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Quality Assurance Report for the 2018-19 National Rivers and Streams Assessment Fish Fillet Tissue Study

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Disclaimer

The U.S. Environmental Protection Agency, Office of Water, Office of Science and Technology has approved this report for publication. Mention of trade names, commercial products, or services does not constitute official EPA approval, endorsement, or recommendation for use.

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Chapter 1
Introduction

This report documents the quality of data gathered during EPA's 2018-19 National Rivers and Streams Assessment (NRSA), a probability-based survey designed to assess the condition of the nation's river and stream resources. The 2018-19 NRSA included collection and analysis of physical, chemical, and biological indicator data that will allow a statistically valid characterization of the condition of the nation's rivers and streams. The Office of Wetlands, Oceans, and Watersheds (OWOW) within the Office of Water (OW) was responsible for the overall planning and implementation of the 2018-19 NRSA.

One component of the 2018-19 NRSA is the Fish Fillet Tissue Study, which is designed to examine national fish contamination trends in U.S. rivers. EPA's Office of Science and Technology (OST) within OW collaborated with the Office of Research and Development Pacific Ecological Systems Division (ORD-PESD) in Corvallis, Oregon, to plan and implement the fish fillet tissue study under the 2018-19 NRSA. By the end of the 2019 field sampling season, whole fish composite samples (for fillet analysis) were collected from 290 sites. This report documents the quality of data gathered during the 2018-19 NRSA Fish Fillet Tissue Study.

Section 1.1 Background

Obtaining statistically representative occurrence data on multiple contaminants in fish tissue is a priority area of interest for EPA. Since 1998, OW has collaborated with ORD to conduct a series of national- and regional-scale assessments of contaminants in fish tissue through statistically based studies of U.S. lakes and rivers. These EPA studies are referred to as the National Lake Fish Tissue Study, the 2008-09 NRSA, the 2013-14 NRSA, the Great Lakes Human Health Fish Tissue Study conducted under the 2010 National Coastal Condition Assessment (NCCA), and the Great Lakes Human Health Fish Fillet Tissue Study conducted under the 2015 NCCA. The 2018-19 NRSA provided additional national data on the occurrence and distribution of contaminants in the fillet tissue from river fish and, through comparison with the 2013-14 NRSA fish tissue results, allowed EPA to examine temporal trends.

Section 1.2 Study Design

OST collaborated with OWOW and with ORD-PESD in Corvallis, Oregon, to plan and implement the fish fillet indicator within the framework of the 2018-19 NRSA. Fish composite samples were collected during June through September of 2018 and extended into November for the 2019 field season at a statistical subset of approximately 290 sites in the NRSA framework (Figure 1). A total of 477 sites were selected and scheduled for sampling; however, due to local restrictions and weather conditions, only 290 sites provided adequate samples for analysis.

The following were the key design components for the 2018-19 NRSA fish fillet tissue study:

- Sampling approximately 290 randomly selected sites during 2018 and 2019, subject to local conditions and restrictions.
- Collecting one fish composite sample for human health applications (i.e., five similarly sized adult fish of the same species that are commonly consumed by humans) from each site.
- Shipping whole fish samples to a commercial laboratory for storage and fish sample preparation, which includes collection of tissue plug samples for mercury analysis before filleting the fish, removing both fillets from each fish, homogenizing the fillet tissue composites, and preparing

fillet tissue aliquots for analysis of mercury, per- and polyfluoroalkyl substances (PFAS) and polychlorinated biphenyls (PCBs).

- Analyzing all the fillet tissue samples for mercury (total), PCBs, and PFAS.

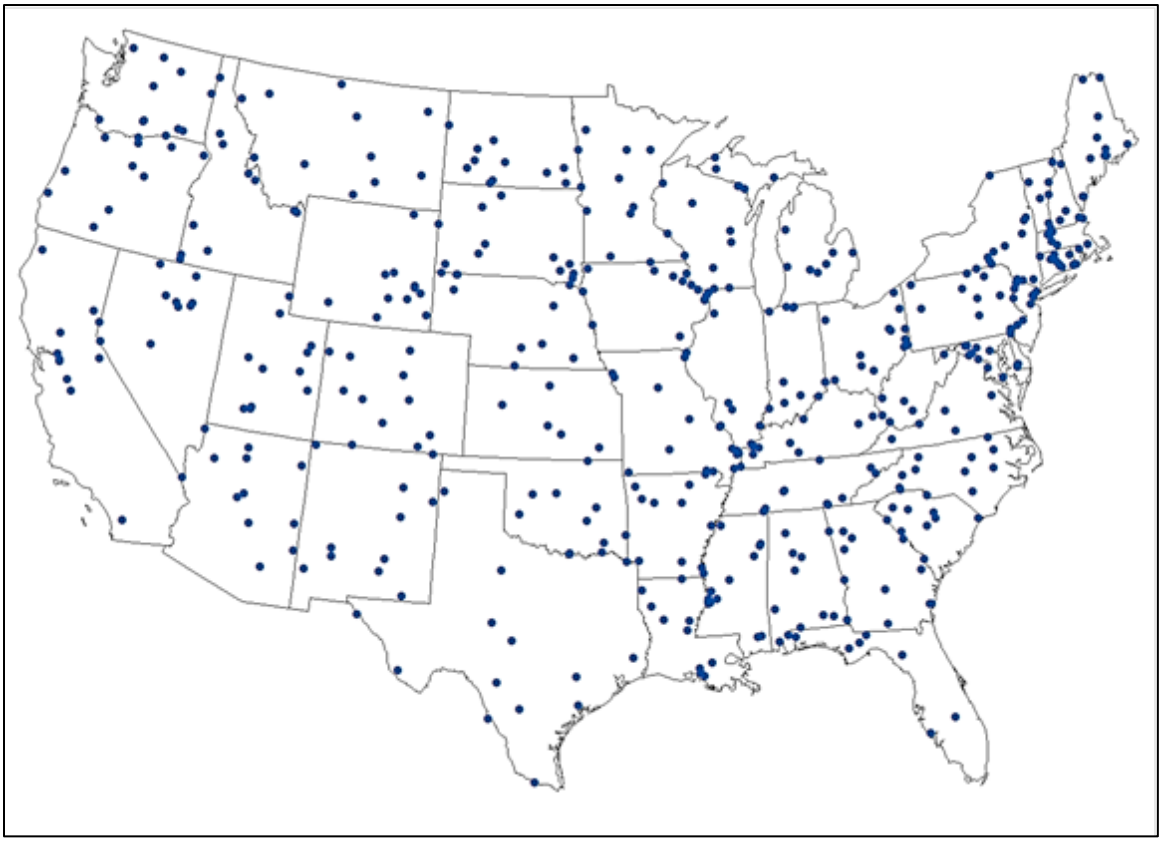

Figure 1. 2018-19 NRSA Fish Fillet Tissue Study sampling locations

EPA stored the 2018-19 NRSA whole fish samples in freezers leased by GDIT at Microbac Laboratories in Baltimore, Maryland, prior to transporting them to the sample preparation laboratory. Tetra Tech's Center for Ecological Sciences in Owings Mills, Maryland, was the sample preparation laboratory preparing the homogenized fish fillet tissue samples and rinsates for analysis as outlined in the fourth bullet above. The sample preparation laboratory prepared aliquots of fillet tissue for mercury, PCBs, PFAS, and archive tissue samples to allow for further analysis of 2018-19 NRSA samples in the future. Commercial environmental laboratories analyzed the 2018-19 NRSA fish fillet tissue samples for mercury, PCB congeners, and PFAS, under project-specific purchase orders issued by GDIT. Procedures for handling and shipping homogenized fish tissue samples to Microbac and the analysis laboratories are described in Appendix B of the *Quality Assurance Project Plan for Preparation of Fish Fillet Tissue Samples for the 2018-19 National Rivers and Streams Assessment* (USEPA 2018b).

