Quality Assurance Project Plan for

Analysis of the National Coastal Condition Assessment 2020 Great Lakes Fish Fillet Samples for Mercury, Per- and Polyfluoroalkyl Substances (PFAS), Polychlorinated Biphenyl (PCB) Congeners, Aroclors, and Fatty Acids

Revision 4

November 19, 2021

Prepared by:

United States Environmental Protection Agency
Office of Water
Office of Science and Technology
Standards and Health Protection Division

Prepared with support from:

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EPA-822-B-24-004

Revision History

November 19, 2021 Revision 4

This revision includes edits to describe the procedures for omega-3 and omega-6 fatty acid analyses of National Coastal Condition Assessment 2020 Great Lakes fillet samples and add them to the mercury, PFAS, PCB congener, and Aroclor analyses that are currently underway. A more detailed list of edits follows below:

- The revision history was updated.
- Section A was updated.
- The fatty acid analysis laboratory was added to Section A3 and to Figure 1.
- Section A7 was updated to refer to the fatty acid QC criteria.
- The details of the analytical procedure for fatty acids and the QC criteria were added in Sections B4.5 and B5.5.
- Section B7 was updated to include the calibration information for fatty acid analysis.
- Sections C1, C1.1, C1.3, and C1.4 were updated to include the information for fatty acids.
- Sections D1.2, D1.3, and D3 were updated to include the fatty acid results in the list of analytical data that will be verified and validated, respectively.
- The Reference section was updated with the citation for the QA manual from the fatty acid analysis laboratory.
- Appendix B was updated to include the fatty acid MDLs and MLs from the laboratory selected for fatty acid analysis.

April 26, 2021 Revision 3

This revision includes edits to describe the procedures for Aroclor analyses of National Coastal Condition Assessment 2020 Great Lakes fillet samples, as planned for the future, and add them to the mercury, PFAS, and PCB congener analyses that are currently underway. A more detailed list of edits follows below:

- The revision history was updated.
- Section A was updated.
- The Aroclor analysis laboratory was added to Section A3 and to Figure 1.
- Section A6 was updated to include shipping fillet tissue samples from the 2020 GLHHFFTS for Aroclor analyses.
- Section A7 was updated to refer to the Aroclor QC criteria.
- The placeholder text for Aroclors in Sections B4.4 and B5.4 was replaced with the actual details.
- Section B7 was updated to include the calibration information for Aroclor analysis.
- Sections C1, C1.1, C1.3, and C1.4 were updated to include the information for Aroclors.
- Section D1.2 and D1.3 were updated to include the Aroclor results in the list of analytical data that will be verified and validated, respectively.
- The Reference section was updated with the citation for the QA manual from the Aroclor analysis laboratory and the citations for the Aroclor methods.
- Appendix B was updated to include the Aroclor MDLs and MLs from the laboratory selected for Aroclor analysis.

March 12, 2021 Revision 2

This revision includes edits to describe the procedures for PCB congener analyses of National Coastal Condition Assessment 2020 Great Lakes fillet samples, as planned for the future, and add them to the mercury and PFAS analyses that are currently underway. A more detailed list of edits follows below:

- The revision history was updated.
- Section A was updated.
- The PCB congener analysis laboratory was added to Section A3 and to Figure 1.
- Section A7 was updated to refer to the PCB congener QC criteria.
- The placeholder text for PCB congeners in Sections B4.3 and B5.3 was replaced with the actual details.
- Section B7 was updated to include the calibration information for PCB congener analysis.
- Sections C1, C1.1, C1.3, and C1.4 were updated to include the information for PCB congeners.
- The Reference section was updated with the citation for the QA manual from the PCB congener analysis laboratory and the citations for the PCB congener method.
- Appendix B was updated to include the PCB congener MDLs and MLs from the laboratory selected for PCB congener analysis.
- Appendix D, with the PCB congener QC acceptance criteria, was added.

February 26, 2021 Revision 1

This revision includes edits to describe the procedures for PFAS analyses of National Coastal Condition Assessment 2020 Great Lakes fillet samples and aqueous QC samples, as planned for the future, and add them to the mercury analyses that are currently underway. A more detailed list of edits follows below:

- The revision history was added.
- Section A was updated.
- The PFAS laboratory was added to Section A3 and to Figure 1.
- Section A7 was updated to refer to the PFAS QC criteria.
- The placeholder text for PFAS in Sections B4.2 and B5.2 was replaced with the actual details.
- Section B7 was updated to include the calibration information for PFAS.
- Sections C1, C1.1, C1.3, C1.4, and C2 were updated to include the information for PFAS.
- The Reference section was updated with the citation for the QA manual from the PFAS laboratory and the citations for the PFAS methods.
- Appendix B was updated to include the PFAS MDLs and MLs from the laboratory selected for these analyses.
- Appendix C was added to include the QC acceptance criteria for the PFAS analyses.

December 16, 2020 - Original QAPP (Revision 0) signed

Quality Assurance Project Plan for Analysis of the National Coastal Condition Assessment 2020 Great Lakes Fish Fillet Samples for Mercury, Per- and Polyfluoroalkyl Substances (PFAS), Polychlorinated Biphenyl (PCB) Congeners, Aroclors, and Fatty Acids

A. PROJECT MANAGEMENT

The U.S. Environmental Protection Agency's (EPA's) Office of Science and Technology (OST) within the Office of Water (OW) prepared this Quality Assurance Project Plan (QAPP) with support from CSRA under EPA Contract No. EP-C-17-024. It presents objectives, performance requirements, and acceptance criteria for the analyses of National Coastal Condition Assessment (NCCA) 2020 Great Lakes Human Health Fish Fillet Tissue Study (GLHHFFTS) and Office of Research and Development (ORD)-Duluth 2020 Great Lakes special study fillet samples for mercury (Revision 0), fillet samples and aqueous QC samples for per- and polyfluoroalkyl substances (PFAS) (Revision 1), fillet samples for the full complement of 209 polychlorinated biphenyl (PCB) congeners (Revision 2), fillet samples for PCBs as Aroclors (Revision 3), and fillet samples for omega-3 and omega-6 fatty acids (Revision 4).

This QAPP does not address fish sample preparation because OST developed a separate QAPP in July 2020 that presents objectives, procedures, performance requirements, and acceptance criteria for the preparation of fillet tissue samples from whole fish composite samples collected from designated Great Lakes nearshore sites for the 2020 GLHHFFTS and from Lake Michigan enhancement sites for the ORD-Duluth 2020 Great Lakes special study by field crews during the NCCA 2020 field sampling season (USEPA 2020a). (Note: Some field crews will need to continue the NCCA 2020 Great Lakes field sampling season in 2021 due to travel restrictions related to the coronavirus pandemic in 2020.) This QAPP also does not address fish sample collection because that information is included in separate documents (USEPA 2020a and USEPA 2020b) prepared by EPA's OW/Office of Wetlands, Oceans, and Watersheds (OWOW) with support from OST.

This QAPP was prepared in accordance with the most recent version of EPA QA/R-5, *EPA Requirements for Quality Assurance Project Plans* (USEPA 2001a), which was reissued in 2006. In accordance with EPA QA/R-5, this QAPP is a dynamic document that is subject to change as project activities progress. Changes to procedures in this QAPP must be reviewed by the OST Project Manager for the 2020 NCCA and by the EPA Standards and Health Protection Division (SHPD) Quality Assurance Coordinator to determine whether the changes will impact the technical and quality objectives of the project. If so, the QAPP will be revised accordingly, circulated for approval, and forwarded to all project participants listed in the QAPP distribution list (Section A3). Key project personnel and their roles and responsibilities are discussed in the QAPP section to follow (Section A4), and information on project background and description is provided in Sections A5 and A6, respectively.

A1. Approvals

Leanne Stahl

Leanne Stahl, OST Project Manager, EPA — November 19, 2021
Date

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Shari Barash, Chief, National Branch, EPA — Date

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Bill Kramer, SHPD QA Coordinator, EPA — Date

JOSEPH BEAMAN Digitally signed by JOSEPH BEAMAN Date: 2021.12.02 08:30:03 -05'00'

Joe Beaman, OST QA Officer, EPA — Date

Harry McCarty Digitally signed by Harry McCarty Date: 2021.12.02 10:03:22 -05'00'

Harry McCarty, CSRA Project Leader — Date

Yildiz Chambers_Velarde Digitally signed by Yildiz Chambers_Velarde Date: 2021.12.02 09:47:42 -05'00'

Yildiz Chambers-Velarde, CSRA Work Assignment Manager — Date

Marguerite E. Jones Digitally signed by Marguerite E. Jones Date: 2021.12.02 09:41:44 -05'00'

Marguerite Jones, CSRA QA Manager — Date

A2. Table of Contents

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APPENDICES

LIST OF ACRONYMS AND ABBREVIATIONS

CCV	Continuing calibration verification
EPA	Environmental Protection Agency
GLHHFFTS	Great Lakes Human Health Fish Fillet Tissue Study
HRGC	High resolution gas chromatography
HRMS	High resolution mass spectrometry
ID	Identification
LCS	Laboratory control sample (also known as an OPR)
MDL	Method detection limit
ML	Minimum level (also referred to as the quantitation limit)
MS	Matrix spike sample
MSD	Matrix spike duplicate sample
NCCA	National Coastal Condition Assessment
OPR	Ongoing precision and recovery sample
OST	Office of Science and Technology
OW	Office of Water
OWOW	Office of Wetlands, Oceans, and Watersheds
PCB	Polychlorinated biphenyl
PFAS	Per- and polyfluoroalkyl substances
QA	Quality assurance
QAPP	Quality Assurance Project Plan
QC	Quality control
QCS	Quality control sample
QSA	Quality system audit
RPD	Relative percent difference
RSD	Relative standard deviation
SHPD	Standards and Health Protection Division
SOP	Standard operating procedure
SPE	Solid-phase extraction
VER	Verification

A3. Distribution List

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A4. Project/Task Organization

This current study of contaminants in Great Lakes fish is referred to as the 2020 Great Lakes Human Health Fish Fillet Tissue Study (GLHHFFTS). The EPA project team for the 2020 GLHHFFTS consists of managers, scientists, and QA personnel in OST and the Great Lakes National Program Office (GLNPO), along with statisticians in the Pacific Ecological Systems Division within EPA's ORD Center for Public Health and Environmental Assessment (Corvallis, Oregon). The EPA project team receives scientific, technical, and logistical support from contractors at Tetra Tech and at CSRA, a General Dynamics Information Technology company. Tetra Tech provides primarily fisheries support (e.g., fish sampling and fish sample preparation) and CSRA provides analytical support for the project team.

Members of the project team technically and/or financially responsible for fish fillet sample analysis include the OST Project Manager and Work Assignment Contracting Officer Representative (WACOR), the OST Alternate WACOR (Alt-WACOR), the OST Quality Assurance (QA) Officer, the SHPD QA Coordinator, the GLNPO Project Manager, the GLNPO QA Manager, the CSRA Work Assignment Manager, the CSRA Project Leader, and the CSRA QA Manager who collectively provide scientific, technical, logistical, and quality control (QC) support for the study. The project team organization provides the framework for conducting fish sample analysis to meet study objectives. The organization structure and function also facilitate project performance and adherence to QC procedures and QA requirements. The project organizational chart is presented in Figure 1. It identifies individuals serving in key roles and the relationships and lines of communication among these project team members. Responsibilities for key members of the project team are described below.

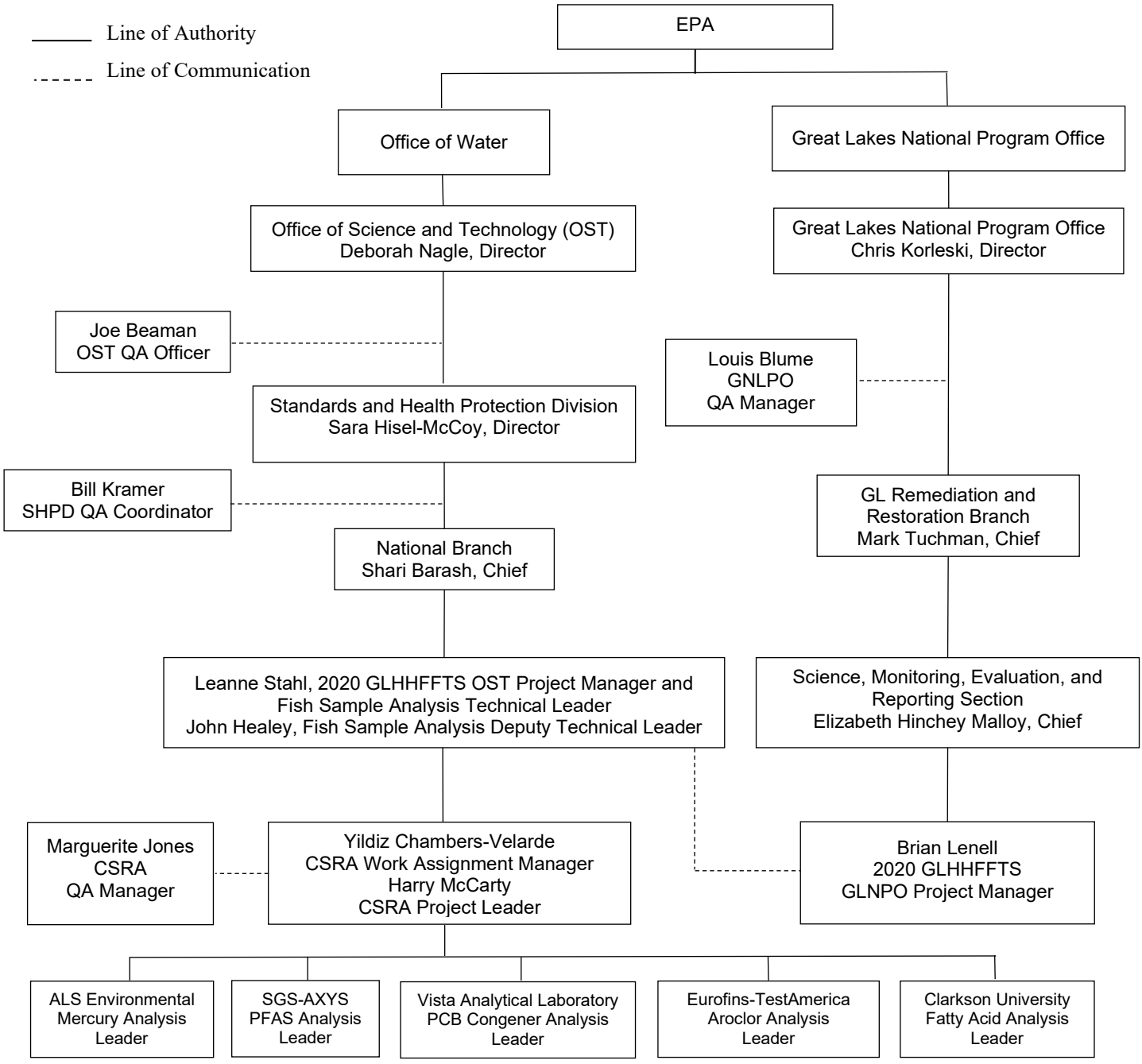

Figure 1. 2020 GLHHFFTS project team organizations from fish fillet sample analyses

Leanne Stahl of OST is the **OST Project Manager** who is providing overall direction for planning and implementation of the 2020 GLHHFFTS being conducted under the NCCA. She is also serving as the **Fish Sample Analysis Technical Leader** to provide technical and work assignment management support for 2020 Great Lakes fish fillet sample analysis and related analytical activities. Both roles involve the following 2020 GLHHFFTS responsibilities:

- developing technical information for whole fish sample collection for fillet analysis that includes preparation of the fish sampling protocols and coordination with the NCCA Project Leader in OWOW to integrate field sampling technical information for the 2020 GLHHFFTS into NCCA documents and training materials (this technical information also applies to the ORD-Duluth 2020 Great Lakes special study)
- providing technical support to conduct training on the 2020 GLHHFFTS field sampling requirements in coordination with the NCCA Project Leader in OWOW (this training also applies to the ORD-Duluth 2020 Great Lakes special study)
- developing the fish sample preparation procedures and requirements in coordination with John Healey who serves additionally as the 2020 GLHHFFTS Fish Sample Preparation Technical Leader as described in the Quality Assurance Project Plan for National Coastal Condition Assessment (NCCA) 2020 Great Lakes Human Health Fish Sample Preparation (USEPA 2020c). (These fish sample preparation procedures and requirements also apply to the ORD-Duluth 2020 Great Lakes special study.)
- managing analysis of 2020 Great Lakes fish fillet samples for target chemicals and related analytical support activities, including developing and managing a work assignment to provide CSRA support for analyzing the 2020 Great Lakes fillet samples, directing development of the initial NCCA 2020 Great Lakes fillet sample analysis QAPP and subsequent QAPP revisions, providing for QA review of the analytical results, developing the data files for statistical analysis of the data, reviewing and approving the final analytical QA report, and providing oversight for development of separate databases to store 2020 GLHHFFTS and ORD-Duluth 2020 Great Lakes special study fillet sample analysis results (this series of fillet sample analysis and related analytical activities also applies to the ORD-Duluth 2020 Great Lakes special study)
- facilitating communication among 2020 GLHHFFTS project team members and coordinating with all of these individuals to ensure technical quality and adherence to QA/QC requirements (this responsibility for communicating and coordinating with project team members also applies to the ORD-Duluth 2020 Great Lakes special study)
- developing and managing other work assignments and/or task orders under OST or other EPA contracts to provide technical support for the 2020 GLHHFFTS, providing oversight of contractor activities, and reviewing and approving study deliverables for each work assignment and task order (contractor support for 2020 Great Lakes human health fish sample collection and analysis activities also applies to the ORD-Duluth 2020 Great Lakes special study)
- scheduling and leading meetings and conference calls with 2020 GLHHFFTS project team members for planning study activities, reporting progress on study tasks, and discussing and resolving technical issues related to the study (this responsibility also applies to the ORD-Duluth 2020 Great Lakes special study)

- working with QA staff to identify corrective actions necessary to ensure that study quality objectives are met for both Great Lakes studies involving human health fish sample collection and analysis
- managing the development of and/or reviewing and approving all major work products associated with the 2020 GLHHFFTS and various other fish tissue studies, including products prepared by OWOW
- leading the Fish Tissue Study Team for reporting the 2020 GLHHFFTS human health fish fillet indicator results and various other fish tissue study results in technical journal articles and federal technical reports (this responsibility includes collaborating with the ORD-Duluth Great Lakes special study project team for reporting 2020 Great Lakes human health fish fillet analysis results)
- coordinating with John Healey (Task Order Contracting Officer Representative or TOCOR) to obtain Tetra Tech support through the task order for preparing fish study briefings and presentations and for providing general technical support; concurring on approval of task order deliverables
- presenting 2020 GLHHFFTS and other fish tissue study briefings for EPA managers and delivering fish tissue study presentations in various forums (e.g., scientific conferences, government meetings, and webinars)

John Healey of OST is serving as the **Fish Sample Analysis Deputy Technical Leader** to assist in providing technical and work assignment management support for 2020 Great Lakes fish fillet sample analysis and related analytical activities. He is also serving as the **Fish Sample Preparation Technical Leader**. Both roles involve the following 2020 GLHHFFTS responsibilities:

