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2023 – 2027 Fish Tissue Contaminants Monitoring Program

Quality Assurance Program Plan (QAPP)

B-060-OWQ-WAP-TGM-23-Q-R0

Agency Indiana Department of Environmental Management

Office Water Quality

Branch Watershed Assessment and Planning

Section Targeted Monitoring

Contract Number Professional Services Contract #53618

Principal Investigator Tim Fields

Effective Date September 26, 2023

Date Revised New

Revision Number 0

Review Cycle 5 year

QAPP Author Tim Fields

Indiana Department of Environmental Management

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A. Program Management

Elements A.1. through A.9. address basic program management (e.g., program history and objectives, roles, and responsibilities of participants, etc.). The elements ensure the program has a defined goal, the participants understand the goal and the planned approach, and planning outputs are documented.

A.1. Title and Approval Sheet	
Ali Meils, Section Chief Targeted Monitoring Section	9/20/23 Date
Charles Hostetter, Program Quality Assurance Officer Technical and Logistical Services Section	<u>9/27/23</u> Date
Timothy Bowner Timothy Bowren, Program Quality Assurance Officer Technical and Logistical Services Section	9/27/2023 Date
David Jordan David Jordan, Program Quality Assurance Officer Technical and Logistical Services Section	$\frac{9/26/23}{\text{Date}}$
Kristen Arnold, Branch Chief, Branch QA Coordinator Watershed Assessment and Planning Branch	<u>9/36/23</u> Date
The IDEM quality assurance staff participated in the development of assurance program plan.	of this quality
Patrick Colcord	9/28/23
Quality Assurance Staff Office of Program Support	Date

Quality Assurance Program Plan Summary:

The Fish Tissue Contaminants Monitoring Program's quality assurance program plan (QAPP) outlines environmental data collection from biological tissues as part of the Watershed Assessment and Planning Branch (WAPB), Office of Water Quality (OWQ), Indiana Department of Environmental Management (IDEM) water quality monitoring programs. The QAPP provides WAPB staff involved in the fish tissue monitoring program a format for environmental data collection targeted to achieve specific program data quality objectives (DQOs) and data usability identified by assigning one of four data quality assessment (DQA) levels for regulatory decisions. The QAPP describes procedures to implement for obtaining environmental data of known quality and adequate for decision-making.

The QAPP serves as a guide to WAPB program officers, field staff, and quality assurance (QA) staff charged with the collection and review of contaminants data in biological tissues. The QAPP also provides guidance for the contract laboratory charged with the analysis of OWQ environmental samples and provision of results meeting the DQOs for the individual program. The QAPP satisfies United States Environmental Protection Agency (U.S. EPA) requirements for environmental data collection programs funded in whole or in part by U.S. EPA grants. Successful collection of precise, accurate, and complete data provides IDEM and U.S. EPA with defensible data to make decisions for implementation of programs and improving and maintaining water quality in the state of Indiana.

QAPP Contact Information

Program office:

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Principal investigator:

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Pace Analytical Services, LLC Tod Noltemeyer 1241 Bellevue Street Green Bay, WI 54302 Phone (608) 232-2200 Tod.Noltemeyer@pacelabs.com

Laboratory accreditation and performance testing information:

The Pace Analytical Services, LLC (Pace) Green Bay, Wisconsin (GB) laboratory offers organic and inorganic analysis, and a broad range of specialty services. Services include low level mercury, and U.S. EPA Contract Lab Program (CLP) level packages and electronic deliverables. In addition to routine environmental matrices, the Pace GB laboratory has expertise in sediment work, biological tissue analysis, and emergency response capabilities. The laboratory holds a broad base of analytical certifications in numerous programs (e.g., National Environmental Laboratory Accreditation Program (NELAP), and is certified in Wisconsin, Illinois, and other states).

The Pace Minneapolis, Minnesota (MN) laboratory performs Dioxin, PFAS and PCB congener analysis.

The Pace Duluth, Minnesota laboratory performs methyl mercury analysis using Method 1630 (EPA 2007b). No certification is available for the method.

No certification is currently available for PFAS.

All labs maintain multiple accreditations including accreditation to ISO 17025:2005 by the American Association for Laboratory Accreditation, NELAP, or both. Pace's certifications cover the following U.S. EPA test methods

- Method 8082A (EPA 2007a)
- Method 8081B (EPA 2007c)
- Method 8270D
- Method 6020A (EPA 2007)
- Method 1631E (EPA 2002)
- Method 1613B (EPA 1994a)
- Method 1668B (EPA 2008)
- Method PFAS DoD 35 (Pace 2020)

Pace Labs annually participate in Performance Studies using water and soil medias. The Pace GB laboratory National Environmental Laboratory Accreditation Conference (NELAC) certification, issued by the Florida Department of Health Certificate Number E87948, expires June 30, 2024.

The Pace GB laboratory State of Wisconsin Department of Natural Resources Accreditation under NR 149 FID 405132750, expires on August 31, 2023. The Pace MN laboratory NELAP certification, issued by the Minnesota Department of Health, Certificate Number 2446997, expires December 31, 2023.

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A.3. Distribution List

The IDEM staff listed in the table below will be notified by email and copied on the most recent version of this QAPP <u>each</u> time:

- This QAPP is revised and replaced by a more up-to-date version.
- This QAPP (effective for multiple years and to be repeated at numerous times and/or locations) has passed its expiration date so that it is either replaced by a reauthorized version of this same QAPP or a revised QAPP, or the program under which this QAPP was implemented, is ended.

Table 1: QAPP Distribution List

Name	QAPP Task(s)/Roles	Email
Kristen Arnold	WAPB Branch Chief	KArnold@idem.IN.gov
Ali Meils	Targeted Monitoring Section Chief	AMeils@idem.IN.gov
Caleb Rennaker	WAPB Technical and Logistical Section	CRennake@idem.IN.gov
Tim Fields	IDEM Principal Investigator (PI)	TFields@idem.IN.gov
Todd Davis	IDEM crew leaders – sample collection and data management	TDavis@idem.IN.gov
Tim Bowren Charles Hostetter David Jordan	WAPB QA staff – WAPB QA Manager – Review laboratory data results	TBowren@idem.IN.gov CJHostet@idem.IN.gov DJordan@idem.IN.gov
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Mike Mettler Grace Bassett	Indiana Department of Health (IDOH) – Fish tissue data user	MMettler@isdh.IN.gov GBassett@isdh.IN.gov
Pat Colcord	IDEM QA manager – QAPP review and approval	PCOLCORD@idem.IN.gov

A.4. Program Organization

Program Area Management

The WAPB branch chief, as program manager, is the final authority and responsible for managing all WAPB surface water quality monitoring programs and projects. Each section chief, as program officer, guides and supervises planning of environmental data collection programs and is responsible for quality control (QC) procedure implementation and program data collection activities.

Quality Assurance

The WAPB QA manager (QAM) coordinates all QA and laboratory activities and assigns QA officers (QAOs) to programs. QAOs coordinate and audit QA and QC (QA/QC) activities, review program QAPPs, act as liaison to external

laboratories, and report a program's QA aspects to management. QA staff perform data validation review, data assessment, data qualification, and internal performance and system audits for assigned programs under the direction of the QAM. Assigned QA staff review all work plans and standard operating procedures (SOPs) for compliance with the QAPP and verify DQOs are addressed by the respective work plan. Each laboratory performing data analysis for OWQ is responsible for data validation of results before reporting to WAPB. IDEM Requests for Quotation and Requests for Proposal (RFPs) require each contract analytical laboratory to appoint a QAO and have a written QA plan.

Field

The program manager is responsible for sampling and data collection efforts and assigns section staff to data collection duties. The program officer ensures section staff follow the QAPP during sampling and data collection activities, reports any nonconformities to the appropriate section chief, and documents and addresses the nonconformity through the corrective action process. The program officer notifies the QAO of the nonconformity and status of corrective action. The section chief typically assigns a technical staff member to document the nonconformity and institute required corrective actions.

Laboratory and Analytical Laboratories

IDEM RFPs set forth requirements and technical specifications for contract laboratory analysis of sediment and fish tissue samples for various contaminants or pollutants. Laboratories must have and maintain a documented QA/QC program capable of demonstrating data are a specified degree of precision and reliability. The Handbook for Analytical Quality Control in Water and Wastewater Laboratories, U.S. EPA 600/4-79/019 (EPA 1979) serves as a pattern for an acceptable QA/QC program. Laboratories must have the ability to validate each method used and each analysis performed using the method through the QA/QC program. Documenting QA/QC measures is a requirement. Laboratories must maintain all documentation and make available to OWQ for five years after the expiration date of the contract. Submission of QA/QC documentation is a requirement. Laboratories must maintain and document continual evaluation of the accuracy and precision of an analytical procedure, and the ability of individual analysts to perform the laboratory procedure.

Table 2 identifies individuals or organizations with a role in this QAPP.

Table 2: Key QAPP Individuals

Role	Name	Affiliation	Phone	Email
Program area manager	Kristen Arnold	IDEM	317-308-3142	KArnold@idem.IN.gov
PI	Tim Fields	IDEM	317-308-3184	TFields@idem.IN.gov
Program area QAM	Tim Bowren	IDEM	317-308-3181	TBowren@idem.IN.gov

QAPP development team	Tim Fields	IDEM	317-308-3184	TFields@idem.IN.gov
QAPP field oversight lead	Tim Fields	IDEM	317-308-3184	TFields@idem.IN.gov
Lead field auditor	Ali Meils	IDEM	317-308-3204	AMeils@idem.IN.gov
Staff responsible for ensuring all involved staff have access to the most up-to-date version of the QAPP	Tim Fields Pat Colcord	IDEM	317-308-3184 317-234-7134	TFields@idem.IN.gov PCOLCORD@idem.IN.gov
Primary data user or decision-makers	Ali Meils Tim Fields Paul McMurray Grace Bassett	IDEM IDEM IDEM IDOH	317-308-3204 317-308-3184 317-308-3210 317-233-7183	AMeils@idem.IN.gov TFields@idem.IN.gov PMcMurra@idem.IN.gov GBassett@isdh.IN.gov
Lead data reviewer	David Jordan Charles Hostetter	IDEM	317-308-3100 317-308-3369	DJordan@idem.IN.gov CJHostet@idem.IN.gov
Contacts for the contracted laboratory	Mary Christie Tod Noltemeyer	Pace	612-751-1374 608-232-3300	Mary.Christie@pacelabs.com Tod.Noltemeyer@pacelabs.com

IDEM QAPP Lines of Authority (Figure 1) depicts the relationships and the lines of communication among the key program individuals (Table 2) throughout the various stages of QAPP implementation. The QAPP is a product of planning by the PI, and a team of program area staff. Program staff could include any of the staff depicted in Figure 1 or other program area staff with expertise in the topic of the QAPP.

Nearly all standard IDEM data operations (QAPPs) include the same basic staff relationships, with the primary exceptions of either:

- More than one person may staff some positions.
- One person may staff more than one position shown in Figure 1.

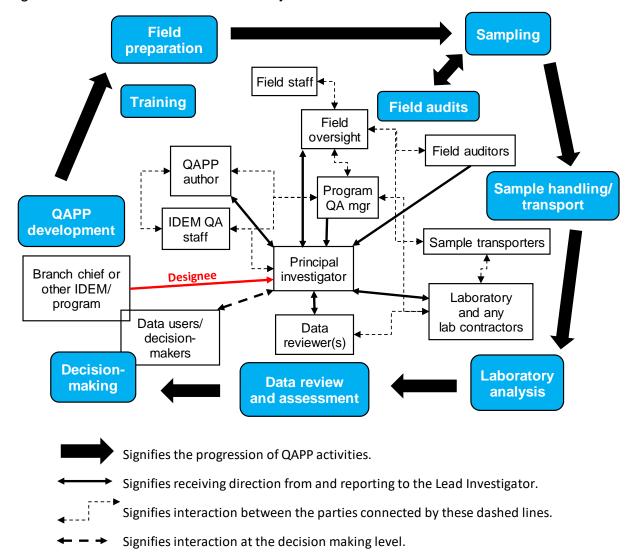


Figure 1 IDEM QAPP Lines of Authority

A.5. Background

A.5.1. The Study

OWQ environmental sample collection and analyses of fish tissues for various pollutants supports ongoing water quality monitoring activities. Indiana is required to assess and determine the designated use attainment status of all waters of the state. "Surface waters of the state are designated for full-body contact recreation and will be capable of supporting a well-balanced, warm water aquatic community" [327 IAC 2-1-3] and [327 IAC 2-1.5-5]. This program gathers data on bioaccumulating contaminants in fish.

Nine major river basins geographically divide the state for the purpose of contaminants monitoring, analysis, and assessment of surface water quality. Each year, one or two major river basins and the associated watersheds are sampled. Every five years, water quality assessments based on biological tissue data for the entire state are updated.