Note: *Unless otherwise modified, all references to "fish" and "samples" in this report refer to homogenized fish fillet tissue samples prepared by Tetra Tech.*

Section 1.3 Study Participants

The 2018-19 NRSA project team consisted of managers, scientists, statisticians, and QA personnel in OST, and ORD-WED, along with contractors providing scientific and technical support to OST from GDIT and Tetra Tech, Inc. (Figure 2). Project team members from OST provided support for developing

and reviewing technical and program information related to all aspects of the study, including training materials, standard operating procedures, QAPPs, analytical QA reports, briefings and reports on study results, and outreach materials. Key members of the project team are listed below.

- Leanne Stahl, OST NRSA fish fillet tissue study Technical Leader and OST Project Manager, provided overall direction for planning and implementation of this fillet tissue study being conducted under the NRSA.
- Marion Kelly, OST Quality Assurance Officer, was responsible for reviewing and approving all QAPPs that involve scientific work being conducted by OST with support from Bill Kramer, the Standards and Health Protection Division QA Coordinator.
- Blaine Snyder, Tetra Tech Project Leader, was responsible for managing all aspects of the technical support being provided by Tetra Tech staff for the NRSA fish fillet tissue study.
- Susan Lanberg was the Tetra Tech QA Officer.
- Yildiz Chambers-Velarde, GDIT Work Assignment Lead, was responsible for managing all aspects of the technical support being provided by GDIT staff for the 2018-19 NRSA fish fillet tissue study.
- Harry McCarty, GDIT Project Leader, was responsible for managing all aspects of the technical support being provided by GDIT staff for the NRSA fish fillet tissue study.
- Marguerite Jones was the GDIT QA Officer.
- Tony Olsen, Senior Statistician at ORD-WED in Corvallis, Oregon, supported the NRSA fish fillet tissue study by providing technical expertise for study planning and implementation.

Two commercial laboratories analyzed the 2018-19 NRSA fish tissue samples for mercury, PCBs, and PFAS, under purchase orders issued by GDIT, as shown below and in Figure 2.

Laboratory	Analysis Type
ALS-Environmental	Mercury
SGS-AXYS Analytical	PCB congeners
SGS-AXYS Analytical	PFAS

Section 1.4 Study Results

EPA posted the final analytical results for all of the samples in this study in MS Excel files at:

https://www.epa.gov/fish-tech/2018-2019-national-rivers-and-streams-assessment-fish-tissue-study

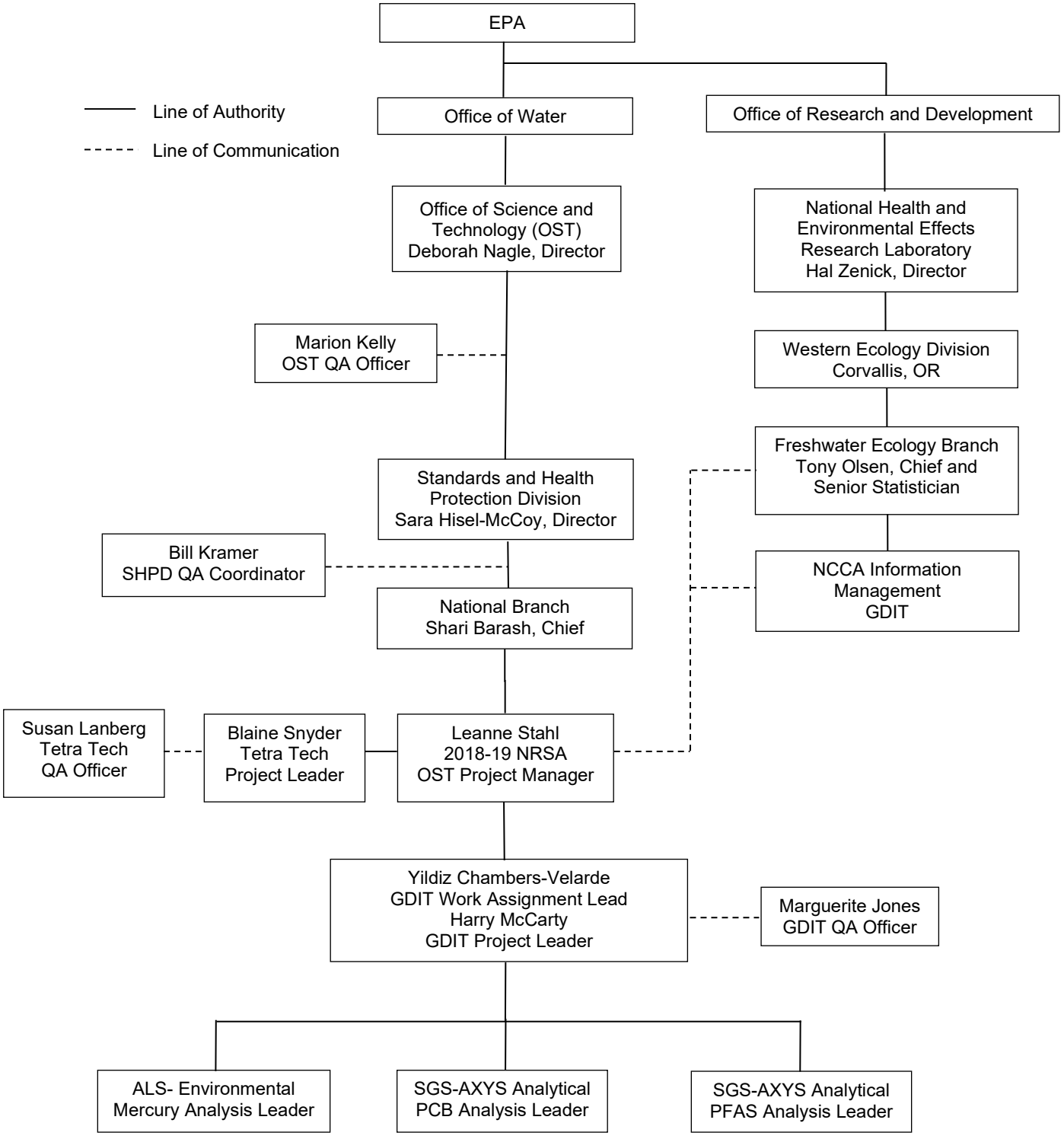

Figure 2. NRSA project team organization

Chapter 2
Quality Assurance Program

At the beginning of the study, EPA managers recognized that data gathered from the study would be used extensively by individuals responsible for making environmental, economic, and policy decisions. Environmental measurements always contain some level of uncertainty. Decision makers, therefore, must recognize (and have the means to assess) the uncertainty associated with the data on which their decisions are based. In recognition of this, the study managers established a quality assurance (QA) program to ensure that data produced under the study would meet defined standards of quality.