- providing support for development of the initial QAPP for analysis of the NCCA 2020 Great Lakes fillet samples and preparation of the subsequent QAPP revisions (this responsibility also applies to the ORD-Duluth 2020 Great Lakes special study)
- providing assistance in managing analysis of 2020 Great Lakes fish fillet samples for target chemicals and related analytical support activities, including assistance for developing and managing a work assignment to provide CSRA support for analyzing the 2020 Great Lakes fillet samples, participating in review of work assignment deliverables, and providing data management and analysis support (this series of fillet sample analysis and related analytical activities also applies to the ORD-Duluth 2020 Great Lakes special study)
- developing and managing a task order to provide technical support for preparation of NCCA 2020 Great Lakes human health fish fillet tissue samples for chemical analysis, which includes ensuring training for laboratory processing of Great Lakes human health fish samples, providing technical direction for and oversight of fish sample preparation activities (e.g., providing oversight for fish sample processing and analysis of fish sample preparation QC samples and single lipid samples), and reviewing and approving task order deliverables (e.g., fish sample preparation weekly progress reports and results for analysis of QC samples) with OST project Manager concurrence (this responsibility also applies to the ORD-Duluth 2020 Great Lakes special study)

- participating in developing, reviewing, and approving the NCCA 2020 Great Lakes Human Health Fish Sample Preparation QAPP (this responsibility also applies to the ORD-Duluth 2020 Great Lakes special study)
- developing and managing a task order to provide Tetra Tech support for preparing 2020 GLHHFFTS and other fish tissue study presentations and briefings and reviewing and approving task order deliverables with OST Project Manager concurrence and EPA management approval
- coordinating with OST QA staff and 2020 GLHHFFTS project team members to ensure technical quality and adherence to QA/QC requirements for task order deliverables
- obtaining training on the 2020 Great Lakes human health fish sampling requirements in coordination with the OST Project Manager
- participating in meetings and conference calls with Fish Tissue Study Team members for planning 2020 GLHHFFTS and other fish tissue study activities, reporting progress on various fish tissue study tasks, and discussing and resolving technical issues related to the 2020 GLHHFFTS and other fish tissue studies
- attending OWOW weekly NARS meetings and reporting information presented in the meeting (particularly information related to the NCCA and NRSA) to the OST Project Manager and SHPD managers
- managing the development of and/or reviewing and approving all major work products associated with the 2020 GLHHFFTS and various other fish tissue studies, including products prepared by OWOW
- providing support for collaborating with Fish Tissue Study Team members for reporting 2020 GLHHFFTS results and results for other fish tissue studies in technical journal articles and federal technical reports
- coordinating with the OST Project Manager to obtain CSRA support for preparing materials for fish tissue study briefings and presentations
- participating in presenting 2020 GLHHFFTS and other fish tissue study briefings for EPA managers and delivering fish tissue study presentations in various forums (e.g., scientific conferences, government meetings, and webinars)

Brian Lenell of GLNPO is the **2020 GLHHFFTS GLNPO Project Manager** who is providing support for planning and implementation of this regional Great Lakes study being conducted under the NCCA. This role involves the following responsibilities related to the 2020 GLHHFFTS:

- reviewing and concurring on technical information developed for 2020 GLHHFFTS fish sample collection
- participating in training on the 2020 Great Lakes human health fish sampling requirements in coordination with OST
- arranging additional support for 2020 GLHHFFTS fish sample collection through GLNPO fisheries contacts
- participating in the review of the fish sample preparation QAPP for the 2020 GLHHFFTS

- managing analysis of fish tissue samples for the fatty acids
- coordinating with 2020 GLHHFFTS project team members to ensure technical quality and adherence to QA/QC requirements
- participating in conference calls with project team members for planning study activities, reporting progress on study tasks, and discussing and resolving technical issues related to the study
- reviewing and concurring on all major work products associated with the 2020 GLHHFFTS
- collaborating with the 2020 GLHHFFTS project team for reporting the study results in technical journal articles and federal technical reports
- participating in preparing and/or reviewing fish tissue study presentations and presenting them in various forums (e.g., scientific conferences, government meetings, and webinars)

Joe Beaman is the **OST Quality Assurance Officer** who is responsible for reviewing and approving all QAPPs that involve scientific work being conducted by OST. Bill Kramer is the **Standards and Health Protection Division (SHPD) QA Coordinator** who is responsible for reviewing and recommending approval of all QAPPs that include scientific work being conducted by SHPD within OST. The OST QA Officer and SHPD QA Coordinator are also responsible for the following QA/QC activities:

- reviewing and approving this QAPP
- reviewing and evaluating the QA/QC requirements and data for all the 2020 GLHHFFTS activities and procedures
- conducting external performance and system audits of the procedures applied for all 2020 GLHHFFTS activities
- participating in Agency QA reviews of the study

Yildiz Chambers-Velarde is the **CSRA Work Assignment Manager** who is responsible for managing all aspects of the technical support being provided by CSRA staff for the 2020 GLHHFFTS fish fillet indicator. Her specific responsibilities include the following:

- monitoring the performance of CSRA staff participating in this study to ensure that they are following all the technical and QA procedures described in this QAPP that are related to CSRA tasks being performed to support this study
- ensuring completion of high-quality deliverables within established budgets and time schedules
- developing monthly progress and financial reports for support provided by CSRA
- participating in meetings and conference calls with project team members for planning study activities, reporting progress on study tasks, and discussing and resolving technical issues related to the study

Harry McCarty is the **CSRA Project Leader** who is primarily providing technical support for the 2020 GLHHFFTS fish fillet tissue indicator. His specific responsibilities include the following:

- providing direct technical support for the following 2020 GLHHFFTS fish fillet tissue indicator activities:
  - preparing information related to technical and quality assurance requirements for chemical analysis of homogenized fish fillet tissue samples for target analytes (e.g., mercury, PFAS, and PCB congeners, Aroclors, and fatty acids), verification and validation of analytical data (data quality review), and development of 2020 GLHHFFTS fish fillet indicator documents (including this QAPP) or characterization of this indicator in other 2020 GLHHFFTS documents
  - obtaining laboratory services to analyze 2020 Great Lake human health fish fillet tissue samples for target analytes (e.g., mercury, PFAS, and PCB congeners, Aroclors, and fatty acids), and providing technical and QA oversight of laboratory operations
  - completing review of the fillet tissue analytical data and developing the analytical data QA report
  - compiling fish fillet tissue analytical data files for statistical analysis and for public release
  - developing and maintaining separate project-specific databases for storing 2020 GLHHFFTS human health fish sample collection information from Great Lakes nearshore sites and fillet sample analysis data and for storing ORD-Duluth 2020 Great Lakes special study human health fish sample collection information from Lake Michigan enhancement sites and fillet sample analysis data, and initiating queries of these databases to respond to data requests from Agency and external users
  - preparing summary project information and graphics for development of project fact sheets, presentations, and other EPA meeting and outreach materials
  - supporting development of text and graphics for technical journal articles and final project reports for reporting 2020 GLHHFFTS data and data from other EPA fish tissue studies
  - obtaining freezer space that meets the requirements for long-term storage of archived fish tissue samples, organizing the archived fish tissue samples by project to facilitate retrieval of the samples, and developing and maintaining an inventory of the archived samples, as required
- participating in meetings and conference calls with project team members for planning study activities, reporting progress on study tasks, and discussing and resolving technical issues related to the study
- serving as the project team member providing technical expertise on any issues related to analytical chemistry and analytical methods for the 2020 GLHHFFTS and other EPA fish tissue studies

Marguerite Jones is the **CSRA QA Manager**, whose primary responsibilities include the following:

- approving this QAPP
- providing oversight for the implementation of QA procedures related to CSRA tasks that are described in this QAPP
- reporting deviations from this QAPP to the CSRA Project Leader and recommending corrective actions to resolve these deviations

A5. Problem Definition/Background

Obtaining statistically representative occurrence data on multiple contaminants in fish tissue is a priority area of interest for EPA. Since 2010, OST has collaborated with the Great Lakes National Program Office (GLNPO), Office of Wetlands, Oceans, and Watersheds (OWOW) within the Office of Water (OW), and with the Office of Research and Development (ORD) to conduct a series of regional-scale assessments of chemical contaminants in Great Lakes fish from nearshore areas as part of EPA's National Coastal Condition Assessment (NCCA). This current study of contaminants in Great Lakes fish is referred to as the 2020 Great Lakes Human Health Fish Fillet Tissue Study (GLHHFFTS). It is the third study of Great Lakes fish contamination conducted by OST and GLNPO under the NCCA. The two previous OST/ GLNPO studies of contaminants in Great Lakes fish were the 2010 Great Lakes Human Health Fish Tissue Study and the 2015 Great Lakes Human Health Fish Fillet Tissue Study.

Overall, the 2020 NCCA is a probability-based survey designed to assess the condition of coastal waters of the United States, which includes coastal waters of the Great Lakes. Building on EPA's experience from the 2010 NCCA and the 2015 NCCA, it includes collection and analysis of physical, chemical, and biological indicator data that will allow a statistically valid characterization of the condition of the Nation's coastal waters. EPA used an unequal probability design to select 725 estuarine sites along the coasts of the contiguous United States, 226 freshwater sites from U.S. nearshore areas throughout the Great Lakes, and 50 enhancement sites in Lake Michigan. OWOW within OW is responsible for managing the planning and implementation of the NCCA.

A6. Project/Task Description

OST and GLNPO began planning and mobilizing for the 2020 GLHHFFTS in 2019. An important new decision during the planning phase for the 2020 GLHHFFTS was to expand human health fish sample collection to the full set of 226 Great Lakes nearshore sites (45 sites per Great Lake except 46 sites in Lake Superior) randomly selected by ORD (Figure 2 and Appendix A). During the previous two Great Lakes human health fish tissue studies in 2010 and 2015, fish sample collection was limited to approximately 150 nearshore sites (about 30 sites per Great Lake). Mobilizing activities for the 2020 GLHHFFTS included updating fish sampling and handling protocols for the NCCA 2020 Field Sampling QAPP (USEPA 2020a) and the Field Operations Manual (USEPA 2020b), along with assembling and shipping human health fish sampling kits to the NCCA central supply distribution center in Traverse City, Michigan. During the mobilization phase, OWOW had to develop and implement a significantly different approach for NCCA 2020 field sampling training due to the coronavirus pandemic. Rather than

conducting a series of up to 14 onsite training workshops across the U.S., OWOW provided NCCA 2020 field sampling training through a series of virtual training workshops that began in late March and continued until late May 2020.

OST and GLNPO also coordinated with EPA scientists at the ORD facility in Duluth, Minnesota (abbreviated as ORD-Duluth) to add collection and analysis of human health fish samples from 50 enhancement sites in Lake Michigan as part of the ORD-Duluth 2020 Great Lakes special study. These enhancements sites include 38 island nearshore sites in northern Lake Michigan and 12 National Park nearshore sites in southern Lake Michigan (Figure 2 and Appendix A). Collection and preparation of human health whole fish samples from the Lake Michigan enhancement sites will involve procedures that are identical to the fish sample collection and preparation procedures for the 2020 GLHHFFTS.

The 2020 GLHHFFTS and ORD-Duluth 2020 Great Lakes special study involve the following key components:

- Collecting human health whole fish samples at 226 randomly selected Great Lakes nearshore sites and at 50 Lake Michigan enhancement sites (Appendix A) during 2020 and into 2021 because field crews were subject to travel restrictions in 2020 due to the coronavirus pandemic. Both types of sites have depths up to 30 meters or distances up to 5 kilometers from the shore.
- Obtaining one fish composite sample from each Great Lakes nearshore site and Lake Michigan enhancement site designated for human health fish sampling, which ideally consists of five similarly sized adult fish of the same species that are commonly consumed by humans.
- Shipping Great Lakes human health whole fish samples to freezers at Microbac Laboratories in Baltimore, MD for interim storage.
- Transferring the whole fish samples to the Tetra Tech facility in Owings Mills, MD for fish sample preparation.
- Preparing fillet tissue samples for chemical analysis by scaling and filleting each fish in the composite sample, homogenizing the fillets from all the fish in the composite sample, and dividing the fillet tissue into aliquots for various chemical analyses and for long-term storage of archived samples in a freezer.
- Shipping fillet tissue samples from both studies to laboratories contracted to analyze these samples for mercury, PFAS, PCB congeners, and lipids. (The fish sample preparation laboratory at the Tetra Tech facility in Owings Mills, Maryland is responsible for this activity in coordination with CSRA to conform to contract analytical laboratory fillet sample analysis schedules.)
- Shipping fillet tissue samples from the 2020 GLHHFFTS to laboratories contracted to analyze those samples for Aroclors and fatty acids, which are not part of the ORD-Duluth 2020 Great Lakes special study.
- Obtaining laboratory services to analyze 2020 Great Lakes fillet samples for target chemicals and monitoring analytical laboratory performance.

- Conducting data quality reviews for fish fillet tissue analytical and QC data and assigning data qualifiers when applicable.
- Developing project-specific databases (i.e., separate databases) for storage and retrieval of biological and analytical data generated during the 2020 GLHHFFTS and the ORD-Duluth 2020 Great Lakes special study.
- Compiling data files for each target chemical or group of related target chemicals for statistical analysis and for public release.
- Preparing summary project information and graphics for meeting materials, public outreach materials, and interim and final data reporting.

This QAPP focuses on fish fillet sample analyses activities for the 2020 GLHHFFTS and ORD-Duluth 2020 Great Lakes special study, which involve the last 5 study components listed above.

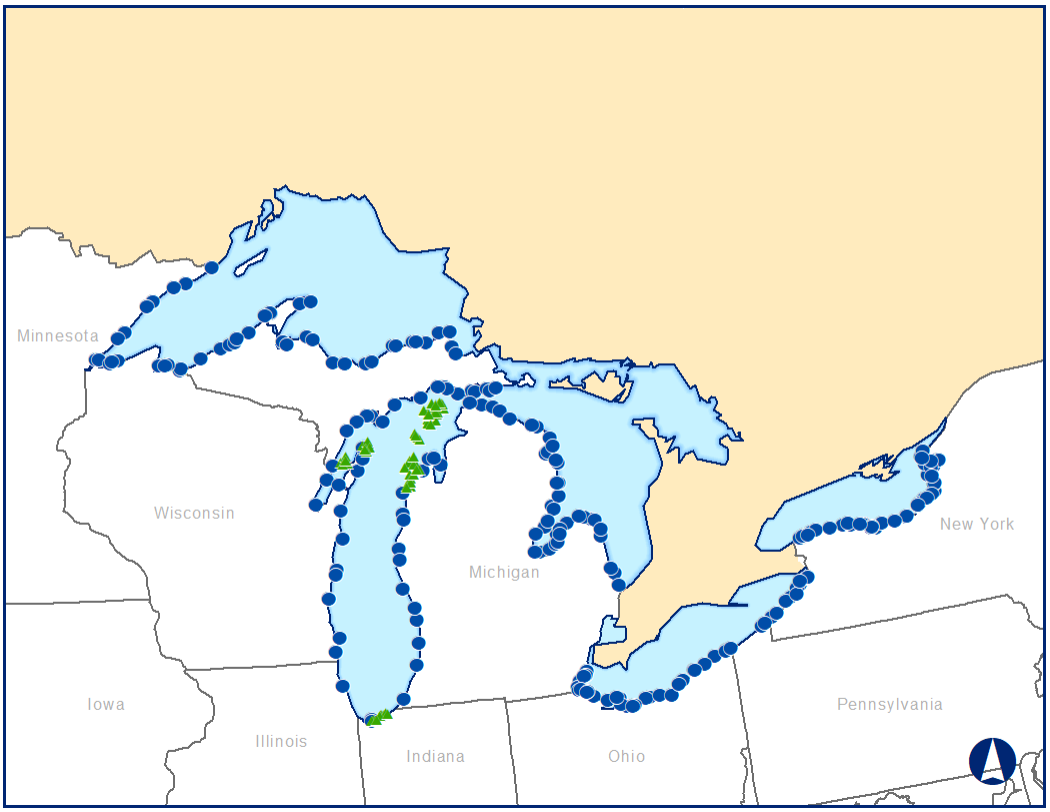

Figure 2. NCCA 2020 Great Lakes human health whole fish sampling locations (226 nearshore sites are blue dots and 50 Lake Michigan enhancement sites are green triangles)

A7. Quality Objectives and Criteria

The overall quality objective for the analysis of the 2020 GLHHFFTS and ORD-Duluth 2020 Great Lakes special study fish fillet tissue samples for mercury, PFAS, PCB congeners, PCBs as Aroclors, and fatty acids (2020 GLHHFFTS fillet samples only) is to obtain a complete set of data for each chemical or chemical group and to produce data of known and documented quality. Analytical completeness is defined as the percentage of valid samples collected in the study for which usable analytical results are produced. The goal for analytical completeness is 95% and it

is calculated at the sample-analyte level, such that an issue with the quality of one analyte out of many does not invalidate the entire sample.

OST specified the use of Method 1631E (USEPA 2002) and its quality control acceptance criteria for analyses of 2020 GLHHFFTS and ORD-Duluth 2020 Great Lakes special study fish fillet tissue samples for mercury. The information describing the analytical method is provided in Section B4 of this QAPP. Data usability for each analysis will be assessed using QC criteria summarized in Section B5.

Because EPA has not formally validated methods for PFAS analyses of fish tissue samples, the laboratory selected for this work proposed the analytical procedures and quality control acceptance criteria that they would use for these analyses. The information describing the fish tissue and rinsate analytical methods is provided in Section B4 of this QAPP. Data usability for each analysis will be assessed using QC criteria summarized in Section B5.

OST specified the use of Method 1668C (USEPA 2010) and its quality control acceptance criteria for analyses of 2020 GLHHFFTS and ORD-Duluth 2020 Great Lakes special study fish fillet tissue samples for PCB congeners. The information describing the analytical method is provided in Section B4 of this QAPP. Data usability for each analysis will be assessed using QC criteria summarized in Section B5.

There are few EPA methods for the analysis of Aroclors in fish tissue. OST had employed EPA Method 1656A in the National Lake Fish Tissue Study because that method was available through an existing laboratory contracting program in the Engineering and Analysis Division (EAD). By the time of the current study, no commercial laboratories were offering that method for analysis of fish tissue samples, so OST reviewed and accepted a series of sample preparation, extraction, cleanup, and determinative procedures from the SW-846 methods manual proposed by the laboratory that will be performing those analyses. The information describing these methods is provided in Section B4 of this QAPP. Data usability for each analysis will be assessed using QC criteria summarized in Section B5.

Because they are not environmental contaminants, EPA has not developed methods for fatty acid analyses of fish tissue samples. Therefore, the laboratory selected for this work proposed the analytical procedures and quality control acceptance criteria that they would use for these analyses. The information describing their fish tissue analytical method is provided in Section B4 of this QAPP. Data usability for each analysis will be assessed using QC criteria summarized in Section B5.

A8. Special Training/Certification

All laboratory staff involved in the analyses of fish tissue samples (and of rinsate samples, which applies to PFAS analyses only) must be proficient in the associated tasks, as required by each analytical laboratory's existing quality system. All contractor staff involved in analytical data review and assessment will be proficient in data review, and no specialized training is required for data reviewers for this project.

A9. Documents and Records

The Statements of Work (SOWs) for the analytical subcontracts provide the specific requirements for laboratory deliverables. The major points are summarized below:

- The laboratory must provide reports of all results required from analyses of environmental and QC samples.
- Summary level data must be submitted in electronic format and must include the following information: EPA sample number, analyte name and CAS number, laboratory sample ID, measured amount, reporting units, sample preparation date, and analytical batch ID (if applicable).
- The laboratory shall provide raw data in the form of direct instrument readouts with each data package. Raw data include:
  - Copy of traffic report, chain-of-custody records, or other shipping information
  - Instrument readouts and quantitation reports for analysis of each sample, blank, standard and QC sample, and all manual worksheets pertaining to sample or QC data or the calculations thereof
  - Copies of bench notes, including preparation of standards and instrumental analyses

The laboratories will maintain records and documentation associated with these analyses for a minimum of three years after completion of the study. Additional copies will be maintained by CSRA for at least five years after completion of the study, and they will be transferred to EPA on request.