The study provides data to support decisions regarding aquatic life use impairment for total PCBs, total mercury, and total selenium based on the benchmarks outlined in Indiana's current Consolidated Assessment and Listing Methodology (CALM) (Tables 3 and 9)(IDEM 2022b). Water quality criteria for selenium is based on the EPA "2021 Revision to: Aquatic Life Ambient Water Quality Criterion for Selenium – Freshwater 2016" (EPA 2021). The Fish Consumption Guidelines (FCG) are based on the "Protocol for a Uniform Great Lakes Sport Fish Consumption Advisory for PCBs" (Anderson et. al. 1993); addendum for mercury (McCann and Anderson 2007); Discussion Paper for a Chlordane Health Protection Value (HPV) (Hornshaw); Potential for Human Exposure to Toxaphene Through Consumption of Great Lakes Fish (Wisconsin Department of Health Services 2015); and the "Best Practice for PFOS Guidelines" (GLC 2019).

The 2022-2026 Water Quality Monitoring Strategy (WQMS) (IDEM 2022c) describes the current program which follows a five-year rotating basin schedule. Twenty-three fixed station program sites from the original fish tissue sampling network exist. The network began operating in the late 1970s in cooperation with the U.S. EPA. Prior to 1997, fish tissue sample collection occurred on a biennial basis for the sites. Post 1997, sampling at the original fish tissue sampling sites occurs once every five years in accordance with the WQMS (IDEM 2022c) rotating basin methodology. In addition to the fish tissue sampling network, other sites are targeted based on historical environmental problems, waterbody access, use for fishing, date of last sampling event, potential contaminant sources, and monitoring recommendations by other agencies and entities.

Members of the QAPP planning team are listed in Table 2 (above).

A.5.2. The Goal

Collect and use fish tissue monitoring data for:

 Determining the aquatic life use impairment for fish consumption, based on concentrations of total PCBs, total mercury, and total selenium. The concentrations are ecological indicators in support of the Performance Partnership Agreement, Clean Water Act (CWA) § 305(b) reporting assessed waters, and CWA § 303(d) (IDEM 2022d) listing and reporting impaired waters. (Primary objective)

- 2. Providing data for use by the Interagency FCG work group supporting IDOH issuance, modification, or removal of fish consumption guidelines for specific Indiana water bodies.
- 3. Developing tools for regional assessment and classification of bioaccumulating contaminants (Table 7) in Indiana waters.
- 4. Providing data supporting the understanding of the risks to piscivorous wildlife (Table 7).
- 5. Evaluating contaminant trends in fish.
- 6. Understanding the presence of emerging contaminants in Indiana wild fish.

Table 3: Target Constituents for Decision-making

Sample Target	Number of Samples	Action Threshold**
Total PCB	1 sample	>20 for ALUS >50 for FCG
Total Mercury	1 sample	>300 for ALUS >50 for FCG (General Population) >160 for FCG (Sensitive Population)
Total Selenium***	1 sample	>15100 for ALUS (Egg-Ovary) >8500 for ALUS (Whole Fish) >11300 for ALUS (Filleted)
PFOS	1 sample	>20 for FCG
Chlordane	1 sample	>150 for FCG
Toxaphene	1 sample	≥111 for FCG (Whole Fish) ≥56 for FCG (Filleted)

^{*} ALUS=Aquatic Life Use Support

Collect an average of 3 to 5 composite or individual fish tissue samples per site. In addition, accept samples from other agencies using the same WAPB techniques to collect, prepare, and preserve samples. Sometimes, other offices or agencies collect fish tissue samples for analysis under IDEM's laboratory services contract (IDOA 2021). Samples are prepared using the whole fish or the edible portion (skin-on or skin-off fillets) of fish. Sample preparation (whole fish or fillets) depends upon the site location, size, and species of fish retrieved. Typically, whole fish are analyzed when processing noncarp minnow species, or fish less than or equal to ten centimeters (four inches) in length.

A.5.3. Inputs to be Used

Fish samples collected from Indiana streams and lakes each year in each chosen major river basin for analysis of bioaccumulating pollutants. Use data from the fish tissue samples to locate and identify contaminant

^{**}micrograms per kilogram wet weight

^{***}micrograms per kilogram dry weight

[®]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.

concentrations in the water column too low for easy detection with routine water sampling and analyses.

Compare data generated by this operation to the Table 4 standards to support the decisions using the data gathered.

The source of the standard or limit upon which each decision is based.

Table 4:Standards for Decision-making

Source of the Standard	Specific Citation	IDEM Derived Criteria values for 303(d) ALUS* Determination (µg/kg** ww)	Indiana FCG decision levels (µg/kg wet weight (ww))
ALUS	IDEM's Appendix G CALM (IDEM 2022b) Indiana Administrative Code ([327 IAC 2-1-3] and [327 IAC 2-1.5-5])	PCB > 20 ppb Mercury > 300 ppb Selenium (µg/kg dw) >15100 for ALUS (Egg-Ovary) >8500 for ALUS (Whole Fish) >11300 for ALUS (Filleted)	NA
PCB Action Level	Protocol For a Uniform Great Lakes Sport Fish Consumption Advisory.(Andersons et.al. 1993)	NA	>50 – 1900 limited consumption >1900 No consumption
Mercury Action Level	A Protocol for Mercury-based Fish Consumption Advice: An Addendum to the 1993 Protocol for a Uniform Great Lakes Sport Fish Consumption Advisory (McCann and Anderson 2007)	NA	>50 – 950 limited consumption for sensitive populations [®] >950 No consumption [®] >160 limited consumption for the general population
Perfluorooctane Sulfonate (PFOS) Action Level	Great Lakes Consortium for Fish Consumption Advisories Best Practices for Perfluorooctane Sulfonate (PFOS) Guidelines (GLC 2019)	NA	>20-200 limited consumption >200 No consumption
Chlordane Action Level	Discussion Paper for a Chlordane Health Protection Value (HPV).(Hornshaw)	NA	>150 – 5620 limited consumption >5620 No consumption
Toxaphene Action Level	Potential for Human Exposure to Toxaphene Through Consumption of Great Lakes Fish.(Wisconsin Department of Health Services)	NA	Whole Fish 111 – 4170 limited consumption >4170 No consumption ——— Filleted 56-2080 limited consumption >2080 No consumption

ppb=parts per billion

Source of the Standard Specific Citation	IDEM Derived Criteria values for 303(d) ALUS* Determination (µg/kg** ww)	Indiana FCG decision levels (µg/kg wet weight (ww))
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^{*} ALUS=Aquatic Life Use Support

In addition to the standards in Table 4, other study inputs (e.g., field conditions, potential sources, etc.) include inputs listed in Table 5.

Table 5 Other Inputs into this Study

Variables to Record During Sample Collection	Record in These Units	Why the Variable is Relevant
Upstream or downstream location in comparison to a potential or historical contamination source	NA	Important for looking at trends and for source investigations

A.5.4. Study Parameters

For the 2023-2027 program years, sampling efforts will follow a five-year rotating basin schedule. The rotation is scheduled as follows (see Figure 2):

2023 – Upper Wabash River Basin

2024 - Lower Wabash River and Upper Illinois River Basins

2025 - Great Lakes Basin and Ohio River Tributaries

2026 – West Fork White River and Patoka River Basins

2027 – East Fork White River and Great Miami River Basins

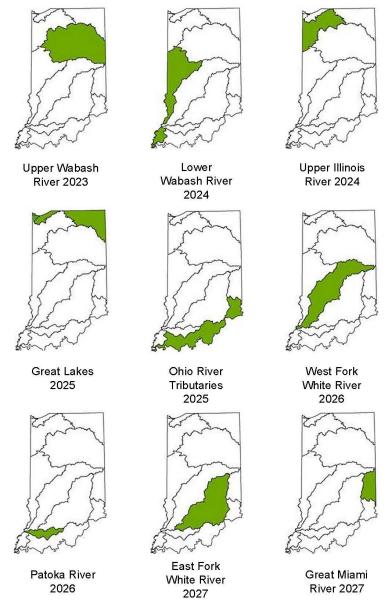
In addition, the IDNR Division of Fish and Wildlife (DFW) annually collects fish tissue samples from the Indiana waters of southern Lake Michigan for processing by the IDEM WAPB.

While the main focus of the study will be in the scheduled basin, the IDEM WAPB may accept or collect samples from basins outside of the selected rotation to fulfil needs/requests by other programs, entities, or the public.

^{**}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.

Figure 2 IDEM Rotating Basin Schedule for Fish Tissue



For this program, the Upper Wabash River Basin (2023 rotation) is geographically within the borders of Indiana contained in the 8-digit Hydrologic Unit Codes (HUCs) 05120101, 05120102, 05120103, 05120104, 05120105, 05120106, and 05120107. The Upper Wabash Basin located in north-central Indiana drains approximately 6918 square miles within Indiana borders. Using the 2019 National Land Cover Database for the Conterminous United States, predominant land uses are cropland (75%), forest (9%), urban (9%), and pasture (3%) (Dewitz 2021).

The Lower Wabash River Basin (2024 rotation) is geographically within the borders of Indiana contained in the 8-digit HUCs 05120108, 05120109, 05120110, 05120111, and 05120113. The Lower Wabash Basin located in western Indiana drains approximately 4799 square miles within Indiana borders. The predominant land uses are cropland (66%), forest (17%) urban (8%), and pasture (4%) (Dewitz 2021).

The Upper Illinois River Basin (2024 rotation) is geographically within the borders of Indiana contained in the 8-digit HUCs 07120001 and 07120002. The Upper Illinois River Basin located in northwest Indiana drains approximately 2986 square miles within Indiana borders. The predominant land uses are cropland (72%), forest (9%), urban (9%), and pasture (4%) (Dewitz 2021).

The Great Lakes Basin (2025 rotation) is geographically within the borders of Indiana contained in the 8-digit HUCs 07120003, 04040001, 04050001, 04100003, 04100004, 04100005, and 04100007. The Great Lakes Basin, located in northern Indiana, drains approximately 3586 square miles within Indiana borders. The predominant land uses are cropland (48%), forest (8%), urban (23%), and pasture (8%) (Dewitz 2021).

The Ohio River Tributaries Basin (2025 rotation) is geographically within the borders of Indiana contained in the 8-digit HUCs 05090203, 05140101, 05140104, 05140201, and 05140202. The Ohio River Trirbutaries Basin located in southern Indiana drains approximately 4249 square miles within Indiana borders. The predominant land uses are cropland (24%), forest (46%), urban (10%), and pasture (16%) (Dewitz 2021).

The White River, West Fork Basin (2026 rotation) is geographically within the borders of Indiana contained in the 8-digit HUCs 05120201, 05120202, 05120203. The White River, West Fork Basin located in southcentral Indiana drains approximately 5599 square miles to the Wabash River. The predominant land uses are cropland (48%), forest (24%), urban (17%), and pasture (7%) (Dewitz 2021).

The Patoka River Basin (2026 rotation) is geographically within the borders of Indiana contained in the 8-digit HUC 05120209. The Patoka River Basin, located in southwestern Indiana, drains approximately 861 square miles to the Wabash River. The predominant land uses are cropland (30%), forest (44%), urban (7%), and pasture (12%) (Dewitz 2021).

The White River, East Fork Basin (2027 rotation) is geographically within the borders of Indiana contained in the 8-digit HUCs 05120204, 05120205, 05120206, 05120207, and 05120208. The White River, East

Fork Basin located in southeastern Indiana drains approximately 5741 square miles to the White River. The predominant land uses are cropland (44%), forest (35%), urban (9%), and pasture (9%) (Dewitz 2021).

The Great Miami River Basin (2027 rotation) is geographically within the area borders of Indiana contained in the 8-digit HUCs 05080001, 05080002, and 05080003. The Great Miami River Basin located in southeastern Indiana drains approximately 1425 square miles within Indiana borders. The predominant land uses are cropland (52%), forest (29%), urban (9%), and pasture (8%) (Dewitz 2021).

Required field activities include collection of whole or edible portions of representative fish tissue samples within the geographical study basins. Samples are collected from August-October to ensure completion of contaminants' depuration during the spawning processes and active storage of fat by fish for the winter months.

Biological samples are collected if the flow is not dangerous for staff to enter the stream (e.g., water levels at or below median base flow); barring any hazardous weather conditions (e.g., thunderstorms or heavy rain in the vicinity); or unexpected physical barriers to accessing the site. The field crew chief makes the final determination as to whether a waterbody is safe to enter.

A.5.5. Analytic Approach

This operation relies on analysis of samples gathered at the sites listed in A.6.2. and Table 6. The contract laboratory uses analytical methods listed in RFP 21-66919 to determine the presence and levels of PCBs, metals, PFAS, pesticides, and PAHs. Section B.4., Table 10, and Table 26 detail additional analytical methods information.

A.6. Program Description

This section provides a detailed overview of all work to perform, products to produce, and the schedule for QAPP implementation necessary for resolution of the problems addressed in A.5.

A.6.1. Sampling Plan

The program currently follows a five-year rotating basin schedule, as described in the WAPB Indiana Surface Water Programs QAPP (IDEM 2023b). The sampling schedule includes twenty-three fixed station program sites as described in the 2022-2026 Water Quality Monitoring Strategy (WQMS) (IDEM 2022c) from the original fish tissue sampling network. In addition to the fish tissue sampling network, other sample sites are selected based on historical environmental problems, waterbody

access, use for fishing, date of last sampling event, potential contaminant sources, and monitoring recommendations by other agencies and entities.

A.6.2. Specific Sampling Locations

a. See Attachments 6-10 for specific sampling locations.