Section 2.1 Quality Assurance Project Plans

Three separate Quality Assurance Project Plans (QAPPs) are associated with this study. In 2018, OWOW developed the *National Rivers and Streams Assessment 2018-2019: Quality Assurance Project Plan* (USEPA 2018a) that contains elements of the overall project management, data quality objectives, measurement and data acquisition, and information management for the NRSA, and is based on the guidelines developed and followed in the Western Environmental Monitoring and Assessment Program (EMAP).

Also in 2018, OST developed the *Quality Assurance Project Plan for Preparation of Fish Fillet Tissue Samples for the 2018-19 National Rivers and Streams Assessment* that described the procedures for preparing composite fish tissue samples (USEPA 2018b).

On August 1, 2019, OST developed the *Quality Assurance Project Plan for Sample Analysis for the 2018-19 National Rivers and Streams Assessment Fish Fillet Indicator* that described the requirements for mercury analysis (USEPA 2019a). On August 27, 2019, the first revision to the OST QAPP was released to include the requirements for PCB analysis (USEPA 2019b). The second and final revision to the QAPP was issued later in November 2019, which added the requirements for PFAS analysis (USEPA 2019c).

Section 2.2 Training

Fish Tissue Sample Preparation

Specialized training was provided for the laboratory technicians who prepared fish tissue fillets, homogenates, and rinsates for the study. Training workshops were conducted at Tetra Tech Biological Research Facility in Owings Mills, Maryland for all laboratory staff involved with 2018-19 NRSA fish tissue sample preparation, to accomplish the following objectives:

- present NRSA fish tissue preparation, homogenization and distribution procedures described in Appendix B to the QAPP,
- demonstrate filleting and homogenizing techniques with fish from invalid 2018-19 NRSA samples, and
- provide hands-on opportunities for fish preparation laboratory staff to become proficient at filleting and homogenizing fish samples.

Analysis of Fish Tissue Samples

All laboratory staff involved in the analysis of fish tissue samples were required to be proficient in the associated tasks, as required by each analytical laboratory's existing quality system. All GDIT staff

involved in analytical data review and assessment were already proficient in data review, therefore no specialized training was required for data reviewers for this project.

Section 2.3 Sample Preparation and Analysis QA/QC

EPA integrated various QA/QC activities into the study to ensure data comparability and generate analytical data of known quality during preparation and analysis of the fish tissue samples and evaluation of analytical data quality. There were separate QA/QC activities associated with the preparation of the fish fillet samples and the analyses of those samples.

Following is a summary of the critical QA/QC components associated with the sample preparation process:

- Development and implementation of the sample preparation activities QAPP (USEPA 2018b)
- Use of one laboratory for sample preparation (filleting, tissue homogenization, and preparation of tissue aliquots)
- Requirement for triplicate lipid analyses to test for tissue homogeneity during sample preparation
- Requirement for preparation equipment rinsate samples and solvent blanks with each batch fish fillet tissue samples prepared
- Requirement for analyses of the rinsate samples and solvent blanks for mercury, selected PCB congeners, and PFAS
- Review and acceptance of mercury and PCB rinsate results by EPA before proceeding with preparation of additional samples

Following is a summary of the critical QA/QC components associated with the sample analysis process:

- Development and implementation of the analytical activities QAPP (USEPA 2019a, 2019b, and 2019c)
- Use of one laboratory for the analyses of a given class of analytes
- Identification of quantifiable measurement quality objectives
- Use of pure and traceable reference standards
- Demonstration of instrument calibration and system performance
- Periodic calibration verification
- Analysis of QC samples to assess performance of analytical methods
- Specification of method detection limits (MDLs) and method/chemical QC acceptance criteria that applied throughout the study
- Use of a standardized data quality assessment process

The general measurement quality objective (MQO) for the study was to satisfy method-specific performance criteria. The analytical activities QAPP provides a summary of the method performance criteria and specifies MQOs and QC acceptance criteria to assess the bias and precision associated with the analytical methods used for this study. Chapter 4 of this report describes the process for data quality assessment and presents the results of these assessments, which includes data from the following laboratory QC samples or measures: blanks, recoveries for isotopically labeled compounds spiked into field-based tissue samples, matrix spike (MS) samples, laboratory duplicate samples, laboratory control samples, and calibration verifications. Chapter 4 also includes a discussion of data completeness for the study.

Section 2.4 QA Oversight of Laboratory Operations

The GDIT Project Leader scheduled and tracked all analytical work performed by laboratories for mercury, PCB, and PFAS analyses. The GDIT Project Leader also coordinated with staff at Tetra Tech regarding fish tissue sample shipments.

When samples were shipped to an analytical laboratory, the GDIT Project Leader contacted designated laboratory staff by email to notify them of the forthcoming shipment(s) and request that they contact GDIT if the shipments did not arrive intact, as scheduled. Within 24 hours of scheduled sample receipt, GDIT contacted the laboratory to verify that the samples arrived in good condition, and if problems were noted, GDIT worked with the laboratory and EPA to resolve any problems as quickly as possible to minimize data integrity problems.

GDIT communicated periodically with laboratory staff by telephone or email to monitor the progress of analytical sample preparation, sample analysis, and data reporting. If any technical problems were encountered during sample preparation and analysis, GDIT identified a technical expert within GDIT to assist in resolving the problem, and work with EPA to identify and implement a solution to the problem. In cases in which the laboratory failed to deliver data on time, or if the laboratory notified GDIT of anticipated reporting delays, GDIT notified the EPA Project Manager. To the extent possible, GDIT adjusted schedules and shifted resources within GDIT as necessary to minimize the impact of any laboratory delays on EPA schedules. GDIT also immediately notified the Project Manager of any laboratory delays that were anticipated to affect EPA schedules.

Finally, the GDIT Project Leader monitored the progress of the data quality audits (data reviews) and database development to ensure that each laboratory data submission was reviewed in a timely manner. If dedicated staff were not able to meet EPA schedules, GDIT identified additional staff who were qualified and capable of reviewing the data so that EPA schedules could be met. In cases when such resources could not be identified, and if training new employees was not feasible, GDIT met with the EPA Project Manager to discuss an appropriate solution.

