyzed for PCBs, organochlorine pesticides, and metals of concern. Of those, the majority contained quantifiable levels of mercury. Criteria for PCBs and mercury assessments in the Indiana FCA were developed from recommendations by the Great Lakes Sport Fish Advisory Task Force for PCBs in 1993 (Anderson et. al. 1993); and for mercury in 2007 (McCann and Anderson 2007). In 2017, Indiana began analyzing for PFAS on close to twenty percent of the samples collected annually. In 2019, Indiana issued first FCA based on PFOS concentrations in the edible portion of fish. Criteria for PFOS assessments in the Indiana FCA were developed from the Best Practice for PFOS Guidelines written by the Great Lakes Consortium for Fish Consumption Advisories in 2019 (Great Lakes Consortium, 2019). The Best Practice document is based on the 2016 U.S. EPA Drinking Water Health Advisory reference dose (RfD) of $2x10^{-5}$ milligrams per kilogram per day (mg/kg/day). Currently PCBs, mercury, and PFOS are the only three bioaccumulating fish tissue contaminants used to determine FCA listings.

A.5.4. The Budget

Sampling costs are detailed in Table 3.

Table 3. Laboratory Charges by Analytical Task Group

Tasks	Cost Per Sample	# Samples	Lab Duplicates	MS/MSDs	Total
PFAS	$385	94	6	12	$43,120
Metals	$75	51	3	3	$4,275
Lipids, PCBs, Pesticides	$205	52	3	6	$12,405
Total					$59,800

A.6. Project Description

A.6.1. Overview of Proposed Work

IDEM's WAPB will conduct field sampling activities to collect fish tissue from 49 locations in the Lake Michigan and Lake Erie basins. Samples will then be analyzed by professional service contractors (Pace) for the following:

- Lipids
- Solids
- PFAS
- Metals (chromium, mercury, lead and selenium)
- PCBs
- Pesticides

A.6.2. Project Locations

The central focus regions are the tributaries to the Great Lakes basins in Indiana. For the purpose of this project, the tributaries to the Great Lakes basins are defined as all streams, rivers, reservoirs, and natural lakes discharging to or within the defined watershed boundaries of the Lake Michigan and Lake Erie basins. This includes tributaries to the St. Joseph River, Little Calumet River, Grand Calumet River, and Lake Michigan proper in the Lake Michigan basin, and tributaries to the St. Mary's River, Maumee River, and St. Joseph River in the Lake Erie basin. IDEM OWQ WAPB sampling of the target basins portion of this project begins in July and continues through October. The sampling basins also include a portion of the Grand Calumet River Area of Concern (AOC).

The Great Lakes basin (Figure 3) is geographically defined as within the borders of Indiana contained in the 8-digit Hydrologic Unit Codes (HUC) Chicago - 07120003, Little Calumet-Galien - 04040001, St. Joseph (Lake Michigan) - 04050001, St. Joseph (Lake Erie) - 04100003, Auglaize - 04100007, St. Marys - 04100004 and Upper Maumee - 04100005. The Great Lakes basin, located in northern Indiana, drains approximately 3200 square miles within Indiana borders. Using the 2018 Crop Data Layer, predominant land uses are cropland (38%), urban (22%) forest (10%), and pasture (15%) (Homer et. al. 2015) (Figure 2). The 49 specific sampling locations are shown in Table 4 and Figure 3.

Figure 2. Great Lakes Basin Land Use

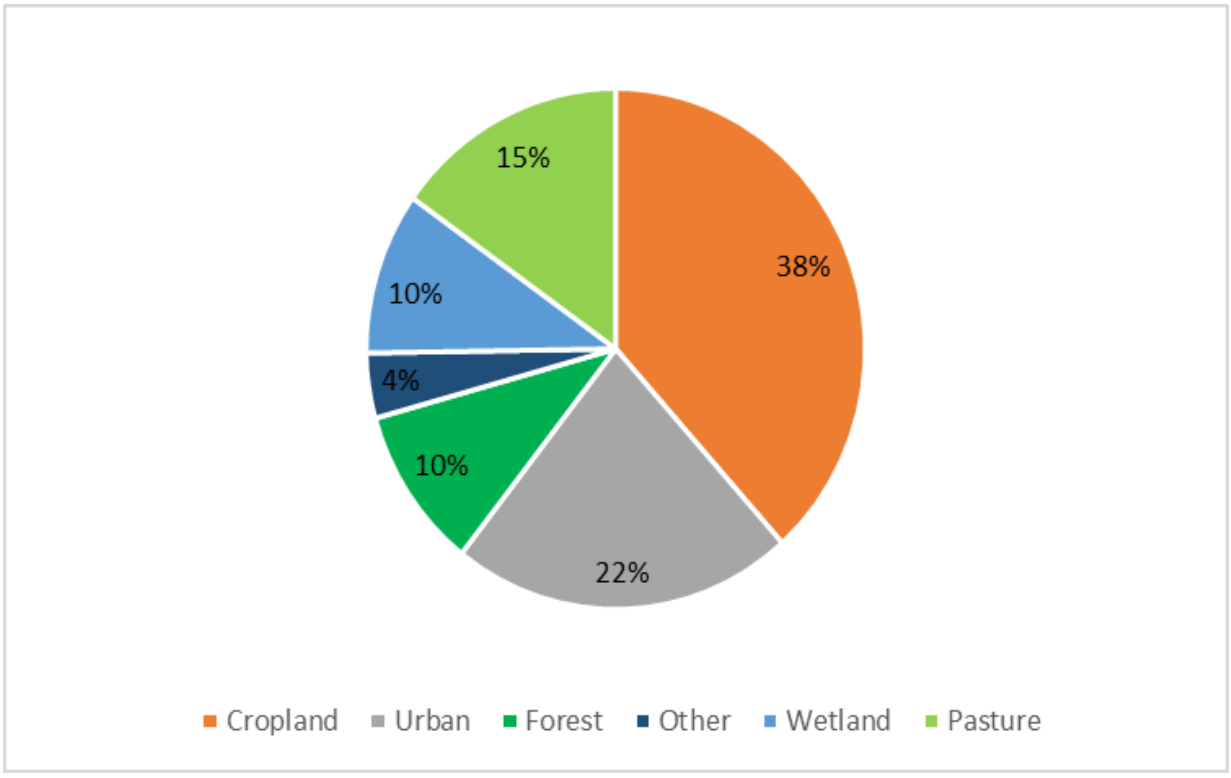
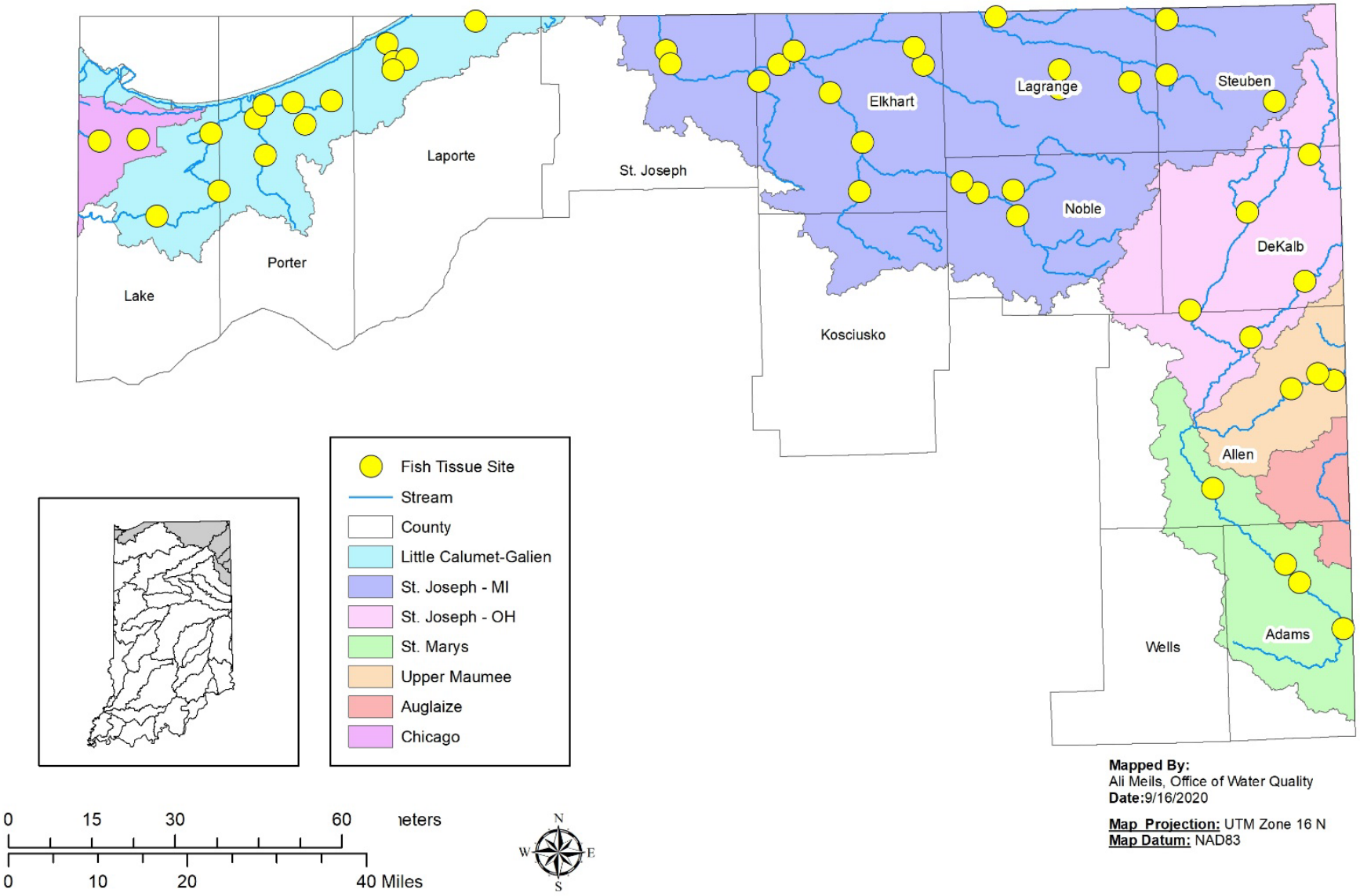