B. DATA GENERATION AND ACQUISITION

B1. Sampling Process Design (Experimental Design)

The target population for the 2020 GLHHFFTS consists of all 226 of the Great Lakes nearshore sites randomly selected for 2020 NCCA sampling. Additionally, there are 50 ORD-Duluth 2020 Great Lakes special study enhancement sites in Lake Michigan identified for human health fish collection. Together, these 226 nearshore sites and 50 enhancement sites are designated as the NCCA 2020 Great Lakes human health whole fish sampling sites. The sample collection goal is to collect enough specimens to provide a composite sample consisting of a minimum of 75 grams of fillet tissue from each NCCA 2020 Great Lakes human health whole fish sampling site. The design for selecting the human health whole fish sampling sites incorporated the following objectives:

- Statistically representative data on the concentrations of mercury, PCBs, and PFAS in Great Lakes fish commonly consumed by humans.
- Information on the potential for PFAS to bioaccumulate in fish fillet tissue.
- Data to answer questions concerning the occurrence of PFAS in the fillets of fish and the potential for human exposure through fish consumption.

- Species-specific information on fatty acid content of Great Lakes fish that are commonly targeted by fishermen and consumed by humans. Fatty acid analyses are being limited to the fillet samples from the 2020 GLHHFFTS.

Fish fillet tissue data from the 2020 GLHHFFTS will also provide EPA with the opportunity to evaluate changes in the levels of Great Lakes fish contamination over time by comparing 2020 GLHHFFTS fillet tissue results to the fillet tissue data generated during the 2015 GLHHFFTS and the 2010 GLHHFTS.

Sampling at the 2020 GLHHFFTS nearshore sites and ORD-Duluth 2020 Great Lakes special study enhancement sites in Lake Michigan involves collection of whole fish samples for analysis of fillet tissue samples for mercury, PFAS, PCB congeners, PCBs as Aroclors, lipids, and fatty acids. (Analyses of fillet samples from Lake Michigan enhancement sites will not include PCBs as Aroclors or fatty acids. The lipid analyses of all samples are being conducted as part of the fillet sample preparation under USEPA 2020c.) To meet the study objectives, one fish sample is collected from each nearshore and enhancement site. Ideally, each fish sample is a routine fish composite sample that consists of five fish of adequate size to provide the required amount of fillet tissue for analysis (USEPA 2020c). Fish are selected for each composite sample by applying the following criteria:

- All are of the same species.
- All satisfy legal requirements of harvestable size (or weight) for the sampled site, or at least be of consumable size if no legal harvest requirements are in effect.
- All are of similar size, so that the smallest fish specimen in a composite sample is no less than 75% of the total length of the largest specimen.
- All are collected at the same time, i.e., collected as close to the same time as possible, but no more than one week apart. (Note: Individual fish may have to be frozen until all fish to be included in the composite sample are available for delivery to the designated laboratory.)

Accurate taxonomic identification is essential in preventing the mixing of closely related target species. Under no circumstances are specimens from different species used in a human health fish composite sample.

The sample collection goal at each NCCA 2020 Great Lakes site designated for whole fish sample collection is to obtain a composite sample of fish that provides a minimum of 75 grams of fillet tissue for chemical analysis. Field crews collected the majority of human health fish samples between June and September during the 2020 field season, but they require additional time in 2021 to complete fish sampling due to logistical constraints imposed on field crews in 2020 because of the coronavirus pandemic.

B2. Fish Sampling and Fillet Sample Preparation Methods

B2.1 Fish Sampling Methods

Sampling method procedures and requirements for collection of human health fish samples are detailed in EPA's *National Coastal Condition Assessment 2020 Quality Assurance Project Plan* (USEPA 2020a) and *National Coastal Condition Assessment 2020 Field Operations Manual* (USEPA 2020b). These sampling procedures and requirements, which apply to human health whole fish sample collection at both the 2020 GLHHFFTS nearshore sites and the ORD-Duluth 2020 Great Lakes special study Lake Michigan enhancement sites, are summarized below.

The sampling objective is for field crews to obtain one representative human health whole fish composite sample from each nearshore and enhancement site. Collecting fish composite samples is a cost-effective means of estimating average chemical concentrations in the tissue of target species, and compositing fish ensures adequate sample mass for analysis of multiple chemicals. The sampling procedures specify that each human health fish composite sample should consist of five similarly sized adult fish of the same species. OST developed a recommended fish species list with GLNPO concurrence that contains 25 primary (priority) target fish species and 18 secondary alternative fish species (see Table 1). Field teams use this list as the basis for selecting appropriate fish species for the NCCA 2020 Great Lakes human health fish samples. The method applied for fish collection is left to the discretion of the field team, but it typically involves angling or gillnetting and occasionally trawling.

In preparing Great Lakes human health whole fish samples for shipping, field teams record sample number, species name, specimen length, sampling location, and sampling data and time on an electronic Human Health Fish Collection Form in the NCCA 2020 app. Each fish is wrapped in solvent-rinsed, oven-baked aluminum foil, with the dull side in using foil sheets provided by EPA. Individual foil-wrapped specimens are placed into a length of food-grade polyethylene tubing, each end of the tubing is sealed with a plastic cable tie, and a fish specimen label is affixed to the outside of the food-grade tubing with clear tape. All of the wrapped fish in the sample from each site are placed in a large plastic bag and sealed with another cable tie, then placed immediately on dry ice for shipment to Microbac Laboratories in Baltimore, Maryland.

Table 1. Primary Target Fish Species and Secondary Alternative Fish Species for the 2020 GLHHFFTS and ORD-Duluth 2020 Great Lakes Special Study

Primary Fish Species Scientific Name*	Primary Fish Species Common Name		Secondary Fish Species Scientific Name*[a]	Secondary Fish Species Common Name
Ambloplites rupestris	Rock bass		*Carpiodes cyprinus*	Quillback
Micropterus dolomieu	Smallmouth bass		*Catostomus catostomus*	Longnose sucker
Micropterus salmoides	Largemouth bass		*Catostomus commersonii*	White sucker
Pomoxis annularis	White crappie		*Hypentelium nigracans*	Northern hogsucker
Pomoxis nigromaculatus	Black crappie		*Ictiobus cyprinellus*	Bigmouth buffalo
Cyprinus carpio	Common carp		*Ictiobus niger*	Black buffalo
Esox lucius	Northern pike		*Lepomis cyanellus*	Green Sunfish
Esox masquinongy	Muskellunge		*Lepomis gibbosus*	Pumpkinseed
Esox niger	Chain pickerel		*Lepomis gulosus*	Warmouth
Ictalurus punctatus	Channel catfish		*Lepomis macrochirus*	Bluegill
Lota lota	Burbot		*Lepomis megalotis*	Longear Sunfish
Morone americana	White perch		*Ameiurus melas*	Black bullhead

Table 1. Primary Target Fish Species and Secondary Alternative Fish Species for the 2020 GLHHFFTS and ORD-Duluth 2020 Great Lakes Special Study

Primary Fish Species Scientific Name*	Primary Fish Species Common Name
Morone chrysops	White bass
Perca flavescens	Yellow perch
Sander canadensis	Sauger
Sander vitreus	Walleye
Coregonus clupeaformis	Lake whitefish
Oncorhynchus gorbuscha	Pink salmon
Oncorhynchus kisutch	Coho salmon
Oncorhynchus tshawytscha	Chinook salmon
Oncorhynchus mykiss	Rainbow trout
Salmo salar	Atlantic salmon
Salmo trutta	Brown trout
Salvelinus namaycush	Lake trout
Aplodinotus grunniens	Freshwater drum

Secondary Fish Species Scientific Name*[a]	Secondary Fish Species Common Name
Ameiurus natalis	Yellow bullhead
Ameiurus nebulosus	Brown bullhead
Coregonus artedi	Cisco/ lake herring
Coregonus hoyi	Bloater
Prosopium cylindraceum	Round whitefish
Salvelinus fontinalis	Brook trout

* Minimum acceptable length is 190 mm, TL

[a] Only send if preferred species are not available

* Minimum acceptable length is 190 mm, TL

[a] Only send if preferred species are not available

Field crews are directed to pack fish samples on dry ice in sufficient quantities to keep samples frozen for up to 48 hours (i.e., 50 pounds), and to ship them via priority overnight delivery service (i.e., FedEx), so that they arrive at Microbac Laboratories in less than 24 hours from the time of sample collection. Alternatively, field crews may transport Great Lakes human health whole fish samples on wet or dry ice (depending on the distance) to an interim facility where the fish samples are frozen and stored for up to two weeks before overnight shipping to Microbac Laboratories on dry ice as described above.

B2.2 Fillet Sample Preparation Methods

The laboratory at Tetra Tech's Biological Research Facility in Owings Mills, MD, is the fish sample preparation laboratory (prep lab) for the NCCA 2020 Great Lakes human health fish samples and all of the sample preparation methods described here are governed by a separate QAPP (USEPA 2020c). Prior to initiating fish sample preparation, Tetra Tech coordinates with CSRA for transfer of NCCA 2020 GLHHFFTS and ORD-Duluth 2020 whole fish samples from Microbac Labs (Baltimore, Maryland) to the Tetra Tech lab, where a sample custodian checks in the whole fish samples before storing them in a freezer at a temperature of ≤ -20º Celsius (C). Whole fish sample check-in procedures involve (1) verifying that all associated paperwork stored with the samples is complete, legible, and accurate, (2) comparing the information on the label for each fish specimen to the fish sample preparation batch spreadsheet containing fish sample processing instructions, (3) reporting problems involving sample paperwork, sample integrity, or fish sample label and processing instruction information inconsistencies to the Fish Sample Preparation Technical Leader and the OST Project Manager via email, and (4) coordinating with the Fish Sample Preparation Technical Leader and OST Project Manager to resolve any discrepancies before beginning fish sample processing.

Fish Sample Preparation Batches

Each NCCA 2020 Great Lakes human health fish sample preparation batch generally consists of 20 whole fish samples. Whole fish samples from the 2020 GLHHFFTS and the ORD-Duluth

2020 Great Lakes special study are assigned to separate batches (labeled Batch 1, Batch 2, etc. for the 2020 GLHHFFTS and Batch ORD1, Batch ORD2, etc., for the ORD-Duluth 2020 Great Lakes special study). The number of whole fish samples in the final fish sample preparation batch (or two) for each of these series may be adjusted to include a few more than 20 or fewer than 20, depending on what fraction of 20 whole fish samples are left for assignment to a batch. Tetra Tech staff develop fish sample preparation instructions that include all valid fish samples available for processing. Processing may not begin until the Fish Sample Preparation Technical Leader and the OST Project Manager review the draft instructions and the Fish Sample Preparation Technical Leader approves the final instructions and batch assignments and the OST Project Manager concurs with the approvals.

Homogenized Fillet Sample Preparation

The homogenized fillet sample preparation process begins with removing the fillet (with skin on and "belly flap" or ventral muscle attached) from both sides of each valid fish in the whole fish sample. The combined fillets from all valid fish in the whole fish sample are weighed separately to the nearest gram (wet weight) before they are homogenized together. An electric meat grinder is used to prepare each homogenized fillet sample. The entire set of fillets (with skin and belly flap) from both sides of every valid fish in the whole fish sample (i.e., ideally 5 fish per sample) are homogenized, and the entire homogenized volume is used to prepare the fillet tissue sample aliquots. Grinding of the fillet tissue is repeated until the tissue consists of a uniform color and finely ground texture. Homogeneity is confirmed by conducting triplicate analyses of the lipid content in one fish sample from each fish sample preparation batch (generally one in 20 fish samples). The collective weight of the homogenized fillet tissue from the whole fish sample is recorded to the nearest gram (wet weight) after processing. Tetra Tech lab technicians prepare fillet tissue sample aliquots for chemical analysis and archive according to specifications in Table 1 of Appendix B of the NCCA 2020 Great Lakes Human Health Fish Sample Preparation QAPP (USEPA 2020c).

B3. Sample Receipt and Inspection

This section describes the sample receipt and inspection procedures that apply to the shipment of 2020 NCCA Great Lakes human health homogenized fillet tissue samples to the analytical laboratories selected for analysis of these samples.

In coordination with CSRA, Tetra Tech staff initiate packing and shipping the 2020 NCCA Great Lakes human health homogenized fillet tissue samples from their fish sample preparation laboratory in Owings Mills, Maryland, to the analytical laboratories designated for analysis of these fillet samples, following procedures described in Appendix B of the NCCA 2020 Great Lakes Human Health Fish Sample Preparation QAPP (USEPA 2020c). CSRA staff prepare sample tracking paperwork that is included in each shipment, notify the laboratories in advance of each shipment, track the progress of each shipment, and identify and resolve any delays that arise during shipment of the fillet samples.

When coolers are received at each analytical laboratory, the fillet tissue samples are inspected for damage, logged into the laboratory, and placed into freezers immediately after the laboratory measures and records the temperature of each cooler. Homogenized fillet tissue samples are

stored frozen at ≤ -20º C until analyzed. Because the samples are shipped frozen, typical temperature blanks consisting of a bottle of water are not practical (they may break due to expansion), so they are not required. The laboratory measures and records the temperature of the coolers containing fillet samples on receipt using an infrared temperature sensor or other suitable device. Each laboratory notifies the CSRA Project Leader about the receipt of the fillet tissue samples by email, and the CSRA Project Leader advises the OST Project Manager of fillet sample receipt on the day of delivery. Any questions from the analytical laboratory regarding sample paperwork or sample condition are sent to CSRA and routed to OST or Tetra Tech, as appropriate, before CSRA sends the answers back to the laboratory.

B4. Analytical Methods

B4.1 Mercury Analysis of Fillet Tissue

ALS Environmental Lab prepares (a process involving tissue digestion and oxidation prior to tissue analysis) and analyzes fillet tissue samples 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 2001b), and Revision E of Method 1631 (1631E) for the analysis of mercury in fish tissue samples (USEPA 2002). This method requires approximately 1 g of tissue for the analysis. The sample is digested with a combination of nitric and sulfuric acids. The mercury in the sample is oxidized with bromine monochloride (BrCl) and analyzed by cold-vapor atomic fluorescence spectrometry.

Tissue sample results are reported based on the wet weight of the tissue sample, in nanograms per gram (ng/g). The mercury method detection limit (MDL) and Minimum Level (quantitation limit) are listed in Appendix B.

B4.2 PFAS Analysis of Fillet Tissue and Rinsate Samples

There are no formal analytical methods from EPA or any voluntary consensus standards bodies for the PFAS analyses of tissue samples. Therefore, fish tissue samples will be analyzed by SGS-AXYS Analytical Services, Ltd. (Sidney, BC, Canada), using procedures developed, tested, and documented in that laboratory. CSRA reviewed the SOP during the solicitation process and will maintain a copy of the SGS-AXYS SOP on file that will be made available to EPA for review on request.

The analytical procedures are briefly described below, based on information in the SOP. The 40 target PFAS analytes are shown in Appendix B.

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, all of the target analyte concentrations are corrected for the recovery of the labeled standards, thus accounting for extraction efficiencies and losses during cleanup.

Approximately 2 g of fish tissue are required for analysis. (If matrix-related analytical problems are identified during the analysis of a given fish tissue sample, a sample aliquot of 1 g may be used to minimize those problems.) The sample is spiked with 24 isotopically labeled standards and extracted by shaking the tissue 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 is centrifuged to remove the solids, and the supernatant liquid is diluted with reagent water and processed by solid-phase extraction (SPE) on a weak anion exchange sorbent. The PFAS analytes are eluted from the SPE cartridge, and the eluant is spiked with additional labeled recovery standards and analyzed by high performance liquid chromatography with tandem mass spectrometry.

The aqueous rinsate samples will be analyzed for the 40 PFAS analytes using the same isotope dilution procedure as used for the fish tissue samples, but with an extraction step based on EPA Method 537 from the Office of Ground Water and Drinking Water (USEPA 2009). The 250-mL aqueous rinsate sample is spiked with the labeled standards and processed by SPE, in a similar manner as is used for the tissue samples. The PFAS analytes are eluted from the SPE cartridge and the eluant is spiked with additional labeled recovery standards and analyzed by high performance liquid chromatography with tandem mass spectrometry.

Tissue sample results are reported based on the wet weight of the tissue sample, in nanograms per gram (ng/g). Method detection limits and Minimum Levels (quantitation limits) for PFAS analytes are listed in Appendix B. Aqueous rinsate results are reported based on the volume of the rinsate sample, in nanograms per liter (ng/L).

B4.3 PCB Congener Analysis of Fillet Tissue

Fish tissue samples are being prepared and analyzed by Vista Analytical Laboratory (El Dorado Hills, California), in general accordance with Revision C of EPA Method 1668, Chlorinated Biphenyl Congeners in Water, Soil, Sediment, Biosolids, and Tissue by HRGC/HRMS (USEPA 2010). The samples are being analyzed for all 209 PCB congeners and reported as either individual congeners or coeluting groups of congeners. The following method modifications have been reviewed, found to be within the allowance for flexibility in Section 9.1.2 of Method 1668C, supported by performance data that are maintained on file at the laboratory, and have been approved for use in this study:

- Section 7.6.4: Vista uses sodium sulfate as the reference matrix for QC samples associated with tissue analyses rather than vegetable oil because they have not found a source of vegetable oil that did not have traces of PCBs in it.
- Sections 7.10.1 and 15.4.2.1: Vista uses a CS-3 (mid-level calibration) standard that contains all 209 of the PCB congeners, rather than the subset of congeners listed in the method. Therefore, they do not run a separate standard containing all 209 congeners during the calibration verification process in Section 15.4.2.1.
- Section 12.5: Vista uses sodium hydroxide to adjust the pH of the solution in the back-extraction procedure, rather than potassium hydroxide.

- Table 3: Vista adds 44 ^{13}C-labeled compounds to each sample, 17 more than the 27 labeled compounds specified in the method, and monitors the recoveries of all of these standards in each sample.

Note: Given the large number of target analytes involved, the final list of PCB congeners and coelutions is provided in Appendix B of this QAPP, along with their MDLs and MLs.

Tissue sample results are reported based on the wet weight of the tissue sample in units of picograms per gram (pg/g).

B4.4 PCBs as Aroclors Analysis of Fillet Tissue

Fish fillet tissue samples are being prepared and analyzed by Eurofins-TestAmerica (Pittsburgh, PA), using sample extraction, cleanup and determinative procedures from the SW-846 methods manual. The laboratory will extract approximately 10 g of tissue for the analysis. The samples are being analyzed for seven common Aroclor mixtures (i.e., 1016 to 1260), plus Aroclor 1268. The laboratory proposed and EPA accepted the following analytical scheme:

- Extraction by Method 3541 (automated Soxhlet)
- Cleanups by Methods 3660B (sulfur), 3665A (sulfuric acid-permanganate), and 3640A (gel-permeation chromatography)
- Analysis by Method 8082A (gas chromatography with electron capture detection)

Tissue sample results are reported based on the wet weight of the tissue sample in units of nanograms per gram (ng/g). Method detection limits and Minimum levels (quantitation limits) for each of the Aroclors are listed in Appendix B.