Chapter 3
Preparation and Analysis Methods

To control variability among tissue sample results, all samples collected during the study were analyzed by a single set of methods, and all analyses performed with a given method were performed by only one laboratory. Further control of variability was ensured by utilizing a single laboratory to prepare, composite, homogenize, and aliquot samples in a strictly controlled, contaminant-free environment. The methods employed by the sample preparation laboratory and by the three analysis laboratories are described below.

Section 3.1 Preparation of Fish Tissue Samples

Tetra Tech served as the fish sample preparation laboratory for the study. In this role, Tetra Tech was responsible for filleting each valid fish sample, homogenizing the fillet tissue, preparing the required number of fish tissue aliquots for analysis and archive, shipping the fish tissue aliquots for each analysis to the designated analytical laboratory, and storing archived fish tissue samples in a freezer at its facility. The specific procedures for all 2018-19 NRSA fish sample preparation activities are described in Appendix B of the 2018-19 NRSA fish fillet sample preparation QAPP (USEPA 2018b).

Fish were prepared by trained technicians, using thoroughly cleaned utensils and cutting boards (cleaning procedures are detailed in the sample preparation QAPP for the study). Each fish was weighed to the nearest gram wet weight, rinsed with deionized water, and filleted on a glass cutting board. Fillets from both sides of each fish were prepared with scales removed, skin on, and belly flap (ventral muscle and skin) attached. Fillets were composited using the "batch" method, in which all of the individual specimens that comprise the sample were homogenized together, regardless of each individual specimen's proportion to one another (as opposed to the "individual" method, in which equal weights of each specimen are added together).

An electric meat grinder was used to homogenize the fillet tissue. Entire fillets (with skin and belly flap) from both sides of each fish were homogenized, and the entire homogenized volume of all fillets from the fish sample was used to prepare the tissue sample. Tissues were mixed thoroughly until they were completely homogenized as evidenced by a fillet homogenate that consisted of a fine paste of uniform color and texture. Homogeneity was confirmed by conducting triplicate analyses of the lipid content in one of every twenty samples. The collective weight of the homogenized tissue from each sample was recorded to the nearest gram (wet weight) after processing. Tetra Tech prepared fillet tissue aliquots according to the specifications listed in the fish sample preparation procedures in Appendix B of the 2018-19 NRSA fish fillet sample preparation QAPP (USEPA 2018b).

Section 3.2 Analysis of Fish Tissue Samples for Mercury

Fish tissue samples were prepared and analyzed by ALS-Environmental (Kelso, WA), using Procedure I from "Appendix to Method 1631, Total Mercury in Tissue, Sludge, Sediment, and Soil by Acid Digestion and BrCl Oxidation" from Revision B of Method 1631 (1631B) for sample preparation (USEPA 2001), and Revision E of Method 1631 (1631E) for the analysis of mercury in fish tissue samples (USEPA 2002). This laboratory utilized approximately 0.5 g of tissue for analysis. The sample was digested with a combination of nitric and sulfuric acids. The mercury in the sample was oxidized with bromine monochloride (BrCl) and analyzed by cold-vapor atomic fluorescence spectrometry. Tissue sample results were reported based on the wet weight of the tissue sample, in nanograms per gram (ng/g).

Section 3.3 Analysis of Fish Tissue Samples for PCBs

The PCB samples were prepared and analyzed by SGS-AXYS Analytical Services Ltd. (Sidney, BC, Canada), in general accordance with EPA Method 1668C (USEPA 2010) and as detailed in the laboratory's SOP. The samples were analyzed for all 209 PCB congeners and reported as either individual congeners or coeluting groups of congeners. The SGS-AXYS SOP deviated from the published EPA method in several aspects, including:

- An anthropogenic isolation column was not used for cleanup
- The Soxhlet extraction apparatus was cleaned with toluene instead of dichloromethane
- The CALVER solution contained all 209 PCB congeners instead of only the 27 PCB congeners in Table 4 of Method 1668C.

The entire list of modifications is presented in detail in the 2018-19 NRSA sample analysis QAPP. These changes fall within the method's established allowance for flexibility, and EPA accepted these deviations from Method 1668C for the purposes of the study.

The laboratory utilized approximately 10 g of tissue for the analysis. The samples were extracted with methylene chloride and analyzed by high resolution gas chromatography-mass spectrometry (HRGC/MS). Tissue sample results were reported based on the wet weight of the tissue sample, in nanograms per gram (ng/g).

Section 3.4 Analysis of Fish Tissue and Rinsate Samples for PFAS

At the time of this study, there were no formal analytical methods from EPA or any voluntary consensus standard bodies for the PFAS analyses of tissue samples. Therefore, fish tissue samples were analyzed by SGS-AXYS (Sidney, BC, Canada) using procedures developed, tested, and documented in that laboratory. The SOP for the procedure is considered proprietary by the laboratory. However, the SOP was reviewed by GDIT, and the analytical procedure is briefly described below.

Approximately 2 g of fish tissue was required for analysis. If matrix-related analytical problems were identified during the analysis of a given fish tissue sample, a sample aliquot of 1 g was used to minimize those problems. The samples were spiked with 24 isotopically labeled standards and extracted by shaking in a caustic solution of methanol, water, potassium hydroxide, and acetonitrile. The hydroxide solution breaks down the tissue and allows the PFAS analytes to be extracted into the solution.

After extraction, the solution was centrifuged to remove the solids and the supernatant liquid diluted with reagent water and processed by solid-phase extraction (SPE) on a weak anion exchange sorbent. The PFAS analytes were eluted from the SPE cartridge and the eluant spiked with additional labeled recovery standards and analyzed by high performance liquid chromatography with tandem mass spectrometry.

The concentration of each PFAS analyte is determined using the responses from one of the ^{13}C- or deuterium-labeled standards added prior to sample extraction, applying the technique known as isotope dilution. As a result, the target analyte concentrations are corrected for the recovery of the labeled standards, thus accounting for extraction efficiencies and losses during cleanup. Tissue sample results were reported based on the wet weight of the tissue sample, in nanograms per gram (ng/g).

Section 3.5 Analysis of Rinsates and Solvent Blanks

As noted in Section 2.3, Tetra Tech prepared equipment rinsate samples with each batch of fish fillet tissue samples. Aqueous rinsates were prepared for mercury and PFAS, and hexane rinsates were prepared for PCBs. Paired rinsate and solvent blank samples were analyzed for mercury and PCBs by

subcontract laboratories under the control of Tetra Tech. ALS (Kelso, WA) analyzed the rinsate and solvent blank samples for mercury using EPA Method 245.1 (USEPA 1983), while ALS (Burlington, Ontario) analyzed the rinsate and solvent blank samples for PCBs using EPA Method 1668C. Results for mercury were reported in micrograms per liter (µg/L), and PCBs were reported in picograms per milliliter (pg/mL), which is equivalent to nanograms per liter (ng/L).