Specific sampling location selection schedule:

2023 Fish Tissue Project (Attachment 6) - Spring 2023

2024 Fish Tissue Project (Attachment 7) – Spring 2024

2025 Fish Tissue Project (Attachment 8) – Spring 2025

2026 Fish Tissue Project (Attachment 9) - Spring 2026

2027 Fish Tissue Project (Attachment 10) – Spring 2027

Sites will be selected on an annual basis for each basin in the five-year rotating basin schedule. When specific sites are selected for the 2023, 2024, 2025, 2026, and 2027 rotations they will be added to this Quality Assurance Program Plan and included in Attachments 6-10. Sites are selected/scheduled closer to the time of sampling to accommodate for changing needs and specific requests for fish tissue contaminant data.

b. Map of Sampling Location

See Figure 3 in Attachments 6-10 for specific sampling locations and maps.

c. Sample Collection Locations in Table 34.

See Attachments 6-10 for specific sampling locations.

Tables 7 through 10 identify the numerous variables of interest to the user for each sampling site.

d. Constituents or Characteristics to Sample in Table 35.

See Attachments 6-10 for constituents or characteristics to be sampled at specific sampling locations.

e. Sampling Specifics in Table 36.

See Attachments 6-10 for sampling specifics related to specific sampling locations.

f. Sample Collection Schedule in Table 37.

See Attachments 6-10 for sample collection schedule for specific sampling locations.

g. Method of Analysis Employed in the Lab in Table 38.

See Attachments 6-10 for method of analysis employed in the lab for specific sampling locations.

A.7. Quality Objective and Criteria

The program does not collect the minimum three samples per site needed to calculate a sampling location average or is NOT intended to measure or address a measure of the results compared to an action level by a statistical method. The QAPP instead drives the following "if/then" decision statements.

If the total PCBs concentration from any sample collected at a site is above 20 μ g/kg ww (A.5.2., Tables 3 and 4, and established in the CALM), then IDEM will recommend the waterbody as not meeting the ALUS for fish consumption and list in the CWA Section 303(d) List of Impaired Waterbodies of Indiana.

If the total mercury trophic level consumption weighted arithmetic mean concentration at a site is above 300 μg/kg ww, (A.5.2., Tables 3 and 4, and established in the CALM), then IDEM will recommend the waterbody as not meeting the ALUS for fish consumption and list in the CWA Section 303(d) List of Impaired Waterbodies of Indiana.

If the total selenium concentration at a site is above 15,100 μ g/kg dw for an eggovary sample, above 8,500 μ g/kg dw for a whole fish sample, or above 11,300 μ g/kg dw for a filleted sample, (A.5.2., Tables 3 and 4, and established in the CALM), then IDEM will recommend the waterbody as not meeting the ALUS for fish consumption and list in the CWA Section 303(d) List of Impaired Waterbodies of Indiana.

If the total PCBs concentration at a site is above 50 µg/kg ww, (A.5.2., Tables 3 and 4, and established in <u>Protocol For a Uniform Great Lakes Sport Fish</u> <u>Consumption Advisory</u>), then IDEM will recommend the waterbody as having limited fish consumption on the Indiana FCG.

If the total mercury concentration at a site is above 50 µg/kg ww (A.5.2., Tables 3 and 4, and as established in <u>A Protocol for Mercury-based Fish Consumption</u>

<u>Advice: An Addendum to the 1993 Protocol for a Uniform Great Lakes Sport Fish</u>

<u>Consumption Advisory</u> (McCann and Anderson 2007)), then IDEM will recommend the waterbody as having limited fish consumption on the Indiana FCG.

If the PFOS concentration at a site is above 20 µg/kg ww (A.5.2., Tables 3 and 4, and established in the <u>Great Lakes Consortium for Fish Consumption Advisories</u> <u>Best Practices for PFOS Guidelines</u> (GLC 2019)), then IDEM will recommend the waterbody as having limited fish consumption on the Indiana FCG.

If the total chlordane concentration at a site is above 150 µg/kg ww (A.5.2., Tables 3 and 4, and as established in <u>Discussion Paper for a Chlordane Health Protection Value (HPV)</u> (Hornshaw)), then IDEM will recommend the waterbody as having limited fish consumption on the Indiana FCG.

If the toxaphene concentration at a site is above 111 µg/kg ww for a whole fish sample or above 56 µg/kg ww for a filleted sample (A.5.2., Tables 3 and 4, and as established in <u>Potential for Human Exposure to Toxaphene Through Consumption of Great Lakes Fish</u> (Wisconsin Department of Health Services)), then IDEM will recommend the waterbody as having limited fish consumption on the Indiana FCG.

Based on data results, take the following actions:

- If the concentration of total PCBs is above the threshold (20 µg/kg ww) in any sample from a site, report the stream reach as impaired in the integrated report to U.S. EPA.
- If the weighted site arithmetic average concentration of total mercury is above the threshold (300 μg/kg ww), report the stream reach as impaired in the integrated report to U.S. EPA.
- If the concentration of total selenium is above 15,100 μg/kg dw for an eggovary sample, above 8,500 μg/kg dw for a whole fish sample, or above 11300 μg/kg dw for a filleted sample from a site, report the stream reach as impaired in the integrated report to U.S. EPA.
- Provide data for use by the Interagency FCG work group supporting IDOH issuance, modification, or removal of FCGs for specific Indiana water bodies.
- Use estimates to assess trends of contaminants in fish.
- Use estimates to understand emerging contaminants.

Site specific ALUS for fish consumption includes program specific controls which identify analysis errors. Controls include laboratory blanks and duplicates, matrix spike and matrix spike duplicate (MS/MSD), laboratory control spikes (LCS), ongoing laboratory performance evaluations, and analytical chemistry data qualifiers and flags specified in the program RFP 21-66919 (IDEM 2021), the analytical services contract (IDOA 2021), the QC section of the WAPB Indiana Surface Water Programs QAPP (IDEM 2023b, B.5., pp. 91-94), and the agency Quality Management Plan (IDEM 2018, p. 38). Tables 28 and 29 of the WAPB Indiana Surface Water Programs QAPP (IDEM 2023b, p. 106-107) outlines analytical chemistry data qualifiers and flags.

Use any data flagged as estimated on a case-by-case basis. Reanalyze fish tissue samples flagged as not meeting minimum QC requirements using the preserved sample material stored at the contract laboratory. Do not use any data rejected, due to analytical problems or errors, for water quality assessment decisions or in FCG determinations. Conduct further investigation in response to consistently rejected data to determine the source of error. If IDEM identifies a trend of consistently rejected data and if corrective action is warranted, the WAPB QA chemist directs the analytical laboratory to make corrections. Field techniques used during sample collection and preparation, and laboratory procedures are subject to periodic evaluation by both WAPB QA and field staff.

A.7.1. Precision

Precision measures the degree to which two or more measurements agree. Calculate relative percent difference (RPD) for each pair of duplicates using:

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

Where:

S = First sample value (original or MS value)

D = Second sample value (duplicate or MSD value)

This calculation applies to RPD calculations for lab duplicates, and MS/MSD QC samples.

The laboratories 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	μg/kg ww	RPD ≤ 40%
(NMeFOSE)		

Measurement	Units	Precision Objective
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	μg/kg ww	RPD ≤ 40%
(NMeFOSAA)		
N-ethyl perfluorooctanesulfonamidoacetic acid - br/lin	μg/kg ww	RPD ≤ 40%
(NEtFOSAA)		
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	μg/kg ww	RPD ≤ 40%
(9CI-PF3ONS)		
11-chloroeicosafluoro-3-oxaundecane-1-sulfonic acid	μg/kg ww	RPD ≤ 40%
(11CI-PF3OUdS)		
Metals	1 /1	DDD 1000/
Chromium	μg/kg ww	RPD ≤ 20%
Lead	μg/kg ww	RPD ≤ 20%
Mercury	μg/kg ww	RPD ≤ 20%
Selenium	μg/kg ww	RPD ≤ 20%
Arsenic	μg/kg ww	RPD ≤ 20%
PCBs	1 , 1	555 + 400/
Total PCBs	μg/kg ww	RPD ≤ 40%
Organochlorine Pesticides	1 /1	DDD 1 100/
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%

Measurement	Units	Precision Objective
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%
PAHs		
Naphthalene	μg/kg ww	RPD ≤ 19%
1-Methyl Naphthalene	μg/kg ww	RPD ≤ 24%
2-Methyl Naphthalene	μg/kg ww	RPD ≤ 12%
Acenaphthylene	μg/kg ww	RPD ≤ 21%
Acenaphthene	μg/kg ww	RPD ≤ 16%
Fluorene	μg/kg ww	RPD ≤ 12%
Phenanthrene	μg/kg ww	RPD ≤ 10%
Anthracene	μg/kg ww	RPD ≤ 16%
Chrysene	μg/kg ww	RPD ≤ 17%
Fluoranthene	μg/kg ww	RPD ≤ 17%
Pyrene	μg/kg ww	RPD ≤ 15%
Benzo (a) anthracene	μg/kg ww	RPD ≤ 15%
Benzo (b) fluoranthene	μg/kg ww	RPD ≤ 14%
Benzo (k) fluoranthene	μg/kg ww	RPD ≤ 13%
Benzo (a) pyrene	μg/kg ww	RPD ≤ 13%
Dibenzo (a,h) anthracene	μg/kg ww	RPD ≤ 15%
Benzo (g,h,i) perylene	μg/kg ww	RPD ≤ 14%
Indeno (1,2,3-c,d) pyrene	μg/kg ww	RPD ≤ 13%

A.7.2. Accuracy

Accuracy is the degree to which an observed value and an accepted reference value agree. Calculate percent reference standard recovery (%R) using:

$$%R = [(A - B) \times 100] / C$$

Where:

A = Analyte concentration determined experimentally with known quantity of reference material added.

B = Background determined in the laboratory by separate (unspiked) sample analysis, or in the field from a blank.

C = True value of reference standard added.

Use accuracy (% Recovery) wherever comparing a measured value to a reference value.

QC samples using %R include LCS or LFB, CCV, ICV, SS, certified reference standards, MS (LFM), and MSD (LFMD).

Laboratories use different accuracy goal ranges based on QC sample type (i.e., LCS, LFB, CV, CR, SS, STDs, MS/MSDs). Therefore, the accuracy objectives may vary based on the QC sample type. In this work plan use MS/MSDs to determine accuracy.

Table 12 contains accuracy goals with acceptance limits for applicable analytical parameters.

Table 7 Accuracy Objectives by Measurement Type

Measurement 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

Measurement	Units	Accuracy
		Objective
N-methylperfluorooctane sulfonamide	μg/kg ww	70 ≤ %R ≤ 130
(NMeFOSA)	,	70 +0/ 5 + 400
N-ethylperfluorooctane sulfonamide	μg/kg ww	70 ≤ %R ≤ 130
(NEtFOSA)	- // -	70 + 0/ D + 400
N-methyl perfluorooctanesulfonamidoacetic	μg/kg ww	70 ≤ %R ≤ 130
acid - br/lin (NMeFOSAA) N-ethyl perfluorooctanesulfonamidoacetic acid	μg/kg ww	70 ≤ %R ≤ 130
- 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 (4:2 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 (0.2 FTS)	μg/kg ww	70 ≤ %R ≤ 130
4,8-dioxa-3H-perfluorononanoic acid (ADONA)	μg/kg ww	70 ≤ %R ≤ 130 70 ≤ %R ≤ 130
9-Chlorohexadecafluoro-3-oxanone-1-sulfonic	μg/kg ww	70 ≤ %R ≤ 130 70 ≤ %R ≤ 130
acid (9CI-PF3ONS)	μg/kg ww	70 3 7010 3 130
11-chloroeicosafluoro-3-oxaundecane-1-	μg/kg ww	70 ≤ %R ≤ 130
sulfonic acid (11CI-PF3OUdS)	pg/kg ww	70 = 7010 = 100
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
Arsenic	μg/kg ww	75 ≤ %R ≤ 125
PCBs	pg/kg ww	70 = 7011 = 120
Total PCBs	μg/kg ww	70 ≤ %R ≤ 130
Organochlorine Pesticides	F9/1.9 1111	70 - 7011 - 100
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
	Ma/ Na WW	1.0 = 7010 = 100

Measurement	Units	Accuracy
		Objective
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
PAHs		
Naphthalene	μg/kg ww	31 ≤ %R ≤ 146
1-Methyl Naphthalene	μg/kg ww	16 ≤ %R ≤ 162
2-Methyl Naphthalene	μg/kg ww	43 ≤ %R ≤ 116
Acenaphthylene	μg/kg ww	18 ≤ %R ≤ 146
Acenaphthene	μg/kg ww	32 ≤ %R ≤ 129
Fluorene	μg/kg ww	32 ≤ %R ≤ 106
Phenanthrene	μg/kg ww	49 ≤ %R ≤ 111
Anthracene	μg/kg ww	16 ≤ %R ≤ 113
Chrysene	μg/kg ww	24 ≤ %R ≤ 123
Fluoranthene	μg/kg ww	27 ≤ %R ≤ 126
Pyrene	μg/kg ww	33 ≤ %R ≤ 123
Benzo (a) anthracene	μg/kg ww	23 ≤ %R ≤ 112
Benzo (b) fluoranthene	μg/kg ww	30 ≤ %R ≤ 116
Benzo (k) fluoranthene	μg/kg ww	32 ≤ %R ≤ 112
Benzo (a) pyrene	μg/kg ww	34 ≤ %R ≤ 110
Dibenzo (a,h) anthracene	μg/kg ww	24 ≤ %R ≤ 114
Benzo (g,h,i) perylene	μg/kg ww	27≤ %R ≤ 109
Indeno (1,2,3-c,d) pyrene	μg/kg ww	33 ≤ %R ≤ 113

Like precision, accuracy is susceptible to variations in technique. Minimize such variation by using SOPs; correct field and laboratory technique; and qualified individuals (A.8.).