B4.5 Fatty Acid Analysis of Fillet Tissue

Because they are not environmental contaminants, there are no formal EPA methods for the analysis of fatty acids in any matrix. Therefore, fish tissue samples will be analyzed by Clarkson University (Potsdam, NY), using procedures developed, tested, and documented in that laboratory and currently employed under GLNPO Grant No. GL 00E02957. The laboratory's SOP was reviewed by CSRA during the solicitation process, along with the supporting materials. A copy of the Clarkson SOP will be maintained on file at CSRA and will be made available to EPA for review on request.

The analytical procedures are briefly described below. The 38 target fatty acid analytes are shown in Appendix C.

Approximately 2 g of homogenized fish tissue is spiked with a surrogate solution (nonadecacanoic acid, C19:0) and extracted with 2:1 mixture of chloroform and methanol using ultrasonic extraction. The extract is centrifuged to separate the water from the chloroform, and concentrated to approximately 20 mL. A 10-µL aliquot of the extract is transferred to a clean autosampler vial, purged for 30 seconds with nitrogen, capped, and then placed on the instrument for derivatization and injection.

The automated instrument adds 100 µL of deuterated C18:0 (as an internal standard) and 250 µL of 12% boron trifluoride (BF3) in methanol. The solution is mixed and heated to 70 °C for 50 minutes. After heating, 25 µL of water is added to quench the derivatization reaction and the derivatized extract is mixed, followed by the addition of 0.65 mL of hexane and further mixing to separate the fatty acid methyl esters from the aqueous solution.

An aliquot of the hexane extract is analyzed by gas chromatography, with flame ionization detection (GC/FID), using a 100 m x 250 µm x 0.2 µm HP-88 column. The concentration of each fatty acid is calculated based on a multi-point calibration curve and reported based on the wet weight of the tissue sample, in micrograms per gram (µg/g). Method detection limits and quantitation limits for the fatty acids are listed in Appendix B.

B5. Analytical Quality Control

The analytical procedures being applied by the laboratories designated for analysis of 2020 NCCA Great Lakes human health homogenized fillet tissue samples include many of the traditional EPA analytical quality control (QC) activities. For example, all samples are analyzed in batches and each batch includes:

- up to 20 field samples and the associated QC samples
- blanks – at least 5% of the samples within a batch are method blanks (with higher percentages specified in some analytical methods)

Other common quality control activities vary by the analysis type. The QC activities associated with the chemical analyses of fillet samples for target chemicals are described in Subsection B5.1 for mercury, Subsection B5.2 for PFAS, Subsection B5.3 for PCB congeners, and Subsection B5.4 for PCBs as Aroclors.

B5.1 Mercury Analysis QC Criteria

Quality control samples associated with each batch of fillet tissue samples analyzed for mercury are summarized in Table 2 below.

The cold-vapor atomic fluorescence instrument is calibrated daily, as described in Method 1631E and the laboratory's SOP. At least five calibration standards and a blank are used for calibration, and the variability in the calibration factors for the five standards must have a relative standard deviation (RSD) less than or equal to 15%. The calibration is verified after every 20 samples by the analysis of the ongoing precision and recovery (OPR) standard, or the laboratory control sample (LCS). The results for the OPR/LCS standard must fall within the limits in Table 2.

Table 2. QC Samples and Acceptance Criteria for Mercury Analysis of Fish Tissue

QC Operation	Frequency*	Acceptance Limit	Corrective Action
Bubbler blank or System blank (depending on instrument configuration)	3 blanks run during calibration and with each analytical batch of up to 20 field samples	50 picograms (pg) of mercury	If the bubbler or system blank is above 50 pg, take corrective action to reduce the blank level to below 50 pg, and reanalyze any samples in the affected batch.
OPR/LCS	Prepared once per batch of up to 20 field samples, analyzed *once prior to* the analysis of any field samples, *and again at* the end of each analytical batch, spiked at 4.0 ng	70 - 130% recovery (5.6 –10.4 ng/g)	If the OPR recovery is not within the QC acceptance limits, • take corrective action and repeat the OPR analysis, beginning with a fresh aliquot, reanalyze all samples in the affected analytical batch.
Method blank	3 method blanks per batch of up to 20 field samples, with analyses interspersed among the samples in the analysis batch	0.4 nanograms (ng) (400 pg) of mercury, or Less than one tenth the concentration of an associated sample	If any of the three method blank results is above 0.4 nanograms, • take corrective action to reduce the blank level to below 0.4 ng, • reanalyze any samples in the affected batch with results less than 10 times the observed results for any of the three blanks, and • flag sample results greater than 10 times the observed blank level to advise the data user of the potential contamination.
QC sample	Once per batch of up to 20 field samples	Per the provider of the QCS *or* 75 - 125% recovery if no criteria provided by the supplier	If the QCS results are not within the provider's acceptance limits, • take corrective action and repeat the QCS analysis, beginning with a fresh aliquot, • reanalyze all samples in the affected analytical batch.
MS/MSD	Once per every 10 field samples (e.g., twice per 20 samples in a preparation batch) See note below this table regarding spiking levels and the use of a sample from a previous analysis batch for preparation of the MS/MSD aliquots.	70 - 130% recovery and RPD ≤ 30%	If either the MS or MSD recovery is not within the QC acceptance limits, • take corrective action and repeat the MS/MSD analysis, beginning with fresh aliquots, • reanalyze all samples in the affected analytical batch. If the RPD exceeds the acceptance limit, the laboratory will reanalyze the MS/MSD samples: • If the reanalysis results meet the RPD limit, then the laboratory will reanalyze all of the associated field and QC samples.

* The term "field sample" refers to homogenized fillet tissue samples provided to the analytical laboratory for mercury analysis.

Note: Provision of useful MS/MSD data is highly dependent on selection of an appropriate spiking level relative to the background concentration of mercury in the unspiked sample. After the first batch of samples, the MS/MSD sample may be prepared from excess volume of tissue from a sample in the previous batch, such that the background level is known. Spiking should be performed at approximately 3 to 5 times the background concentration.

B5.2 PFAS Analysis QC Criteria

The high performance liquid chromatograph/tandem mass spectrometer is calibrated as described in the laboratory's SOP. Seven calibration standards are used for calibration, using a weighted linear regression. The correlative coefficient for the regression must be ≥ 0.95. The calibration is verified every 12 hours through the analysis of the calibration verification standard. The results for the calibration verification must meet the requirements in Appendix C of this QAPP.

Quality control samples associated with each batch of tissue samples or rinsate samples analyzed for PFAS are summarized in Table 3 below.

Table 3. QC Samples and Acceptance Criteria for PFAS Analysis of Tissue and Rinsates

QC Operation	Frequency*	Acceptance Limit	Corrective Action
Labeled compounds	Spiked into every sample before extraction	Per Appendix C of this QAPP	Evaluate failure and impact on samples. If sample results are non-detects for analytes which have a high labeled compound recovery, report non-detect results with case narrative comment. For detected analytes with low labeled compound recovery, extract and analyze a smaller sample aliquot.
Calibration Verification	Every 12 hours, before sample analysis.	Per Appendix C of this QAPP	• Evaluate failure and impact on samples. If sample results are non-detects for analytes which have a high bias, report non-detect results with case narrative comment. *or* Immediately analyze two additional consecutive verification standards. If both pass, samples may be reported without reanalysis. If either fails, take corrective action(s) and re-calibrate; then reanalyze all affected samples since the last acceptable verification standard.
Lab Control Sample (LCS)	Once per batch of up to 20 field samples	Per Appendix C of this QAPP	• Reanalyze LCS once. If acceptable, report. Evaluate samples for detections, and LCS for high bias. If LCS has high bias, and sample results are non-detects, report with case narrative comment. If LCS has low bias, or if there are detected analytes with failures, evaluate and reprepare and reanalyze the LCS and all samples in the associated prep batch for failed analytes.
Method blank	Once per batch of up to 20 field samples	Less than or equal to the MDLs in Appendix B of this QAPP	All results, including blanks, are reported down to the method detection limit (MDL). • If the method blank result for any PFAS is above the MDL, but below the laboratory's nominal quantitation limit, the laboratory will flag all associated tissue sample and rinsate results as having a detectable method blank for that analyte. (Subsequent validation of the results by EPA or its contractors will evaluate the potential contribution of the blank to such sample results.) • If the method blank result is above the quantitation limit, the laboratory will reanalyze the method blank. - If the method blank reanalysis result is below the quantitation limit, then the laboratory will reanalyze all of the associated tissue or rinsate samples and QC samples. - If the method blank reanalysis result is still above the quantitation limit, then the laboratory will re-extract and reanalyze all tissue or rinsate samples with original results above the MDL.
Laboratory duplicate	Once per batch of up to 20 field samples	The relative percent difference (RPD) of the duplicate measurements must be < 40%	Evaluate the data, and re-extract and reanalyze the original sample and duplicate: • If the reanalysis results meet the RPD limit, then the laboratory will reanalyze all of the associated field and QC samples. • If the reanalysis result still does not meet the RPD limit, then the laboratory will re-extract and reanalyze all field samples with original results above the MDL.

* The term "field sample" refers to homogenized fillet tissue samples provided to the analytical laboratory for PFAS analysis.

B5.3 PCB Congener Analysis QC Criteria

The high-resolution gas chromatograph/high-resolution mass spectrometer (HRGC/HRMS) is calibrated periodically as described in Method 1668C and the laboratory's SOP. At least five calibration standards are used for calibration, and the variability in the response factors for the five standards must have a relative standard deviation (RSD) less than or equal to 20%. The

calibration is verified every 12 hours by the analysis of the calibration verification (VER) standard. The results for the VER must meet the requirements in Appendix D.

Quality control samples associated with each batch of tissue samples analyzed for PCBs are summarized in Table 4, below, and are based on the QC requirements of Method 1668C, with the project-specific addition of one laboratory duplicate sample per batch.

Table 4. QC Samples and Acceptance Criteria for PCB Analysis of Fish Tissue

QC Sample	Frequency*	Acceptance Limit	Corrective Action
Laboratory control sample	One per sample batch	Per Appendix D	Per Method 1668C
Calibration verification (VER)	At the beginning of every 12-h analytical shift	Per Appendix D	Per Method 1668C
Method blank	Once per batch of up to 20 field samples	5x MDL for each congener (As noted elsewhere, all results, including blanks, are reported down to the MDL.)	If the method blank result is above 5x MDL, the laboratory will reanalyze the method blank extract to confirm the presence of the blank contaminants. If the reanalysis result is still above 5x MDL, then the laboratory will compare the results in the method blank to the results in all of the associated field samples in the batch and take corrective action as follows: • If the result for a congener (or group of coeluting congeners) that is present in the method blank at 5x MDL or higher is *not present* in the field sample, then the result for that field sample may be reported without corrective actions. The result must be flagged with a "B" flag that indicates the presence of the analyte in the associated blank and the data package narrative must discuss the comparison of the blank and sample results for that sample. • If the result for the congener in the field sample is more than 10 times the level found in the method blank, then the result for that field sample also may be reported without corrective actions. The result must be flagged with a "B" flag that indicates the presence of the analyte in the associated blank and the data package narrative must discuss the comparison of the blank and sample results for that sample. • If the result for the congener in the field sample is less than or equal to 10 times the level found in the method blank, then re-extraction and reanalysis of the affected sample is **required** (but not samples that meet the conditions in #1 and #2 above) in conjunction with a new method blank and all other method-specified QC samples. CSRA will work with the laboratory to schedule any required reanalyses in a manner that does not delay analyses of subsequent batches of field samples. • If the results of the re-extraction and reanalysis of the field sample do not resolve the problem, i.e., the background levels in the method blank are still a concern, CSRA will require that the laboratory provide information on historical levels of blank contaminants for similar matrices. CSRA and EPA will evaluate those historical results and the reanalysis results on a case-by-case basis to determine if there is a pattern of blank contamination that is indicative of a broader problem and if any further corrective actions are required by the laboratory.

Table 4. QC Samples and Acceptance Criteria for PCB Analysis of Fish Tissue

QC Sample	Frequency*	Acceptance Limit	Corrective Action
Laboratory duplicate	Once per batch of up to 20 field samples	The RPD of the duplicate measurements must be: • < 50% for sample concentrations greater than or equal to 5 times the MDL, and • <100% for sample concentrations less than 5 times the MDL. (When comparing the sample concentration to the MDL, use the lower of the two concentrations in the paired samples.)	If the RPD exceeds the acceptance limit, the laboratory will reanalyze the laboratory duplicate extract: • If the reanalysis result meets the RPD limit, then the laboratory will reanalyze all of the associated field and QC samples. • If the reanalysis result still does not meet the RPD limit, then the laboratory will re-extract and reanalyze all field samples with original results above the MDL.

* The term "field sample" refers to homogenized fillet tissue samples provided to the analytical laboratory for PCB congener analysis.

B5.4 PCBs as Aroclors Analysis QC Criteria

The gas chromatograph is calibrated periodically as described in Method 8082A and the laboratory's SOP. At least five calibration standards are used for calibration, and the variability in the response factors for the five standards must have a relative standard deviation (RSD) less than or equal to 20%. The calibration is verified at least once every 12-hour shift during which analyses are performed by the analysis of the calibration verification standard. The results for the calibration verification must meet the requirements in Table 5.

Quality control samples associated with each batch of fillet tissue samples analyzed for Aroclors are summarized in Table 5 below.

Table 5. QC Samples and Acceptance Criteria for Aroclor Analysis of Fish Tissue

QC Operation	Frequency*	Acceptance Limit	Corrective Action
Lab Control Sample - *Using Aroclor 1016 and 1260*	Once per batch of up to 20 field samples	34 - 118%	Reanalyze LCS once. If acceptable, report. Evaluate samples for detections, and LCS for high bias. If LCS has high bias, and samples non-detect, report with case narrative comment. If LCS has low bias, or if there are detections, evaluate and re-prepare and reanalyze the LCS and all samples in the associated prep batch for failed analytes
Calibration Verification (CV) - *Using Aroclor 1016 and 1260*	Every 12 hours, before sample analysis.	80 - 120%	Evaluate failure and impact on samples. If samples non-detect for analytes which have a high bias, report non-detect results with narrative comment. For analytes with low bias, or samples with detected analytes, use the approach below. Immediately analyze two additional consecutive CVs. If both pass, samples may be reported without reanalysis. If either fails, take corrective action(s) and re-calibrate; then reanalyze all affected samples since the last acceptable CV.

Table 5. QC Samples and Acceptance Criteria for Aroclor Analysis of Fish Tissue

QC Operation	Frequency*	Acceptance Limit	Corrective Action
Method blank	Once per batch of up to 20 field samples	3x MDL for each Aroclor (As noted elsewhere, all results, including blanks, are reported down to the MDL.)	If the method blank result is above 3x MDL, the laboratory will reanalyze the method blank extract to confirm the presence of the blank contaminants. If the reanalysis result is still above 3x MDL, then the laboratory will compare the results in the method blank to the results in all of the associated field samples in the batch and take corrective action as follows: • If the result for an Aroclor that is present in the method blank at 3x MDL or higher is *not present* in the field sample, then the result for that field sample may be reported without corrective actions. The result must be flagged with a "B" flag that indicates the presence of the analyte in the associated blank and the data package narrative must discuss the comparison of the blank and sample results for that sample. • If the result for an Aroclor in the field sample is more than 10 times the level found in the method blank, then the result for that field sample also may be reported without corrective actions. The result must be flagged with a "B" flag that indicates the presence of the analyte in the associated blank and the data package narrative must discuss the comparison of the blank and sample results for that sample. • If the result for an Aroclor in the field sample is less than or equal to 10 times the level found in the method blank, then re-extraction and reanalysis of the affected sample is **required** (but not samples that meet the conditions in #1 and #2 above) in conjunction with a new method blank and all other method-specified QC samples. CSRA will work with the laboratory to schedule any required reanalyses in a manner that does not delay analyses of subsequent batches of field samples. • If the results of the re-extraction and reanalysis of the field sample do not resolve the problem, i.e., the background levels in the method blank are still a concern, CSRA will require that the laboratory provide information on historical levels of blank contaminants for similar matrices. CSRA and EPA will evaluate those historical results and the reanalysis results on a case-by-case basis to determine if there is a pattern of blank contamination that is indicative of a broader problem and if any further corrective actions are required by the laboratory.
MS/MSD - *Using Aroclor 1016 and 1260*	Once per batch of up to 20 field samples	34 - 118% recovery and RPD ≤ 30%	If either the MS or MSD recovery is not within the QC acceptance limits, • Take corrective action and repeat the MS/MSD analysis, beginning with fresh aliquots, • Reanalyze all samples in the affected analytical batch. If the RPD exceeds the acceptance limit, the laboratory will reanalyze the MS/MSD samples: • If the reanalysis results meet the RPD limit, then the laboratory will reanalyze all of the associated field and QC samples
Surrogates	Two surrogates added to each field and QC sample	20 - 125%	Extract and analyze a smaller aliquot of tissue sample. • If the results of the re-extraction and reanalysis of the field sample resolve the problem, then report only the analytical results from the analysis with acceptable surrogate recovery. • Otherwise, report both sets of results and discuss in the narrative.

* The term "field sample" refers to homogenized fillet tissue samples provided to the analytical laboratory for Aroclor analysis.

B5.5 Fatty Acid Analysis QC Criteria

The gas chromatograph is calibrated periodically, as described in the laboratory's SOP. At least five calibration standards are used for calibration, and the variability in the response factors for the five standards must have a relative standard deviation (RSD) less than or equal to 20%. The calibration is verified at least once every 12-hour shift during which analyses are performed by the analysis of the calibration verification standard. The results for the calibration verification must meet the requirements in Table 6.

Quality control samples associated with each batch of fillet tissue samples analyzed for fatty acids are summarized in Table 6 below.

Table 6. QC Samples and Acceptance Criteria for Fatty Acid Analysis of Fish Tissue

QC Operation	Frequency*	Acceptance Limit	Corrective Action
Method blank	1 per analysis batch (10 field samples, plus QC samples)	Method detection limit (MDL)	If any of the analytes are present in the method blank above the MDL, • take corrective action to reduce the blank level to below the MDL, • reanalyze any samples in the affected batch with results less than 10 times the observed results for the blank, and • flag sample results greater than 10 times the observed blank level to advise the data user of the potential contamination.
Calibration linearity (5 points)	Twice a year	$r^2 > 0.95$	Do not analyze samples until linear calibration is achieved
Calibration verification	Every 10 samples	70 to 130% recovery	If the recovery of any analyte is outside the acceptance limits, recalibrate the instrument and reanalyze samples in the affected batch.
Reference sample	Analyze 1 aliquot of the Lake Superior reference tissue sample per analysis batch	50 to 150% of the certified value	If the results are not within the acceptance limits, • take corrective action and repeat the reference sample analysis, beginning with a fresh aliquot, • reanalyze all samples in the affected analytical batch.
Laboratory duplicate sample	1 per every two analysis batches (10 field samples, plus QC samples)	RPD ≤ 50%	If the RPD exceeds the acceptance limit, the laboratory will reanalyze the duplicate sample: • If the reanalysis results meet the RPD limit, then the laboratory will reanalyze all of the associated field and QC samples.
Surrogate	Added to every field and QC sample	50 to 150% recovery	If the recovery of the surrogate is not within the acceptance criteria, reanalyze the affected samples.
Internal Standard	Added to every field and QC sample	Not applicable	Not applicable

* The term "field sample" refers to homogenized fillet tissue samples provided to the analytical laboratory for fatty acid analysis.

B6. Instrument/Equipment Testing, Inspection, and Maintenance

All analytical instrumentation associated with the fillet tissue sample analyses will be inspected and maintained as described in the respective analysis methods and laboratory SOPs.