Tetra Tech stored the aqueous rinsate and solvent blank samples for PFAS analyses until EPA obtained the funding for the tissue analysis laboratories. PFAS rinsate and solvent blank samples were analyzed by AXYS Analytical Services (Sydney, BC, Canada) at the same time as the analyses of the fish fillet tissue samples, but with an extraction step based on EPA Method 537 from the Office of Ground Water and Drinking Water (USEPA 2009). Results for PFAS were reported in ng/L.

Section 3.6 Quality Control Procedures

Fish Tissue Analyses

The analytical procedures applied by the laboratories designated for analysis of 2018-19 NRSA fish tissue samples included many of the traditional EPA analytical quality control activities. For example, all samples were analyzed in batches and each batch included:

- up to 20 samples, including both field samples and QC samples
- blanks – 5% of the samples within a batch are method blanks

Other quality control activities for fish tissue samples varied by the analysis type, as described in Table 1.

Table 1. Quality Control Activities for Analysis of Fish Tissue Samples

Analyte Type	Quality Control Sample	Frequency
Mercury	Bubbler blank	3 blanks run during calibration and with each analytical batch of up to 20 field samples
	Method blank	3 method blanks per batch of up to 20 field samples, with analyses interspersed among the samples in the analysis batch
	Laboratory control sample	Once per batch of up to 20 field samples, prior to the analysis of any field samples, and again at the end of each analytical batch, spiked at 4.0 ng
	Matrix spike and matrix spike duplicate samples	Once per every 10 field samples (e.g., twice per 20 samples in a preparation batch)
PCBs	Method blank	One per sample batch
	Laboratory control sample	One per sample batch
	Duplicate sample	One per sample batch
	Labeled compounds	Spiked into every field sample and QC sample
PFAS	Method blank	One per sample batch
	Laboratory control sample	One per sample batch
	Duplicate sample	One pair per sample batch
	Labeled compounds	Spiked into every field and QC sample

Rinsate and Solvent Blank Analyses

The quality control activities associated with the rinsate and solvent blank analyses were generally similar to those for the tissue analyses, with the following exceptions. First, the rinsate analyses for mercury and PCBs were prepared and analyzed individually, not in batches of up to 20, in order to provide timely feedback on the cleanliness of the homogenization equipment. The rinsates for PFAS were held for later analyses and therefore were grouped together in batches, each with its own associated QC activities.

Secondly, because the rinsates for PCBs were prepared in solvent, there were no sample extraction procedures required, so the typical QC procedures relevant to the sample extraction procedure were modified. The common quality control activities for rinsate samples are described in Table 2.

Table 2. Quality Control Activities for Analysis of Rinsates

Analyte Type	Quality Control Sample	Frequency
Mercury	Instrument blank	With each rinsate sample
	Laboratory control sample	With each rinsate sample
PCBs	Instrument blank	With each rinsate sample
	Labeled compounds	Added to every rinsate sample
PFAS	Method blank	With each batch of rinsate samples
	Laboratory control sample	With each batch of rinsate samples
	Labeled compound recovery	Every rinsate sample

Because the mercury rinsates and the PFAS rinsates were prepared in reagent water, there was little chance of a "matrix effect" and the laboratory control sample, which was also prepared in reagent water, provided sufficient information on the performance of the method and the laboratory, so a separate matrix spike sample was not required.

Because the rinsates for PCBs were prepared from hexane and no sample extraction was required, "matrix effects" were not possible. Therefore, matrix spike and duplicate samples were not required for the rinsate samples.

GDIT reviewed the results for the mercury and PCB rinsates as soon as they were available from Tetra Tech and its subcontracted laboratories and relayed the review findings to EPA within hours of receipt of the results. Mercury was never detected above the laboratory's MDL in any of the rinsate or aqueous (solvent) blank samples from the study. However, in making its assessments of the rinsate results, GDIT took a conservative approach and assumed that mercury could be present in the rinsate sample at exactly the MDL. Based on this assumption, GDIT calculated the total mass of mercury that theoretically might be transferred to the smallest bulk homogenized tissue sample in the sample batch (due to inadequate cleaning of the homogenization equipment). That "worst case" estimate was then compared to the MDL for mercury in tissues and was always at least 6 times lower than the tissue sample MDL. Therefore, in no instance was there any risk that the mercury reported in the fish tissue samples was the result of inadequate equipment cleaning, and EPA authorized Tetra Tech to continue processing fish samples.

A similar review approach was utilized for the PCB rinsates and solvent blanks. Because the PCB rinsates and blanks were analyzed using the very sensitive procedures in EPA Method 1668C (USEPA 2010a), each of the 10 of the PCB congeners of interest was detected sporadically among the 15 pairs of rinsates and solvent blanks. The amounts reported in the rinsates and solvent blanks generally were hundreds to thousands of times below the concentration that might be detected in a tissue sample.

The PFAS rinsate and solvent blank samples were analyzed after the end of the preparation of all of the fish samples and thus were not used to determine if Tetra Tech could proceed with preparing additional batches of fish. The only PFAS detected in any of the rinsates and solvent blanks was 6:2 FTS, which was reported in 1 of the 15 of the solvent blanks, but not in the associated rinsate sample. Therefore, in no instance was there any risk that the PFAS results reported in the fish tissue samples were the result of inadequate equipment cleaning.

Overall, the rinsate results demonstrate that the equipment cleaning procedures employed for the study were more than adequate to ensure that cross contamination between tissue samples was not occurring during processing.

Chapter 4
Data Quality Assessment

Section 4.1 Data Review

All the data from the study were subjected to two levels of review. First, all laboratory results and calculations were reviewed by the respective laboratory manager for that analysis prior to submission. Any errors identified during this peer review were returned to the analyst for correction prior to submission of the data package. Following correction of the errors, the laboratory manager verified that the final package was complete and compliant with the contract, and then signed each data submission to certify that the package was reviewed and determined to be in compliance with the terms and conditions of the GDIT subcontract.

For the second level of review, GDIT data reviewers examined the results for each field-based tissue sample and the available quality control data to assess and document the quality of the data relative to the objectives of the study. Each data package was thoroughly reviewed by GDIT to ensure the following:

- All samples were analyzed, and results were provided for each sample analyzed, including results for any dilutions and re-analyses, and for all associated QC samples.
- All required QC samples were analyzed, and these QC samples met specified acceptance criteria.
- Data reporting forms and/or electronically formatted data were provided for each of the field-based tissue samples and/or associated QC analyses.
- Raw data associated with each field-based tissue sample and QC sample were provided with each data package, and the instrument output (peak height, area, or other signal intensity) was traceable from the raw data to the final result reported.
- Any problems encountered and corrective actions taken were clearly documented.

When anomalies were identified, GDIT contacted the laboratory and asked them to provide the missing data, clarifications, and/or explanations so that a comprehensive data review could be performed to verify the quality of their results.

GDIT developed a database to capture results for each sample and entered results of the data reviews directly in the database through the application of standardized data qualifier flags and descriptive comments concerning the reliability of the flagged results. Table 3 contains the individual data qualifiers that were applied to results from the study and provides an explanation of the implications of each qualifier for the use of the data.