A.7.3. Representativeness

Unlike precision and accuracy, representativeness tends to be a qualitative measurement. Essentially, the measurement describes how similar the analytical data are in essential characteristics to the parent population of interest. Many factors influence how representative a sampled area's analytical results are. The factors include:

- 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 a quantitative measure of the number of valid items versus the total number of items processed. (e.g., number of samples collected versus number of samples planned; number of samples producing valid results versus the planned number of samples to analyze). The percent completeness for both field and laboratory analyses is:

% Completeness = (number of valid measurements) x 100 (number of measurements planned)

Where: "valid measurements" refers to numbers of investigational samples obtained or to be obtained for a specific purpose, or to satisfy a particular program objective.

In this case, the overall completeness goal is 100% for samples analyzed for the program. Therefore, collect all planned samples, analyze all samples collected, and all samples should yield analytical data usable for the intended purpose.

If a result is flagged as rejected, do not consider it a valid measurement for this calculation.

A.7.5. Comparability

Comparability is a qualitative measure of dataset equivalency. If two datasets are not readily comparable, using the datasets may make drawing inferences or making comparisons difficult. To assure comparability, use common variables, standardized collection and analysis techniques, and satisfy the requirements of the other measurement quality objectives. Program comparability is largely addressed due to all sample collection utilizing the exact same OWQ collection methods and laboratory analytical methods to collect and analyze Indiana fish tissue data.

A.7.6. Sensitivity

Sensitivity is related to the reporting limit (RL). 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's concern is whether the instrument or method is sensitive enough to quantitate a result less than or equal to one-third times the standard (≤1/3*standard). In general, RLs are less than the

applicable standard or screening level. Do not use analytical results for samples which are nondetect for a particular analyte, and when the instrument's, or method's RLs are greater than the applicable standards or screening levels to demonstrate compliance with the applicable standards or screening levels.

Table 8 Sensitivity Objectives by Measurement Type Expressed as IDEM RL

Measurement	Units	Reporting Limit
General Chemistry ⁵		
Lipid	%	0.1
Moisture	%	0.1
PFAS ⁶		
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

Measurement	Units	Reporting Limit
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	μg/kg ww	0.232
(9CI-PF3ONS)		
11-chloroeicosafluoro-3-oxaundecane-1-sulfonic acid (11CI-PF3OUdS)	μg/kg ww	0.235
Metals ⁷		
Chromium ⁷	μg/kg ww	10
Lead ⁷	μg/kg ww	70
Mercury ¹⁰	μg/kg ww	50
Selenium ⁷	μg/kg ww	110
Arsenic ⁷	μg/kg ww	1000
PCBs ⁸	µg/Ng WW	1000
Total PCBs	μg/kg ww	50
Organochlorine Pesticides ⁹	μg/ng ww	
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

Measurement	Units	Reporting Limit
Pentachloroanisole	μg/kg ww	2.5
Toxaphene	μg/kg ww	10
PAHs		
Naphthalene	μg/kg ww	100
1-Methyl Naphthalene	μg/kg ww	16
2-Methyl Naphthalene	μg/kg ww	16
Acenaphthylene	μg/kg ww	67
Acenaphthene	μg/kg ww	33
Fluorene	μg/kg ww	3.3
Phenanthrene	μg/kg ww	3.3
Anthracene	μg/kg ww	3.3
Chrysene	μg/kg ww	3.3
Fluoranthene	μg/kg ww	5
Pyrene	μg/kg ww	3.3
Benzo (a) anthracene	μg/kg ww	3.3
Benzo (b) fluoranthene	μg/kg ww	3.3
Benzo (k) fluoranthene	μg/kg ww	3.3
Benzo (a) pyrene	μg/kg ww	3.3
Dibenzo (a,h) anthracene	μg/kg ww	6.7
Benzo (g,h,i) perylene	μg/kg ww	6.7
Indeno (1,2,3-c,d) pyrene	μg/kg ww	6.7

⁵ ASTM D2974-87 and Pace Analytical Lipid Method

A.8. Specialized Training and Certification

A.8.1. through A.8.3. and Table 14 describe required specialized trainings, qualifications, or certifications required to perform program work. All contractors are chosen through a bidding process. The process only considers contractors with the ability to adequately achieve all requirements in the program specifications including documented staff biographies and past program accomplishments. All contractors must comply with the terms of the RFP, associated contract, and the QAPP.

A.8.1. IDEM PI

Tim Fields, B.S. in Chemistry from Indiana University, has experience in environmental measurement, data QC, fish tissue collection, and has overseen a variety of IDEM monitoring projects for 10 years.

A.8.2. IDEM QA reviewer

⁶ Pace Analytical DoD 36 (Pace 2020)

⁷ EPA Preparation 3540C (EPA 1996a) and EPA Method 8082A (EPA 2007a)

⁸ EPA Method 6020A (EPA 2007)

⁹ EPA Method 8081B (EPA 2007c)

¹⁰EPA Method 1631, Revision E (EPA 2002, EPA 2001)

Pat Colcord holds a BS in Aquatic Biology from Ball State University, a BS in Bioengineerng from IUPUI, and has 38 years of experience from a variety of environmental programs at IDEM. He has assisted in the writing of numerous QAPPs, SOPs, TSOPs and other QA documents. He possesses nearly two years of experience as a QA manager.

A.8.3. IDEM OWQ field staff

All training is conducted in-house, utilizing existing SOPs and equipment operating manuals. OWQ typically conducts on-the-job training where an experienced staff mentor or field crew chief accompanies new or less experienced staff.

Specialized training or certifications needed by staff to successfully complete the specific program or task identified in the QAPP include:

Table 9 Specialized Training or Certifications

Table 9 Specialized 11	anning or Certificat	.10113	
Role	Training and Experience Requirement	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 2010)	-Staff lacking 4 hours of in-service training or appropriate certification require accompaniment in the field by WAPB staff meeting Health and Safety Training requirements
	-Personal Protective Equipment Policy	-IDEM 2008	-Indiana Code 14-8-2- 27 requires a high intensity whistle and Safety of Life at Sea (SOLAS) certified strobe
	-Memorandum "Use of Personal Flotation Devices (PFD) by Branch Personnel" dated February 29, 2000	-February 29, 2000 WAPB internal memorandum regarding use of approved PFDs	light when working on cojurisdictional waters or during hours of darkness
	-IDEM Injury and Illness Resulting from Occupational Exposure Policy (IDEM 2016)	-Hazard Communication Manual (IDEM 2019)	
	-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 require accompaniment in the field by WAPB staff meeting the boating safety requirements

Role	Training and Experience Requirement	Training References	Training Notes
Field crew chief	-DNR issued scientific collectors permit		-At least one staff member on a field crew must obtain a permit

A.9. Documents and Records

IDEM QA staff post and maintain the most up-to-date versions of each agency QAPP and SOP in the SharePoint IDEM QA Library. All program managers direct staff participating in data operations or QAPP implementation to the IDEM QA Library. Also, the most up-to-date versions of all active agencywide and program SOPs documenting QAPP activities are available through the agency SharePoint page links, <u>Standards</u>, <u>Policies</u>, <u>and Mailcodes</u>, under <u>Standard Operating Procedures (SOPs) links to the IDEM QA Library</u>. Table 2 contains Key QAPP Individuals contact information.

A.9.1. The QAPP Report

Table 15 contains information associated with implementation of the QAPP:

- The QAPP for a completed program, covering work performed under the QAPP, is stored in the IDEM QA library archives.
- SOPs referenced in the QAPP are similarly stored. Current or future interested parties may contact the appropriate IDEM program area QAM or the IDEM QAM for date specific copies of the QAPP or any referenced SOPs.
- All completed forms generated during implementation of the data operation are cataloged in a QAPP, or data report.

Table 10 Records Associated with 2023-27 Fish Tissue Contaminant Monitoring

Table 10 Records 7.330clated with 2020 27 1 1311 1133de Contaminant Montoning		
Record Type	Where Records are Stored	
Completed program QAPP	S:\IGCN\OWQ\WSP\OWM\Biological Studies\Fish tissue and Sediments\Sampling Plans\2023 Also located on SharePoint – WAPB – Project Documents: 2023-2027 Fish Tissue Contaminants Monitoring Quality Assurance Program Plan	
Sample collection forms	AIMS database	
Field recon forms	AIMS database	
Chain of custody forms	S:\IGCN\OWQ\WSP\OWM\Biological Studies\Fish tissue and Sediments\ SMPLELOG	
EDI file from laboratory	S:\IGCN\OWQ\AIMS\EDIFiles\EnChem	
Related laboratory contracts	S:\IGCN\OWQ\WSP\OWM\Biological Studies\Fish tissue and Sediments\ RFP\2021	

Record Type	Where Records are Stored
Laboratory reports	S:\IGCN\OWQ\WSP\OWM\Biological Studies\Fish tissue and Sediments\ SMPLELOG
QA reports	S:\IGCN\OWQ\WSP\OWM\Biological Studies\Fish tissue and Sediments\SMPLELOG

A.9.2. Field Activities

Attachment 1 contains a copy of the Field Record for Biological Tissue Contaminants Monitoring Program. Scan completed field sheets and save as an attachment to the program in AIMS. Blank field sheets are on the IDEM SharePoint site. QA staff audit field data reduction, validation, and reporting procedures as a component of performance audits described in WAPB Indiana Surface Water Programs QAPP (IDEM 2023b) Section C, Assessment and Oversight.

A.9.3. Laboratory Activities

IDEM's OWQ receives the analytical results from Pace. Per the WAPB Indiana Surface Water Programs QAPP (IDEM 2023b), the data is subject to the Laboratory Reporting Requirements including receipt of data in the electronic data interface specified in EDI Format Description (IDEM 2021b). Pace submits a laboratory report in electronic portable document format (PDF) for each batch of samples (sample set) which consists of Contract Laboratory Chain of Custody Form (Attachment 2), spreadsheets of results, and the QC report in accordance with the contract requirements. In addition, Pace submits an electronic data import (EDI) file containing laboratory data and lab QC for each sample set. The EDI file must comply with the EDI Format Description (IDEM 2021b). WAPB uploads the EDI files into the AIMS database. Data results in the laboratory reports shall meet requirements of the WAPB Indiana Surface Water Programs QAPP (IDEM 2023b, pp. 105-106) DQA Level 4. Submit reports to the Technical and Logistical Services Section for review. Section D.3.1 contains additional information.

A.9.4. QA Records and Reports

The IDEM principal program investigator ensures the appropriate program staff have the most current approved version of the QAPP. Keep QA, program, and site managers up to date on any revisions and edits made to the QAPP during the term of the program. The data report package shall be available in hard copy and electronic formats. As with other program reports, store any QA records or reports generated in electronic format on the IDEM server shared drive (S Drive).

A.9.5. Retention Time and Location of Records and Reports

Retain all hard copy files of data and reports, for a minimum of three years, in accordance with the State of Indiana Records Retention Policy (General State of Indiana Schedule and the IDEM specific schedule). Maintain hard copy records at the IDEM Shadeland office currently located at 2525 N Shadeland Avenue, Indianapolis, IN 46219. Store an electronic copy of all data files on the IDEM server shared drive (S Drive) in the program folder (S:\IGCN\OWQ\WSP\OWM\Biological Studies\Fish tissue and Sediments\SMPLELOG) for a period of not less than three years after the conclusion of the program.

B. Data Generation and Acquisition

The elements of B. Data Generation and Acquisition address all aspects of program design and implementation. Implementation of the elements ensures employment and proper documentation of appropriate methods for sampling, measurement and analysis, data collection or generation, data handling, and QC activities.

B.1. Sampling Process Design

The design of site selection captures profiles of major rivers and tributaries located within the five-year rotating basin schedule. Selected sites include historical sites for trend analysis and new site locations to increase information availability in the basins. Targets are larger tributaries to ensure the collection of filletable size fish. The number of sites and the samples collected from each site are highly variable and based on availability of WAPB resources and funding for the laboratory service contract. No collection of field duplicates nor carrying of field blanks will occur during the fish tissue sampling and sample preparation processes. The site selection process criteria:

- Historical sampling locations
- Location in relation to municipalities
- Current FCG status
- New locations of interest with no previous sampling
- Public access locations
- Known contaminated areas
- Sampling requests by other programs within and external to IDEM
- Special studies

The Fish Tissue Contaminants Monitoring program follows a five-year basin rotation design. The rotating basin approach facilitates a more comprehensive estimation of an impairment's extent within the basin. The number of sites and the samples collected from each site are highly variable and based on resources and funds, WAPB Indiana Surface Water Programs QAPP (IDEM 2023b, p. 23). Sampling may also occur in watersheds outside the targeted basins to support IDEM ALUS assessment, or other agency programs. Other targeted locations may include special studies for other program areas. In addition, WAPB accepts

samples collected during IDNR DFW study projects. The agency partnership increases sampling efficiency, while also reducing the stress on biological communities caused by multiple sampling events on the same waterbody. This includes samples from natural lakes, streams, or Indiana waters of Lake Michigan. The locations may not necessarily be in the target basin of the 2022 – 2026 WQMS (IDEM 2022c). To handle and process the fish, IDNR staff training includes the U.S EPA guidance (EPA 2000), the Sport Fish Advisory Protocols (Anderson et. al. 1993), the IDEM Fish Community Field Collection Procedures (IDEM 2023), and the procedures described in this QAPP.