B7. Instrument/Equipment Calibration and Frequency

All analytical instrumentation associated with the fillet tissue sample analyses will be calibrated as described in the respective analysis methods. The mercury analysis method for tissue

samples, Method 1631E, specifies calibration with at least five calibration standards and multiple blanks, as described in Section B5.1 above. The PFAS analysis procedures from SGS-AXYS for tissue and rinsate analyses specifies calibration with at least seven calibration standards, as described in Section B5.2 above. The PCB congener analysis method for tissue samples, Method 1668C, specifies calibration with at least five calibration standards (refer to Section B5.3 above). The Aroclor analysis method for tissue samples, Method 8082A, specifies calibration with at least five calibration standards (refer to Section B5.4 above). The fatty acid laboratory's SOP specifies calibration with at least five calibration standards (refer to Section B5.5 above).

B8. Inspection/Acceptance of Supplies and Consumables

The inspection and acceptance of any laboratory supplies and consumables associated with the fillet tissue sample analyses are addressed in the individual laboratory operating procedures to be used, and/or in the laboratory's existing overall quality system documentation. There are no additional requirements specific to this project, and therefore, none are described here.

B9. Non-direct Measurements

Non-direct measurements are not required for this project. (The analytical results from the previous fish tissue studies conducted under the NCCA (e.g., the 2010 NCCA and 2015 NCCA) to which any new data are to be compared are primary data that EPA generated under an approved QAPP for that study.)

B10. Data Management

Data management practices employed in this study will be based on standard data management practices used for EPA's National Lake Fish Tissue Study and other EPA fish contamination studies (e.g., NCCA 2015 GLHHFFTS). The data management (i.e., sample tracking, data tracking, data inspection, data quality assessment, database development) procedures have been regularly applied to other technical studies by CSRA. These procedures are being employed because they are effective, efficient, and have successfully withstood repeated internal and external audits, including internal review by EPA Quality Staff, public review and comment, judicial challenge, and an audit by the Government Accountability Office. These procedures, as implemented for the 2020 NCCA Great Lakes human health fish fillet indicator, are summarized below.

- All laboratories performing analyses for this project are required to maintain all records and documentation associated with the analyses of the fish tissue samples for a minimum period of three years after completion of the study.
- All required reports and documentation, including raw data, must be sequentially paginated and clearly labeled with the laboratory name, and associated sample numbers. Any electronic media submitted must be similarly labeled.
- Each laboratory will adhere to a comprehensive data management plan that is consistent with the principles set forth in Good Automated Laboratory Practices, EPA Office of Administration and Resources Management (USEPA 1995) or with commonly employed data management procedures approved by the National Environmental Laboratory

Accreditation Conference (NELAC). Each laboratory's data management plan is incorporated in its overall quality system documentation, e.g., its quality management plans, copies of which will be maintained on file at CSRA.

C. ASSESSMENT AND OVERSIGHT

C1. Assessments and Response Actions

The laboratory contracts prepared to support analysis of Great Lakes human health homogenized fillet tissue samples for the 2020 GLHHFFTS and ORD-Duluth 2020 Great Lakes special study will stipulate that each laboratory has a comprehensive QA program in place and operating at all times during the performance of their contract, and that in performing laboratory work for this study, the laboratory shall adhere to the requirements of that QA program. These materials (ALS, 2020; SGS-AXYS, 2020; Vista, 2020; Eurofins-TestAmerica, 2020, and Clarkson, 2021) were reviewed by CSRA during the laboratory solicitations, as part of an assessment of laboratory capabilities. A copy of each QA plan will be maintained on file at CSRA and will be made available to EPA for review on request.

Sections C1.1 through C1.6 describe other types of assessment activities and corresponding response actions identified to ensure that data gathering activities in the 2020 GLHHFFTS and ORD-Duluth 2020 Great Lakes special study are conducted as prescribed and that the performance criteria defined for this study are met.

C1.1 Surveillance

The CSRA Project Leader will schedule and track all analytical work performed by the laboratories designated for mercury, PFAS, PCB congener, Aroclor, and fatty acid analyses. The Project Leader will coordinate with Tetra Tech staff at the fish sample preparation laboratory regarding fillet tissue sample shipments to the analytical laboratory.

When samples are shipped to the analytical laboratories for mercury, PFAS, PCB congener, Aroclor, or fatty acid analysis, the CSRA Project Leader will contact designated laboratory staff by email to notify them of the forthcoming shipment(s) and request that they contact CSRA on the scheduled day of delivery if the shipments do not arrive intact. Within 24 hours of scheduled sample receipt, CSRA will contact the laboratory to verify that the samples arrived in good condition, and if problems are noted, will work with the laboratory and EPA to resolve the problems as quickly as possible to minimize data integrity problems.

The laboratory designated for mercury analysis of 2020 GLHHFFTS and ORD-Duluth 2020 Great Lakes special study fillet tissue samples will be permitted to work one batch ahead of the CSRA-EPA review of the QC results associated with the fillet tissue sample analyses. CSRA will also immediately notify the OST Project Manager of any mercury laboratory delays that are anticipated to impact EPA schedules.

The laboratory designated for PFAS analysis of 2020 GLHHFFTS and ORD-Duluth 2020 Great Lakes special study fillet tissue samples will be permitted to work two batches ahead of the CSRA/EPA review of the QC results associated with the fillet tissue sample analyses. CSRA

will also immediately notify the OST Project Manager of any PFAS laboratory delays that are anticipated to impact EPA schedules.

The laboratory designated for PCB congener analysis of 2020 GLHHFFTS and ORD-Duluth 2020 Great Lakes special study fillet tissue samples will be permitted to work two batches ahead of the CSRA/EPA review of the QC results associated with the fillet tissue sample analyses. CSRA will also immediately notify the OST Project Manager of any PCB congener analysis laboratory delays that are anticipated to impact EPA schedules.

The laboratory designated for Aroclor analysis of 2020 GLHHFFTS fillet tissue samples will be permitted to work two batches ahead of the CSRA/EPA review of the QC results associated with the fillet tissue sample analyses. CSRA will also immediately notify the OST Project Manager of any Aroclor analysis laboratory delays that are anticipated to impact EPA schedules.

The laboratory designated for fatty acid analysis of 2020 GLHHFFTS fillet tissue samples will be permitted to work two batches ahead of the CSRA/EPA review of the QC results associated with the fillet tissue sample analyses. CSRA will also immediately notify the OST Project Manager of any fatty acid analysis laboratory delays that are anticipated to impact EPA schedules.

Finally, the CSRA Project Leader will monitor the progress of the data quality audits (data reviews) and database development to ensure that the laboratory data submission is reviewed in a timely manner. In the event that dedicated staff are not able to meet EPA schedules, CSRA will identify additional staff who are qualified and capable of reviewing the data in a timely manner. If such resources cannot be identified, and if training new employees is not feasible, CSRA will meet with the OST Project Manager to discuss an appropriate solution.

C1.2 Product Review

Product reviews for validated analytical data packages will be performed within CSRA to verify that the CSRA data reviews are being performed consistently over time and across data reviewers, that the review findings are technically correct, and that the reviews are being performed in accordance with this QAPP. Product reviewers will be charged with evaluating the completeness of the original CSRA data review, the technical accuracy of the reviewer's findings, and the technical accuracy of the separate analytical databases developed to store results associated with the 2020 GLHHFFTS and ORD-Duluth 2020 Great Lakes special study data packages, respectively. Product reviews will be conducted on at least 10% of the data packages. Qualified product reviewers will include any staff members that have been trained in CSRA data review procedures, are experienced in reviewing data similar to those being reviewed and are familiar with the requirements of this QAPP. To ensure the findings of each data review are documented in a consistent and technically accurate manner, CSRA staff will review 100% of the data qualifier flags entered into each project database.

The 2020 GLHHFFTS and ORD-Duluth 2020 Great Lakes special study data files prepared by CSRA for statistical analysis of the data will be reviewed internally by CSRA staff and independently by the OST Project Manager with support from Tetra Tech.

C1.3 Quality Systems Audit

A quality system audit (QSA) is used to verify, by examination and evaluations of objective evidence, that applicable elements of the quality system are appropriate and have been developed, documented, and effectively implemented in accordance and in conjunction with specified requirements. The focus of these assessments is on the quality system processes – not on evaluating the quality of specific products or judging the quality of environmental data or the performance of personnel or programs. The SHPD QA Coordinator may perform a QSA of the 2020 GLHHFFTS Fish Tissue Study mercury, PFAS, PCB congener, Aroclor, or fatty acid analyses.

C1.4 Readiness Review

A readiness review of each analysis laboratory's capability to produce acceptable sample results begins with a review of materials submitted by the laboratory during the solicitation process and continues during a kick-off conference call with each laboratory (ALS Environmental for mercury, SGS-AXYS for PFAS, Vista Analytical Laboratory for PCB congeners, Eurofins-TestAmerica for Aroclors, and Clarkson University for fatty acids). The requested materials include information about the laboratory's capacity, past experience with tissue analyses, and accreditations or certifications for mercury, PFAS, PCB congener, Aroclor, or fatty acid analyses in tissue and other matrices. These materials are reviewed during the solicitation process to assess the laboratory's competency and will be kept on file by CSRA.

Readiness reviews are performed by CSRA data reviewers. If problems are identified during these reviews, CSRA staff will work with the laboratory, to the extent possible, to resolve the problem prior to awarding an analysis contract. If the problem cannot be resolved within the time frame required by EPA, the CSRA Project Leader will notify the OST Project Manager immediately. Records of these reviews and any corrective actions are maintained by CSRA separate from the analytical results for the field samples. CSRA staff will document their findings and recommendations concerning the readiness review as part of a written analytical QA report to EPA.

C1.5 Technical Systems Audit

The laboratory contracts will require that the laboratory be prepared for and willing to undergo an on-site audit or technical systems audit of its facilities, equipment, staff, sample processing, tissue sample analysis, training, record keeping, data validation, data management, and data reporting procedures. An audit will be conducted only if the results of the readiness reviews, data quality audits, and surveillance suggest serious or chronic laboratory problems that warrant on-site examinations and discussion with laboratory personnel.

If such an audit is determined to be necessary, a standardized audit checklist may be used to facilitate an audit walkthrough and document audit findings. Audit participants may include the OST Project Manager and/or the SHPD QA Coordinator (or a qualified EPA staff member designated by the OST QA Officer) and a CSRA staff member experienced in conducting laboratory audits. One audit team member will be responsible for leading the audit and conducting a post-audit debriefing to convey significant findings to laboratory staff at the

conclusion of the audit. Another audit team member will be responsible for gathering pre-audit documentation of problems that necessitated the audit, customizing the audit checklist as necessary to ensure that those problems are addressed during the audit, documenting audit findings on the audit checklist during the audit, and drafting a formal report of audit findings for review by EPA.

C1.6 Data Quality Assessment

Upon completion of data verification and validation procedures (see Section D1), CSRA staff will create an analytical database that contains all fillet tissue and QC sample results from the 2020 GLHHFFTS and a separate analytical database that contains all fillet tissue and QC sample results from the ORD-Duluth 2020 Great Lakes special study. At selected intervals and upon completion of the study, the CSRA Project Leader will perform analyses to verify the accuracy of each database. The procedures will be directed at evaluating the overall quality of each database against data quality objectives established for the respective studies and in identifying trends in fillet tissue sample results derived from field samples and QC results obtained during each of the studies. CSRA staff will document their findings and recommendations concerning this data quality assessment and provide them to EPA.

C2. Reports to Management

CSRA will track the receipt of data submissions for the homogenized fish fillet tissue analyses (and aqueous sample analyses for PFAS only) and advise the OST Project Manager of progress on a monthly basis.

Following data verification and validation of all project-specific analytical data, CSRA will apply data qualifier flags, where needed, to the fillet tissue results in each project database that describe data quality limitations and recommendations concerning data use. The data qualifier flags are based on those developed for the National Lake Fish Tissue Study and the complete list of qualifier flags used and their implications for data use will be summarized in a report to EPA at or near the end of the data assessment process.

The CSRA Project Leader will provide a monthly report to the OST Project Manager that describes the status of all current analysis and data review activities, during each month in which analyses and data review are conducted.

D. DATA VALIDATION AND USABILITY

This QAPP addresses the generation of data from homogenized fish fillet tissue samples prepared from 2020 Great Lakes human health fish samples (and from aqueous QC samples for PFAS analyses only). Sections D1, D2, and D3 of this QAPP apply to all of the analytical data generation for the 2020 GLHHFFTS and the ORD-Duluth 2020 Great Lakes special study.

D1. Data Review, Verification, and Validation

The data review, verification, and validation aspects of the homogenized fish fillet tissue analyses and aqueous QC sample analyses are described below for all of the analytical data generated for the 2020 GLHHFFTS and the ORD-Duluth 2020 Great Lakes special study.

D1.1 Data Review

All laboratory results and calculations will be reviewed by the Laboratory Manager prior to data submission. Any errors identified during this peer review will be returned to the analyst for correction prior to submission of the data package. Following correction of the errors, the Laboratory Manager will verify that the final package is complete and compliant with the contract, and will sign each data submission to certify that the package was reviewed and determined to be in compliance with the terms and conditions of the contract.

D1.2 Data Verification

The basic goal of data verification is to ensure that project participants know what data were produced, if they are complete, if they are contractually compliant, and the extent to which they meet the objectives of the NCCA 2020 Great Lakes human health fish tissue studies. Every laboratory data package submitted for these studies (2020 GLHHFFTS and ORD-Duluth 2020 Great Lakes special study) will be subjected to data verification by qualified CSRA staff who have been trained in procedures for verifying data and who are familiar with the laboratory methods used to analyze the samples. This includes all of the mercury, PFAS, PCB congener, Aroclor, and fatty acid data generated under this QAPP and any subsequent QAPP revisions. The verification process is designed to identify and correct data deficiencies as early as possible in order to maximize the amount of usable data generated during the studies. The CSRA Project Leader will verify the summary level results for these analytical data, determine if they meet the project objectives in this QAPP, and report the verification findings to OST.

D1.3 Data Validation

Data validation is the process of evaluating the quality of the results relative to their intended use. Data need not be "perfect" to be usable for a particular project, and the validation process is designed to identify data quality issues uncovered during the verification process that may affect the intended use. One goal of validation is to answer the "So what?" question with regard to any data quality issues. CSRA data review staff will validate all of the mercury, PFAS, PCB congener, Aroclor, and fatty acid analysis results to be generated under this QAPP and any subsequent QAPP revisions.

D2. Verification and Validation Methods

D2.1 Verification Methods

In the first stage of the data verification process, the CSRA data review chemists will perform a "Data Completeness Check" in which all elements in each laboratory submission will be evaluated to verify that results for all specified samples are provided, that data are reported in the

correct format, and that all relevant information, such as preparation and analysis records, are included in the data package. Corrective action procedures will be initiated if deficiencies are noted.

The second stage of the verification process will focus on an "Instrument Performance Check" in which the CSRA data review chemists will verify that calibrations, calibration verifications, standards, and calibration blanks were analyzed at the appropriate frequency and met method or study performance specifications. If errors are noted at this stage, corrective action procedures will be initiated immediately.

Stage three of the verification process will focus on a "Laboratory Performance Check" in which the CSRA data review chemists will verify that the laboratory correctly performed the required analytical procedures and was able to demonstrate a high level of precision and accuracy. This stage includes evaluation of QC elements such as the laboratory control samples, method blanks, matrix spike samples and/or reference samples, where applicable. Corrective action procedures will be initiated with the laboratories to resolve any deficiencies identified.

In stage four of the verification process, the CSRA data review chemists will perform a "Method/Matrix Performance Check" to discern whether any QC failures are a result of laboratory performance or difficulties with the method or sample matrix. Data evaluated in this stage may include matrix spike and reference sample results. The CSRA data review chemists also will verify that proper sample dilutions were performed and that necessary sample cleanup steps were taken. If problems are encountered, the CSRA data review chemists will immediately implement corrective actions.

D2.2 Validation Methods

CSRA data review chemists will perform a data quality and usability assessment in which the overall quality of data is evaluated against the performance criteria (see Section B5 for a description of performance criteria). This assessment will strive to maximize use of data gathered in this study based on performance criteria established for these 2020 Great Lakes human health fish tissue studies. This will be accomplished by evaluating the overall quality of a particular data set rather than focusing on individual QC failures. Results of this assessment will be documented in project-specific QA reports developed after all of the results have been evaluated, and before they are used in any final decision making.

During this assessment, data qualifier flags are applied to project results to identify any results that did not meet the method- or project-specific requirements; CSRA data review chemists still may also apply additional qualifiers that indicate an assessment of the impact of the problem. For example, individual sample results are often qualified based on the presence of the analyte in a method blank associated with samples prepared together (e.g., extracted or digested in the same batch). While it is important to identify any result associated with the presence of the analyte in the blank, the relative significance of the potential for sample contamination will be assessed using commonly accepted "rules." In instances where the amount of the analyte found in the method blank has very limited potential to affect the field sample result, an additional data qualifier will be applied to that field sample result to indicate that the result was not affected by the observed blank contamination. Similar assessments made for other data quality concerns

may result in the application of additional flags that reconcile the observed data quality concerns with the user requirements and warn the end user of any limitations to the results (i.e., potential low or high bias, blank contamination, etc.). All of the data qualifiers will be included in the data file along with summary level comments that explain the implication in relatively plain English.

Where data quality concerns suggest that no valid result was produced for a given analyte, the result for the analyte will be flagged for exclusion in the project-specific databases, and the comments will provide the rationale for the exclusion. The final report of fish tissue study results generated from each database and provided to EPA will not include such invalid results, although the records marked for exclusion will be retained in the database for transparency. As noted earlier, the overall verification and validation process is designed to maximize the amount of usable data for each fish tissue study, so flagging results for exclusion in each final fish tissue study database is intended as a last resort.

D3. Reconciliation with User Requirements

The QC results for the analyses of the homogenized fish fillet tissue samples for mercury, PFAS, PCB congeners, Aroclors, and fatty acids will be assessed against the QC acceptance criteria for those respective analyses. CSRA will track laboratory performance, notify the OST Project Manager of any issues, initiate corrective actions, and track progress by each sample analysis laboratory.