Note: *The presence of data qualifiers is not intended to suggest that data are not useable; rather, the qualifiers are intended to caution the user about an aspect of the data that does not meet the acceptance criteria established in the project QAPP.*

Table 3. Individual SCC Codes Applied to the 2018-19 NRSA Results		
SCC Code	**Comments**	**Implication**
B, RMAX	Blank Contamination, Result is a Maximum Value	Blank contamination was observed, and the target analyte was reported in the sample at a concentration between 5 and 10 times higher than the blank value. The result was considered to be of acceptable quality, but data users are cautioned that it may be a maximum value due to possible influence of contamination.
B, RNAF	Blank Contamination, Result is Not Affected	Blank contamination was present but was not considered to adversely impact the sample result. The presence of the analyte in the blank is not considered to adversely affect the data in cases where the sample results are more than 10 times the associated blank results or where the analyte is not detected in associated samples.
B, RNON	Blank Contamination, Result Reported as Non-detect	When the sample result is less than five times the blank result, there are no means by which to ascertain whether or not the presence of the analyte may be attributed to contamination. Therefore, the result is reported in the database as a non-detect at the MDL, adjusted for sample size and dilution.
HLBL, J	High Labeled Compound Recovery, Estimated	The labeled analog of the target analyte was recovered above acceptance criteria, suggesting the possible presence of matrix interferences. Isolated instances of high recovery are not uncommon, and patterns across multiple samples are more of a concern.
HLBL, RNAF	High Labeled Compound Recovery, Result is Not Affected	The labeled analog of the target analyte was recovered above acceptance criteria, suggesting the possible presence of matrix interferences. Isolated instances of high recovery are not uncommon, and patterns across multiple samples are more of a concern. If the analyte was not detected in a field sample, there is no concern and the RNAF is added to the HLBL flag.
HLCS	High Lab Control Sample Recovery	The lab control sample (LCS) was a clean reference matrix. If recovery in the LCS was high, there may be a high bias for that analyte.
HLCS, RNAF	High Lab Control Sample Recovery, Result is Not Affected	The recovery in the LCS was high, but the analyte was not detected in the associated tissue sample, so there was no high bias concern and the RNAF flag was applied.
HRPD, J	High RPD, Estimated	The relative percent difference (RPD) between the results in the parent sample and the laboratory duplicate is above the acceptance limit. This may be due to inhomogeneity in the bulk sample or analytical variability. When high RPD was observed for an analyte, all the detected results for that analyte in any of the samples in the batch with the duplicate sample were qualified as estimated values.
HRPD, RNAF	High RPD, Result is Not Affected	The relative percent difference (RPD) between the results in the parent sample and the laboratory duplicate is above the acceptance limit. This may be due to inhomogeneity in the bulk sample or analytical variability. However, when high RPD was observed for an analyte, the non-detected results for that analyte were not affected, and the RNAF flag was applied.
HVER, J	High CALVER, Estimated	The results for the calibration verification associated with the analyte were above the acceptance limit, suggesting a possible high bias. Detected analytes also are considered estimated values.
J	Estimated	When applied alone, this code indicates that the result is at or above the MDL, but below the QL. This flag also may be applied in conjunction with other flags to indicate the potential for greater uncertainty.

Table 3. Individual SCC Codes Applied to the 2018-19 NRSA Results		
SCC Code	**Comments**	**Implication**
LLBL	Low Labeled Compound Recovery	The labeled analog of the target analyte was recovered below acceptance criteria, suggesting the possible presence of matrix interferences or incomplete recovery of both the labeled compound and target analyte during the extract cleanup processes used in the analytical procedure. The use of isotope dilution quantitation automatically corrects the results for the target analyte. (Non-detects)
LLBL, J	Low Labeled Compound Recovery, Result is an Estimate	The labeled analog of the target analyte was recovered below acceptance criteria, suggesting the possible presence of matrix interferences or incomplete recovery of both the labeled compound and target analyte during the extract cleanup processes used in the analytical procedure. The use of isotope dilution quantitation automatically corrects the results for the target analytes. For detects, results considered an estimate.
LLCS	Low LCS result	The lab control sample (LCS) was a clean reference matrix. If recovery in the LCS was low, there may be a low bias for that analyte. When low LCS recovery was observed for an analyte, the results for that analyte were qualified in all of the samples in the batch with the LCS.
LMI, J	Lock-Mass Interference, Result Estimated	There was an interference with the lock ion mass. The detected result is considered an estimated value.
LMI, RNAF	Lock-Mass Interference, Result Not Affected	There was an interference with the lock ion mass. The associated target compound was not detected; therefore, the result is not affected.
LND, NQ	Labeled compound Not Detected, Not Quantitated	The labeled compound associated with the target compound was not detected; therefore, the target compound could not be quantitated.
NDP, RNON	No definitive peak shoulder for co-eluter, Result Reported as Non-detect	The coeluting peak did not have a definitive peak shoulder. The result was reported as a non-detect.
RRT, J	RRT outside of window, Estimated	Relative retention time is outside the acceptable range, the result is considered an estimated value.
RTI, RMAX	Retention Time Interference, Result is a Maximum Value	There was an interference within the target compound retention time. The result is considered a maximum value.

Section 4.2 Analysis of Blanks

Blanks are used to verify the absence of contamination that may occur at any point in the measurement process. The data reviewers evaluated each sample result in comparison to the result for that analyte in the method blank prepared in the same extraction batch. For those analytes reported as present in the method blank, the data reviewers applied the 5x and 10x rules (described in the first three SCC codes of Table 3) to determine the potential impact of the blank contamination on the study results. The impacts of blank contamination are discussed separately for each analyte class in Sections 4.2.1 to 4.2.3.

4.2.1 Blanks for Mercury Analysis

Mercury was never detected above the QC acceptance limit of 0.4 nanograms (ng) in any of the three method blanks associated with each batch of samples. Therefore, no method blank qualifiers were applied to the mercury results for the study.

4.2.2 Blanks for PCB Analysis

As shown in Figure 3, more than 99% of the PCB results were not affected by blank contamination, either because the analyte was not detected in the blank (93.93%) or because the concentration in the sample was more than 10 times the level observed in the blank (4.81%). For 0.35% of the results, the data reviewers judged that the sample result is likely a maximum value (RMAX) because there is some chance that the sample result was inflated by the background contamination from the laboratory that is evident in the blank. Only 0.91% of the PCB results were changed to non-detects (RNON) due to concerns about blank contamination.