Sampling Design and Rationale, also references A.6.1. and A.7.

Table 11 Sample Schedule and Characteristics

Sampling schedule	Fish tissue collections can occur year-round. Although the most desirable sampling period is from late summer to early fall (i.e., August through October). For the study, plan to collect samples from August through October. Many species lipid content, which is a reservoir for many lipophilic bioaccumulating organic pollutants, is generally highest during this period. Also, water levels are typically lower during this time, thus simplifying collection procedures (EPA 2000).	
Sampling frequency	One sampling event per site	
Constituent to be sampled	Lipids, moisture, total PCBs, metals, pesticides, PFAS, PAHs	
Sampling matrix	Fish tissue	
Sampling procedure	Attachment C	
Sample volume	Examine fish collected and select a predetermined number of samples for the site, generally between three and five samples. One to 12 fish of the same species may comprise a sample, depending on size. For total lengths, the preferred length difference between the largest and smallest is 90% of largest individuals of any composite sample. To obtain an adequate composite sample, a minimum difference in lengths as low as 75% is tolerable.	
Preservation measures	If processing samples in the field, place samples in the dry ice chest and cover with dry ice for tissue preservation. 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 processing samples in the laboratory, place all processed tissue samples in the analytical-grade freezer or the upright commercial-grade freezer. Once prepared for individual or composite fish tissue samples, store 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 requests analysis and reporting to occur within 90 days from the initial shipping date.	

B.2. Sampling Methods

Gather the required samples using SOPs or standard scientific methodologies. Sample containers, preservatives, and maximum holding times must comply with the requirements of the applicable laboratory test method.

Table 12 Sampling SOPs or Standard Scientific Methodologies
Tables 17 through 20 contain numerous variables of user's interest for each method or SOP.

Method or SOP #6	Name of Method or SOP	Doc Version Number	Document Location
B-001-OWQ-WAP- XXX-22-T-R0	Global Positions System (GPS) Data Creation	R0	Global Positioning System Data Creation B-001-OWQ-WAP-XXX-22-T- R0.docx(IDEM 2022e)
S-004-OWQ-WAP- TL-20-T-R0	EDI Import to AIMS II	R0	EDI Import to AIMS II S-004-OWQ-WAP- TL-20-T-R0.docx(IDEM 2020)
CSP2C01	Principles and Techniques of Electrofishing	NA	https://www.fws.gov/training/CSP2C01- principles-and-techniques-of- electrofishing-online (U.S. FWS 1998)
Sample Collection Procedure	Sample Collection Procedure	NA	Attachment 3
EPA Guidance Document	Guidance for Assessing Chemical Contaminant	Volume 1	https://www.epa.gov/sites/default/files/201 5-06/documents/volume1.pdf (EPA 2000)

Table 13 Sampling SOPs or Standard Scientific Methodologies History and Related Information

Method or SOP # (Table 17 continuation)	Method or SOP Source	Date Approved
B-001-OWQ-WAP-XXX-22-T-R0	IDEM	2022
S-004-OWQ-WAP-TL-20-T-R0	IDEM	2020
CSP2C01	U.S. FWS	2018
Sample Collection Procedure	IDEM	2021
EPA Guidance Document	U.S. EPA	2000

Table 14 Sampling SOPs or Standard Scientific Methodologies, Related Details

Method or SOP # (Table 17 continuation)	Equipment Requiring Decontamination	Decontamination Process	Method of Decontamination Product Disposal
B-001-OWQ-WAP-XXX-22- T-R0	NA	NA	NA
S-004-OWQ-WAP-TL-20-T- R0	NA	NA	NA
CSP2C01	Watercraft, anodes, cathodes, nets, electrofishing gloves, PPE	Spray with 2% bleach solution	Safe to pour down the drain
Sample Collection Procedure	Knives, cutting boards, scalers, buckets, scale, measuring board, table	Wash equipment with Alconox	Safe to pour down the drain
EPA Guidance Document	Watercraft, anodes, cathodes, nets, electrofishing gloves, PPE	Spray with 2% bleach solution	Safe to pour down the drain

Table 15 Sampling SOPs or Standard Scientific Methodologies; Sampling Details

Method or SOP # (Table 17 continuation)	Sample Volume	Sample Preservation Type	Holding Time to Analysis
B-001-OWQ-WAP-XXX-22-T-R0	NA	NA	NA
S-004-OWQ-WAP-TL-20-T-R0	NA	NA	NA
CSP2C01	NA	NA	NA
Sample Collection Procedure	100 grams tissue material	Ice in the field and solid state at <32 degrees Celsius in the laboratory	1 year*
EPA Guidance Document	200 grams tissue material	Ice in the field and solid state at <32 degrees Celsius in the laboratory	1 year*

These holding times are based on guidance that is sometimes administrative rather than technical in nature; there are no promulgated holding time criteria for tissues (EPA 1995).

Table 16 Types of Samples Collected

Constituents	Type of Samples	Number of Samples	Frequency of Sampling	Type and Number of QC Samples ¹¹
Fish Tissue	Composite	TBD	1 sampling event per site	1 per 20

¹¹The general rule is that 10 percent of all samples should be QC samples.

If a failure, disruption, or other breakdown occurs during the implementation of this QAPP, take corrective action. Any technical staff may identify a nonconformity. Once identified, the individual section chief, program officer, or QAO is responsible for corrective action for the one or more programs within their responsibility. The section chief, program officer, or QAO is responsible for documenting the nonconformity, working with relevant staff members to develop corrective actions, and notifying the QAM of corrective action progress. Depending on the nonconformity and associated corrective actions either the section chief or the QAO approves the final corrective action.

B.3. Sample Handling and Custody

Chain of custody is the chronological documentation or paper trail, showing the seizure, custody, control, transfer, analysis, and disposition of physical or electronic evidence. To accomplish full documentation, keep a combination of field and laboratory records demonstrating possession and transfer of custody.

WAPB requires each laboratory performing analytical tests to provide copies of SOPs upon request. Confirm availability of laboratory SOPs through laboratory proposal review and onsite audit during the RFP process and annually thereafter. Require 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. A.7.6. Table 13 lists the RLs and acceptable U.S. EPA analytical test methods for each analyte.

Tables 22 through 25 contain the requirements for sample handling and custody in the field, transport, and laboratory.

Table 17 Sample Handling in the Field

Sample Collection Location	Sample Constituent of Concern	Container Type and Size	Package Temperature or Special Condition
All	All	48-quart cooler	Buried in wet ice

Table 18 Sample Transport Details

Carrier	Means of Transport	Allowable Time from Carrier Pick- up to Lab	Days of the Week when Shipping is Unavailable
Pace supplies a commercial carrier to transport samples directly to the laboratory. Carrier is Special Dispatch, Inc.	Carrier vans	24 hours	None

Table 19 Sample Handling in the Laboratory

Lab Location	Lab Contact Info	Time Span Lab is Open for Sample Receipt	Sample Storage – Where and How, Until Analysis
Pace – GB	Tod Noltemeyer – tod.noltemeyer@pacelabs.com	Monday through Wednesday	The laboratories store all samples at a maximum temperature of -10°C

The laboratory will preserve all samples, as agreed in the analytical services contract, until IDEM chemists and WAPB QAO approve all sample data for the sampling year.

Attachment 3 contains agency or program area SOPs which include additional information on sample package handling.

The analytical laboratory provides laboratory chain of custody forms. Forms are available at Forms: S:\IGCN\OWQ\WSP\OWM\Biological Studies\Fish tissue and Sediments\SMPLELOG

The laboratory contact for information on sample receipt, storage, handling, and preparation is Tod Noltemeyer, Tod.Noltemeyer@pacelabs.com.

Table 20 Sample Handling System Responsible person or laboratory group

Sample Collection, Packaging, and Shipment
Sample collection: Tim Fields
Sample packaging: Tim Fields
Coordination of shipment: Tim Fields; Tod. Noltemeyer – Pace
Type of shipment: Courier – Kristi Howard or Tammy Sipes for Special Dispatch, Inc.
Sample Receipt and Analysis

Responsible organization: Pace

Sample receipt: Pace

Sample custody and storage: Pace

Sample preparation: Pace

Sample determinative analysis: Pace

Sample Archiving

Field sample storage: Tim Fields

Sample extract or digestate Storage: Pace

Sample Disposal

Responsible organization: Pace

Responsible staff: Pace

B.4. Analytical Methods

Table 10 identifies analytical methods by number, date, and regulatory citation.

The contract laboratory QA Manual describes analytical methods-related variables and may be proprietary.

B.5. Quality Controls

A.7.1. Table 11 identifies the needed field sampling QC activities to follow.

Table 28 contains a list of major equipment used for field measurement. Do not collect QC samples in the field.

The WAPB's expectation for each contract laboratory providing analytical services is to meet the statement of work and technical specifications of the IDEM RFP in compliance with contract requirements. Find laboratory QC checks for biological samples in the IDEM RFP Technical Specifications (Appendix H6 of IDEM RFP 21-66919 (IDEM 2021)). Table 27 summarizes the laboratory QC checks for test method procedures. Refer to individual test methods for specific requirements.

Table 21 Quality Controls for Laboratory Analysis

Sample Site ID #	Matrix	Analytical Parameter	Analytical Method or SOP Reference
All	Tissue	Metals	6020A
All	Tissue	Hg	1631E
All	Tissue	Pesticides	8270D, 8081B
All	Tissue	PCBs	1668B, 8270D, 8082A
All	Tissue	PFAS	DoD QSM 5.3, WDNR PFAS Guidance, DoD Guidance for PFAS Analysis in Biota, and USEPA Method 537.1
All	Tissue	PAHs	8270D

Sample Site ID #	Matrix	Analytical Parameter	Analytical Method or SOP Reference
All	Tissue	PBDEs	1614A
All	Tissue	Dioxins and Furans	1613

Table 22 Quality Controls for Laboratory Analysis

Parameters	Calibration ¹² or Verification	Sample Lab Duplicate*	MS/MSD ¹³	LCS	Method Blank	External QC Standard	Surrogate	Serial Dilution	Interference Check
Metals	1/10	1/20	1/20	1/20	1/run	1/20	n/a	1/run w/dilution	2/run
Pesticides	every day	1/20	1/10	1/10	1/20 or 1/extract batch	4/day	every sample	n/a	n/a
PCBs	every day	1/20	1/10	1/10	1/20 or 1/extract batch	4/day	every sample	n/a	n/a
PFAS	every day	1/20	1/10	1/10	1/20 or 1/extract batch	4/day	every sample	n/a	n/a
PAHs	every day	1/20	1/10	1/10	1/20 or 1/extract batch	4/day	every sample	n/a	n/a
PBDEs	every day	1/20	1/10	1/10	1/20 or 1/extract batch	4/day	every sample	n/a	n/a
Dioxins and Furans	every day	1/20	1/10	1/10	1/20 or 1/extract batch	4/day	every sample	n/a	n/a
General Chemistry	n/a	1/20	n/a	1/run	1/run	n/a	n/a	n/a	n/a

¹²Continuing calibration verification standards (CCVs) shall be run according to the test method or at the beginning and end of a run batch and at a rate of 5%, whichever is greater.

B.6. Instrument and Equipment Testing, Inspection, and Maintenance

All equipment receives a visual inspection on every day of use to ensure maintenance of crew safety and efficient sampling effort. Inspect, maintain, and test instruments and equipment for implementation of the data operation (Table 28).

¹³Laboratories shall analyze MS/MSDs at a rate of 1 per batch or 5%, whichever is greater.

^{*}IDEM selects the Lab Duplicate by denoting desired sample with adequate mass on the COC.

Table 23 Equipment Testing, Inspection, and Maintenance

Equipment Type	Inspection	Maintenance	Frequency	Testing
Boat	Day of use	Maintenance or repair	As needed	Annually
Boat motor	Day of use	Maintenance of engine oil, lower gear lube, propellers, state of turn engine	Annually	Annually
Boat trailer	Day of use	Inspection, repack wheel bearings, check lights, bunks, and tires	Annually	Annually
Electroshock box	Day of use	Factory calibration	Every two years	Annually
Electroshock backpack	Day of use	Factory calibration	Every two years	Annually
Fire extinguisher	Day of use	Inspection	Annually	Annually
Generator	Day of use	Maintenance	Annually	Annually
GPS unit – Trimble	Day of use	Update software	As new software becomes available	Annually
Weighing scale	Day of use	Calibration	Every two years	Every two years

OWQ requires each analytical laboratory, in accordance with IDEM RFP, to have preventive maintenance procedures with set frequencies for all analytical instruments and measurement equipment used in the performance of analytical services. The IDEM RFP Technical Specifications require each contract laboratory to have written SOPs demonstrating the contract laboratory's capability to provide the services requested. Preventive maintenance is an element of laboratory system audits, testing, and inspection.