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Appendix A

Target Lists of NCCA 2020 Great Lakes Human Health Whole Fish Sampling Locations

2020 Great Lakes Human Health Fish Fillet Tissue Study
Nearshore Target Sampling Locations (226)[1]

Lake	State	Site ID	Latitude	Longitude
Lake Erie	MI	NGL20_MI-10001	41.855488	-83.371811
Lake Erie	MI	NGL20_MI-10002	41.978389	-83.226068
Lake Erie	MI	NGL20_MI-10003	41.775408	-83.424598
Lake Erie	MI	NGL20_MI-10004	41.928531	-83.250994
Lake Erie	MI	NGL20_MI-10005	41.920233	-83.297047
Lake Erie	MI	NGL20_MI-10006	41.739698	-83.421714
Lake Erie	NY	NGL20_NY-10001	42.732124	-78.970972
Lake Erie	NY	NGL20_NY-10002	42.538288	-79.275335
Lake Erie	NY	NGL20_NY-10003	42.681456	-79.086131
Lake Erie	NY	NGL20_NY-10004	42.504435	-79.383478
Lake Erie	NY	NGL20_NY-10005	42.753035	-78.929682
Lake Erie	NY	NGL20_NY-10006	42.645265	-79.138888
Lake Erie	NY	NGL20_NY-10007	42.771058	-78.910310
Lake Erie	NY	NGL20_NY-10008	42.318041	-79.692515
Lake Erie	NY	NGL20_NY-10009	42.358800	-79.581425
Lake Erie	NY	NGL20_NY-10010	42.566027	-79.158291
Lake Erie	NY	NGL20_NY-10011	42.730634	-79.073758
Lake Erie	OH	NGL20_OH-10001	41.746250	-83.379173
Lake Erie	OH	NGL20_OH-10002	41.510475	-82.139115
Lake Erie	OH	NGL20_OH-10003	41.500632	-82.214543
Lake Erie	OH	NGL20_OH-10004	41.975683	-80.615216
Lake Erie	OH	NGL20_OH-10005	41.633941	-83.168247
Lake Erie	OH	NGL20_OH-10006	41.428897	-82.581778
Lake Erie	OH	NGL20_OH-10007	41.488651	-81.741749
Lake Erie	OH	NGL20_OH-10008	41.566691	-82.765201
Lake Erie	OH	NGL20_OH-10009	41.779146	-81.271315
Lake Erie	OH	NGL20_OH-10010	41.919821	-80.859975
Lake Erie	OH	NGL20_OH-10011	41.712038	-83.249070
Lake Erie	OH	NGL20_OH-10012	41.552818	-82.700842
Lake Erie	OH	NGL20_OH-10013	41.472286	-82.692721
Lake Erie	OH	NGL20_OH-10014	41.837711	-81.051557
Lake Erie	OH	NGL20_OH-10015	41.426038	-82.438730
Lake Erie	OH	NGL20_OH-10016	41.549394	-82.924082
Lake Erie	OH	NGL20_OH-10017	41.961265	-80.629612
Lake Erie	OH	NGL20_OH-10018	41.516712	-81.951277
Lake Erie	OH	NGL20_OH-10019	41.569624	-82.744472
Lake Erie	OH	NGL20_OH-10020	41.668449	-81.492888
Lake Erie	OH	NGL20_OH-10021	41.495360	-81.733241
Lake Erie	OH	NGL20_OH-10022	41.422845	-82.470374
Lake Erie	OH	NGL20_OH-10023	41.994433	-80.541792
Lake Erie	OH	NGL20_OH-10024	41.693706	-83.281403
Lake Erie	OH	NGL20_OH-10025	41.773549	-81.262087
Lake Erie	OH	NGL20_OH-10026	41.629780	-81.589718
Lake Erie	PA	NGL20_PA-10001	42.216060	-79.908285
Lake Erie	PA	NGL20_PA-10002	42.248563	-79.833229
Lake Huron	MI	NGL20_MI-10020	43.661172	-83.813745
Lake Huron	MI	NGL20_MI-10021	43.246154	-82.464205
Lake Huron	MI	NGL20_MI-10022	45.750362	-84.563951
Lake Huron	MI	NGL20_MI-10023	44.839345	-83.242500
Lake Huron	MI	NGL20_MI-10024	43.691878	-83.607161
Lake Huron	MI	NGL20_MI-10025	45.963069	-84.714304
Lake Huron	MI	NGL20_MI-10026	45.378104	-83.647971
Lake Huron	MI	NGL20_MI-10027	44.005051	-83.227579
Lake Huron	MI	NGL20_MI-10028	45.939652	-84.669392
Lake Huron	MI	NGL20_MI-10029	45.006597	-83.359057
Lake Huron	MI	NGL20_MI-10030	43.879908	-83.436643
Lake Huron	MI	NGL20_MI-10031	44.013500	-82.767393
Lake Huron	MI	NGL20_MI-10032	45.960709	-84.419150
Lake Huron	MI	NGL20_MI-10033	45.186507	-83.333887
Lake Huron	MI	NGL20_MI-10034	44.262727	-83.475720
Lake Huron	MI	NGL20_MI-10035	45.365339	-83.558984
Lake Huron	MI	NGL20_MI-10036	43.933393	-83.375104
Lake Huron	MI	NGL20_MI-10037	44.058141	-82.890460
Lake Huron	MI	NGL20_MI-10038	44.975283	-83.442236

2020 Great Lakes Human Health Fish Fillet Tissue Study
Nearshore Target Sampling Locations (226)[1]

Lake	State	Site ID	Latitude	Longitude
Lake Huron	MI	NGL20_MI-10039	43.987738	-83.643622
Lake Huron	MI	NGL20_MI-10040	43.762320	-82.615251
Lake Huron	MI	NGL20_MI-10041	45.700899	-84.357468
Lake Huron	MI	NGL20_MI-10042	44.380129	-83.305156
Lake Huron	MI	NGL20_MI-10043	43.998848	-82.682268
Lake Huron	MI	NGL20_MI-10044	45.637261	-84.224843
Lake Huron	MI	NGL20_MI-10045	44.220559	-83.436314
Lake Huron	MI	NGL20_MI-10046	44.560749	-83.269783
Lake Huron	MI	NGL20_MI-10047	43.325180	-82.522957
Lake Huron	MI	NGL20_MI-10048	45.931044	-84.370602
Lake Huron	MI	NGL20_MI-10049	43.747200	-83.482180
Lake Huron	MI	NGL20_MI-10050	43.069218	-82.418883
Lake Huron	MI	NGL20_MI-10051	45.510818	-84.053375
Lake Huron	MI	NGL20_MI-10052	44.995087	-83.402484
Lake Huron	MI	NGL20_MI-10053	43.763117	-83.450645
Lake Huron	MI	NGL20_MI-10054	43.866042	-83.384851
Lake Huron	MI	NGL20_MI-10055	45.399225	-83.640746
Lake Huron	MI	NGL20_MI-10056	44.881911	-83.263937
Lake Huron	MI	NGL20_MI-10057	43.672811	-83.905286
Lake Huron	MI	NGL20_MI-10058	44.082561	-82.990724
Lake Huron	MI	NGL20_MI-10059	44.563966	-83.305080
Lake Huron	MI	NGL20_MI-10060	43.915173	-83.840608
Lake Huron	MI	NGL20_MI-10061	43.857134	-82.597289
Lake Huron	MI	NGL20_MI-10062	44.062607	-83.575569
Lake Huron	MI	NGL20_MI-10063	45.079141	-83.300411
Lake Huron	MI	NGL20_MI-10064	45.961854	-84.254088
Lake Michigan	IL	NGL20_IL-10001	42.141568	-87.745414
Lake Michigan	IN	NGL20_IN-10001	41.663612	-87.266719
Lake Michigan	IN	NGL20_IN-10002	41.626684	-87.268386
Lake Michigan	MI	NGL20_MI-10088	45.937953	-84.994048
Lake Michigan	MI	NGL20_MI-10089	45.769089	-86.742179
Lake Michigan	MI	NGL20_MI-10090	43.375394	-86.462772
Lake Michigan	MI	NGL20_MI-10091	45.000710	-85.477045
Lake Michigan	MI	NGL20_MI-10092	44.944033	-85.840715
Lake Michigan	MI	NGL20_MI-10093	44.396029	-86.308820
Lake Michigan	MI	NGL20_MI-10094	45.888090	-86.257438
Lake Michigan	MI	NGL20_MI-10095	43.918899	-86.457437
Lake Michigan	MI	NGL20_MI-10096	42.944195	-86.246774
Lake Michigan	MI	NGL20_MI-10097	45.797503	-84.792273
Lake Michigan	MI	NGL20_MI-10098	43.102397	-86.271767
Lake Michigan	MI	NGL20_MI-10099	45.934508	-85.719973
Lake Michigan	MI	NGL20_MI-10100	45.097762	-85.699194
Lake Michigan	MI	NGL20_MI-10101	44.312549	-86.299545
Lake Michigan	MI	NGL20_MI-10102	46.051344	-85.241341
Lake Michigan	MI	NGL20_MI-10103	44.678209	-86.261122
Lake Michigan	MI	NGL20_MI-10104	45.128687	-87.553117
Lake Michigan	MI	NGL20_MI-10105	45.772779	-86.809326
Lake Michigan	MI	NGL20_MI-10106	42.327680	-86.334775
Lake Michigan	MI	NGL20_MI-10107	46.023363	-85.195092
Lake Michigan	MI	NGL20_MI-10108	45.708931	-87.015603
Lake Michigan	MI	NGL20_MI-10109	45.107563	-85.596550
Lake Michigan	MI	NGL20_MI-10110	42.629003	-86.255722
Lake Michigan	MI	NGL20_MI-10111	45.667295	-86.518665
Lake Michigan	MI	NGL20_MI-10112	46.068962	-85.381571
Lake Michigan	MI	NGL20_MI-10113	45.590173	-87.221718
Lake Michigan	MI	NGL20_MI-10114	43.778261	-86.456345
Lake Michigan	MI	NGL20_MI-10115	41.884623	-86.630807
Lake Michigan	WI	NGL20_WI-10001	45.029147	-87.093383
Lake Michigan	WI	NGL20_WI-10002	42.614701	-87.810577
Lake Michigan	WI	NGL20_WI-10003	43.328918	-87.864071
Lake Michigan	WI	NGL20_WI-10004	44.948027	-87.698607
Lake Michigan	WI	NGL20_WI-10005	45.336088	-86.956190
Lake Michigan	WI	NGL20_WI-10006	43.719069	-87.657049
Lake Michigan	WI	NGL20_WI-10007	43.331739	-87.865814

2020 Great Lakes Human Health Fish Fillet Tissue Study
Nearshore Target Sampling Locations (226)[1]

Lake	State	Site ID	Latitude	Longitude
Lake Michigan	WI	NGL20_WI-10008	44.513063	-87.479199
Lake Michigan	WI	NGL20_WI-10009	44.863781	-87.481783
Lake Michigan	WI	NGL20_WI-10010	44.132949	-87.500974
Lake Michigan	WI	NGL20_WI-10011	45.188723	-86.979138
Lake Michigan	WI	NGL20_WI-10012	43.653379	-87.685584
Lake Michigan	WI	NGL20_WI-10013	42.798074	-87.717127
Lake Michigan	WI	NGL20_WI-10014	44.621561	-87.961059
Lake Ontario	NY	NGL20_NY-10032	43.968268	-76.115404
Lake Ontario	NY	NGL20_NY-10033	43.913595	-76.183412
Lake Ontario	NY	NGL20_NY-10034	43.358199	-78.702733
Lake Ontario	NY	NGL20_NY-10035	43.337974	-77.673215
Lake Ontario	NY	NGL20_NY-10036	43.506222	-76.487717
Lake Ontario	NY	NGL20_NY-10037	43.912188	-76.284124
Lake Ontario	NY	NGL20_NY-10038	43.311974	-78.888999
Lake Ontario	NY	NGL20_NY-10039	43.254800	-77.488727
Lake Ontario	NY	NGL20_NY-10040	43.587591	-76.250649
Lake Ontario	NY	NGL20_NY-10041	44.075882	-76.376997
Lake Ontario	NY	NGL20_NY-10042	43.381275	-78.085324
Lake Ontario	NY	NGL20_NY-10043	43.431453	-76.627178
Lake Ontario	NY	NGL20_NY-10044	43.803382	-76.251820
Lake Ontario	NY	NGL20_NY-10045	44.005321	-76.185961
Lake Ontario	NY	NGL20_NY-10046	43.361377	-77.930974
Lake Ontario	NY	NGL20_NY-10047	43.319131	-76.879006
Lake Ontario	NY	NGL20_NY-10048	43.334502	-78.808970
Lake Ontario	NY	NGL20_NY-10049	43.256623	-77.569462
Lake Ontario	NY	NGL20_NY-10050	43.470100	-76.579153
Lake Ontario	NY	NGL20_NY-10051	43.380115	-78.595466
Lake Ontario	NY	NGL20_NY-10052	43.294845	-77.351292
Lake Ontario	NY	NGL20_NY-10053	43.684236	-76.239633
Lake Ontario	NY	NGL20_NY-10054	43.313321	-78.918995
Lake Ontario	NY	NGL20_NY-10055	43.985167	-76.060517
Lake Ontario	NY	NGL20_NY-10056	43.822188	-76.317569
Lake Ontario	NY	NGL20_NY-10057	43.340606	-77.703826
Lake Ontario	NY	NGL20_NY-10058	43.331946	-78.852610
Lake Ontario	NY	NGL20_NY-10059	43.684749	-76.220903
Lake Ontario	NY	NGL20_NY-10060	43.972557	-76.335932
Lake Ontario	NY	NGL20_NY-10061	43.288035	-77.553622
Lake Ontario	NY	NGL20_NY-10062	43.373254	-78.324593
Lake Ontario	NY	NGL20_NY-10063	43.546523	-76.314592
Lake Ontario	NY	NGL20_NY-10064	44.144023	-76.327305
Lake Ontario	NY	NGL20_NY-10065	43.516782	-76.433882
Lake Ontario	NY	NGL20_NY-10066	43.330161	-78.766097
Lake Ontario	NY	NGL20_NY-10067	43.913117	-76.242845
Lake Ontario	NY	NGL20_NY-10068	43.278913	-77.545576
Lake Ontario	NY	NGL20_NY-10069	43.332665	-76.863334
Lake Ontario	NY	NGL20_NY-10070	43.794633	-76.246004
Lake Ontario	NY	NGL20_NY-10071	43.986909	-76.206235
Lake Ontario	NY	NGL20_NY-10072	43.436884	-76.619956
Lake Ontario	NY	NGL20_NY-10073	43.387118	-77.986445
Lake Ontario	NY	NGL20_NY-10074	43.347117	-77.755232
Lake Ontario	NY	NGL20_NY-10075	44.059480	-76.369895
Lake Ontario	NY	NGL20_NY-10076	43.298707	-77.100180
Lake Superior	MI	NGL20_MI-10130	47.388639	-87.924763
Lake Superior	MI	NGL20_MI-10131	46.532907	-87.389569
Lake Superior	MI	NGL20_MI-10132	46.887188	-88.324722
Lake Superior	MI	NGL20_MI-10133	47.283798	-88.517410
Lake Superior	MI	NGL20_MI-10134	46.685295	-86.169696
Lake Superior	MI	NGL20_MI-10135	46.924509	-87.843784
Lake Superior	MI	NGL20_MI-10136	46.793415	-85.233590
Lake Superior	MI	NGL20_MI-10137	47.042887	-88.981274
Lake Superior	MI	NGL20_MI-10138	46.512006	-87.148603
Lake Superior	MI	NGL20_MI-10139	46.589141	-85.020580
Lake Superior	MI	NGL20_MI-10140	46.487506	-86.740911
Lake Superior	MI	NGL20_MI-10141	46.720289	-85.762836

2020 Great Lakes Human Health Fish Fillet Tissue Study
Nearshore Target Sampling Locations (226)[1]

Lake	State	Site ID	Latitude	Longitude
Lake Superior	MI	NGL20_MI-10142	46.730769	-89.968199
Lake Superior	MI	NGL20_MI-10143	46.846226	-89.573089
Lake Superior	MI	NGL20_MI-10144	46.686942	-85.506656
Lake Superior	MI	NGL20_MI-10145	46.582070	-90.406321
Lake Superior	MI	NGL20_MI-10146	46.898957	-89.388481
Lake Superior	MI	NGL20_MI-10147	46.708512	-85.708076
Lake Superior	MI	NGL20_MI-10148	46.918146	-89.272481
Lake Superior	MI	NGL20_MI-10149	47.393787	-87.701592
Lake Superior	MI	NGL20_MI-10150	47.296755	-88.548888
Lake Superior	MI	NGL20_MI-10151	46.508743	-87.146502
Lake Superior	MI	NGL20_MI-10152	46.604595	-90.378582
Lake Superior	MI	NGL20_MI-10153	46.839705	-88.262902
Lake Superior	MI	NGL20_MI-10154	46.485322	-84.958745
Lake Superior	MI	NGL20_MI-10155	46.689560	-86.114568
Lake Superior	MI	NGL20_MI-10156	46.967084	-89.234385
Lake Superior	MI	NGL20_MI-10157	46.876538	-87.730033
Lake Superior	MI	NGL20_MI-10158	46.790886	-85.026494
Lake Superior	MI	NGL20_MI-10159	47.259431	-88.643379
Lake Superior	MI	NGL20_MI-10160	46.494254	-86.608618
Lake Superior	MN	NGL20_MN-10001	47.141140	-91.450357
Lake Superior	MN	NGL20_MN-10002	47.556276	-90.867735
Lake Superior	MN	NGL20_MN-10003	46.790489	-92.044779
Lake Superior	MN	NGL20_MN-10004	47.771646	-90.180874
Lake Superior	MN	NGL20_MN-10005	47.480891	-90.983105
Lake Superior	MN	NGL20_MN-10006	47.973099	-89.635062
Lake Superior	MN	NGL20_MN-10007	46.793050	-91.995742
Lake Superior	MN	NGL20_MN-10008	47.062637	-91.592607
Lake Superior	MN	NGL20_MN-10009	47.727800	-90.426800
Lake Superior	WI	NGL20_WI-10022	46.770510	-91.622243
Lake Superior	WI	NGL20_WI-10023	46.672803	-90.816963
Lake Superior	WI	NGL20_WI-10024	46.729253	-91.787980
Lake Superior	WI	NGL20_WI-10025	46.680720	-90.632506
Lake Superior	WI	NGL20_WI-10026	46.754410	-91.731229
Lake Superior	WI	NGL20_WI-10027	46.666176	-90.692698

[1] This list of sites is subject to change as the project proceeds. For example, access to some sites may not be granted by property owners. Other sites may not yield fish of suitable size or species. OST maintains the list of valid sites, and this QAPP will **not** be revised just to address changes in the list of sites.

ORD-Duluth 2020 Great Lakes Special Study
Lake Michigan Enhancement Target
Sampling Locations (50)[1]

Lake	State	Site ID	Latitude	Longitude
Lake Michigan	MI	ISA20-01	45.022576	-85.955361
Lake Michigan	MI	ISA20-02	44.992944	-86.143602
Lake Michigan	MI	ISA20-03	45.810411	-85.536973
Lake Michigan	MI	ISA20-04	45.732114	-85.586710
Lake Michigan	MI	ISA20-05	45.100510	-86.075701
Lake Michigan	MI	ISA20-06	45.754047	-85.396307
Lake Michigan	MI	ISA20-07	45.744411	-85.518414
Lake Michigan	MI	ISA20-08	45.406036	-85.865609
Lake Michigan	MI	ISA20-09	45.373191	-86.950673
Lake Michigan	MI	ISA20-10	45.806034	-85.336755
Lake Michigan	MI	ISA20-11	45.690690	-85.460965
Lake Michigan	MI	ISA20-12	45.457843	-85.912753
Lake Michigan	MI	ISA20-13	45.386882	-86.829426
Lake Michigan	MI	ISA20-14	45.821972	-85.334852
Lake Michigan	MI	ISA20-15	45.627184	-85.633541
Lake Michigan	MI	ISA20-16	45.161108	-87.409663
Lake Michigan	MI	ISA20-17	44.994629	-86.163159
Lake Michigan	MI	ISA20-18	45.740626	-85.583030
Lake Michigan	MI	ISA20-19	45.071318	-86.101963
Lake Michigan	MI	ISA20-20	45.733368	-85.438622
Lake Michigan	MI	ISA20-21	45.780002	-85.549848
Lake Michigan	MI	ISA20-22	45.367833	-85.835910
Lake Michigan	MI	ISA20-23	45.418739	-86.835769
Lake Michigan	MI	ISA20-24	45.803657	-85.395611
Lake Michigan	MI	ISA20-25	45.600707	-85.478461
Lake Michigan	MI	ISA20-26	45.243421	-87.297889
Lake Michigan	MI	ISA20-27	45.333378	-86.809511
Lake Michigan	MI	ISA20-28	45.798403	-85.577589
Lake Michigan	MI	ISA20-29	45.748655	-85.684087
Lake Michigan	MI	ISA20-30	45.164952	-87.285285
Lake Michigan	MI	ISA20-31	45.032575	-86.005719
Lake Michigan	MI	ISA20-32	45.758626	-85.313298
Lake Michigan	MI	ISA20-33	45.791583	-85.527481
Lake Michigan	MI	ISA20-34	45.577667	-85.641267
Lake Michigan	MI	ISA20-35	45.037425	-85.997924
Lake Michigan	MI	ISA20-36	45.742621	-85.459929
Lake Michigan	MI	ISA20-37	45.321337	-86.892611
Lake Michigan	MI	ISA20-38	45.829346	-85.393017
Lake Michigan	MI	NPA20-01	44.914807	-86.099659
Lake Michigan	MI	NPA20-02	44.799368	-86.090591
Lake Michigan	IN	NPA20-03	41.646178	-87.217218
Lake Michigan	IN	NPA20-04	41.699221	-87.039672
Lake Michigan	MI	NPA20-05	44.859089	-86.088796
Lake Michigan	MI	NPA20-06	44.749306	-86.096894
Lake Michigan	IN	NPA20-07	41.632908	-87.216226
Lake Michigan	IN	NPA20-08	41.699639	-87.078886
Lake Michigan	MI	NPA20-09	44.772349	-86.171933
Lake Michigan	MI	NPA20-10	44.971318	-85.896002
Lake Michigan	IN	NPA20-11	41.635451	-87.266269
Lake Michigan	IN	NPA20-12	41.699258	-87.087526

[1] This list of sites is subject to change as the project proceeds. For example, access to some sites may not be granted by property owners. Other sites may not yield fish of suitable size or species. OST maintains the list of valid sites, and this QAPP will **not** be revised just to address changes in the list of sites.