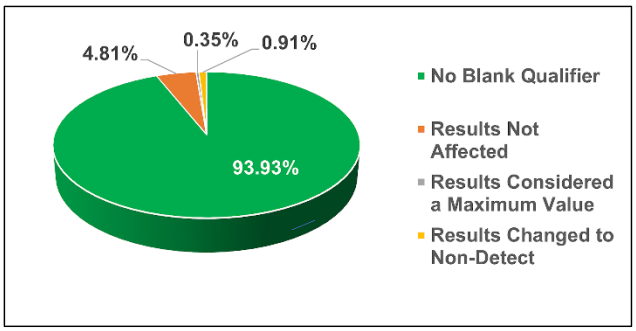

Figure 3. Impacts of Blank Contamination on the PCB Results

4.2.3 Blanks for PFAS Analysis

Overall, there were few data quality issues with the blanks from the PFAS analyses. As shown in Figure 4, more than 99% of the PFAS results were not affected by blank contamination, either because the analyte was not detected in the blank (99.33%) or because the concentration in the sample was more than 10 times the level observed in the blank (0.15%). For 0.05% of the results, the data reviewers judged that the sample result is likely a maximum value (RMAX) because there is some chance that the sample result was inflated by the background contamination from the laboratory that is evident in the blank. The remaining 0.47% of the PFAS results were changed to non-detects (RNON) because of concerns about blank contamination. Given that such small percentage of results were affected by blank contamination, a pie chart has not been included in this section because the tiny sliver of affected results would not be visible.

Section 4.3 Analysis of Laboratory Control Samples

A laboratory control sample (LCS) is a mass or volume of a clean reference matrix into which the laboratory spikes the analytes of interest. In some EPA methods, LCS is also known as the ongoing precision and recovery (OPR) sample. The laboratory analyzes the LCS or OPR using the same sample preparation and analysis techniques that are applied to the field samples and compares the results to method- or project-specific acceptance criteria to demonstrate that the laboratory can perform the analysis acceptably in the absence of matrix-specific interferences.

The QAPP for the 2018-19 NRSA study required that each laboratory performing analyses of fish tissue samples prepared and analyzed one LCS for each batch of 20 or less field samples. The impacts of LCS results are discussed separately for each analyte class in Sections 4.3.1 to 4.3.3.

4.3.1 Mercury LCS Results

The LCS results associated with each batch of samples analyzed for mercury met the QC acceptance limit. Therefore, no LCS qualifiers were applied to the mercury results for the study.

4.3.2 PCB LCS Results

The LCS results associated with each batch of samples analyzed for PCBs met the QC acceptance limit. Therefore, no LCS qualifiers were applied to the PCB results for the study.

4.3.3 PFAS LCS Results

There were a few data quality issues with the LCS results for the PFAS analyses. However, as shown in Figure 5, 96.31% of the PFAS results not affected by LCS issues. Another 1.96% were qualified due to high LCS results; however, the compounds were not detected in the associated samples and therefore those results were not affected. A total of 1.72% of the PFAS results were qualified because of a low LCS result that might reflect a low bias in the results, while 0.10% of the results were qualified due to a high LCS result that might reflect a high bias in the results.

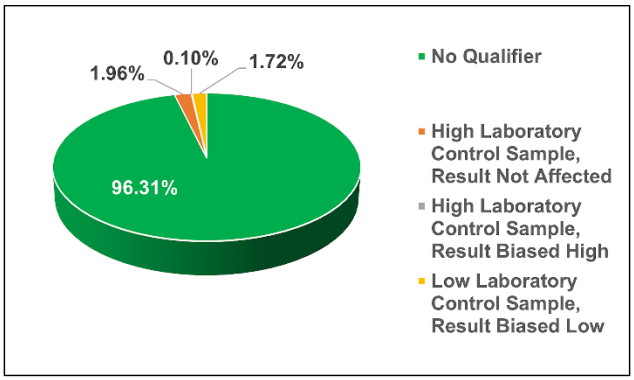

Figure 4. Impacts of LCS Recoveries on the PFAS Results

Section 4.4 Analysis of Matrix Spike and Laboratory Duplicate Samples

A matrix spike sample (MS) is a mass or volume of a field sample into which the laboratory spikes the analytes of interest. The laboratory analyzes the MS using the same sample preparation and analysis techniques that are applied to the field samples and compares the results to method- or project-specific acceptance criteria to provide information on the effects of the sample matrix on method performance.

A laboratory duplicate sample is a second aliquot of one field sample that is prepared and analyzed to provide information on the precision of the analytical method. Laboratory duplicate samples are routinely used for analytes such as metals that are expected to be found in most or all samples. However, other types of analytes, particularly organic contaminants, are not detected as frequently in field samples, and the analysis of an unspiked duplicate sample often will not yield useful data on analytical precision when both the original sample and the duplicate are reported as "not detected." Therefore, EPA methods for organic contaminants often require that a second spiked aliquot of the sample matrix be prepared as a matrix spike duplicate (MSD). By spiking the analytes into both, the MS and MSD aliquots, there is a greater chance of generating useful data on method and laboratory precision.

Alternatively, some EPA methods, such as the one used for the PCBs, and the procedure used for the PFAS, call for spiking labeled compounds into every sample and the results for those labeled compounds provide sample-specific data on method performance, as opposed to the batch-specific data generated from one MS/MSD pair per batch. For these methods, only a laboratory duplicate sample per batch is analyzed. A duplicate sample is a second aliquot of a field sample that is prepared and analyzed to provide information on the precision of the analytical method by comparing the results of the original analysis of the sample and the analysis of the laboratory duplicate sample.

The analytical QAPP for the study (USEPA 2019a) required that the laboratories performing analyses of fish tissue samples prepare and analyze MS/MSD or duplicate samples with each batch of field samples as follows.

Table 4. Matrix Spike and Duplicate Sample Requirements by Analysis Type		
Analysis Type	**MS/MSD**	**Duplicate**
Mercury	X	
PCBs		X
PFAS		X

The data reviewers evaluated the results for each MS/MSD and laboratory duplicate sample. The impacts are discussed separately for each analyte class in Sections 4.4.1 to 4.4.3.

4.4.1 Mercury Matrix Spike and Matrix Spike Duplicate Sample Results

The matrix spike and matrix spike duplicate sample results associated with each batch of samples analyzed for mercury met the QC acceptance limit. Therefore, no data qualifiers for recovery or precision were applied to the mercury results for the study.

4.4.2 PCB Duplicate Sample Results

The laboratory duplicate sample results associated with each batch of samples analyzed for PCBs met the QC acceptance limit. Therefore, no duplicate sample qualifiers were applied to the PCB results for the study.

4.4.3 PFAS Duplicate Sample Results

As shown in Figure 6, the PFAS laboratory duplicate analyses exhibited excellent precision, with 99.78% of the PFAS results not affected by duplicate issues, with 0.33% of those results where the associated target compound was not detected. Only 0.22% of results were qualified due to high RPD values, with the results considered estimates.