Table 4. Tentative target sites for monitoring in the Great Lakes basins

Site ID	Waterbody	Location	Latitude	Longitude
LMJ-21-0013	Baugo Creek	Creekwood Terrace	41.65190	-86.05619
LEM-01-0016	Black Creek	Ehle Road	41.16129	-84.86434
LES-04-0013	Blue Creek	SR 124	40.74678	-84.82250
LEJ-06-0011	Cedar Creek	CR 28	41.42617	-85.00663
LMJ-14-0001	Christiana Creek	Willowdale Park	41.70080	-85.97922
LMG-04-0052	Coffee Creek	Coffee Creek Preserve	41.58622	-87.03742
LMG-05-0047	Deep River	Bicentennial Park Public Access	41.57100	-87.24026
LMG-05-0049	Deep River	Deep River County Park	41.47700	-87.22230
LMG-04-0051	E Arm Little Calumet	SR 149	41.61699	-87.12577
LMG-04-0050	E Arm Little Calumet River	Chesterton, IN	41.62074	-87.06224
LMG-04-0027	E Arm Little Calumet River	CR 450 E	41.62387	-86.98054
LMG-01-0031	East Branch Trail Creek	CR 600 W	41.69150	-86.81637
LMJ-18-0016	Elkhart River	N. Calvin Street	41.46587	-85.58674
LMJ-18-0017	Elkhart River	CR 1025 W	41.48385	-85.62054
LMJ-19-0009	Elkhart River	W. Kercher Road	41.55085	-85.83402
LMJ-19-0010	Elkhart River	Elkhart County (Oxbow) Park	41.63175	-85.90181
LMJ-08-0111	Fawn River	Fawn River Fish Hatchery	41.74147	-85.17131
LMJ-08-0110	Fawn River	CR 750 W	41.75077	-85.54060
LEJ-04-0021	Fish Creek	CR4A	41.51725	-84.87032
LMJ-11-0038	Fly Creek	CR S 100 E	41.63205	-85.40677
LMJ-11-0039	Fly Creek	CR E 150 N	41.66332	-85.40613
LMG-02-0005	Galena River	CR 1000 N	41.75318	-86.66795
UMC-03-0004	Hart Ditch	Wicker Memorial Park	41.55783	-87.48072
LEJ-07-0019	Little Cedar Creek	CR 327	41.26880	-85.13509
LMJ-12-0011	Little Elkhart River	Riverbend Park	41.67466	-85.69979
LMJ-12-0012	Little Elkhart River	CR 10	41.70373	-85.72009
LMG-05-0048	Main Beaver Dam Ditch	Center Ross Road	41.43597	-87.35642
LEM-01-0015	Marsh Ditch	Gustin Road	41.14954	-84.82948

Site ID	Waterbody	Location	Latitude	Longitude
LMJ170-0014	North Branch Elkhart River	CR N 450 W	41.46946	-85.51025
LMJ-10-0163	Pigeon Creek	E. Hanselmen Road	41.60456	-84.94245
LMG-04-0053	Pigeon River	Pigeon River FWA	41.65135	-85.17437
LMJ-22-0006	Saint Joseph River	Elkhart, IN	41.67809	-86.01287
LMJ-22-0007	Saint Joseph River	Keller Park Landing	41.70331	-86.25569
LMJ-22-0008	Saint Joseph River	E. Madison Street (East Race)	41.68141	-86.24732
LEJ-08-0016	Saint Joseph River	CR 63	41.31192	-84.88664
LEJ-08-0017	Saint Joseph River	Cedarville, IN;	41.22264	-85.00565
LES-05-0055	Saint Marys River	Decatur, IN	40.85335	-84.94325
LES-06-0013	Saint Marys River	Bostick Road	40.97918	-85.09475
LES-04-0012	Saint Marys River	Kekionga Park	40.82360	-84.91371
LMG-03-0024	Salt Creek	CR 700 N	41.53573	-87.12267
LMG-03-0025	Salt Creek	Sandalwood Street	41.59547	-87.14380
LMJ-16-0077	South Branch Elkhart River	CR W 525 N	41.42774	-85.50174
LMG-01-0029	Trail Creek	Springland Ave	41.71664	-86.85979
LMG-01-0030	Trail Creek	US Highway 20	41.68766	-86.84476
LMJ-10-0162	Turkey Creek	US 20	41.64109	-85.25444
LMJ-17-0067	Turkey Creek	CR 50	41.47121	-85.84146
UMC-03-0003	W Arm Little Calumet River	Lake Etta County Park	41.56070	-87.39700
LMG-01-0032	West Branch Trail Creek	Wozniak Road	41.67418	-86.84635
LEM-01-0017	Wilbur Ditch	Irving Road	41.13801	-84.92155

Figure 3. Tentative Target Sites in the Great Lakes Basin



A.6.3. Milestones and Timeline

The project will take place over the course of two years, with project year one consisting of project preparation, two sampling episodes, and some beginning data analysis. Project year two will consist entirely of data analysis and reporting.

Table 5. Project Timetable

Item	Time Frame	Responsible Staff
QAPP Development	Aug. 2020 – Oct. 2020	Principal Investigator
IDEM OWQ amends Laboratory Contract	Aug. 2020 – Oct. 2020	IDEM OWQ Contract Manager
IDEM sample collection	Sept. 2020 – Oct. 2020	Principal Investigator, IDEM OWQ Staff
Fish tissue sample analysis	Dec. 2020 – Jun. 2021	Professional Services Contractor
Data post-processing	Mar. 2021 - Jun. 2021	Professional Services Contractor, IDEM OWQ Staff
Data Analysis	Jun. 2021-Mar. 2022	Principal Investigator, IDEM OWQ Staff
Progress Report 1 to USEPA	Apr. 30, 2021	Principal Investigator
Progress Report 2 to USEPA	Oct. 30, 2021	Principal Investigator
Progress Report 3 to USEPA	Apr. 30, 2022	Principal Investigator
Draft Final Report	Oct. 15, 2022	Principal Investigator, IDEM OWQ Staff
Final Report to USEPA	Oct. 31, 2022	Principal Investigator

A.6.4. Monitoring and Oversight

The IDEM PI will have primary oversight responsibilities for this project. As described in Section A.9., all collected data will be submitted to the IDEM PI for record keeping and reporting to the U.S. EPA project officer. All data will be maintained on the IDEM server. The PI is also responsible for ensuring project data collected meets project goals.

A.6.5. Results, Outputs, and Outcomes:

Outputs

- Determine the extent and magnitude of chemicals of mutual concern, specifically PFAS in fish tissue.
- Protection of Great Lakes and Great Lakes tributary fish consumers from harmful chemicals like PFOS, PCBs and mercury using scientific evidence to issue advisory information.

Expected Outcomes:

- Increase knowledge about PFAS in the region, a “Chemicals of Mutual Concern” identified in the Great Lakes Water Quality Agreement’s Annex 3 and a priority chemical with the potential to negatively impact the ecological or public health of the Great Lakes.
- Share information on the risks and benefits of consuming Great Lakes fish with consumers through assessment activities.

A.7. Quality Objectives and Criteria

The project is an estimation project and all values will be reported to U.S. EPA.

A.7.1. Precision

Precision is a measure of the degree to which two or more measurements are in agreement. Relative percent difference (RPD) is calculated for each pair of duplicates as indicated below:

$$RPD = \frac{|(S - D)| \times 100}{(S + D) / 2}$$

Where: S = First sample value (original or matrix spike value)
D = Second sample value (duplicate or matrix spike duplicate value)

Field quality objectives and criteria are contained in the 2020 Fish Tissue WP. The laboratories will use the following quality indicators for analysis.

Table 6. Precision objectives by measurement type

Measurement	Units	Precision Objective
General Chemistry		
Lipid	**%**	RPD ≤ 20%
Moisture	**%**	RPD ≤ 10%
PFAS		
Perfluoro-1-octanesulfonate (C8, PFOS)	µg/kg ww	RPD ≤ 40%
Perfluoro-1-butanesulfonate (C4, PFBS)	µg/kg ww	RPD ≤ 40%
Perfluoro-1-hexanesulfonate (C6, PFHxS)	µg/kg ww	RPD ≤ 40%
Perfluoro-n-octanoic acid (C8, PFOA)	µg/kg ww	RPD ≤ 40%
Perfluoro-n-butanoic acid(C4 PFBA)	µg/kg ww	RPD ≤ 40%
Perfluoro-n-pentanoic acid (C5, PFPeA)	µg/kg ww	RPD ≤ 40%
Perfluoro-n-hexanoic acid (C6, PFHxA)	µg/kg ww	RPD ≤ 40%
Perfluoro-n-heptanoic acid (C7, PFHpA)	µg/kg ww	RPD ≤ 40%
Perfluoro-n-nonanoic acid (C9, PFNA)	µg/kg ww	RPD ≤ 40%
Perfluoro-n-decanoic acid (C10, PFDA)	µg/kg ww	RPD ≤ 40%
Perfluoro-n-undodecanoic acid (C11, PFUnA)	µg/kg ww	RPD ≤ 40%
Perfluoro-n-dodecanoic acid (C12, PFDoA)	µg/kg ww	RPD ≤ 40%
Perfluoro-1-octanesulfonamide (PFOSA)	µg/kg ww	RPD ≤ 40%
Perfluorotridecanoic acid (PFTrDA)	µg/kg ww	RPD ≤ 40%
Perfluorotetradecanoic acid (PFTeDA)	µg/kg ww	RPD ≤ 40%
Perfluorohexadecanoic acid (PFHxDA)	µg/kg ww	RPD ≤ 40%
Perfluorooctandecanoic acid (PFODA)	µg/kg ww	RPD ≤ 40%
Perfluoropentanesulfonic acid (PFPeS)	µg/kg ww	RPD ≤ 40%
Perfluoroheptanesulfonic acid (PFHpS)	µg/kg ww	RPD ≤ 40%
Perfluorononanesulfonic acid (PFNS)	µg/kg ww	RPD ≤ 40%
Perfluorodecanesulfonic acid (PFDS)	µg/kg ww	RPD ≤ 40%

Perfluorododecanesulfonic acid (PFDoS)	µg/kg ww	RPD ≤ 40%
N-methylperfluorooctane sulfonamidoethanol (NMeFOSE)	µg/kg ww	RPD ≤ 40%
N-ethylperfluorooctane sulfonamidoethanol (NEtFOSE)	µg/kg ww	RPD ≤ 40%
N-methylperfluorooctane sulfonamide (NMeFOSA)	µg/kg ww	RPD ≤ 40%
N-ethylperfluorooctane sulfonamide (NEtFOSA)	µg/kg ww	RPD ≤ 40%
N-methyl perfluorooctanesulfonamidoacetic acid - br/lin (NMeFOSAA)	µg/kg ww	RPD ≤ 40%
N-ethyl perfluorooctanesulfonamidoacetic acid - br/lin (NEtFOSAA)	µg/kg ww	RPD ≤ 40%
4:2 Fluorotelomer sulfonic acid (4:2 FTS)	µg/kg ww	RPD ≤ 40%
6:2 Fluorotelomer sulfonic acid (6:2 FTS)	µg/kg ww	RPD ≤ 40%
8:2 Fluorotelomer sulfonic acid (8:2 FTS)	µg/kg ww	RPD ≤ 40%
10:2 Fluorotelomer sulfonic acid (10:2 FTS)	µg/kg ww	RPD ≤ 40%
4,8-dioxa-3H-perfluorononanoic acid (ADONA)	µg/kg ww	RPD ≤ 40%
9-Chlorohexadecafluoro-3-oxanone-1-sulfonic acid (9Cl-PF3ONS)	µg/kg ww	RPD ≤ 40%
11-chloroeicosafluoro-3-oxaundecane-1-sulfonic acid (11Cl-PF3OUdS)	µg/kg ww	RPD ≤ 40%
Metals		
Chromium	µg/kg ww	RPD ≤ 20%
Lead	µg/kg ww	RPD ≤ 20%
Mercury	µg/kg ww	RPD ≤ 20%
Selenium	µg/kg ww	RPD ≤ 20%
PCBs		
Total PCBs	µg/kg ww	RPD ≤ 40%
Organochlorine Pesticides		
Aldrin	µg/kg ww	RPD ≤ 43%
BHC, alpha-	µg/kg ww	RPD ≤ 40%
BHC, beta-	µg/kg ww	RPD ≤ 40%
BHC, delta-	µg/kg ww	RPD ≤ 40%
BHC, gamma-	µg/kg ww	RPD ≤ 40%
Chlordane, gamma	µg/kg ww	RPD ≤ 40%
Chlordane, alpha	µg/kg ww	RPD ≤ 40%
DDD, o,p'-	µg/kg ww	RPD ≤ 40%
DDD, p,p'-	µg/kg ww	RPD ≤ 40%
DDE, o,p'-	µg/kg ww	RPD ≤ 40%
DDE, p,p'-	µg/kg ww	RPD ≤ 40%
DDT, o,p'-	µg/kg ww	RPD ≤ 40%
DDT, p,p'-	µg/kg ww	RPD ≤ 50%
Dieldrin	µg/kg ww	RPD ≤ 38%
Endosulfan I	µg/kg ww	RPD ≤ 40%
Endosulfan II	µg/kg ww	RPD ≤ 40%