Appendix B

2020 NCCA
Detection and Quantitation Limits for Great Lakes Human Health Fish Fillet Tissue Analyses

Method Detection Limits (MDLs) and Minimum Levels (MLs) for 2020 NCCA Great Lakes Fish Fillet Tissue Target Analytes

Mercury MDL and ML *(based on a 0.5-g sample)*

MDL[1] (ng/g)	**ML** (ng/g)
0.2	1

[1] The MDL is based on the EPA procedure described at 40 CFR 136, Appendix B, Revision 2, from August 2017.

The PFAS analytes to be determined in this project are listed in the table below, along with their common abbreviations. The method detection and quantitation limits (also referred to as minimum levels) were provided by the laboratory as part of its bid submission.

PFAS MDLs and MLs *(based on a 2-g tissue sample and a 250-mL rinsate sample)*

		Tissue Samples (ng/g)		**Rinsate Samples (ng/L)**	
Name	**Abbreviation**	**MDL**[1]	**ML**	**MDL**[1]	**ML**
Perfluorobutanoic acid	PFBA	0.593	0.8	0.661	6.4
Perfluoropentanoic acid	PFPeA	0.083	0.4	0.392	3.2
Perfluorohexanoic acid	PFHxA	0.096	0.2	0.636	1.6
Perfluoroheptanoic acid	PFHpA	0.088	0.2	0.443	1.6
Perfluorooctanoic acid	PFOA	0.086	0.2	0.604	1.6
Perfluorononanoic acid	PFNA	0.160	0.2	0.442	1.6
Perfluorodecanoic acid	PFDA	0.124	0.2	0.666	1.6
Perfluoroundecanoic acid	PFUnA	0.152	0.2	0.527	1.6
Perfluorododecanoic acid	PFDoA	0.130	0.2	0.758	1.6
Perfluorotridecanoic acid	PFTrDA	0.086	0.2	0.476	1.6
Perfluorotetradecanoic acid	PFTeDA	0.185	0.2	0.527	1.6
Perfluorobutanesulfonic acid	PFBS	0.070	0.2	0.491	1.6
Perfluoropentanesulfonic acid	PFPeS	0.032	0.2	0.407	1.6
Perfluorohexanesulfonic acid	PFHxS	0.083	0.2	0.434	1.6
Perfluoroheptanesulfonic acid	PFHpS	0.043	0.2	0.274	1.6
Perfluorooctanesulfonic acid	PFOS	0.294	0.3	0.654	1.6
Perfluorononanesulfonic acid	PFNS	0.114	0.2	0.606	1.6
Perfluorodecanesulfonic acid	PFDS	0.101	0.2	0.668	1.6
Perfluorododecanesulfonic acid	PFDoS	0.177	0.2	0.358	1.6
1H, 1H, 2H, 2H-perfluorohexane sulfonic acid	4:2 FTS	0.740	0.8	4.561	6.4
1H, 1H, 2H, 2H-perfluorooctane sulfonic acid	6:2 FTS	1.149	1.3	7.946	8.7
1H, 1H, 2H, 2H-perfluorodecane sulfonic acid	8:2 FTS	0.373	0.8	3.132	6.4
2H, 2H, 3H, 3H-perfluorohexanoic acid	3:3 FTCA	0.247	0.8	1.441	6.4
2H, 2H, 3H, 3H-perfluorooctanoic acid	5:3 FTCA	1.537	5.0	10.133	40.0
2H, 2H, 3H, 3H-perfluorodecanoic acid	7:3 FTCA	0.845	5.0	11.885	40.0
Perfluorooctanesulfonamide	PFOSA	0.094	0.2	0.454	1.6
N-Methylperfluorooctanesulfonamide	N-MeFOSA	0.161	0.2	0.392	1.8
N-Ethylperfluorooctanesulfonamide	N-EtFOSA	0.169	0.5	1.169	4.0

PFAS MDLs and MLs *(based on a 2-g tissue sample and a 250-mL rinsate sample)*

Name	Abbreviation	Tissue Samples (ng/g)		Rinsate Samples (ng/L)	
		MDL[1]	ML	MDL[1]	ML
N-Methylperfluoro-1-octanesulfonamidoacetic acid	N-MeFOSAA	0.930	0.2	0.650	0.0
N-Ethylperfluoro-1-octanesulfonamidoacetic acid	N-EtFOSAA	0.138	0.2	2.040	12.0
N-Methylperfluoro-1-octanesulfonamidoethanol	N-MeFOSE	9.977	11.0	2.383	16.0
N-Ethylperfluoro-1-octanesulfonamidoethanol	N-EtFOSE	1.500	1.7	2.043	12.0
2,3,3,3-Tetrafluoro-2-(1,1,2,2,3,3,3-heptafluoropropoxy)propionic acid	HFPO-DA	0.161	0.8	0.811	6.1
4,8-dioxa-3H-perfluorononanoic acid	ADONA	0.082	0.8	1.558	6.4
Perfluoro-3,6-dioxaheptanoic acid	NFDHA	0.294	0.4	2.768	3.2
Perfluoro-3-methoxypropanoic acid	PFMPA	0.070	0.4	0.354	3.2
Perfluoro-4-methoxybutanoic acid	PFMBA	0.069	0.2	0.233	1.6
9-chlorohexadecafluoro-3-oxanonane-1-sulfonic acid	9Cl-PF3ONS	0.152	0.8	1.742	6.4
11-chloroeicosafluoro-3-oxaundecane-1-sulfonic acid	11Cl-PF3OUdS	0.312	0.8	1.639	6.4
Perfluoro(2-ethoxyethane)sulfonic acid	PFEESA	0.045	0.2	0.274	1.6

[1] The MDL is based on the EPA procedure described at 40 CFR 136, Appendix B, Revision 2, from August 2017.

PFAS analytes in NARS fish tissue studies from 2008 to 2015
PFAS analytes new for 2018-19 NRSA Fish Tissue Study
PFAS analytes new to this study (2020 GLHHFFTS)

The PCB congeners to be determined in this project are listed in the table below. The method detection and quantitation limits (also referred to as minimum levels) were provided by the laboratory as part of its bid submission.

PCB MDLs and MLs in pg/g
(in elution order, based on a 10-g sample)

Analyte	MDL[1]	ML
PCB-1	0.14	1
PCB-2	0.13	1
PCB-3	0.22	1
PCB-4/10	0.29	1
PCB-5/8	0.39	1
PCB-6	0.21	1
PCB-7/9	0.21	1
PCB-11	1.13	1
PCB-12/13	0.24	1
PCB-14	0.14	1
PCB-15	0.11	1
PCB-16/32	0.34	1
PCB-17	0.19	1
PCB-18	0.30	1
PCB-19	0.28	1
PCB-20/21/33	0.74	2
PCB-22	0.21	1
PCB-23	0.20	1

PCB MDLs and MLs in pg/g

(in elution order, based on a 10-g sample)

Analyte	MDL[1]	ML
PCB-24/27	0.39	1
PCB-25	0.18	1
PCB-26	0.25	1
PCB-28	0.36	1
PCB-29	0.21	1
PCB-30	0.20	1
PCB-31	0.41	1
PCB-34	0.17	1
PCB-35	0.17	1
PCB-36	0.14	1
PCB-37	0.15	1
PCB-38	0.14	1
PCB-39	0.18	1
PCB-40	0.24	1
PCB-41/64/71/72	0.62	2
PCB-42/59	0.40	1
PCB-43/49	0.28	1
PCB-44	0.17	1
PCB-45	0.28	1
PCB-46	0.19	1
PCB-47	0.64	1
PCB-48/75	0.21	1
PCB-50	0.25	1
PCB-51	0.20	1
PCB-52/69	0.42	1
PCB-53	0.31	1
PCB-54	0.15	1
PCB-55	0.24	1
PCB-56/60	0.25	1
PCB-57	0.08	1
PCB-58	0.10	1
PCB-61/70	0.24	1
PCB-62	0.26	1
PCB-63	0.18	1
PCB-65	0.29	1
PCB-66/76	0.30	1
PCB-67	0.29	1
PCB-68	0.16	1
PCB-73	0.16	1
PCB-74	0.28	1
PCB-77	0.14	1
PCB-78	0.13	1
PCB-79	0.12	1
PCB-80	0.19	1
PCB-81	0.13	1
PCB-82	0.32	1

PCB MDLs and MLs in pg/g

(in elution order, based on a 10-g sample)

Analyte	MDL[1]	ML
PCB-83	0.29	1
PCB-84/92	0.40	1
PCB-85/116	0.46	1
PCB-86	0.43	1
PCB-87/117/125	0.52	2
PCB-88/91	0.66	1
PCB-89	0.22	1
PCB-90/101	0.32	1
PCB-93	0.52	1
PCB-94	0.26	1
PCB-95/98/102	0.59	2
PCB-96	0.32	1
PCB-97	0.25	1
PCB-99	0.29	1
PCB-100	0.17	1
PCB-103	0.24	1
PCB-104	0.17	1
PCB-105	0.16	1
PCB-106/118	0.41	1
PCB-107/109	0.31	1
PCB-108/112	0.46	1
PCB-110	0.37	1
PCB-111/115	0.31	1
PCB-113	0.24	1
PCB-114	0.24	1
PCB-119	0.16	1
PCB-120	0.18	1
PCB-121	0.41	1
PCB-122	0.21	1
PCB-123	0.15	1
PCB-124	0.24	1
PCB-126	0.16	1
PCB-127	0.13	1
PCB-128/162	0.33	1
PCB-129	0.13	1
PCB-130	0.23	1
PCB-131/133	0.32	1
PCB-132/161	0.29	1
PCB-134/143	0.39	1
PCB-135	0.30	1
PCB-136	0.17	1
PCB-137	0.22	1
PCB-138/163/164	0.61	2
PCB-139/149	0.72	1
PCB-140	0.31	1
PCB-141	0.22	1

PCB MDLs and MLs in pg/g

(in elution order, based on a 10-g sample)

Analyte	MDL[1]	ML
PCB-142	0.22	1
PCB-144	0.18	1
PCB-145	0.35	1
PCB-146/165	0.31	1
PCB-147	0.17	1
PCB-148	0.43	1
PCB-150	0.25	1
PCB-151	0.16	1
PCB-152	0.21	1
PCB-153	0.37	1
PCB-154	0.35	1
PCB-155	0.34	1
PCB-156	0.14	1
PCB-157	0.12	1
PCB-158/160	0.32	1
PCB-159	0.10	1
PCB-166	0.14	1
PCB-167	0.17	1
PCB-168	0.14	1
PCB-169	0.22	1
PCB-170	0.37	1
PCB-171	0.35	1
PCB-172	0.26	1
PCB-173	0.20	1
PCB-174	0.29	1
PCB-175	0.16	1
PCB-176	0.14	1
PCB-177	0.21	1
PCB-178	0.26	1
PCB-179	0.22	1
PCB-180	0.49	1
PCB-181	0.23	1
PCB-182/187	0.36	1
PCB-183	0.18	1
PCB-184	0.16	1
PCB-185	0.18	1
PCB-186	0.18	1
PCB-188	0.17	1
PCB-189	0.22	1
PCB-190	0.29	1
PCB-191	0.16	1
PCB-192	0.26	1
PCB-193	0.27	1
PCB-194	0.15	1
PCB-195	0.22	1
PCB-196/203	0.55	1

PCB MDLs and MLs in pg/g
(in elution order, based on a 10-g sample)

Analyte	MDL[1]	ML
PCB-197	0.24	1
PCB-198	0.37	1
PCB-199	0.42	1
PCB-200	0.35	1
PCB-201	0.21	1
PCB-202	0.22	1
PCB-204	0.23	1
PCB-205	0.16	1
PCB-206	0.23	1
PCB-207	0.21	1
PCB-208	0.19	1
PCB-209	0.27	1

[1] The Vista Analytical Laboratory MDLs are based on Revision 2 of the MDL procedure published in August 2017.

The Aroclors to be determined in this project are listed in the table below. The method detection and quantitation limits (also referred to as minimum levels) were provided by the laboratory as part of its bid submission.

Aroclor MDLs and MLs in ng/g
(based on a 10-g sample)

Analyte	MDL[1]	ML[2]
Aroclor 1016	0.5568	2.5
Aroclor 1221	0.8868	2.5
Aroclor 1232	0.6063	2.5
Aroclor 1242	0.3642	2.5
Aroclor 1248	0.6057	2.5
Aroclor 1254	0.7476	2.5
Aroclor 1260	0.7437	2.5
Aroclor 1268	0.8778	2.5

[1] The Eurofins-TestAmerica MDLs are based on Revision 2 of the MDL procedure published in August 2017.
[2] The Eurofins-TestAmerica ML values are based on the concentration of the lowest calibration standard analyzed.

The fatty acids to be determined in this project are listed in the table below, along with the method detection and quantitation limits that were provided by the laboratory as part of its bid submission. The quantitation limits (QLs) in the table below are three times the MDL and rounded to two decimal places.

Fatty Acid MDLs and QLs in µg/g
(based on a 0.5-g sample)

Fatty Acid	Lipid Number Abbreviation*	MDL	QL
Decanoic acid	C10:0	3.00	9.00
Undecanoic acid	C11:0	3.00	9.00
Dodecanoic acid	C12:0	3.00	9.00
Tridecanoic acid	C13:0	0.09	0.27
Myristic acid	C14:0	1.89	5.67
Myristoleic acid	C14:1	0.18	0.54
Pentadecanoic acid	C15:0	0.36	1.08
cis-10-Pentadecenoic acid	C15:1	0.09	0.27
Palmitic acid	C16:0	6.72	20.16
cis-7-Hexadecenoic acid	C16:1	7.14	21.42
Heptadecanoic acid	C17:0	0.33	0.99
ccis-10-Heptadecenoic acid	C17:1	0.09	0.27
Stearic acid	C18:0	0.69	2.07
Elaidic acid	C18:1n9 *trans*	0.30	0.90
Oleic acid	C18:1n9 *cis*	10.6	31.8
cis-vaccenic acid	C18:1w7	2.26	6.78
Linolelaidic acid	C18:2 *trans*	0.93	2.79
Linoleic acid	C18:2n6 *cis*	1.60	4.80
gamma-Linolenic acid	C18:3n6	0.34	1.02
Arachidic acid	C20:0	0.09	0.27
Linolenic acid	C18:3n3	3.50	10.50
Eicosenoic acid	C20:1n9	3.50	10.50
Octadecatetraenoic acid	C18:4n3	3.50	10.50
Heneicosanoic acid	C21:0	0.12	0.36
Eicosadienoic acid	C20:2	0.16	0.48
Dihomo-*gamma*-linolenic acid	C20:3n6	0.32	0.96
Behenic acid	C22:0	0.09	0.27
Eicosatrienoic acid	C20:3n3	2.04	6.12
Arachidonic acid	C20:4n6	1.67	5.01
Cetoleic acid	C22:1n11	0.14	0.42
Erucic acid	C22:1n9	0.14	0.42
Tricosanoic acid	C23:0	0.15	0.45
13,16-Docosadienoic acid	C22:2	1.84	5.52
Eicosapentaenoic acid	C20:5n3	0.20	0.60
Lignoceric acid	C24:0	0.09	0.27
Nervonic acid	C24:1n9	0.63	1.89
Docosapentaenoic acid	C22:5n3	2.49	7.47
Docosahexaenoic acid	C22:6n3	2.49	7.47

* Lipid numbers take the form C:DnX, where C is the number of carbon atoms in the fatty acid and D is the number of double bonds. Where applicable, the fatty acid double bond location is identified by nX, where X is the carbon number of the first double bond relative to the terminal alkyl end of the fatty acid.