Section 4.5 Labeled Compounds

The methods for PCBs and PFAS use analogs of the target analytes that contain a stable (nonradioactive) isotope of one or more of the atoms that make up the contaminant. These compounds are referred to as "labeled compounds" and often incorporate multiple atoms of naturally occurring, but less common isotopes such as ^{13}C, ^{18}O, or ^{37}Cl. For example, because ^{13}C makes up 1.1% of the carbon in nature, some PCBs in the environment may contain a single occurrence of ^{13}C among the 12 carbon atoms that make up the basic PCB structure. However, if the labeled compound is synthesized with all 12 atoms of the more common isotope ^{12}C replaced by ^{13}C, there is virtually no chance that the $^{13}C_{12}$-labeled compound will be present in an environmental sample. Therefore, the labeled compound is ideally suited for use as a quantitation reference standard during the analysis of PCBs.

The labeled compounds in such methods serve two functions. First, their responses can be used to quantify the responses for the unlabeled target analytes in each sample through the technique known as isotope dilution. Secondly, the measured recovery of each labeled compound provides information about the overall extraction and analysis process applied to each sample. Other labeled compounds are often added to each sample extract before any cleanup steps to provide information on the performance of those cleanups as well.

The PCB laboratory added known amounts of 32 ^{13}C-labeled PCBs to each sample before extraction. The PFAS laboratory added known amounts of 18 ^{13}C-labeled PFAS and six deuterium-labeled PFAS standards to each sample before extraction. The QAPP for the study includes acceptance criteria for the

recoveries of the various labeled compounds. The impacts of the labeled compound results are discussed separately for each analyte class in Sections 4.5.1 and 4.5.2.

No labeled compounds were employed for the mercury analyses.

4.5.1 PCB Labeled Compound Recoveries

Virtually all (99.61%) of the labeled compound recoveries for the PCB samples met the QC acceptance limits. Of the 0.39% of the results that were affected by low labeled compound recoveries, 0.13% did not have the associated target compounds detected and therefore were technically not affected, while 0.22% of the results were detected and were considered estimates. There were also 17 instances (0.037%) in which the labeled compounds were not detected due to matrix interferences; therefore, the target congeners associated with these labeled compounds could not be quantified. A pie chart has not been included in this section because the tiny sliver of affected results would not be visible.

4.5.2 PFAS Labeled Compound Recoveries

Overall, 96% of the labeled compound recoveries for the PFAS samples met the QC acceptance limits. As shown in Figure 7, 1.89% of the results were affected by high labeled compound recoveries. Of those, 1.86% did not have the target analytes associated with those labeled compounds detected in the samples and therefore, the results were not affected, while 0.031% of the results had the associated target compounds detected and those results were considered estimated values. Approximately 2.08% of the results were affected by low labeled compound recoveries. Of those, 0.91% did not have the target analytes associated with those labeled compounds detected in the samples and therefore the results were not affected, while 1.17% of the results had the associated target compound detected and those results were considered estimated values. A total of 0.031% of the results (17 instances) had an issue where the labeled compound was not detected; therefore, the associated target compounds could not be quantified.

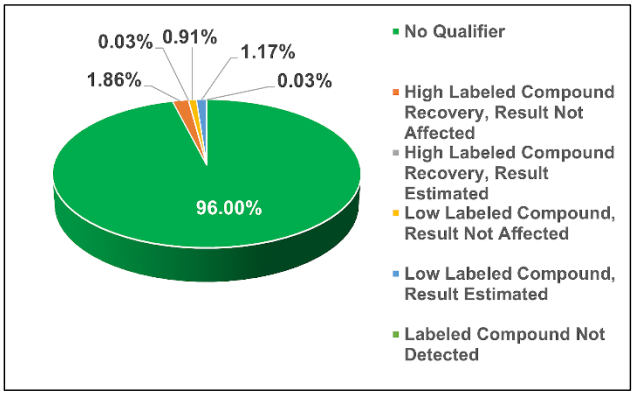

Figure 5. Impacts of Labeled Compound Recovery on the PFAS Results

Section 4.6 Ion Abundance Ratios

The instruments used for PCBs and PFAS analyses monitor the signals from two ions produced for each analyte. The resolution of the mass spectrometer is sufficient to distinguish ions that differ in mass by a few ten-thousandths of an atomic mass unit. The ratio of the abundances of these two ions is used as one of four criteria to identify the analyte. The methods include QC acceptance criteria for the ion abundance ratio (IAR) for each target analyte that are based on the theoretical occurrence of each of the component atoms in nature, plus and minus some percentage (e.g., ± 15%).

In some cases, the observed IAR may fall outside of the consensus-based acceptance limit. That does not mean that the analyte is not present, but it suggests that there may be some contribution to the response from an ion with a very similar mass produced by an interference. A higher-than-expected IAR suggests an interference with the ion in the pair for the target analyte with the smaller mass, while a lower-than-expected IAR suggests an interference with the ion in the pair for the target analyte with the larger mass.

When the exceedance from the acceptance limit is small (e.g., a few percent), the method for PCB allows the analyst to report the results in such instances with a qualifier flag that alerts the data user to the situation.

During the data review process, any results reported with an IAR issue are reviewed in more depth. If all other identification criteria in the method are met, the results are reported for the analyte with the appropriate qualifier flag. The impacts of the IAR are discussed separately for each analyte class in Sections 4.6.1 and 4.6.2.

4.6.1 PCB Ion Abundance Ratios

Overall, 99.14% of the PCB results were not qualified because of ion abundance ratio concerns. As shown in Figure 8, higher-than-expected IARs were observed on 0.75% of the results while lower-than-expected IARs were observed in 0.11% of the results. The slightly higher distribution of high ion ratios suggests that the interferences being extracted from the fish tissue for the affected samples systematically influenced the numerical results in one direction more than the other.

4.6.2 PFAS Ion Abundance Ratios

The IARs for sample results associated with each batch of samples analyzed for PFAS met the acceptance limit. Therefore, no IAR qualifiers were applied to the PFAS results for the study.

Section 4.7 Other QC parameters

As evidenced by the list of individual SCC data qualifier codes in Table 3, the data review effort identified instances where the calibration verifications for the PCB analyses did not always meet the acceptance criteria (see Table 3). However, the frequencies were very low, with only 0.026% of the PCB calibration verification results falling outside of the acceptance criteria. Given these very low occurrences, a pie chart has not been included in this section because the tiny slivers of affected results would not be visible.

Section 4.8 Completeness

Completeness is a measure of the amount of data that are collected and deemed to be acceptable for use the intended purpose. The completeness goal established in the QAPP in this study is to obtain valid measurements from 95% of the samples analyzed. For multi-analyte methodologies, analytical completeness is best calculated based on the number of possible sample/analyte combinations. Otherwise, a problem with a single analyte could be seen as invalidating an entire field sample.

Combining the number of target analytes for the three types of analyses (mercury, PCBs, and PFAS) yields a total of 193 measured results for each sample (based on 159 results that cover all 209 PCB congeners). For the 290 field samples analyzed, the total number of sample/analyte combinations would be 55,970.

Despite the data quality concerns outlined in this report, all the available and intended samples were successfully analyzed for all the target analytes. Following an intensive review of the project data, none of the results were excluded from consideration based on data quality concerns. Therefore, analytical completeness is 100%, and OST met its completeness goal.

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