Endosulfan sulfate	µg/kg ww	RPD ≤ 40%
Endrin	µg/kg ww	RPD ≤ 45%
Endrin aldehyde	µg/kg ww	RPD ≤ 40%
Endrin ketone	µg/kg ww	RPD ≤ 40%
Heptachlor	µg/kg ww	RPD ≤ 31%
Heptachlor epoxide	µg/kg ww	RPD ≤ 40%
Hexachlorobenzene	µg/kg ww	RPD ≤ 40%
Methoxychlor	µg/kg ww	RPD ≤ 40%
Mirex	µg/kg ww	RPD ≤ 40%
cis- Nonachlor	µg/kg ww	RPD ≤ 40%
trans- Nonachlor	µg/kg ww	RPD ≤ 40%
Oxychlordane	µg/kg ww	RPD ≤ 40%
Pentachloroanisole	µg/kg ww	RPD ≤ 40%
Toxaphene	µg/kg ww	RPD ≤ 40%

A.7.2. Accuracy

Accuracy describes the agreement between an observed value and an accepted reference or true value. PFAS is evaluated using surrogate spikes. Analytical accuracy is often reported as a percent recovery (%R) calculated as follows:

$$\%\boldsymbol{R} = \frac{\boldsymbol{X_{observed}}}{\boldsymbol{X_{True}}} * \mathbf{100},$$

where $\boldsymbol{X_{observed}}$ is the observed value and
$\boldsymbol{X_{True}}$ is the reference or "true" value.

Table 7. Accuracy objectives by measurement type

Measurement	Units	Accuracy Objective
General Chemistry		
Lipid	**%**	70 ≤ %R ≤ 130
Moisture	**%**	70 ≤ %R ≤ 130%
PFAS		
Perfluoro-1-octanesulfonate (C8, PFOS)	µg/kg ww	70 ≤ %R ≤ 130
Perfluoro-1-butanesulfonate (C4, PFBS)	µg/kg ww	70 ≤ %R ≤ 130
Perfluoro-1-hexanesulfonate (C6, PFHxS)	µg/kg ww	70 ≤ %R ≤ 130
Perfluoro-n-octanoic acid (C8, PFOA)	µg/kg ww	70 ≤ %R ≤ 130
Perfluoro-n-butanoic acid(C4 PFBA)	µg/kg ww	70 ≤ %R ≤ 130
Perfluoro-n-pentanoic acid (C5, PFPeA)	µg/kg ww	70 ≤ %R ≤ 130
Perfluoro-n-hexanoic acid (C6, PFHxA)	µg/kg ww	70 ≤ %R ≤ 130
Perfluoro-n-heptanoic acid (C7, PFHpA)	µg/kg ww	70 ≤ %R ≤ 130
Perfluoro-n-nonanoic acid (C9, PFNA)	µg/kg ww	70 ≤ %R ≤ 130
Perfluoro-n-decanoic acid (C10, PFDA)	µg/kg ww	70 ≤ %R ≤ 130

Perfluoro-n-undodecanoic acid (C11, PFUnA)	µg/kg ww	70 ≤ %R ≤ 130
Perfluoro-n-dodecanoic acid (C12, PFDoA)	µg/kg ww	70 ≤ %R ≤ 130
Perfluoro-1-octanesulfonamide (PFOSA)	µg/kg ww	70 ≤ %R ≤ 130
Perfluorotridecanoic acid (PFTrDA)	µg/kg ww	70 ≤ %R ≤ 130
Perfluorotetradecanoic acid (PFTeDA)	µg/kg ww	70 ≤ %R ≤ 130
Perfluorohexadecanoic acid (PFHxDA)	µg/kg ww	70 ≤ %R ≤ 130
Perfluorooctandecanoic acid (PFODA)	µg/kg ww	70 ≤ %R ≤ 130
Perfluoropentanesulfonic acid (PFPeS)	µg/kg ww	70 ≤ %R ≤ 130
Perfluoroheptanesulfonic acid (PFHpS)	µg/kg ww	70 ≤ %R ≤ 130
Perfluorononanesulfonic acid (PFNS)	µg/kg ww	70 ≤ %R ≤ 130
Perfluorodecanesulfonic acid (PFDS)	µg/kg ww	70 ≤ %R ≤ 130
Perfluorododecanesulfonic acid (PFDoS)	µg/kg ww	70 ≤ %R ≤ 130
N-methylperfluorooctane sulfonamidoethanol (NMeFOSE)	µg/kg ww	70 ≤ %R ≤ 130
N-ethylperfluorooctane sulfonamidoethanol (NEtFOSE)	µg/kg ww	70 ≤ %R ≤ 130
N-methylperfluorooctane sulfonamide (NMeFOSA)	µg/kg ww	70 ≤ %R ≤ 130
N-ethylperfluorooctane sulfonamide (NEtFOSA)	µg/kg ww	70 ≤ %R ≤ 130
N-methyl perfluorooctanesulfonamidoacetic acid - br/lin (NMeFOSAA)	µg/kg ww	70 ≤ %R ≤ 130
N-ethyl perfluorooctanesulfonamidoacetic acid - br/lin (NEtFOSAA)	µg/kg ww	70 ≤ %R ≤ 130
4:2 Fluorotelomer sulfonic acid (4:2 FTS)	µg/kg ww	70 ≤ %R ≤ 130
6:2 Fluorotelomer sulfonic acid (6:2 FTS)	µg/kg ww	70 ≤ %R ≤ 130
8:2 Fluorotelomer sulfonic acid (8:2 FTS)	µg/kg ww	70 ≤ %R ≤ 130
10:2 Fluorotelomer sulfonic acid (10:2 FTS)	µg/kg ww	70 ≤ %R ≤ 130
4,8-dioxa-3H-perfluorononanoic acid (ADONA)	µg/kg ww	70 ≤ %R ≤ 130
9-Chlorohexadecafluoro-3-oxanone-1-sulfonic acid (9Cl-PF3ONS)	µg/kg ww	70 ≤ %R ≤ 130
11-chloroeicosafluoro-3-oxaundecane-1-sulfonic acid (11Cl-PF3OUdS)	µg/kg ww	70 ≤ %R ≤ 130
Metals		
Chromium	µg/kg ww	75 ≤ %R ≤ 125
Lead	µg/kg ww	75 ≤ %R ≤ 125
Mercury	µg/kg ww	75 ≤ %R ≤ 125
Selenium	µg/kg ww	75 ≤ %R ≤ 125

PCBs		
Total PCBs	µg/kg ww	70 ≤ %R ≤ 130
Organochlorine Pesticides		
Aldrin	µg/kg ww	34 ≤ %R ≤ 142
BHC, alpha-	µg/kg ww	70 ≤ %R ≤ 130
BHC, beta-	µg/kg ww	70 ≤ %R ≤ 130
BHC, delta-	µg/kg ww	70 ≤ %R ≤ 130
BHC, gamma-	µg/kg ww	70 ≤ %R ≤ 130
Chlordane, gamma	µg/kg ww	70 ≤ %R ≤ 130
Chlordane, alpha	µg/kg ww	70 ≤ %R ≤ 130
DDD, o,p'-	µg/kg ww	70 ≤ %R ≤ 130
DDD, p,p'-	µg/kg ww	70 ≤ %R ≤ 130
DDE, o,p'-	µg/kg ww	70 ≤ %R ≤ 130
DDE, p,p'-	µg/kg ww	70 ≤ %R ≤ 130
DDT, o,p'-	µg/kg ww	70 ≤ %R ≤ 130
DDT, p,p'-	µg/kg ww	23 ≤ %R ≤ 134
Dieldrin	µg/kg ww	31 ≤ %R ≤ 134
Endosulfan I	µg/kg ww	70 ≤ %R ≤ 130
Endosulfan II	µg/kg ww	70 ≤ %R ≤ 130
Endosulfan sulfate	µg/kg ww	70 ≤ %R ≤ 130
Endrin	µg/kg ww	42 ≤ %R ≤ 139
Endrin aldehyde	µg/kg ww	70 ≤ %R ≤ 130
Endrin ketone	µg/kg ww	70 ≤ %R ≤ 130
Heptachlor	µg/kg ww	35 ≤ %R ≤ 130
Heptachlor epoxide	µg/kg ww	70 ≤ %R ≤ 130
Hexachlorobenzene	µg/kg ww	70 ≤ %R ≤ 130
Methoxychlor	µg/kg ww	70 ≤ %R ≤ 130
Mirex	µg/kg ww	70 ≤ %R ≤ 130
cis- Nonachlor	µg/kg ww	70 ≤ %R ≤ 130
trans- Nonachlor	µg/kg ww	70 ≤ %R ≤ 130
Oxychlordane	µg/kg ww	70 ≤ %R ≤ 130
Pentachloroanisole	µg/kg ww	70 ≤ %R ≤ 130
Toxaphene	µg/kg ww	70 ≤ %R ≤ 130

Like precision, accuracy is susceptible to variations in technique. Such variation is minimized through the use of SOPs, and correct field and laboratory technique by qualified individuals. (Section A.8.)

A.7.3. Representativeness

Unlike precision and accuracy, representativeness tends to be a qualitative measurement. Essentially, describing how similar the analytical data is in essential characteristics to the parent population of interest. Many factors can influence how representative the analytical results are for an area sampled. These factors include:

- The selection of appropriate analytical procedures.

- The sampling plan.
- Matrix heterogeneity.
- The procedures and protocols used to collect, preserve, and transport samples.

In this case, choice of sampling locations and techniques and use of OWQ SOPs provide confidence in the representativeness of the results.

A.7.4. Completeness

Completeness is another quantitative measure, used to evaluate how many valid analytical data were obtained in comparison to the amount planned. In this case, the completeness goal is 100% for samples analyzed in this project. Therefore, all samples must be collected, analyzed, and yield analytical data usable for the intended purpose.

A.7.5. Comparability

Comparability is a qualitative measure of dataset equivalency. If two datasets are not readily comparable, using those data may make drawing inferences or making comparisons difficult. Comparability is assured through the use of common variables, standardized collection and analysis techniques, and satisfying the requirements of the other measurement quality objectives. In this case, comparability is largely addressed due to all sampling utilizing the exact same IDEM OWQ collection and analysis methods used to collect other Indiana fish tissue data.

A.7.6. Sensitivity

Sensitivity is related to the reporting limit. In this context, sensitivity refers to the capability of a method or instrument to detect a given analyte at a given concentration and reliably quantitate the analyte concentration. The investigator should be concerned the instrument or method can detect and provide an accurate analyte concentration not greater than an applicable standard or screening level. In general, RLs should be less than the applicable standard or screening level. Analytical results for samples which are non-detect for a particular analyte with RLs greater than the applicable standards or screening levels cannot be used to demonstrate compliance with the applicable standards or screening levels.