B.7. Instrument and Equipment Calibration and Frequency

Measurement equipment require periodic calibration or standardization to reliably produce accurate results. IDEM requires contract labs to follow this QAPP and WAPB RFP 21-66919 (IDEM 2021). Both documents require a quality system which includes standards for calibration and corrective actions. In addition, IDEM may elect to request pertinent QA data, including calibration standards.

Contract laboratories (Pace) providing analytical services to the WAPB water quality monitoring programs must document calibration procedures and frequency in compliance with WAPB RFP 21-66919 (IDEM 2021). The requirements specify a QA system is in place and QA/QC data related to the program must be available to IDEM.

Table 24 Instrument and Equipment Calibration and Frequency

Equipment Type	Calibration Procedure	Frequency of Calibration	Acceptance Criteria	Responsible Staff
Weighing Scale	Coordinate with Weights and Measures	Every two years	Approval by IN Weights and Measures	WAPB Staff

B.8. Inspection and Acceptance of Critical Supplies

To reliably produce accurate results requires QA of consumable supplies, IDEM requires contract laboratories follow this QAPP and WAPB RFP 21-66919 (IDEM 2021). Both documents require a quality system 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.

No critical field elements require inspection or approval prior to the use for sample collection activities.

Contract laboratories (Pace), providing analytical services to the WAPB water quality monitoring programs, demonstrate proficiency through past performance and response to RFP 21-66919 (IDEM 2021). In addition, all contract laboratories must have a QA plan which includes acceptance criteria for consumables and supplies. The IDEM PI reviews invoices submitted from the contract laboratories for reasonableness, including monitoring for unwarranted or excessive usage of consumable supplies.

B.9. Nondirect Measurements

Pool historical IDEM fish tissue data collected in the target basins with 2023-2027 data for FCG determinations. Keep the data in the WAPB AIMS II electronic database.

The City of Elkhart's fish tissue data is stored in the WAPB AIMS II electronic database and combined with IDEM WAPB data for FCG determinations; as they follow the IDEM WAPB protocols for fish tissue collection and processing and their samples are processed by the same lab and QC measures as IDEM WAPB.

Historical Indiana fish tissue data from Lake Michigan is combined with data from Illinois, Michigan, and Wisconsin for FCG determinations and to promote consistent consumption advice on shared waterbodies.

The QAPP will not use any other existing data.

B.10. Data Management

The Indiana Office of Technology determines state hardware and software requirements outlined in Section 6 of the IDEM Quality Management Plan (IDEM

2018). Section A.9. Documents and Records and Section D contain more detailed information.

Preserve records generated by the data operation as described in Section A.9. (Table 15).

Table 25 Mechanism for Avoiding Errors

Potential Data Errors	Mechanism for Avoiding Errors and Data Loss
Database uploads	Per 6.4.4. Data Security Standards , of the U.S. EPA R5 approved IDEM 2018 Quality Management Plan (IDEM 2018), the Indiana Office of Information Technology provides an Information Security Network which secures all IDEM information assets.
Hand-entered data	All data entered goes through two rounds of QC to minimize errors in the transcription process.
Lab reports	WAPB chemists review the data packages to ensure reports are complete and meet all DQOs.

C. Assessment and Oversight

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

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

Precision
 RPD between field duplicate measurements

Accuracy %R of field references

Completeness % planned samples collected, analyzed, reported, and useable for each present

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

- Reviewing sampling work plans, including DQOs and target parameters
- Equipment calibration, maintenance, and frequency
- Field data collection procedures
- Sample collection and chain of custody procedures

Conduct field audits to ensure sampling activities adhere to approved SOPs. WAPB managers systematically conduct audits which include all WAPB staff engaged in field sampling activities. Managers trained in the associated sampling SOPs and in the processes related to conducting an audit, evaluate WAPB field staff involved with sample collection and preparation. Produce an evaluation report documenting each audit for review by field staff audited, as well as WAPB management. As a result of the audit process, communicate corrective actions to field staff which implement corrective action.

Audit analytical laboratory results for performance using QC checks. WAPB QA staff audit reported results, which include data deliverables, at 100% frequency. Review each sample set and determine data usability. 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

QAO or designee performs laboratory system audits at the beginning of a laboratory contract and at least once a year during the contract. Report results to the WAPB QAO. The system audit includes any or all operational QC elements of the laboratory's QA system. Address all applicable elements and the laboratory contract requirements including, but not limited to, activities in Table 31.

Table 31 identifies the planned assessment activities to track implementation of the data operation to ensure the prescribed plan's implementation.

Table 26 Data Operation Tracking

Targeted Assessment Activities	Type of Planned Assessment ¹⁴ (What it will consist of)	Assessor ¹⁵	Number of Assessments	Report Findings to?	Corrective Actions (CAs) Anticipated
Field performance and system audits	Sample collection and handling	WAPB Manager	1 per 2 years	Field crew chief	Section C.1.
Contract laboratory performance and system audits	Sample handling; sample analysis, record keeping, preventative maintenance, proficiency testing, staff requirements, and training	Pace program expert	1 per year	PI	Section C.1.

¹⁴The assessment types may be supervisory surveillance, management systems reviews, readiness reviews, technical systems audits, audits of data quality, or DQAs.

C.1. Assessments and Response Actions

Any WAPB technical staff may identify and report a field or laboratory nonconformity. Once reported, the program 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, a WAPB Section Chief or the QAO may need to approve the final corrective action.

For field corrective actions, the field crew chief assigned to the sampling event is responsible for all field decisions, including corrective action. Bring any unusual or unexpected occurrence during data or sample collection to the attention of the crew chief. The crew chief decides the necessary immediate actions to take and the follow up actions, if any. The field crew chief has discretion on field corrective actions and documents the action upon return to the office. The Section Chief assigns a staff member to follow up and document any further required action.

¹⁵Assessors must be independent of the group with interest in the data operation.

If a failure, disruption, or other breakdown occurs in the laboratory analytical system take corrective action. Each analytical or contract laboratory must maintain a corrective action program as indicated in the Technical Specifications of IDEM Requests for Quotation, or RFP. The laboratory must document any corrective actions taken because of problems during the handling, preparation, analysis, or reporting of analytical data to the WAPB. Document corrective actions 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.

Laboratory corrective actions require each analytical or contract laboratory, conducting analyses for OWQ, to maintain a corrective action program as indicated in the technical specifications of WAPB RFP 21-66919 (IDEM 2021). The laboratory must document any resulting corrective actions taken for problems during the handling, preparation, analysis, or reporting of analytical data to the WAPB. Document corrective actions in the case narrative section of the report for each sample set. Problems, indicating the laboratory QA system may be out of control, trigger a system audit by the QAO or a designee.

Report significant nonconformities to the IDEM program PI within 14 days, in accordance with the contract. Once identified, the program PI is responsible for ensuring implementation of the corrective action. If the contractor and the IDEM PI cannot come to an agreement on corrective actions or program progress is irreparably harmed, IDEM may refuse payment or conduct other corrective actions through the contract agreement. Several clauses in the state of Indiana contract legal boilerplate language apply and are paraphrased:

- 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 endangering performance of the contract.
 - Perform any other contract provisions.
- Waiver of Rights 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 The contractor shall apply the highest professional and technical guidelines and standards. Further, if the state becomes dissatisfied with the work product of or the working relationship with staff assigned to

work on the contract, it may request in writing the replacement of any or all such staff.

Finally, in the event any problem is identified with QA, or any changes are necessary to the QAPP, make recommendations to the PI and QAM. Communicate any necessary changes to the program team.

C.2. Reports to Management

WAPB QAO submits QA reports, upon completion of the dataset data validation to the program manager (program PI). This ensures investigation and correction of problems arising during the sampling and analysis phases of the program. 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 QC task problems.
- Recommended solutions, and status of corrective actions.
- Status of the extent to which program DQOs are satisfied.

Bring problems arising during data assessment and qualification due to any contract laboratory or QA actions to the program PI's attention. The PI will work with other staff as necessary to determine whether immediate corrective action is required. Implement laboratory corrective actions according to the respective IDEM RFP and contract requirements.

The QAM, relevant WAPB Section Chief, program PI, any technical staff working on corrective actions, and QA staff receive copies of the progress reports when new developments arise. Store corrective actions progress reports along with the program correspondence in IDEM's Virtual File Cabinet which provides availability to any interested parties.

Table 27 Distribution of Assessment Reports and Corrective Actions

Assessment Report Recipient	Why Recipient is Receiving the Assessment Results
Targeted Monitoring Section Chief	Oversight of program
Program PI	Oversight of program and directly responsible for annual project
QA staff	Oversight of laboratory data

D. Determining Data Usability

Upon return of data packets from the laboratory after the data collection or generation phase of the program, WAPB staff performs data review, verification, and validation.

D.1. Data Verification

Data Review

The data review precedes evaluation (data verification, validation, and assessment) of this program and includes review of the items in Table 33.

Table 28 Date Review

Principal Actions, Steps, or Processes to Review Which Could Impact Data Quality	Documents or Forms to Review to Determine Deficiencies or Missing or Incomplete Data
Entering datasheets into AIMS	Field Record for Biological Tissue Contaminants Monitoring Program – Attachment 1
EDI review and import to AIMS per "EDI Import to AIMS II S-004-OWQ-WAP-TL-20-T-R0" and the AIMS User Guide.	MS Access used to export data to a MS Excel spreadsheet for further data validation and use in the QA Review Report.
QA Review of each contract lab report	A MS Word QA review report is created for each lab report. A checklist of items reviewed are provided in each QA report. Deficiencies are communicated with the lab program manager for corrections if possible.

The data verification process for this data operation includes review of the items in Table 34.

Table 29 Data Verification 16

Factors or Characteristics to use for Verification	Specifications to Verify Performance	How to Note Errors
Two rounds of QC for datasheet entry into database	100% data transcription accuracy	Errors are corrected in the AIMS database

¹⁶Additional information covered in the <u>AIMS User Guide</u> (IDEM 2022a) and <u>EDI Import to AIMS II S-004-OWQ-WAP-TL-20-T-R0</u> (IDEM 2020).

Table 30 Data Verifiers

Staff Assigned to Verify Data	Other Roles They May Have with the Program
PI and other technical WAPB field staff	Sample collection and oversight
WAPB QA staff	Program and project QAPP review

D.2. Verification and Validation Methods

Data validation is the process of qualifying analytical or measurement data on the performance of the field and laboratory QC measures incorporated into the sampling and analysis procedures.

WAPB field staff apply several levels of verification to the program data. Upon field data capture, one staff takes the observation and reads the result aloud to

the other staff, who records the data. The recorder then verifies the result is correctly recorded by reading the value aloud back to the observer.

Prior to entering data into AIMS II database, run a completeness check on the field data sheets. The check also verifies filling of all applicable fields and legibility to both field staff conducting observations. Finally, double key the data into the AIMS II database from the original field data sheets. Then resolve any discrepancies. In addition, data collection in the field is subject to the QC checks described in B.5 and the calibration checks described in B.7.

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. Conduct this independent validation 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, QC measures, and calculations. Only report data acquired in the field after the responsible technical staff perform reduction and validation. Report WAPB contract laboratory analyses data, after the laboratory reports the data by reviewing, assessing for QA, and determining the data usability identified by assigning one of four DQA Levels to the data. A.9. contains a list of program records and documents.

D.3. Laboratory Results for Data Validation Process

In lieu of the data validation table identifying the analytes to validate and the DQIs to use to compare how analyte results aligned with measurement quality objectives, as would be done using the data quality objective process. The Fish Contaminants Monitoring Program uses the following data qualifiers to establish data usability.

Table 31 Data Validators

Staff Assigned to Validate the Data	How They are Independent of the Program
WAPB Chemists	WAPB chemists do not participate in planning, sampling, or issuing FCG activities for this program.

QA staff review and qualify laboratory data by using U.S. EPA CLP guidance for data validation. Data flags consist of two parts, a cause (U, Q, D, B, or H) and an action (R or J). For WAPB projects, assign data qualifiers and flags and enter them into AIMS II. Use flags for both the individual test result and QA/QC Review Reports. Table 37 lists data qualifiers and Table 38 lists data quality flags for analytical results.

Table 32 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 are determined on a
	case-by-case basis.
UJ	Estimated (Between MDL and RL). The parameter result is above the MDL but
	below the Laboratory RL and are estimates.