Table 8. Sensitivity objectives by measurement type, expressed as IDEM reporting limit

Measurement	Units	Reporting Limit
General Chemistry[1]		
Lipid	**%**	0.1

Moisture	%	0.1
PFAS[2]		
Perfluoro-1-octanesulfonate (C8, PFOS)	µg/kg ww	0.231
Perfluoro-1-butanesulfonate (C4, PFBS)	µg/kg ww	0.221
Perfluoro-1-hexanesulfonate (C6, PFHxS)	µg/kg ww	0.227
Perfluoro-n-octanoic acid (C8, PFOA)	µg/kg ww	0.250
Perfluoro-n-butanoic acid(C4 PFBA)	µg/kg ww	0.250
Perfluoro-n-pentanoic acid (C5, PFPeA)	µg/kg ww	0.250
Perfluoro-n-hexanoic acid (C6, PFHxA)	µg/kg ww	0.250
Perfluoro-n-heptanoic acid (C7, PFHpA)	µg/kg ww	0.250
Perfluoro-n-nonanoic acid (C9, PFNA)	µg/kg ww	0.250
Perfluoro-n-decanoic acid (C10, PFDA)	µg/kg ww	0.250
Perfluoro-n-undodecanoic acid (C11, PFUnA)	µg/kg ww	0.250
Perfluoro-n-dodecanoic acid (C12, PFDoA)	µg/kg ww	0.250
Perfluoro-1-octanesulfonamide (PFOSA)	µg/kg ww	0.250
Perfluorotridecanoic acid (PFTrDA)	µg/kg ww	0.250
Perfluorotetradecanoic acid (PFTeDA)	µg/kg ww	0.250
Perfluorohexadecanoic acid (PFHxDA)	µg/kg ww	0.250
Perfluorooctandecanoic acid (PFODA)	µg/kg ww	0.250
Perfluoropentanesulfonic acid (PFPeS)	µg/kg ww	0.250
Perfluoroheptanesulfonic acid (PFHpS)	µg/kg ww	0.237
Perfluorononanesulfonic acid (PFNS)	µg/kg ww	0.240
Perfluorodecanesulfonic acid (PFDS)	µg/kg ww	0.241
Perfluorododecanesulfonic acid (PFDoS)	µg/kg ww	0.242
N-methylperfluorooctane sulfonamidoethanol (NMeFOSE)	µg/kg ww	0.250
N-ethylperfluorooctane sulfonamidoethanol (NEtFOSE)	µg/kg ww	0.250
N-methylperfluorooctane sulfonamide (NMeFOSA)	µg/kg ww	0.250
N-ethylperfluorooctane sulfonamide (NEtFOSA)	µg/kg ww	0.250
N-methyl perfluorooctanesulfonamidoacetic acid - br/lin (NMeFOSAA)	µg/kg ww	0.250
N-ethyl perfluorooctanesulfonamidoacetic acid - br/lin (NEtFOSAA)	µg/kg ww	0.250
4:2 Fluorotelomer sulfonic acid (4:2 FTS)	µg/kg ww	0.233
6:2 Fluorotelomer sulfonic acid (6:2 FTS)	µg/kg ww	0.237
8:2 Fluorotelomer sulfonic acid (8:2 FTS)	µg/kg ww	0.241
10:2 Fluorotelomer sulfonic acid (10:2 FTS)	µg/kg ww	0.241
4,8-dioxa-3H-perfluorononanoic acid (ADONA)	µg/kg ww	0.236
9-Chlorohexadecafluoro-3-oxanone-1-sulfonic acid (9Cl-PF3ONS)	µg/kg ww	0.232

11-chloroeicosafluoro-3-oxaundecane-1-sulfonic acid (11Cl-PF3OUdS)	µg/kg ww	0.235
Metals[3]		
Chromium	µg/kg ww	10
Lead	µg/kg ww	70
Mercury	µg/kg ww	50
Selenium	µg/kg ww	110
PCBs[4]		
Total PCBs	µg/kg ww	50
Organochlorine Pesticides[5]		
Aldrin	µg/kg ww	8
BHC, alpha-	µg/kg ww	8
BHC, beta-	µg/kg ww	8
BHC, delta-	µg/kg ww	8
BHC, gamma-	µg/kg ww	8
Chlordane, gamma	µg/kg ww	8
Chlordane, alpha	µg/kg ww	8
DDD, o,p'-	µg/kg ww	10
DDD, p,p'-	µg/kg ww	10
DDE, o,p'-	µg/kg ww	10
DDE, p,p'-	µg/kg ww	10
DDT, o,p'-	µg/kg ww	10
DDT, p,p'-	µg/kg ww	10
Dieldrin	µg/kg ww	10
Endosulfan I	µg/kg ww	20
Endosulfan II	µg/kg ww	20
Endosulfan sulfate	µg/kg ww	20
Endrin	µg/kg ww	10
Endrin aldehyde	µg/kg ww	10
Endrin ketone	µg/kg ww	10
Heptachlor	µg/kg ww	8
Heptachlor epoxide	µg/kg ww	8
Hexachlorobenzene	µg/kg ww	10
Methoxychlor	µg/kg ww	20
Mirex	µg/kg ww	5
cis- Nonachlor	µg/kg ww	8
trans- Nonachlor	µg/kg ww	8
Oxychlordane	µg/kg ww	8
Pentachloroanisole	µg/kg ww	2.5
Toxaphene	µg/kg ww	10

[1] ASTM D2974-87 and Pace Analytical Lipid Method

[2] **Pace Analytical DoD 36 (Pace 2020)**

[3]EPA Preparation 3540C (U.S. EPA 1996a) and EPA Method 8082A (U.S. EPA 2007)

[4]EPA Method 6020A (U.S. EPA 2004)

[5]EPA Method 8081B (U.S. EPA 2007c)

A.8. Special Training Requirements or Certification

Descriptions of required trainings, qualifications, or certifications which are required to perform the project work are described below. All contractual work will be done by contractors chosen through a bid process which have the ability to adequately achieve all requirements in the project specifications through documentation of staff biographies and past project accomplishments. All contractors will be required to comply with the terms of the RFQ, associated contract, and this QAPP.

A.8.1. IDEM Principal Investigator

Alison Meils, B.S. in Fisheries and Aquatic Sciences from Purdue University, has experience in environmental measurement, data quality control, fish tissue collection, and has overseen the IDEM Contaminants Monitoring Program for 6 years.

A.8.2. IDEM Quality Assurance Reviewer

James Bailey possesses a significant amount of experience in quality assurance matters. He has served as the ISO 9001 quality management system coordinator at several companies prior to working at IDEM. To date, James serves as a QA staff member at IDEM and has reviewed 5 QAPPs, 18 QAPP related workplans, 38 TSOPs and nearly 26 SOPs since joining in July 2017. He has also utilized a number of online QA trainings. James has a bachelor's degrees in Biomedical Engineering Technology and in Chemical Engineering from Purdue University.

A.8.3. IDEM Office of Water Quality Field Staff

All training is conducted in-house, utilizing existing SOPs and equipment operating manuals. Typically, on-the-job training is conducted by OWQ, wherein new or less experienced staff accompany an experienced staff mentor or field crew chief.

Table 9. WAPB Project Roles, Experience and Training

Role	Required Training and Experience	Training References	Training Notes
All staff participating in fish tissue sample collections	-Basic First Aid and CPR	-A minimum of 4 hours of in-service training provided by WAPB (IDEM 2010b)	-Staff lacking 4 hours of in-service training or appropriate certification will be accompanied in the field at all times by WAPB staff which meet Health and Safety Training requirements
	-Personal Protective Equipment (PPE) Policy -Memorandum "Use	-IDEM 2008 -February 29, 2000	-Indiana Code 14-8-2-27 requires a high intensity whistle and Safety of Life at Sea (SOLAS) certified

	of Personal Flotation Devices (PFD) by Branch Personnel" dated February 29, 2000	WAPB internal memorandum regarding use of approved PFDs	strobe light when working on co-jurisdictional waters or during hours of darkness
	-IDEM Injury and Illness Resulting from Occupational Exposure Policy	-Hazard Communication Manual (IDEM 1992d)	
	-Compliance with the Biological Studies Section SOPs Manual: Section II		
	-Compliance with Indiana boating safety requirements	-State of Indiana Boating Safety Requirements (U.S. PS 2017) and the DNR approved online Boating Safety Course	-Staff lacking 2 years field experience will be accompanied in the field at all times by WAPB staff which meet the boating safety requirements

A.8.4. Fish Tissue Contract Laboratories

All fish tissue analysis will be conducted by Pace, which has a long history of analyzing biological samples. Once fish tissue samples are collected by IDEM all parameters are analyzed by Pace. Pace contract laboratory conducts QA which includes blanks, duplicates, and calibration checks.

A.9. Documentation and Records

Copies of original documents and records for this project, including field sheets and logs, raw laboratory data, and QA/QC data shall be provided to the IDEM project PI. Fish tissue sample measurements, analysis sets and appropriate sample metadata (e.g., site id, latitude, longitude, date) are entered into the AIMS II Database by WAPB staff.

A.9.1. Field Activities

Fish common name, total length (in millimeters), and mass (in grams) measurements are made on each fish included in their respective composite samples. Information on the fishing method and any identified DELTS will be recorded on the Field Record for Biological Tissue Contaminants Monitoring Program (Appendix 1). Results are documented and reported after quality control verification procedures are performed. QA staff audit field data reduction, validation, and reporting procedures as a component of performance audits described in Section C1.1, WAPB Field Performance and System Audits. No field duplicates are collected, nor field blanks carried during the fish tissue sampling and sample preparation processes.

Table 10. Field Record for Biological Tissue Contaminants Monitoring Program

Type of Report/Document	Indexing
EDI file from Pace (A.9.2)	S:\IGCN\OWQ\AIMS\EDIFiles\EnChem\2020
Contract Laboratory Chain of Custody	S:\IGCN\OWQ\WSP\OWM\Biological Studies\Fish tissue and Sediments\GLRI Grant 2020\COCs
Laboratory Report	S:\IGCN\OWQ\WSP\OWM\Biological Studies\Fish tissue and Sediments\GLRI Grant 2020\Laboratory Report
QC Report	S:\IGCN\OWQ\WSP\OWM\Biological Studies\Fish tissue and Sediments\GLRI Grant 2020\QC Report
Contract Laboratory Quarterly Reports (A.9.3)	S:\IGCN\OWQ\WSP\OWM\Biological Studies\Fish tissue and Sediments\GLRI Grant 2020\EPA Progress Reports
Sample Collection Forms (Appendix 1)	S:\IGCN\OWQ\WSP\OWM\Biological Studies\Fish tissue and Sediments\GLRI Grant 2020\Field Sheets
Field Logs	S:\IGCN\OWQ\WSP\OWM\Biological Studies\Fish tissue and Sediments\GLRI Grant 2020\Blue Book
Photos	S:\IGCN\OWQ\WSP\OWM\Biological Studies\Fish tissue and Sediments\GLRI Grant 2020\Photos
Grant Progress Reports (A.9.4.)	S:\IGCN\OWQ\WSP\OWM\Biological Studies\Fish tissue and Sediments\GLRI Grant 2020\EPA Progress Reports

A.9.2. Laboratory Activities

IDEM's OWQ will receive the analytical results from Pace. Per the Surface Water QAPP (2017a). This data is subject to the Laboratory Reporting Requirements in Table 10, including receipt of data in the electronic data interface specified in Appendix I of the Surface Water QAPP (IDEM 2017a). A report for each batch of samples (sample set) consisting of Contract Laboratory Chain of Custody Form (Appendix 2), spreadsheets of results, and the QC report, will be submitted in electronic (pdf) format in accordance with the contract requirements. In addition, an electronic data import (EDI) file containing laboratory data and lab QC will be submitted for each sample set. The EDI file will be in compliance with the Surface Water QAPP (IDEM 2017a, Appendix I). EDI files will be uploaded into the AIMS database. Reports shall meet requirements of DQA Level 4 in the

Surface Water QAPP, pp 182-183, (IDEM 2017a) and be submitted to the Technical and Logistical Services Section for review. For additional information see Section D.3.1.

A.9.3. Project Quarterly Progress Reports

IDEM's OWQ and the various contract laboratories will provide the IDEM PI with quarterly progress reports indicating all work accomplished during the reporting period, including all pertinent monitoring data, results, and photos. All reports will be stored in electronic format on the IDEM server shared drive (S Drive).

A.9.4. Grant Progress Reports

IDEM will obtain quarterly progress reports from IDEM's OWQ and the contract laboratory and combine the information with budget information obtained with the assistance of IDEM's Accounting Office to provide semiannual progress reports to the U.S. EPA. All reports will be stored in electronic format on the IDEM server shared drive (S Drive).

A.9.5. QA Records/Reports

The IDEM PI for this project, will be responsible for ensuring the appropriate project personnel have the most current approved version of the QAPP. QA, project, and site managers will be kept up to date on any revisions and edits made to the QAPP during the term of the project. The data report package shall include field logs which will be made available in hard copy and electronic formats. As with other project reports, any QA records or reports generated will be stored in electronic format on the IDEM server shared drive (S Drive).

A.9.6. Retention Time and Location of Records and Reports

All hard copy files of data and reports will be retained, for a minimum of three years, in accordance with the State of Indiana Records Retention Policy (see the General State of Indiana Schedule at: http://www.in.gov/iara/files/gr.pdf and the IDEM-specific one at: http://iaraappp01vl.state.in.us/cgibin/appx.sh?ACTIONS_NAME=scheduleReport(SCH)&SCHEDULE_RECORD_ID=47). Hard copy records will be maintained at the IDEM WSP currently located at 2525 N Shadeland Avenue, Indianapolis, IN 46219. An electronic copy of all reports and data files will be stored on the IDEM server shared drive (S Drive) in the GLRI Grant 2020 Folder (S:\IGCN\OWQ\WSP\OWM\Biological Studies\Fish tissue and Sediments\GLRI Grant 2020) for a period of not less than three years after the conclusion of the project.

B. Data Generation or Acquisition

B.1. Sampling Network and Rationale

Site selection for this project is designed to capture PFAS, metals, PCBs and pesticide profiles (Table 6) of major rivers and tributaries located within the Lake Michigan and Lake Erie basins. This will be the first time IDEM has analyzed for PFAS in the Great Lakes basins due to the chemicals being emerging contaminants of concern. Therefore, sites were not selected for comparison purposes or trend analysis, but rather spatial representation. Larger tributaries were targeted to ensure the collection of fish of filletable size. In addition, the 2020 Fish Tissue WP already targeted a number of sites in the basins so different sites were targeted to prevent duplication and increase the collection of new information in the basins. A total of 49 sites have been selected for sample collection. Two samples will be collected from each site which would allow for 98 total samples. The following criteria was taken into consideration during the site selection process:

- Land use
- Location in relation to wastewater treatment plant effluent
- New locations of interest which have never been sampled
- Public access locations
- Rivers and streams having a stream order of 2 or greater

Table 11. WAPB Sampling Procedure

Sampling Schedule	September to November
Constituent to be Sampled	Lipids, Moisture, PFAS, Metals, Total PCBs, Organochlorine Pesticides
Sampling Matrix	Fish Tissue
Sampling Procedure	See Appendix C
Sample Volume	Examine fish collected and select the predetermined number of samples for the site (generally between three and five samples). A sample may be comprised of 1-12 fish of the same species, depending on size. The preferred total lengths of the smallest and largest individuals of any composite sample should be within 90% of each other. Total lengths as low as 75% will be tolerated in order to obtain an adequate composite sample
Preservation Measures	Place the double wrapped and bagged samples in the dry ice chest underneath the dry ice for preservation of the tissue. Upon return to the office, place all processed tissue samples in the chest style analytical-grade freezer located in the IDEM WAPB laboratory or the upright commercial-grade freezer, located in building 41. Once prepared for individual or composite fish tissue samples they are stored at approximately -80°C for long term storage and -26°C for temporary storage in WAPB laboratory room 124 and building 41 laboratory freezers.
Container Size	Drain excess water and **double wrap** the fillets in clean aluminum foil to make a package. For whole fish, all individual

	fish of a composite sample should be double wrapped together if possible.
Maximum Holding Time	IDEM requires analysis to be conducted and reported within 90 days from the initial shipping date.

B.2. Sampling Methods Requirements

Sample containers, preservatives, and maximum holding times shall comply with the requirements of the applicable laboratory test method.

B.2.1. Sample Collection

See Appendix 3.

B.2.2. Sampling Timeframes

Fish tissue collections can occur year-round, although the most desirable sampling period is from late summer to early fall (i.e., July through October). For this study all samples will be collected from September to November. The lipid content of many species (which is a reservoir for many lipophilic bioaccumulating organic pollutants) is generally highest at this time. Also, water levels are typically lower during this time, thus simplifying collection procedures (U.S. EPA 2000).

B.2.3. Related Geospatial Data

At all locations, GPS locations shall be recorded, according to the accuracy DQO listed in Section A.7.1.2. In addition, photographs shall be taken showing: (1) the sampling location, (2) upstream conditions, and (3) downstream conditions. Photographs shall indicate the sampling location and direction in the filename. All geospatial data and photographs shall be provided to the IDEM PI.

B.2.4. Sample Preservation and Holding Time

Fish collected in the field are stored on ice until filleted and packaged in the laboratory. Once prepared for individual or composite fish tissue samples they are stored at approximately -80°C for long term storage and -26°C for temporary storage in WAPB laboratory room 124 and building 41 laboratory freezers. The laboratories are located in the Western Select Building office at 2525 N. Shadeland Ave, Indianapolis, IN 46219 (Shadeland). Samples are stored until relinquished to the contract analytical services laboratory for analysis. Once all sampling for the year is complete, prepared samples are sent to the laboratory.

Once WAPB is prepared to submit a batch of fish tissue samples for analysis, typically in late summer or fall, contact the Pace project manager. Pack samples securely in coolers with dry ice complete with a chain of custody form. A Pace hired courier service picks up the coolers during business hours.

The samples are couriered by Pace staff in the Chicago LabOps Service Center. From Chicago, the samples are shipped to the Green Bay, Wisconsin laboratory for sample preparation, including maceration. Each shipping step uses same day or overnight shipping. IDEM requires analysis to be conducted and reported within 90 days from the initial shipping date.

The analytical laboratory stores all samples at a maximum temperature of -10°C until written permission for disposal is given by IDEM's Fish Tissue Contaminants Monitoring Program manager. Permission is only given when all laboratory data reports, for a given sample year, have been reviewed for QC and accepted as usable data. The laboratory may dispose of excess sample material in accordance with state and federal regulations.

Table 12. Method Holding Times and Conditions

Method	Hold Time	Conditions	Source
ASTM D2974-87	1 year	<-10°C	Uses same value as EPA 1630/1631E. (U.S. EPA 2002)
Pace Lipid	1 year	<-10°C	Uses same value as EPA 1630/1631E. (U.S. EPA 2007b, U.S. EPA 2002)
Pace Analytical DoD 36	1 year	<-20°C	Determination of Selected 36 Per- and Polyfluoroalkyl Substances (PFAS) by LC/MS/MS (Isotope Dilution) (Pace 2020)
EPA Preparation 3540C and EPA Method 8082A	1 year	<-10°C	(U.S. EPA 1996a and U.S. EPA 2007)
EPA Method 6020A	1 year	<-10°C	(U.S. EPA 2004)
EPA Method 8081B	1 year	<-10°C	(U.S. EPA 2007b)

B.3. Custody Procedures

Chain-of-custody is the chronological documentation or paper trail, showing the seizure, custody, control, transfer, analysis, and disposition of physical or electronic evidence. This is accomplished through a combination of field and laboratory records demonstrating possession and transfer of custody. Procedures for chain of custody for laboratory activities and final evidence files are discussed in this section.

B.3.1. WAPB Contract Laboratory Activities

Each contract laboratory performing analyses on behalf of the WAPB water quality monitoring programs (in this case Pace) is responsible for implementing chain-of-custody procedures which meet WAPB chain-of-custody requirements, as defined in the Surface Water QAPP (IDEM

2017a). Compliance with the IDEM RFPs ensures acceptable laboratory chain-of-custody procedures are in place before samples are collected.

B.3.2. WAPB Final Evidence Files

Data collected for water quality assessment and characterization are maintained by the WAPB. Reports are assessed, qualified, and entered into the AIMS II Database. Reports containing raw data are cataloged and stored from future needs. Electronic database files are maintained indefinitely.

B.4. Analytical Procedures

Analytical procedures are written instructions which describe how to prepare a sample for analysis; prepare and calibrate test measurement equipment; perform a test; and calculate results. Field analytical procedures generate analytical data on site to be used in decision-making processes involving sample selection and site screening. Laboratory analytical procedures produce data under more controlled conditions, and therefore, usually provide lower detection limits and greater precision than field data. Both field and laboratory data provide information which help in DQA process for the purpose of meeting defined project DQOs.

B.4.1. Analytical Parameters and Estimated Costs

Specific test parameters and reporting limits for analyses of biological samples are excerpted from the IDEM RFP and provided in Table 8 (Section A.7.6). Sample preservation and holding time requirements are listed in Section B.2.4. DQOs are detailed in Section A.7.1.

B.4.2. Standard Operating and Analytical Procedures

SOPs prescribe agreed upon steps needed to carry out an operation, analysis, or action. In other words, an SOP constitutes the approved method for performing a specific routine function or repetitive task. All contract laboratories are required to provide SOPs to IDEM upon request. SOPs are required to meet the standards set forth in EPA G-6: Guidance on Preparing Standard Operating Procedures. SOPs or quality assurance plans are required to include information describing the analytical, safety, and corrective action procedures

B.4.3. Contract Laboratory Analytical Procedures

Each laboratory performing analytical tests for the OWQ WAPB is required to provide copies of SOPs upon request of the OWQ. Availability of laboratory SOPs is confirmed through laboratory proposal review and onsite audit, during the RFP process and annually thereafter. Written SOPs for all test methods and standard procedures including sample preparation and cleanup methods, if separate from the determinative

SOP; determinative test methods; initial and continuing calibrations and frequencies; and confirmatory methods are required. Table 7 (see Section A.7.1.6) lists the reporting limits and acceptable U.S. EPA analytical test methods for each analyte in this project.

B.5. Quality Control Requirements

B.5.1. Internal Quality Control Checks

WAPB staff utilizes the Preventative Maintenance Program (PMP) to track equipment inventory and related calibration and maintenance schedules to ensure accurate measurements and proper maintenance. A list of major equipment used for field measurement can be found in Table 14. There are no quality control samples collected in the field.