Table 33 WAPB Data Flags

	APB Data Flags
Flags	Description
Q	QC Checks or Criteria. One or more of the QC checks or criteria are out of control
D	RPD for Duplicates. The RPD for a parameter result is outside the acceptable control limits. Consider the parameter an estimate or rejected on the basis listed below:
	If either the sample or duplicate value is less than the RL and the other value exceeds 5 times the method detection limit (MDL), then the sample result is an estimate.
	 If the RPD is outside the established control limits (max. RPD) but below two times the established control limits (max. RPD), then the sample result is an estimate.
	 If the RPD is twice the established control limits (max. RPD) or greater, then reject the sample result.
Н	 Holding Time. The performance of the analysis for this parameter is out of the holding time. Estimate or reject the results on the basis listed below: 1) If performance of the analysis is between the holding time limit and 1.5 times the holding time limit, estimate the result. 2) If performance of the analysis is outside 1.5 times the holding time limit, reject the result.
В	 Blank Contamination This parameter is found in a field or a lab blank. Whether to accept, estimate, or reject the result is 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, reject the result. 1) If the result of the sample is between five and ten times the blank contamination, estimate the result. 2) If the result of the sample is less than the RL or greater than ten times the blank contamination, accept the result.

D.4. Reconciliation with User Requirements

DQA is the process of determining the scientific and statistical quality of data collected to satisfy the project DQOs. Assess field data and laboratory results for usability regarding each specific project DQO (A.7; Section D.1 Data Verification; D.2. Verification and Validation; C.1. Assessments and Response Actions; C.2. Reports to Management; and Section C.). On performance and system audits describe the procedures used to produce data and to evaluate the data production system's effectiveness.

Report data from WAPB contract laboratory analyses after the laboratory reports the data are reviewed, assessed for QA, and the data usability determined and assigned one of four DQA Levels to the data.

- DQA Level 1 Screening Data The results are usually generated onsite with no QC checks. This category includes analytical results with no QC checks, precision or accuracy information, or detection limit calculations. Use is primarily onsite data for presurvey 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. The category includes analytical results with limited QC checks. Detection limits and ranges are set for each analysis. The QC checks information for field or laboratory results and are 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. Method detection limits 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 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, contract-required quantification limit, 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 WAPB Indiana Surface Water Programs QAPP, especially Table 6 (IDEM 2023b), 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.

E. References

- (EPA 1979) <u>Handbook for Analytical Quality Control in Wastewater Laboratories</u>, U.S. Environmental Protection Agency. Washington, DC. EPA/821/B-94/005
- (EPA 1994a) Method 1613 Revision B, Tetra- Through Octa-Chlorinated Dioxins and Furans by Isotope Dilution HRGC/HRMS, U.S. Environmental Protection Agency. Washington, DC. EPA/821/B-94/005
- (EPA 1995) QA/QC Guidance for Sampling and Analysis of Sediments, Water, and <u>Tissues for Dredged Material Evaluations</u>, U.S. Environmental Protection Agency. Washington, DC. EPA 823-B-95-001.
- (EPA 1996a) Method 3540 Revision C, Soxhlet Extraction. U.S. Environmental Protection Agency. Washington, DC.
- (EPA 2000) <u>Guidance for Assessing Chemical Contaminant Data for Use in Fish</u>
 <u>Advisories</u>, Volume 1, Fish Sampling and Analysis Third Edition. U.S. Environmental Protection Agency. Washington, DC.
- (EPA 2001) <u>Appendix to Method 1631</u>, Total Mercury in Tissue, Sludge, Sediment, and Soil by Acid Digestion and BrCl Oxidation. U.S. Environmental Protection Agency. Washington, DC. EPA/821/R/01/013.
- (EPA 2002) <u>Method 1631, Revision E</u>, Mercury in Water by Oxidation, Purge and Trap, and Cold Vapor Atomic Absorption Fluorescence Spectrometry. U.S. Environmental Protection Agency. Washington, DC.
- (EPA 2007) Method 6020A, Inductively Coupled Plasma Mass Spectrometry. U.S. Environmental Protection Agency. Washington, DC.
- (EPA 2007a) Method 8082A, Polychlorinated Biphenyls (PCBs) by Gas Chromatography. U.S. Environmental Protection Agency. Washington, DC.
- (EPA 2007b) Method 1630, Methyl Mercury in Water by Distillation, Aqueous Purge and Trap, and CVAFS U.S. Environmental Protection Agency. Washington, DC. EPA-821-R-01-020, January 2007.

- (EPA 2007c) Method 8081B Revision 2, Organochlorine Pesticides by Gas Chromatography. U.S. Environmental Protection Agency. Washington, DC.
- (EPA 2007d) Method 1614A, Brominated Diphenyl Ethers in Water, Soil, Sediment, and Tissue by HRGC/HRMS. U.S. Environmental Protection Agency. Washington, DC.
- (EPA 2008) Method 1668B, Toxic PCBs by Isotope Dilution HRGC/HRMS. November 2008, Washington, DC; U.S. Environmental Protection Agency, Office of Water. EPA-821-R-08-020.
- (EPA 2017) Method 8270D, Revision 5, Semivolatile Organic Compounds by Gas Chromatography Mass Spectrometry (GC/MS). U.S. Environmental Protection Agency. Washington, DC.
- (EPA 2021) <u>2021 Revision to: Aquatic Life Ambient Water Quality Criterion for Selenium</u> <u>- Freshwater 2016</u>, U.S. Environmental Protection Agency, Office of Water. EPA 822-R-21-006.
- (U.S. FWS 1998) (et. seq.) Principles and Techniques of Electrofising (Online) CSP2C01. Correspondence Course #FID2C01. U.S. Fish and Wildlife Service National Conference Training Center, Branch of Aquatic Resources Training.
- (U.S. PS 2017) United States Power Squadron 2017. <u>State of Indiana: State-Specific Boating Safety Requirements</u> 2017.
- IC <u>Title 14-8-2-27</u> As added by P.L.1-1995, SEC.1. Indiana Code Natural and Cultural Resources, General Provisions and Definitions, Definition of boundary waters.
- IAC (Indiana Administrative Code), <u>Title 327 Water Pollution Control Division</u>, <u>Article 2</u>. <u>Water Quality Standards</u>. Last updated April 26, 2023.
- (IDEM 1992a) revision 1. Section 4, Standard Operating Procedures for Fish Collections, Use of Seines, Electrofishers, and Sample Processing. Biological Studies Section, Surveillance and Standards Branch, Office of Water Management, Indiana Department of Environmental Management, Indianapolis, Indiana.
- (IDEM 2008) <u>Personal Protective Equipment Policy, A-059-OEA-08-P-R0</u>. Indiana Department of Environmental Management, Office of External Affairs.
- (IDEM 2010) IDEM policy, <u>Health and Safety Training Policy, revised October 1 2010</u>, A-030-OEA-10-P-R2, Office of Program Support Health and Safety Director, Indiana Department of Environmental Management, Indianapolis, Indiana
- (IDEM 2016) <u>Injury and/or Illness Resulting from Occupational Exposure Policy, A-034-AW-16-P-R3</u>. IDEM, Office of the Commissioner.
- (IDEM 2018) <u>Indiana Department of Environmental Management 2018 Quality</u>
 <u>Management Plan</u>. Indiana Department of Environmental Management. Office of Water Quality. Indianapolis, Indiana.

- (IDEM 2019) IDEM Hazard Communication (HazCom) Plan. Indiana Department of Environmental Management. Office of Program Support. Indianapolis, Indiana.
- (IDEM 2020) <u>EDI Import to AIMS II</u>. Indiana Department of Environmental Management. Office of Water Quality. Indianapolis, Indiana.
- (IDEM 2021) Request for Proposals 21-66919, Request for Laboratory Analytical Services for the Determination of Contaminants in Fish Tissue and Surficial Aquatic Sediments. Indiana Department of Environmental Management. Indiana Department of Administration. Indianapolis, Indiana.
- (IDEM 2021b) <u>EDI Format Description</u>. Revised May 18, 2021. Indiana Department of Environmental Management. Office of Water Quality. Indianapolis, Indiana.
- (IDEM 2022a) <u>The AIMS II User Guide</u>. Indiana Department of Environmental Management. Office of Water Quality. Indianapolis, Indiana.
- (IDEM 2022b) <u>2022 Indiana Integrated Water Monitoring and Assessment Report to the U.S. EPA. Appendix G: IDEM's 2022 Consolidated Assessment and Listing Methodology (CALM).</u> Indiana Department of Environmental Management, Office of Water Quality, Watershed Assessment and Planning Branch: Indianapolis, Indiana.
- (IDEM 2022c) <u>Indiana Water Quality Monitoring Strategy 2022-2026.</u> Watershed Assessment and Planning Branch, Office of Water Quality. Indiana Department of Environmental Management. Indianapolis, Indiana.
- (IDEM 2022d) <u>2022 Section 303(d) List of Impaired Waters</u>. Approved by U.S. Environmental Protection Agency Pursuant to Clean Water Act Section 303(d). Indiana Department of Environmental Management. Office of Water Quality, Indianapolis, Indiana.
- (IDEM 2022e) Global Positioning System Data Creation. B-001-OWQ-WAP-XXX-22-T-R0. Watershed Assessment and Planning Branch, Office of Water Quality. Indiana Department of Environmental Management. Indianapolis, Indiana.
- (IDEM 2023) <u>Fish Community Field Collection Procedures</u>. B-009-OWQ-WAP-XXX-23-T-R1. Watershed Assessment and Planning Branch, Office of Water Quality. Indiana Department of Environmental Management. Indianapolis, Indiana.
- (IDEM 2023b) <u>WAPB Indiana Surface Water Programs quality Assurance Program Plan</u>. B-001-OWQ-WAP-XX-23-Q-R5. Watershed Assessment and Planning Branch, Office of Water Quality. Indiana Department of Environmental Management. Indianapolis, Indiana.
- (IDOA 2021) Executive Document Summary, Contract #0000000000000000000053618. Indiana Department of Administration, Indianapolis, Indiana.

- (Anderson et.al. 1993) Anderson, Henry A. MD, James R. Amrheim, Pam Shubat, John Hesse. 1993. Protocol For a Uniform Great Lakes Sport Fish Consumption Advisory. Great Lakes Fish Advisory Task Force Protocol Drafting Committee.
- (GLC 2019) Great Lakes Consortium. 2019. <u>Great Lakes Consortium for Fish Consumption Advisories Best Practices for Perfluorooctane Sulfonate (PFOS)</u> Guidelines.
- (McCann and Anderson 2007) McCann, Pat and Henry Anderson, MD. 2007. <u>A Protocol for Mercury-based Fish Consumption Advice: An Addendum to the 1993 Protocol for a Uniform Great Lakes Sport Fish Consumption Advisory</u>. Great Lakes Fish Advisory Task Force Protocol Drafting Committee.
- (Dewitz 2021) Dewitz, J., and U.S. Geological Survey, 2021, National Land Cover Database (NLCD) 2019 Products (ver. 2.0, June 2021): U.S. Geological Survey data release, https://doi.org/10.5066/P9KZCM54
- (Hornshaw) <u>Discussion Paper for a Chlordane Health Protection Value (HPV).</u> Great Lakes Fish Advisory Task Force Protocol Drafting Committee.
- (Pace 2020) Pace Analytical. 2020. PFAS Method DoD 35. Determination of Selected 36 Per- and Polyfluoroalkyl Substances (PFAS) by LC/MS/MS (Isotope Dilution). Pace Analytical, Minneapolis, MI.
- (Sauvé and Desrosiers 2014) Sauvé, S., and Desrosiers, M. 2014. <u>A review of what is</u> an emerging contaminant. Chemistry Central Journal, 8(1), 15.
- (Smith and Young 2009) Smith, H., and Young, R. (2009, April 21). Poisoned Waters.
- (Wisconsin Department of Health Services) <u>Potential for Human Exposure to</u>
 <u>Toxaphene Through Consumption of Great Lakes Fish.</u> Great Lakes Fish Advisory
 Task Force Protocol Drafting Committee.
- * All hyperlinks were current as of 30 May 2023. References not available via hyperlink are stored by the WAPB on the agency shared (S:) drive and backed up by the Indiana Office of Technology. Please contact the branch by telephone at (317) 308-3173 for further information.

F. Attachments

The blank field forms, checklists, and other materials used by staff implementing the QAPP include:

- Attachment 1: Field Record for Biological Tissue Contaminants Monitoring Program
- Attachment 2: Contract Laboratory Chain of Custody Form

Attachment 3: Sample Collection Procedure

Attachment 4: List of Acronyms

Attachment 5: Definitions

Attachment 6: 2023 Fish Tissue Project

Figure 3

Tables 34-37

Attachment 7: 2024 Fish Tissue Project (to be selected Spring 2024)

Attachment 8: 2025 Fish Tissue Project (to be selected Spring 2025)

Attachment 9: 2026 Fish Tissue Project (to be selected Spring 2026)

Attachment 10: 2027 Fish Tissue Project (to be selected Spring 2027)

Field Record for Biological Tissue Contaminants Monitoring Program

	Office of			t of Environme tershed Assess			ranch	
	Field Re	ecord for B	iological I	Tissue Contam	inant Moni	itoring Pro	<u>gram</u>	
Site ID:			9	Sampling Date and	Time:			
Sample ID:_						(mm/dd/yyy	3)	(24hr clock)
SITE LOCA	ATION							
Waterbody N	Vame:							
County			Fipscode	Lat./L	ong.:			
Location:								
				RESER		WETL	AND	
Collection M	[ethod:							
Collector's N	Vame(s):							
Agency:				Phone:()				
Composite S	ample #:			_Number of Indivi	duals:	Lab ID		
Species Nam	1e:							
Sample Prepa	aration: SI	KIN-ON SCA	LELESS	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				_
		100 =			osite mean lei			
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Composite S	ample #:			_Number of Indivi	duals:	Lab ID		
		KIN-ON SCA		SKIN-OFF	WHOLE	OTHER:		
Fish#		Weight(gm)	Sex(M,F)	Fish#	Length(mm)	_	Sex(M,F)	
001	()	/B/	(- /	007			(
002				008				_
003				009				_
004				010				
005				011				
006				012				
(min length/r	nax length)x	100 =	%	•	osite mean lei	ngth	mm	
, -		100=			osite mean we			
Notes (e.g., I	DELT anoma	lies)						

Chain of Custody / Analytical Request Document

CHAIN-OF-CUSTODY / Analytical Request Document
The Chain-of-Custody is a LEGAL DOCUMENT. All relevant fields must be completed accurately.