B.5.2. Contract Laboratory Quality Control Checks

Each contract laboratory (Pace) providing analytical services to the OWQ WAPB is expected to meet the statement of work and technical specifications of the IDEM RFP in compliance with contract requirements. Laboratory quality control checks for biological samples may be found in the Technical Specifications of the IDEM RFP. See Appendix H6 of IDEM RFP 16-88 (IDEM 2016b). Table 13 summarizes the laboratory quality control checks for test method procedures. Refer to individual test methods for specific requirements.

Table 13. Laboratory quality control check frequency

Parameters and Test Procedures	Calibration and/or Verification	Sample Lab Duplicate	MS/MSD	LCS	External QC Standard	Surrogate
% Lipid	daily	1/20	n/a	1/run	n/a	n/a
% Moisture	daily	1/20	n/a	1/run	n/a	n/a
PFAS	daily	1/20	1/20	n/a	4/day	n/a
PCBs	daily	1/20	1/20	1/20	4/day	every sample
Metals	daily*	1/20	1/20	1/20	1/20	n/a
Pesticides	daily	1/20	1/20	1/20	4/day	every sample

* Continuing calibration verification standards (CCVs) shall be run at the beginning and end of a run batch and at a rate of 1/10.

B.6. Preventative Maintenance

Preventive maintenance is the planned upkeep program for measurement instruments which enhances the instrument performance, ensures accurate and precise readings, and prolongs useful life.

B.6.1. Field Equipment Preventative Maintenance

Table 14 summarizes major field equipment and the maintenance frequency. Visual inspection of all equipment occurs on every day of use to ensure crew safety and efficient sampling effort is maintained.

Table 14. Field Equipment Maintenance

Equipment Item	Maintenance Item	Frequency	Inspection	Testing
Boat	Maintenance or Repair	As Needed	Day of use	Annually
Boat Motor	Maintenance of engine oil, lower gear lube, propellers, state of turn engine	Annually	Day of use	Annually
Boat Trailer	Inspection, repack wheel bearings, check lights, bunks and tires	Annually	Day of use	Annually
Electro Shock Box	Factory Calibration	Every two years	Day of use	Annually
Electro Shock Backpack	Factory Calibration	Every two years	Day of use	Annually
Fire Extinguisher	Inspection	Annually	Day of use	Annually
Generator	Maintenance	Annually	Day of use	Annually
GPS Unit – Trimble Juno	Update Software	As new software becomes available	Day of use	Annually
Weighing Scale	Calibration	Every two years	Day of use	Every two years

Maintenance records are kept within the Preventive Maintenance Plan (PMP), an electronic equipment management system used to track equipment inventory, and related calibration and maintenance schedules, for each measurement instrument. It includes the preventive maintenance schedule, their frequency, location of maintenance, parts kept in supply, location of part (if not with instrument). Each section maintains hard copy logs of equipment calibration and equipment status.

B.6.2. Laboratory Equipment Preventative Maintenance

Preventive maintenance procedures at set frequencies for all analytical instruments and measurement equipment used in the performance of analytical services for the OWQ are required of each analytical laboratory in accordance with IDEM RFP. Written SOPs demonstrating the contract

laboratory is capable of providing the services requested in the IDEM RFP, (Technical Specifications), are required of each contract laboratory. Preventive maintenance is an element of laboratory system audits, testing, and inspection.

B.7. Calibration Procedures and Frequency

Measurement equipment requires periodic calibration or standardization in order to reliably produce accurate results. IDEM requires contract labs to follow this QAPP and WAPB RFP 16-88 (IDEM 2016b). Both documents require a quality system to be in place which includes standards for calibration and corrective actions. In addition, IDEM may elect to request pertinent QA data, including calibration standards, upon request.

B.7.1. Contract Laboratory Instrument Calibration

Contract laboratories, providing analytical services to the WAPB water quality monitoring programs (Pace), are required to document calibration procedures and frequency in compliance with WAPB RFP 16-88 (IDEM 2016b). These requirements specify a QA system must be in place and QA/QC data related to this project must be available to IDEM.

B.8. Inspection and Acceptance Requirements for Supplies and Consumables

Quality assurance of consumable supplies is required to reliably produce accurate results. IDEM requires contract laboratories follow this QAPP and WAPB RFP 16-88 (IDEM 2016b). Both documents require a quality system to be in place which includes standards for consumables and corrective actions for nonconformities. In addition, IDEM may elect to request pertinent QA data, including inspection criteria for supplies, upon request.

B.8.1. WAPB Requirements

There are no critical field elements which require inspection or approval prior to the use for sample collection activities.

B.8.2. Contract Laboratory Requirements

Contract laboratories providing analytical services to the WAPB water quality monitoring programs (Pace) have demonstrated proficiency through past performance and response to RFP 16-88 (IDEM 2016b). In addition, all contract laboratories are required to have a QA plan in place which includes acceptance criteria for consumables and supplies. The IDEM PI will review invoices submitted from the contract laboratories for reasonableness, including monitoring for unwarranted or excessive usage of consumable supplies.

B.9. Use of Existing Environmental Data

The IDEM 2020 fish tissue project has also selected sites in the Great Lakes basins from which sites will be analyzed for contaminants identified in this project. This data will be pooled for data analysis in order to maximize spatial coverage of the region. The QAPP will not use any other existing data.

B.10. Data Management

State hardware and software requirements are determined by the Indiana Office of Technology (IOT) and are related in Section 6 of the IDEM Quality Management Plan (IDEM, 2018). Detailed information can be found in Section D of this QAPP.

C. Assessment and Oversight

C.1. Quality Assurance Assessment Actions

IDEM's PI is ultimately responsible for conducting assessments and response actions to ensure the project is being implemented in such a manner as to accomplish the project objectives. The WAPB operates a robust QA system, including a mechanism for performing system audits and reporting and addressing observed nonconformities. This system also extends to laboratory contracts principally overseen by WAPB staff, such as those with Pace.

C.1.1. Field Performance and System Audits

WAPB conducts two kinds of audits to verify quality control procedures are being followed and the QA system is functioning effectively. The performance audit is an independent review of internal quality control checks and procedures. The system audit, on the other hand, is an onsite review and evaluation of facilities, instrumentation, quality control practices, data validation, and documentation practices. Data quality is evaluated by the OWQ WAPB staff after each sampling event in order to assess data usability. Field performance measurements include:

- Precision: RPD between field duplicate measurements.
- Accuracy: %R of field references.
- Completeness: % planned samples which are actually collected, analyzed, reported, and useable for each project.

WAPB QA staff perform a system audit once a year before or at the beginning of the field season. Specific system audits are performed throughout the field season on data collection and sampling procedures to ensure continuity of data acquisition and determine person to person variability. System audits include, but may not be limited to:

- Sampling work plan reviews, including DQOs and target parameters.
- Equipment calibration, maintenance, and frequency.
- Field data collection procedures.
- Sample collection and chain-of-custody procedures.

Field audits are conducted to ensure sampling activities adhere to approved SOPs. Audits are systematically conducted by WAPB QA staff to include all WAPB personnel engaged in field sampling activities. WAPB field staff involved with sample collection and preparation are evaluated by QA staff trained in the associated sampling SOPs, and in the processes related to conducting an audit. QA staff produce an evaluation report documenting each audit for review by field staff audited, as well as WAPB management. Corrective actions are communicated to, and implemented by, field staff as a result of the audit process.

C.1.2. Contract Laboratory Performance and System Audits

Analytical laboratory results are audited for performance using quality control checks. QA staff audit reported results and included data deliverables at 100% frequency. Each sample set is reviewed, and the data usability is determined. Laboratory performance measurements include:

- Precision: RPD of (LCS/LCSD) or (MS/MSD) pairs.
- Accuracy: %R of MSs, LCS, or surrogates.
- Completeness: % of samples delivered to the laboratory which are analyzed, reported, and useable for each project.

Laboratory system audits are performed by the QAO or designee at the beginning of a laboratory contract and at least once a year during the contract. The results are reported to the WAPB QAO. The system audit includes any or all of the operational quality control elements of the laboratory's QA system. All applicable elements of this QAPP and the laboratory contract requirements are addressed including, but not limited to, those listed in Table 15.

Table 15. Laboratory system audit elements

Procedure	Related Activities
Sample handling	Receiving, custody procedures, log in, storage, retention
Sample analysis	Written SOPs consistent with acceptable U.S. EPA test methods or IDM RFP specifications; availability to analysts; inclusions of QC samples specified in the RFP.
Record keeping	Written SOPs for all procedures associated with sample handling, analysis, and reporting.
Preventative maintenance	Written SOPs and records for equipment maintenance.

Proficiency testing	Analysts individually perform U.S. EPA reference sample analyses.
Personnel requirements	Laboratory personnel meet contract required experience requirements.
Training	Documented technical staff training and experience appropriate for tasks assigned.
Workload	Holding times are met and sufficient manpower is available to handle the workload.
Other	Any operational laboratory procedure influencing sample results, reporting, or procedural documentation.

C.2. Quality Assurance Reports to Management

C.2.1. QA Reports

QA reports are submitted by the WAPB QAO, upon completion of the data validation of a dataset, to the program manager, in this case, the project PI. This is done to ensure problems arising during the sampling and analysis phases of the project are investigated and corrected. Each report addresses:

- Data assessment and qualification results since the last report.
- Field and laboratory audits performed since the last report.
- Significant QA system and quality control task problems.
- Recommended solutions, and status of corrective actions.
- Status of the extent to which project DQOs have been satisfied.

The QA manager, relevant WAPB section chief, project PI, any technical staff working on corrective actions, and QA staff receive copies of the progress reports when new developments arise. The corrective actions progress reports are stored in IDEM's Virtual File Cabinet along with the project correspondence and are available for any interested parties.

C.2.2. Reports to the IDEM Principal Investigator

The State of Indiana's contract legal boilerplate language specifically requires professional services contractors to submit written progress reports to the state, upon request, in order to assure work is progressing in line with the schedule, and completion can be reasonably assured on the scheduled date. As such, quarterly project reports and a final report shall be submitted to the PI by the contract laboratories. The quarterly reports shall include all information; work collected and conducted during the reporting quarter; a narrative of the work completed under each task of the individual contracts; and identification of any issues impacting project status or data quality. The PI will review all progress reports prior to

approving invoices for expenses incurred by the contractors. The PI will also compile comprehensive progress reports to be distributed to the U.S. EPA project officer. These reports are due on April 30 and October 30 of each calendar year. The PI will also provide additional updates regarding project progress, as requested by the U.S. EPA. The final report shall consist of all laboratory results, including the report items described in Section A.9. demonstrating the data met the DQOs in Section A.7.1 and the project outputs described in Section A.6.5.

C.2.3. Corrective Actions

A field or laboratory nonconformity may be identified by any WAPB technical staff. Once identified, the project PI is responsible for corrective action in concert with the pertinent WAPB section chief or QAO. The PI works with the section chief, QAO, or other pertinent staff to document the nonconformity, and then develop and implement corrective actions. Depending on the nonconformity and associated corrective actions, the WAPB section chief or the QAO may need to approve the final corrective action.

C.2.3.1. Field Corrective Action

The field crew chief assigned to the sampling event is responsible for all field decisions, including corrective action. Any unusual or unexpected occurrence during data or sample collection is brought to the attention of the crew chief, who decides what actions should be taken immediately and what actions, if any, are necessary as a follow up. Field corrective actions are at the discretion of the field crew chief and are documented by the crew chief upon return to the office. The section chief will assign a staff member to follow up and document any further required action.

C.2.3.2. Laboratory Corrective Action

Each analytical or contract laboratory conducting analyses for OWQ is required to maintain a corrective action program as indicated in the technical specifications of WAPB RFP 16-88 (IDEM 2016b). The laboratory is required to document any corrective actions taken as a result of problems during the handling, preparation, analysis, or reporting of analytical data to the IDEM OWQ WAPB. Corrective actions are documented in the case narrative section of the report for each sample set. Problems indicating the laboratory QA system may be out of control will trigger a system audit by the QAO or a designee.

Significant nonconformities are to be reported to the IDEM project PI within 14 days, in accordance with the contract. Once

identified, the project PI is responsible for ensuring the corrective action is implemented. If the contractor and the IDEM PI cannot come to an agreement on corrective actions or project progress is irreparably harmed, IDEM may refuse payment or conduct other corrective actions through the contract agreement itself. Several clauses in the State of Indiana contract legal boilerplate language apply and are paraphrased below:

- Substantial Performance – The contract is deemed to be substantially performed only when fully performed according to its terms and conditions and any written amendments or supplements.
- Termination for Default – The state may terminate the contract in whole or in part if the contractor fails to:
  - Correct or cure any breach of the contract.
  - Deliver the supplies or perform the services within the time specified in the contract or extension.
  - Make progress so as to endanger performance of the contract.
  - Perform any of the other provisions of the contract.
- Waiver of Rights – States, in part, the contractor shall be liable to the state in accordance with applicable law for all damages to the state caused by the contractor's negligent performance of any of the services furnished.
- Work Standards – States the contractor shall apply the highest professional and technical guidelines and standards. Further states, if the state becomes dissatisfied with the work product of or the working relationship with those individuals assigned to work on the contract, it may request in writing the replacement of any or all such individuals.

Finally, in the event any problem is identified with QA or any changes are necessary to the QAPP, recommendations will be made to the project and QA manager. Any necessary changes will be communicated to the project team.

D. Data Validation and Usability

D.1. Data Review, Verification, and Validation

Data reduction, validation, and reporting, for both field and laboratory activities, are explained in this section. These activities are performed by field staff for data acquired in the field and by the contract laboratory in compliance with IDEM RFP requirements for the samples analyzed.

All data recording on field sheets by the data recorder are mirrored back to the field staff taking the measurements to verify the correct observation is recorded.

All field sheets are reviewed for completeness and legibility. All data entry is verified by two rounds of QA/QC data verification. These data verification rounds are conducted by two different staff members.

Data reduction is the process of converting raw analytical data into final results in proper reporting units. All data reduction for analysis and assessments will be conducted or confirmed in-house as resources allow. Contract laboratories will report the raw data and accompanying quality control information for interpretation on the quality of the data.

WAPB chemists produce QA reports for each analysis set. The QA reports to management, include data validation and usability to ensure good quality data for this project. A QA audit report will be submitted for this project should problems arise, with the contract laboratory which warrant a laboratory audit, and need to be investigated and corrected Surface Water QAPP, pp 177-178 (IDEM 2017a).

Each of the items below are reviewed for field samples and all laboratories to ensure they are complete and acceptable.

- Chain of Custody:
  - Sampler signature
  - Custodian signature
  - Containers
  - Collection date(s)
  - Receiving time(s)
  - Receiving date(s)
  - Samples received and stored at proper temperatures
- Quality Control (QC) Checks and Compliance:
  - Summary data package
  - Approved analytical methods
  - Approved detection and reporting limits
  - Prep dates
  - Analysis dates
  - Holding times
  - Initial, continuing, method, field, and trip blanks (< CRQL, MRL, or control limit)
  - Method duplicate RPDs
  - MSs and MSDs RPDs
  - Surrogates (< CRQL or control limit)
  - Internal standards (70% to 100%)
  - Instrument calibrations (Correlation Coefficient > 0.995)
  - Initial and continuing calibration verification standards (+ 10%)
  - ICP interference check standards (< CRQL or < control limit; + 20%)

- ICP serial dilutions (+ 10%)
- System performance

D.2. Data Verification and Validation Methods

Data validation is the process of qualifying analytical or measurement data on the performance of the field and laboratory quality control measures incorporated into the sampling and analysis procedures. WAPB field staff apply several levels of verification to the project data. Upon field data capture, one staff member will take the observation and read the result aloud to the other staff member, who records the data. The recorder then verifies the result is recorded correctly by reading the value aloud back to the observer. Prior to entering data into AIMS II database a completeness is run on the field data sheets. This includes a verifying all applicable fields are filled in and are legible to both field staff conducting observations. Finally, these data are double-keyed into the AIMS II database from the original field data sheets. Any discrepancies are then resolved. In addition, data collection in the field is subject to the QC checks described in Section B.5.2 and the calibration checks described in Section B.7.1.

Analytical laboratories are responsible for validating data from samples analyzed in the laboratory. WAPB QA staff review laboratory validation results and perform an additional level of data validation for 100% of the data received from a contract laboratory. This independent validation is conducted based on data flags and other QA/QC information obtained from the contract laboratories.

Data reporting is the detailed description of the data deliverables used to completely document the calibration, analysis, quality control measures, and calculations. Data acquired in the field are reported after reduction and validation by the responsible technical staff. Data from WAPB contract laboratory analyses are reported after laboratory reports the data are reviewed, assessed for QA, and the data usability is determined by assigning 1 of 4 DQA Levels to the data. See Section A.9. for a list of records and documents in this project.

D.3. Reconciliation with User Requirements

D.3.1. Data Quality Assessment

DQA is the process of determining the scientific and statistical quality of data collected to satisfy the project DQOs. Field data and laboratory results are assessed for usability with regard to each specific project DQOs (Section A.7.1). Section D.1 on Data Reduction, Validation, and Reporting; and Section C.1.2 on performance and system audits describe the procedures used to produce data and to evaluate the data production system effectiveness.

D.3.2. Data Quality Assessment (DQA) Levels

Data from WAPB contract laboratory analyses are reported after laboratory reports the data are reviewed, assessed for QA, and the data usability is determined by assigning 1 of 4 DQAs Levels to the data.

DQA Level 1 **Screening Data:** The results are usually generated onsite and have no QC checks. Analytical results, which include no QC checks, precision or accuracy information, or detection limit calculations are included in this category. Primarily, onsite data are used for presurveys and for preliminary rapid assessment.

DQA Level 2 **Field Analysis Data:** Data is recorded in the field or laboratory on calibrated or standardized equipment. Field duplicates are measured on a regular periodic basis. Calculations may be done in the field or later at the office. Analytical results with limited QC checks are included in this category. Detection limits and ranges have been set for each analysis. The QC checks information for field or laboratory results is useable for estimating precision, accuracy, and completeness for the project. Data from this category are used independently for rapid assessment and preliminary decisions.

DQA Level 3 **Laboratory Analytical Data:** Analytical results include QC check samples for each batch of samples from which precision, accuracy, and completeness can be determined. MDLs have been determined using 40 CFR Part 136 Appendix B. Additionally, all reporting information required in the laboratory contract, and in the Surface Water QAPP, especially Table A9-1, are included in the analytical data reports. Raw data, chromatograms, spectrograms, and bench sheets are not included as part of the analytical report but are maintained by the contract laboratory for easy retrieval and review upon request from WAPB. Data can be elevated from DQA Level 3 to DQA Level 4 by inclusion of this information in the data report and the QC data are reported using U.S. EPA required contract laboratory program (CLP) forms or CLP format. Data falling under this category are considered as complete, legally defensible, and used for regulatory decisions.

DQA Level 4 Enforcement Data: Analytical results mostly meet the CLP data analysis, CRQL, and validation procedures. QC data are reported on CLP forms or CLP format. Raw data, chromatograms, spectrograms, and bench sheets are included as part of the analytical report. Additionally, all reporting information required in the laboratory contract, and in the Surface Water QAPP, especially Table A9-1 (2017a,) are included in the analytical data reports. Data falling under this category are considered as

complete, legally quantitative in value, and used for regulatory decisions.

D.3.2.1. Data Qualifiers and Flags For WAPB Contract Laboratories Laboratory data is reviewed and qualified by QA staff using U.S. EPA CLP guidance for data validation. Data flags have two parts, a cause (U, Q, D, B, or H) and an action (R or J). For WAPB projects, data qualifiers and flags are assigned and entered into AIMS II. The flags are used for both the individual test result and QA/QC Review Reports. Table 16 lists data qualifiers and Table 17 lists data quality flags used for analytical results.

Table 16. WAPB Data Qualifiers

Qualifier	Description
R	**Rejected.** Result is not acceptable for use in decision making processes.
J	**Estimated.** The use of the result in decision making processes will be determined on a case-by-case basis.
U	**Estimated (Between MDL and RL).** The result of the parameter is above the MDL but below the Laboratory Reporting Limit (RL) and will be estimated.
Q	**QC Checks or Criteria.** One or more of the QC checks or criteria are out of control.

Table 17. WAPB Data Flags

Flags	Description
D	**RPD for Duplicates.** The RPD for a parameter is outside the acceptable control limits. The parameter will be considered estimated or rejected on the basis listed below: 1) If either the sample or duplicate value is less than the RL and the other value exceeds 5 times the MDL, then the sample will be estimated. 2) If the RPD is outside the established control limits (max. RPD) but below two times the established control limits (max. RPD), then the sample will be estimated. 3) If the RPD is twice the established control limits (max. RPD) or greater, then the sample will be rejected.
H	**Holding Time.** The analysis for this parameter was performed out of the holding time. The results will be estimated or rejected on the basis listed below:

	1) If the analysis was performed between the holding time limit and 1.5 times the holding time limit, the result will be estimated. 2) If the analysis was performed outside the 1.5 times the holding time limit, the result will be rejected.
B	**Blank Contamination** This parameter is found in a field or a lab blank. Whether the result is accepted, estimated, or rejected will be based upon the level of contamination listed below: If the result of the sample **is greater than the RL** but **less than five times** the blank contamination, the result will be **rejected**. 1) If **the result of the sample is** between five and ten times **the blank contamination, the result will be** estimated**.** **2) If the result of the sample is less than the RL or greater than ten times the blank contamination, the result will be accepted.**

D.3.2.2. Data Assessment Guidelines
References are used by WAPB QA staff as guidelines in assessing data quality and usability and in assigning data qualifiers. The flags are listed in the Surface Water QAPP, Section D.3.2.2 (IDEM 2017a).

D.3.2.3. Data Assessment and Qualification Corrective Action
Problems arising during data assessment and qualification which are due to any contract laboratory or QA actions are brought to the attention of the project PI who will work with other staff as necessary to determine whether immediate corrective action is required. Laboratory corrective actions are implemented according to the respective IDEM RFP and contract requirements.