Section Requir	n A ed Client Information:	Section Require		ct Infor	mation:						tion C		ın:												Pag	je:			of	
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Sample 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, or 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). Remove scales from both sides of the fish, back, and belly areas, if necessary. Fillet the fish 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.

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

FCG Fish Consumption Guidelines IAC Indiana Administrative Code

IDNR Indiana Department of Natural Resources

IDOH Indiana 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.

Cojurisdictional 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 (EPA 2000).

Waters of the state As defined by IC 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.

2023 Fish Tissue Project

A.6.2. Specific Sampling Locations

Table 34 and Figure 3 describe the tentative 2023 sampling sites.

Tables 35 through 38 identify the numerous variables of interest to the user for each sampling site.

Figure 2 2023 Sampling Locations

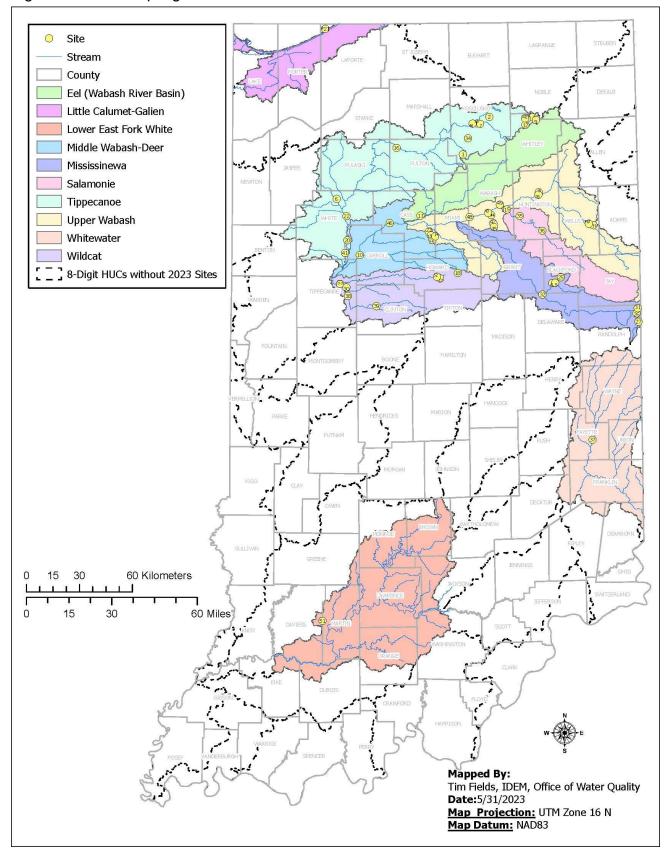


Table 34 Sample Collection Locations

Sample Site ID #1	Site ID	Waterbody	Location ²	Latitude	Longitude
1	WTI-04-0026	Beaver Dam Lake	Open water	41.09157	-85.97524
2	WTI-02-0065	Big Chapman Lake	Open Water	41.28149	-85.79365
3	WTI010-0029	Big Lake	Open water	41.27639	-85.50083
4	WMI-03-0011	Big Lick Creek	CR 100 West Downstream of Hartford City 3M Plant	40.42983	-85.38925
5	WMI-03-0016	Big Lick Creek	SR 3	40.43161	-85.37027
6	WTI110-0002	Big Monon Creek	SR 16	40.86972	-86.82583
7	WTI020-0046	Center Lake	Center of lake	41.24667	-85.85667
8	WUW-11-0001	Clair Lake	Center of lake	40.89288	-85.46871
9	WTI010-0032	Crooked Lake	Center of lake	41.26194	-85.47972
10	WDE050-0022	Deer Creek	Riley Park	40.58417	-86.67222
11	WAE070-0011	Eel River	CR 150 N Northeast of Logansport	40.78233	-86.26450
12	WTI010-0034	Goose Lake	Open water	41.23833	-85.55139
13	WUW-15-0010	Government Ditch	CR 950 East	40.68171	-86.19483
14	WUW-15-0011	Government Ditch	CR 1100 East	40.67658	-86.16666
15	WSA040-0019	Hominy Ridge Lake	Open water	40.80833	-85.68472
16	WTI060-0033	King Lake	Open water	41.12861	-86.42194
17	WAW010-0083	Kokomo Creek	Highland Park	40.46222	-86.13639
18	WAW010-0093	Kokomo Waterworks Reservoir Number Two	Open water	40.48847	-86.01833
19	WUW060-0006	Kunkel Lake	Open water	40.72056	-85.10667
20	WTI140-0004	Lake Freeman	DAM END	40.65861	-86.75472
*21	LMM010-0010	Lake Michigan	Michigan City, Indiana (Trail Creek)	41.73544	-86.90103
22	WTI120-0004	Lake Shafer	DAM END	40.78194	-86.76056
23	WUW-15-0003	Little Deer Creek	CR 450 South	40.70018	-86.20934
24	WUW-15-0008	Little Deer Creek	CR 1000 East	40.65936	-86.18496
25	WMI-03-0012	Little Lick Creek	CR 200 East	40.45891	-85.33161
26	WMI010-0011	Little Mississinewa River	CR 700 North Downstream of Union City Sewage Treatment Plant	40.26611	-84.83306
27	WMI010-0018	Little Mississinewa River	CR 400 North	40.22417	-84.82139
28	WUW120-0006	Little Wabash River	Broadway Street	40.88139	-85.47139
29	WTI010-0041	Loon Lake	Open water	41.26944	-85.54056
30	WAW030-0026	Middle Fork Wildcat Creek	SR 26	40.41861	-86.76194
31	WMI010-0014	Mississinewa River	CR 800 East	40.29347	-84.82458
32	WMI030-0009	Mississinewa River	CR 370 West	40.37250	-85.45639
33	WTI010-0043	Old Lake	Open water	41.27083	-85.55250
34	WTI030-0025	Palestine Lake	Open water	41.17333	-85.94028
35	WSA040-0021	Salamonie Lake	Lost Bridge State Recreation Area East	40.77444	-85.59944
36	WSA040-0018	Salamonie River	CR 900 South	40.69778	-85.44972

Sample Site ID #1	Site ID	Waterbody	Location ²	Latitude	Longitude
37	GMW-04-0039	Smalleys Lake	Connersville Pay Lake	39.62902	-85.14293
38	WAW040-0042	South Fork Wildcat Creek	SR 38	40.37556	-86.75194
39	WAW040-0077	South Fork Wildcat Creek	CR 300 West	40.32306	-86.56222
40	WTI030-0020	Tippecanoe River	Old US 30	41.24806	-85.90722
41	WTI150-0006	Tippecanoe River	SR 18	40.59417	-86.77056
42	WUW-14-0025	Treaty Creek	SR 124	40.73906	-85.77662
43	WUW150-0003	Treaty Creek	Waterworks Road	40.79222	-85.81028
44	WUW-14-0026	Tributary of Treaty Creek	Old Indiana 15	40.78062	-85.79011
45	WUW-14-0027	Tributary of Treaty Creek	CR 100 East	40.72021	-85.77283
46	WDE010-0010	Wabash River	France Park	40.74500	-86.47056
*47	WUW070-0010	Wabash River	River Road	40.72861	-85.13722
48	WUW150-0005	Wabash River	Omar Cole Ramp	40.77278	-85.93528
49	WUW150-0006	Wabash River	Lagro, Indiana	40.83361	-85.73139
50	WTI020-0015	Walnut Creek	Old US 30	41.24842	-85.87527
51	WEL130-0008	West Boggs Lake	DAM END	38.72194	-86.92361
52	WAW-04-0004	Wildcat Creek	Downstream of Kokomo Creek	40.47020	-86.15714
53	WAW050-0009	Wildcat Creek	Wildcat Creek County Park	40.43722	-86.80472

^{*}Historical Core Sampling Site

The IDNR is scheduled to collect the fish tissue samples from the following locations:

- 3 WTI010-0029 Big Lake
- 8 WUW-11-0001 Clair Lake
- 9 WTI010-0032 Crooked Lake
- 18 WAW010-0093 Kokomo Waterworks Reservoir Number Two
- 20 WTI140-0004 Lake Freeman
- 21 LMM010-0010 Lake Michigan (Lake Trout [18 individuals], Freshwater Drum, Burbot)
- 29 WTI010-0041 Loon Lake
- 37 GMW-04-0039 Smalleys Lake
- 51 WEL130-0008 West Boggs Lake

Table 35 Constituents or Characteristics to Sample

Sample Site ID #	Target Constituents or Parameters to Sample, Measure, and Record	Matrix from Which to Take Sample	Units in Which to Measure Constituents	Sampling SOP or Procedure to Follow
ALL	Lipids	Fish Tissue	%	Pace Lipid – uses same value as EPA 1630/1631E. (EPA 2007b, EPA 2002)

¹Sample Site ID # is the number, specific to the QAPP, for each sampling location.

²Access to each site is approved by landowners or considered public access (ex. boat ramps, parks, etc.).

Sample Site ID #	Target Constituents or Parameters to Sample, Measure, and Record	Matrix from Which to Take Sample	Units in Which to Measure Constituents	Sampling SOP or Procedure to Follow
ALL	Moisture	Fish Tissue	%	
ALL	Total PCBs	Fish Tissue	μg/kg ww	U.S. EPA Method 8082A (EPA 2007a) and U.S. EPA Preparation 3540C (EPA 1996a)
ALL	PFAS	Fish Tissue	μg/kg ww	PFAS Method DoD 36 (Pace 2020)
ALL	Metals	Fish Tissue	μg/kg ww	U.S. EPA Method 1631, Revision E (EPA 2002, EPA 2001) and U.S. EPA Method 6020A (EPA 2007)
21, 47, 52	Pesticides	Fish Tissue	μg/kg ww	U.S. EPA Method 8081B (EPA 2007c)
None	PAHs	Fish Tissue	μg/kg ww	U.S. EPA Method 8270D and Method 8310 (EPA 2017 and U.S. EPA 1986)

Table 36 Sampling Specifics

Sample Site ID #	Constituents	Type of Samples	Number of Samples	Frequency of Sampling	Type and Number of QC Samples ³
4, 5, 13, 14, 23, 24, 25, 42, 43, 44, 45	Fish Tissue	Composite	2	1 event	MS/MSD 1 per 20 samples Lab Duplicate 1 per 20 samples
21	Fish Tissue	Composite	7	1 event	MS/MSD 1 per 20 samples Lab Duplicate 1 per 20 samples
All Other Sites	Fish Tissue	Composite	3-5	1 event	MS/MSD 1 per 20 samples Lab Duplicate 1 per 20 samples

For 2023 Lake Michigan sampling (Sample Site ID # 21) the target species are Lake Trout, Freshwater Drum, and Burbot. Salmonidae samples (Lake Trout) are collected from Lake Michigan with the goal of analyzing 3 size classes each consisting of 3 individuals along with a replicate of those samples (18 total individuals) for statistical purposes.

Table 37 Sample Collection Schedule

Sample Site ID #	Constituents	Date
29, 51	Fish Tissue	March - June
All Other Sites	Fish Tissue	August - October

Table 38 Method of Analysis Employed in the Lab*

Sample Site ID #	Parameter	Method of Analysis ⁴	Analysis in Field or Lab?
ALL	Lipids	Pace Lipid - EnChemSVO-59 which is an equivalent to EPA 160.1 for fish tissues	Lab
ALL	Moisture		Lab
ALL	Total PCBs	U.S. EPA Method 8082A (EPA 2007a) and U.S. EPA Preparation 3540C (EPA 1996a)	Lab
ALL	PFAS	PFAS Method DoD 35 (Pace 2020)	Lab
ALL	Metals	U.S. EPA Method 1631, Revision E (EPA 2002, EPA 2001) and U.S. EPA Method 6020A (EPA 2007)	Lab
21,47,52	Pesticides	U.S. EPA Method 8081B (EPA 2007c)	Lab
None	PAHs	U.S. EPA Method 8270D and Method 8310 (EPA 2017 and U.S. EPA 1986)	Lab

⁴ Method of analysis could include lab method, field test kit, or a properly calibrated measurement instrument.

^{*} Reporting limits are detailed in Table 13.

Attachment 7 2024 Fish Tissue Project (to be selected Spring 2024)

Attachment 8 2025 Fish Tissue Project (to be selected Spring 2025)

Attachment 9 2026 Fish Tissue Project (to be selected Spring 2026)

Attachment 10 2027 Fish Tissue Project (to be selected Spring 2027)