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F. Distribution List

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Appendix 1. Field Record for Biological Tissue Contaminants Monitoring Program

Indiana Department of Environmental Management
Office of Water Quality - Watershed Assessment and Planning Branch

Field Record for Biological Tissue Contaminant Monitoring Program

Site ID:_____ Sampling Date and Time:_____
Sample ID:_____ (mm/dd/yyyy) (24hr clock)

SITE LOCATION
Waterbody Name:_____
County_____Fipscode_____Lat./Long.:_____
Location:_____
Waterbody Type: RIVER LAKE RESERVOIR WETLAND
Site Description:_____
_____
Collection Method:_____
Collector's Name(s):_____
Agency:_____Phone:(____) _____

FISH (or other organism) COLLECTED

Composite Sample #:_____Number of Individuals:_____ Lab ID _____
Species Name:_____
Sample Preparation: SKIN-ON SCALELESS SKIN-OFF WHOLE OTHER:_____

Fish#	Length(mm)	Weight(gm)	Sex(M,F)	Fish#	Length(mm)	Weight(gm)	Sex(M,F)
001				007			
002				008			
003				009			
004				010			
005				011			
006				012			

(min length/max length)x 100 = _____% **Composite mean length** _____ **mm**
(min wt/max wt)x 100= _____% **Composite mean weight**_____ **gm**
Notes (e.g., DELT anomalies)_____
_____

Composite Sample #:_____Number of Individuals:_____ Lab ID _____
Species Name:_____
Sample Preparation: SKIN-ON SCALELESS SKIN-OFF WHOLE OTHER:_____

Fish#	Length(mm)	Weight(gm)	Sex(M,F)	Fish#	Length(mm)	Weight(gm)	Sex(M,F)
001				007			
002				008			
003				009			
004				010			
005				011			
006				012			

(min length/max length)x 100 = _____% **Composite mean length** _____ **mm**
(min wt/max wt)x 100= _____% **Composite mean weight**_____ **gm**
Notes (e.g., DELT anomalies)_____

Appendix 2. Contract Laboratory Chain of Custody Form

CHAIN-OF-CUSTODY / Analytical Request Document

The Chain-of-Custody is a LEGAL DOCUMENT. All relevant fields must be completed accurately.

Section A Required Client Information:	Section B Required Project Information:	Section C Invoice Information:
Company:	Report To:	Attention:
Address:	Copy To:	Company Name:
		Address:
Email To:	Purchase Order No.:	Pace Quote Reference:
Phone: Fax:	Project Name:	Pace Project Manager:
Requested Due Date/TAT:	Project Number:	Pace Profile #:

Page: of

REGULATORY AGENCY

☐ NPDES ☐ GROUNDWATER ☐ DRINKINGWATER

☐ UST ☐ RCRA OTHER ☐ _____

Site Location STATE: _____

Requested Analysis Filtered (Y/N)

ITEM #	Section D Required Client Information — SAMPLE ID (A-Z, 0-9 / , -) Sample IDs MUST BE UNIQUE	Valid Matrix Codes: MATRIX / CODE — DRINKING WATER DW WT; WATER WW P; WASTE WATER SL; PRODUCT OL; SOIL/SOLID WP; OIL AR; WIPE OT; AIR TS; OTHER; TISSUE	MATRIX CODE (see valid codes to left)	SAMPLE TYPE (G=GRAB C=COMP)	COLLECTED: COMPOSITE START DATE	COMPOSITE START TIME	COMPOSITE END/GRAB DATE	COMPOSITE END/GRAB TIME	SAMPLE TEMP AT COLLECTION	# OF CONTAINERS	Preservatives: Unpreserved	H_2SO_4	HNO_3	HCl	NaOH	$Na_2S_2O_3$	Methanol	Other	Analysis Test ↓ Y/N	Residual Chlorine (Y/N)	Pace Project No./ Lab I.D.
1																				
2																				
3																				
4																				
5																				
6																				
7																				
8																				
9																				
10																				
11																				
12																				

ADDITIONAL COMMENTS	RELINQUISHED BY / AFFILIATION	DATE	TIME	ACCEPTED BY / AFFILIATION	DATE	TIME	SAMPLE CONDITIONS

SAMPLER NAME AND SIGNATURE		Temp in °C	Received on Ice (Y/N)	Custody Sealed Cooler (Y/N)	Samples Intact (Y/N)
PRINT Name of SAMPLER:					
SIGNATURE of SAMPLER:	DATE Signed (MM/DD/YY):				

Appendix 3. Sampling Collection Procedure

The following is a general summary of procedures for collection, preparation, and preservation of all fish tissue samples collected for contaminant analysis.

Step 1. Sample the fish community using electrofishing gear following sampling equipment SOPs (U.S. FWS 1998, IDEM 1992a). In some cases, lakes will be sampled using gill nets (IDEM 1992a).

Step 2. Examine fish collected and select the predetermined number of samples for the site (generally between three and five samples). A sample may be comprised of 1-12 fish of the same species, depending on size. The preferred total lengths of the smallest and largest individuals of any composite sample should be within 90% of each other. Total lengths as low as 75% will be tolerated in order to obtain an adequate composite sample.

Samples collected from a site preferably consist of: (1) species collected historically from the site, (2) different size classes of predator species, or (3) Common Carp. In addition, species and size classes listed in Indiana Fish Consumption Advisories, should be targeted to support updates to the advisory's information.

Representative samples from sites, with no historical species samples, should target a bottom feeder species (e.g., Common Carp), a predator game fish species (e.g., Largemouth Bass, Channel Catfish, or Flathead Catfish), and a panfish species commonly consumed by humans (e.g., sunfish species, crappie species, Rock Bass).

In addition, samples from sites targeted based on potential or known contamination, should include fish species with small home ranges to ensure the data results are indicative of the conditions in the stream at the site. All other fish captured during the sampling effort should be released back into the water.

Step 3. Fish selected as samples should be placed in a cooler and euthanized by covering with ice to prevent decomposition prior to sample processing. Using a piece of label tape, tag the outside of the cooler with site information, including the sample number (AB Number), site location, date, waterbody, and county information.

Step 4. Determine whether samples are going to be processed in the field or the laboratory (e.g., overnight travel necessitates field processing)?

Step 5. In the laboratory, place fish from one site into the laboratory sink, and sort fish into composite samples by matching total lengths for each species.

- If samples are processed in the field, fish species should be sorted in the cooler by length, as described above.

Step 6. Complete the site ID, event ID, date and time, and site location information on the Field Record for Biological Tissue Contaminant Monitoring Program form (Appendix 1).

Step 7. In the FISH (or other organism) COLLECTED section of the Field Record for Biological Tissue Contaminant Monitoring Program form (Appendix 1), fill out the composite sample number (AB number-taxon ID-species sample number e.g., AB24997-043-01), the number of fish in the composite sample, the species name, and the preparation method (whole fish, skin-on scaleless fillets, skin-on scales-on fillets, skin-off fillets, beheaded and gutted, etc.).

Step 8. Measure and record the total length in millimeters (to the nearest millimeter) and weight in grams (to the nearest gram) of each fish within a composite sample. Also note any individual fish anomalies, such as deformities, eroded fins, lesions, tumors (DELTs).

Step 9. Are there more composite samples?
- Yes, repeat Step 8.
- No, proceed to Step 10.

Step 10. Prepare filleting stations by setting out dedicated food grade plastic low density polyethylene cutting boards or covering workstation with clean aluminum foil. Set out the fish scale removers, scalpel, fish skinning pliers, and stainless-steel fillet knives used for filleting. Sharpen knives as necessary to reduce ragged cuts and slippage, which often occurs when dull knives require increased pressure during cutting. Staff should wear new nitrile or latex gloves for each site in order to minimize the potential of contaminants transferring from hands to tissue samples.

Step 11. Fish fillet samples will be prepared as **skin-on scaleless** fillets, for scaled species and **skin-off** fillets, for scaleless species (Anderson et. al. 1993; IDEM 1992b). Remove scales from both sides of the fish, back, and belly areas, if necessary. Fillet the fish so as to include all flesh from the back of the head to the tail and from the top of the back down to and including the belly flap area of the fish. Fins, tail, head, viscera, and major bones are to be removed. If sampling at a historically contaminated site or investigating possible source of contamination and the fish sample is to be analyzed whole, the composite sample consisting of these fish does not have to be filleted. Place each fillet from the composite sample into the same stainless-steel bucket containing tap water, or ambient water, if processing in the field. The water in the bucket is used to rinse off any soil, scales, or mucous adhering to the sample.

Step 12. Drain excess water and **double wrap** the fillets in clean aluminum foil to make a package. For whole fish, all individual fish of a composite sample should be double wrapped together if possible. Using a blunt

tip black permanent marker, label the outside of the package with the following information:

- Sample number (e.g., AB49005-121-01)
- Number of individuals in the sample
- Species of fish (common name)
- Sample preparation (e.g., scaleless, skin-on fillets; scaleless, skin-off fillets; scaleless, whole fish)
- Waterbody name
- County
- Location description
- Date of collection (format: 01-March-2015)
- Package count if more than one package per sample (e.g., 1 of 2, 2 of 2)

Example:
AB13201-043-01
3-Common Carp, skin-on fillets, scaleless
East Fork White River
Martin Co.
Shoals
21 Aug 2017

Step 13. Each foil package will be placed individually into an appropriate size zip-lock type bag or other food grade plastic bag, and sealed. Using a blunt tip black permanent marker, label the outside of the package with the following information:

- Sample number (e.g., AB49005-121-01)
- Package count if more than one package per sample (e.g., 1 of 2, 2 of 2)

Step 14. Tissue samples need to be frozen as soon as possible. Is the processing taking place in the field?

- If yes, it may be necessary for field staff to bring dry ice for overnight field trips. Use a dry ice chest with dry ice for storage of the fish tissue. Dry ice will be provided by the contracted analytical services laboratory for the project. However, the laboratory needs to be notified at least a week in advance so the contract laboratory can make arrangements for dry ice delivery to the Western Select Building's office location before staff depart for the field. Place the double wrapped and bagged samples in the dry ice chest underneath the dry ice for preservation of the tissue. Upon return to the office, place all processed tissue samples in the chest style analytical-grade freezer located in the IDEM WAPB laboratory or the upright commercial-grade freezer, located in building 41.
- If no, place double wrapped and bagged samples in the chest style analytical-grade freezer located in the IDEM WAPB laboratory room

124 or the upright commercial-grade freezer located in building 41 of the Western Select Building.

Step 15. Are there more composite samples to be processed?
- If yes, proceed to Step 11.
- If no, proceed to Step 16.

Step 16. Wash all stainless-steel buckets, stainless steel filleting knives, scalers, sharpening steels, mass scales, measuring boards, cutting boards, and coolers with ALCONOX® detergent (laboratory quality environmental cleaning product); and a scrub brush. Then thoroughly rinse. Wipe down countertops with an ALCONOX® soaked rag. Throw away used nitrile or latex gloves.

Step 17. Are there more sites to be processed?
- If yes, proceed to Step 5.
- If no, proceed to Step 18.

Step 18. Handling cleaned equipment:

- If processing samples in the laboratory, put all cleaned processing equipment on the drying racks or in the storage bins with lids. Clean out the sink, mop the floor, and take trash bags full of fish carcasses to the dumpster. Leave the laboratory in the condition found so as not to disrupt the function of other staff programs.
- If processing samples in the field, sample processing equipment will be put into the storage bins and placed into the truck. Leave the processing area, usually near the boat ramp or nearshore, in the condition found as much as possible. Pick up as many fish scales as possible. Do not leave fish waste (i.e., entrails, filleted fish carcasses) or trash behind.

Step 19. Field sampling crew leader shall complete any missing information on the Field Record for Biological Tissue Contaminants Monitoring Program form (Appendix 1); calculate the composite length and weight; and the percent ranges for length and weight on all field data forms. Also, the crew leader should log all processed samples into the Nalgene® field sample logbook.

Step 20. The Field Record for Biological Tissue Contaminants Monitoring Program form (Appendix 1) is ready for entry into the AIMS II database.