Appendix C

2020 NCCA Quality Control (QC) Acceptance Criteria for PFAS Analyses of Great Lakes Fish Fillet Tissue Samples and QC Rinsate Samples

Calibration Verification (CCV), LCS, and Labeled Compound Recovery QC Acceptance Criteria for PFAS Analyses

Analyte	CCV (%)	LCS Recovery (%)		Labeled Compound Recovery in Samples (%)	
		Tissues	Rinsates	Tissues	Rinsates
Target Analytes					
PFBA	70 - 130	70 - 130	70 - 130	NA	NA
PFPeA		70 - 130	70 - 130		
PFHxA		70 - 130	70 - 130		
PFHpA		70 - 130	70 - 130		
PFOA		70 - 130	70 - 130		
PFNA		70 - 130	70 - 130		
PFDA		60 - 130	70 - 130		
PFUnA		70 - 140	70 - 130		
PFDoA		70 - 130	70 - 130		
PFTrDA		70 - 130	70 - 130		
PFTeA		70 - 130	70 - 130		
PFBS		60 - 130	70 - 130		
PFPeS		70 - 130	70 - 130		
PFHxS		70 - 130	70 - 130		
PFHpS		70 - 130	70 - 130		
PFOS		70 - 140	70 - 130		
PFNS		60 - 150	70 - 130		
PFDS		40 - 150	70 - 130		
PFDoS		70 - 140	60 - 130		
4:2 FTS		40 - 150	70 - 130		
6:2 FTS		70 - 130	70 - 130		
8:2 FTS		70 - 170	70 - 130		
3:3 FTCA		70 - 130	65 - 130		
5:3 FTCS		70 - 180	70 - 130		
7:3 FTCA		70 - 130	70 - 130		
PFOSA		70 - 130	70 - 130		
N-MeFOSA		50 - 140	70 - 130		
N-EtFOSA		70 - 130	70 - 130		
N-MeFOSAA		60 - 160	70 - 130		
N-EtFOSAA		60 - 160	70 - 130		
N-MeFOSE		70 - 150	70 - 130		
N-EtFOSE		70 - 130	70 - 130		
HFPO-DA		70 - 130	70 - 130		
ADONA		70 - 130	70 - 130		
NFDHA		60 - 130	65 - 140		
PFMPA		70 - 130	70 - 130		
PFMBA		70 - 130	70 - 130		
9Cl-PF3ONS		70 - 130	70 - 130		
11Cl-PF3OUDS		60 - 130	70 - 130		
PFEESA		70 - 130	70 - 130		

Analyte	CCV (%)	LCS Recovery (%)		Labeled Compound Recovery in Samples (%)	
		Tissues	Rinsates	Tissues	Rinsates
Labeled Compounds					
$^{13}C_4$-PFBA	50 - 150	50 - 150	50 - 150	50 - 150	50 - 150
$^{13}C_5$-PFPeA		50 - 150	50 - 150	50 - 150	50 - 150
$^{13}C_5$-PFHxA		50 - 150	50 - 150	50 - 150	50 - 150
$^{13}C_4$-PFHpA		50 - 150	50 - 150	50 - 150	50 - 150
$^{13}C_6$-PFOA		50 - 150	50 - 150	50 - 150	50 - 150
$^{13}C_9$-PFNA		50 - 150	50 - 150	50 - 150	50 - 150
$^{13}C_6$-PFDA		50 - 180	50 - 150	50 - 180	50 - 150
$^{13}C_7$-PFUnA		50 - 150	50 - 150	50 - 150	50 - 150
$^{13}C_2$-PFDoA		50 - 150	50 - 150	50 - 150	50 - 150
$^{13}C_2$-PFTeA		50 - 150	50 - 150	50 - 150	50 - 150
$^{13}C_3$-PFBS	50 - 150	50 - 150	50 - 150	50 - 150	50 - 150
$^{13}C_3$-PFHxS		50 - 150	50 - 150	50 - 150	50 - 150
$^{13}C_8$-PFOS		50 - 150	50 - 150	50 - 150	50 - 150
$^{13}C_2$-4:2 FTS		50 - 220	50 - 150	50 - 220	50 - 150
$^{13}C_2$-6:2 FTS		50 - 180	50 - 150	50 - 180	50 - 150
$^{13}C_2$-8:2 FTS		50 - 300	50 - 150	50 - 300	50 - 150
$^{13}C_8$-PFOSA		50 - 150	50 - 150	50 - 150	50 - 150
D_3-N-MeFOSA		5 - 150	30 - 150	20 - 200	30 - 150
D_5-N-EtFOSA		10 - 150	20 - 150	20 - 200	20 - 150
D_3-N-MeFOSAA		50 - 180	50 - 150	20 - 200	50 - 150
D_5-N-EtFOSAA		50 - 250	50 - 150	20 - 200	50 - 150
D_7-N-MeFOSE		2 - 150	30 - 150	20 - 200	30 - 150
D_9-N-EtFOSE		2 - 150	30 - 150	20 - 200	30 - 150
$^{13}C_3$-HFPO-DA		50 - 150	50 - 150	50 - 150	50 - 150

Appendix D

2020 NCCA Quality Control (QC) Acceptance Criteria for PCB Congener Analysis of Great Lakes Fish Fillet Tissue Samples

Calibration Verification Limits (%), Laboratory Control Sample Recovery Limits (%), and Labeled Compound Recovery Limits (%) for PCB Congener Analyses

QC Acceptance Criteria for VER[1], OPR[2], and Labeled Compounds[3] in Samples				
Congener Name	**Congener Number**	**VER (%)**	**OPR Recovery (%)**	**Labeled Compound Recovery in Samples (%)**
2-MonoCB	1	75-125	60-135	
3-MonoCB	2	75-125	60-135	
4-MonoCB	3	75-125	60-135	
2,2'-DiCB/2,6-DiCB	4/10	75-125	60-135	
2,3-DiCB/2,4'-DiCB	5/8	75-125	60-135	
2,3'-DiCB	6	75-125	60-135	
2,4-DiCB/2,5-DiCB	7/9	75-125	60-135	
3,3'-DiCB	11	75-125	60-135	
3,4-DiCB/3,4'-DiCB	12/13	75-125	60-135	
3,5-DiCB	14	75-125	60-135	
4,4'-DiCB	15	75-125	60-135	
2,2',3-TrCB/2,4',6-TrCB	16/32	75-125	60-135	
2,2',4-TrCB	17	75-125	60-135	
2,2',5-TrCB	18	75-125	60-135	
2,2',6-TrCB	19	75-125	60-135	
2,3,3'-TrCB/2,3,4-TrCB/2',3,4-TrCB	20/21/33	75-125	60-135	
2,3,4'-TrCB	22	75-125	60-135	
2,3,5-TrCB	23	75-125	60-135	
2,3,6-TrCB/2,3',6-TrCB	24/27	75-125	60-135	
2,3',4-TrCB	25	75-125	60-135	
2,3',5-TrCB	26	75-125	60-135	
2,4,4'-TrCB	28	75-125	60-135	
2,4,5-TrCB	29	75-125	60-135	
2,4,6-TrCB	30	75-125	60-135	
2,4',5-TrCB	31	75-125	60-135	
2,3',5'-TrCB	34	75-125	60-135	
3,3',4-TrCB	35	75-125	60-135	NA
3,3',5-TrCB	36	75-125	60-135	
3,4,4'-TrCB	37	75-125	60-135	
3,4,5-TrCB	38	75-125	60-135	
3,4',5-TrCB	39	75-125	60-135	
2,2',3,3'-TeCB	40	75-125	60-135	
2,2',3,4-TeCB/2,3,4',6-TeCB/2,3',4',6-TeCB/2,3',5,5'-TeCB	41/64/71/72	75-125	60-135	
2,2',3,4'-TeCB/2,3,3',6-TeCB	42/59	75-125	60-135	
2,2',3,5-TeCB/2,2',4,5'-TeCB	43/49	75-125	60-135	
2,2',3,5'-TeCB	44	75-125	60-135	
2,2',3,6-TeCB	45	75-125	60-135	
2,2',3,6'-TeCB	46	75-125	60-135	
2,2',4,4'-TeCB	47	75-125	60-135	
2,2',4,5-TeCB/2,4,4',6-TeCB	48/75	75-125	60-135	
2,2',4,6-TeCB	50	75-125	60-135	
2,2',4,6'-TeCB	51	75-125	60-135	
2,2',5,5'-TeCB/2,3',4,6-TeCB	52/69	75-125	60-135	
2,2',5,6'-TeCB	53	75-125	60-135	
2,2',6,6'-TeCB	54	75-125	60-135	
2,3,3',4-TeCB	55	75-125	60-135	
2,3,3',4'-TeCB/2,3,4,4'-TeCB	56/60	75-125	60-135	
2,3,3',5-TeCB	57	75-125	60-135	
2,3,3',5'-TeCB	58	75-125	60-135	
2,3,4,5-TeCB/2,3',4',5-TeCB	61/70	75-125	60-135	
2,3,4,6-TeCB	62	75-125	60-135	
2,3,4',5-TeCB	63	75-125	60-135	
2,3,5,6-TeCB	65	75-125	60-135	

QC Acceptance Criteria for VER[1], OPR[2], and Labeled Compounds[3] in Samples				
Congener Name	**Congener Number**	**VER (%)**	**OPR Recovery (%)**	**Labeled Compound Recovery in Samples (%)**
2,3',4,5-TeCB	67	75-125	60-135	
2,3',4,5'-TeCB	68	75-125	60-135	
2,3',4',5-TeCB	70	75-125	60-135	
2,3',5',6-TeCB	73	75-125	60-135	
2,4,4',5-TeCB	74	75-125	60-135	
2',3,4,5-TeCB/2,3',4,4'-TeCB	76/66	75-125	60-135	
3,3',4,5-TeCB	77	75-125	60-135	
3,3',4,5'-TeCB	78	75-125	60-135	
3,3',5,5'-TeCB	79	75-125	60-135	
3,4,4',5-TeCB	80	75-125	60-135	
2,2',3,3',4-PeCB	81	75-125	60-135	
2,2',3,3',5-PeCB	82	75-125	60-135	
2,2',3,3',5-PeCB	83	75-125	60-135	
2,2',3,3',6-PeCB/2,2',3,5,5'-PeCB	84/92	75-125	60-135	
2,2',3,4,4'-PeCB/2,3,4,5,6-PeCB	85/116	75-125	60-135	
2,2',3,4,5-PeCB	86	75-125	60-135	
2,2',3,4,5'-PeCB/2,3,4',5,6-PeCB/2',3,4,5,6'-PeCB	87/117/125	75-125	60-135	
2,2',3,4,6-PeCB/2,2',3,4',6-PeCB	88/91	75-125	60-135	
2,2',3,4,6'-PeCB	89	75-125	60-135	
2,2',3,4',5-PeCB/2,2',4,5,5'-PeCB	90/101	75-125	60-135	
2,2',3,5,6-PeCB	93	75-125	60-135	
2,2',3,5,6'-PeCB	94	75-125	60-135	
2,2',3,5',6-PeCB/2,2',3',4,6-PeCB/2,2',4,5,6'-PeCB	95/98/102	75-125	60-135	
2,2',3,6,6'-PeCB	96	75-125	60-135	
2,2',3,4',5-PeCB	97	75-125	60-135	
2,2',4,4',5-PeCB	99	75-125	60-135	
2,2',4,4',6-PeCB	100	75-125	60-135	
2,2',4,5',6-PeCB	103	75-125	60-135	
2,2',4,4,6'-PeCB	104	75-125	60-135	NA
2,3,3',4,4'-PeCB	105	75-125	60-135	
2,3',4,4',5-PeCB/2,3,3',4,5-PeCB	118/106	75-125	60-135	
2,3,3',4',5-PeCB/2,3,3',4,6-PeCB	107/109	75-125	60-135	
2,3,3',4,5'-PeCB/2,3,3',5,6-PeCB	108/112	75-125	60-135	
2,3,3',4',6-PeCB	110	75-125	60-135	
2,3,3',5,5'-PeCB/2,3,4,4',6-PeCB	111/115	75-125	60-135	
2,3,3',5',6-PeCB	113	75-125	60-135	
2,3,4,4',5-PeCB	114	75-125	60-135	
2,3',4,4',6-PeCB	119	75-125	60-135	
2,3',4,5,5'-PeCB	120	75-125	60-135	
2,3',4,5',6-PeCB	121	75-125	60-135	
2,3,3',4',5'-PeCB	122	75-125	60-135	
2,3',4,4',5'-PeCB	123	75-125	60-135	
2,3',4',5,5'-PeCB	124	75-125	60-135	
3,3'4,4',5-PeCB	126	75-125	60-135	
3,3',4,5,5'-PeCB	127	75-125	60-135	
2,2',3,3',4,4'-HxCB/2,3,3',4',5,5'-HxCB	128/162	75-125	60-135	
2,2',3,3',4,5-HxCB	129	75-125	60-135	
2,2',3,3',4,5'-HxCB	130	75-125	60-135	
2,2',3,3',4,6-HxCB	131	75-125	60-135	
2,2',3,3',4,6'-HxCB/2,3,3',4,5',6-HxCB	132/161	75-125	60-135	
2,2',3,3',5,5'-HxCB/2,2',3,4,5,6-HxCB	133/142	75-125	60-135	
2,2',3,3',5,6-HxCB/2,2',3,4,5,6'-HxCB	134/143	75-125	60-135	
2,2',3,3',5,6'-HxCB	135	75-125	60-135	
2,2',3,3',6,6'-HxCB	136	75-125	60-135	
2,2',3,4,4',5-HxCB	137	75-125	60-135	
2,2',3,4,4',5'-HxCB/2,3,3',4',5,6-HxCB/2,3,3',4',5',6-HxCB	138/163/164	75-125	60-135	
2,2',3,4,4',6-HxCB/2,2',3,4',5',6-HxCB	139/149	75-125	60-135	

QC Acceptance Criteria for VER[1], OPR[2], and Labeled Compounds[3] in Samples				
Congener Name	**Congener Number**	**VER (%)**	**OPR Recovery (%)**	**Labeled Compound Recovery in Samples (%)**
2,2',3,4,4',6'-HxCB	140	75-125	60-135	
2,2',3,4,5,5'-HxCB	141	75-125	60-135	
2,2',3,4,5',6-HxCB	144	75-125	60-135	
2,2',3,4,6,6'-HxCB	145	75-125	60-135	
2,2',3,4',5,5'-HxCB/2,3,3',5,5',6-HxCB	146/165	75-125	60-135	
2,2',3,4',5,6-HxCB	147	75-125	60-135	
2,2',3,4',5,6'-HxCB	148	75-125	60-135	
2,2',3,4',6,6'-HxCB	150	75-125	60-135	
2,2',3,5,5',6-HxCB	151	75-125	60-135	
2,2',3,5,6,6'-HxCB	152	75-125	60-135	
2,2',4,4',5,5'-HxCB	153	75-125	60-135	
2,2',4,4',5,6'-HxCB	154	75-125	60-135	
2,2',4,4',6,6'-HxCB	155	75-125	60-135	
2,3,3',4,4',5-HxCB	156	75-125	60-135	
2,3,3',4,4',5'-HxCB	157	75-125	60-135	
2,3,3',4,4',6-HxCB/2,3,3',4,5,6-HxCB	158/160	75-125	60-135	
2,3,3',4,5,5'-HxCB	159	75-125	60-135	
2,3,4,4',5,6-HxCB	166	75-125	60-135	
2,3',4,4',5,5'-HxCB	167	75-125	60-135	
2,3',4,4',5',6-HxCB	168	75-125	60-135	
3,3',4,4',5,5'-HxCB	169	75-125	60-135	
2,2',3,3',4,4',5-HpCB	170	75-125	60-135	
2,2',3,3',4,4',6-HpCB	171	75-125	60-135	
2,2',3,3',4,5,5'-HpCB	172	75-125	60-135	
2,2',3,3',4,5,6-HpCB	173	75-125	60-135	
2,2',3,3',4,5,6'-HpCB	174	75-125	60-135	
2,2',3,3',4,5',6-HpCB	175	75-125	60-135	
2,2',3,3'4,6,6'-HpCB	176	75-125	60-135	
2,2',3,3',4',5,6-HpCB	177	75-125	60-135	NA
2,2',3,3',5,5',6-HpCB	178	75-125	60-135	
2,2',3,3',5,6,6'-HpCB	179	75-125	60-135	
2,2',3,4,4',5,5'-HpCB	180	75-125	60-135	
2,2',3,4,4',5,6-HpCB	181	75-125	60-135	
2,2',3,4,4',5,6'-HpCB/2,2',3,4',5,5',6-HpCB	182/187	75-125	60-135	
2,2',3,4,4',5',6-HpCB	183	75-125	60-135	
2,2',3,4,4',6,6'-HpCB	184	75-125	60-135	
2,2',3,4,5,5',6-HpCB	185	75-125	60-135	
2,2',3,4,5,6,6'-HpCB	186	75-125	60-135	
2,2',3,4',5,6,6'-HpCB	188	75-125	60-135	
2,3,3',4,4',5,5'-HpCB	189	75-125	60-135	
2,3,3',4,4',5,6-HpCB	190	75-125	60-135	
2,3,3',4,4',5',6-HpCB	191	75-125	60-135	
2,3,3',4,5,5',6-HpCB	192	75-125	60-135	
2,3,3',4',5,5',6-HpCB	193	75-125	60-135	
2,2',3,3',4,4',5,5'-OcCB	194	75-125	60-135	
2,2',3,3',4,4',5,6-OcCB	195	75-125	60-135	
2,2',3,3',4,4',5,6'-OcCB/2,2',3,4,4',5,5',6-OcCB	196/203	75-125	60-135	
2,2',3,3',4,4',6,6'-OcCB	197	75-125	60-135	
2,2',3,3',4,5,5',6-OcCB	198	75-125	60-135	
2,2',3,3',4,5,5',6'-OcCB	199	75-125	60-135	
2,2',3,3',4,5,6,6'-OcCB	200	75-125	60-135	
2,2',3,3',4,5',6,6'-OcCB	201	75-125	60-135	
2,2',3,3',5,5',6,6'-OcCB	202	75-125	60-135	
2,2',3,4,4',5,6,6'-OcCB	204	75-125	60-135	
2,3,3',4,4',5,5',6-OcCB	205	75-125	60-135	
2,2',3,3',4,4',5,5',6-NoCB	206	75-125	60-135	
2,2',3,3',4,4',5,6,6'-NoCB	207	75-125	60-135	

QC Acceptance Criteria for VER[1], OPR[2], and Labeled Compounds[3] in Samples				
Congener Name	**Congener Number**	**VER (%)**	**OPR Recovery (%)**	**Labeled Compound Recovery in Samples (%)**
2,2',3,3',4,5,5',6,6'-NoCB	208	75-125	60-135	NA
DeCB	209	75-125	60-135	
Labeled Compounds				
$^{13}C_{12}$-2-MonoCB	1L	50-145	15-145	5-145
$^{13}C_{12}$-4-MonoCB	3L	50-145	15-145	5-145
$^{13}C_{12}$-2,2'-DiCB	4L	50-145	15-145	5-145
$^{13}C_{12}$-2,5-DiCB	9L	50-145	15-145	5-145
$^{13}C_{12}$-3,3'-DiCB	11L	50-145	15-145	5-145
$^{13}C_{12}$-2,2',6-TrCB	19L	50-145	15-145	5-145
$^{13}C_{12}$-2,4,4'-TrCB	28L	50-145	15-145	5-145
$^{13}C_{12}$-2,4',6-TrCB	32L	50-145	15-145	5-145
$^{13}C_{12}$-3,4,4'-TrCB	37L	50-145	15-145	5-145
$^{13}C_{12}$-2,2',4,4'-TeCB	47L	50-145	15-145	5-145
$^{13}C_{12}$-2,2',5,5'-TeCB	52L	50-145	15-145	5-145
$^{13}C_{12}$-2,2',6,6'-TeCB	54L	50-145	15-145	5-145
$^{13}C_{12}$-2,3',4',5-TeCB	70L	30-135	15-145	10-145
$^{13}C_{12}$-3,3',4,4'-TeCB	77L	50-145	40-145	10-145
$^{13}C_{12}$-3,4,4',5-TeCB	80L	50-145	40-145	10-145
$^{13}C_{12}$-3,3',4,4'-TeCB	81L	50-145	40-145	10-145
$^{13}C_{12}$-2,2',3,5',6-PeCB	95L	50-145	40-145	10-145
$^{13}C_{12}$-2,2',3,4',5-PeCB	97L	50-145	40-145	10-145
$^{13}C_{12}$-2,2',4,5,5'-PeCB	101L	50-145	40-145	10-145
$^{13}C_{12}$-2,2',4,6,6'-PeCB	104L	50-145	40-145	10-145
$^{13}C_{12}$-2,3,3',4,4'-PeCB	105L	50-145	40-145	10-145
$^{13}C_{12}$-2,3,4,4',5-PeCB	114L	50-145	40-145	10-145
$^{13}C_{12}$-2,3',4,4',5-PeCB	118L	50-145	40-145	10-145
$^{13}C_{12}$-2',3,4,4',5-PeCB	123L	50-145	40-145	10-145
$^{13}C_{12}$-3,3',4,4',5-PeCB	126L	50-145	40-145	10-145
$^{13}C_{12}$-3,3',4,5,5'-PeCB	127L	50-145	40-145	10-145
$^{13}C_{12}$-2,2',3,4,4',5'-HxCB	138L	50-145	40-145	10-145
$^{13}C_{12}$-2,2',3,4,5,5'-HxCB	141L	50-145	40-145	10-145
$^{13}C_{12}$-2,2',4,4',5,5'-HxCB	153L	50-145	40-145	10-145
$^{13}C_{12}$-2,2',4,4',6,6'-HxCB	155L	50-145	40-145	10-145
$^{13}C_{12}$-2,3,3',4,4',5-HxCB	156L	50-145	40-145	10-145
$^{13}C_{12}$-2,3,3',4,4',5'-HxCB	157L	50-145	40-145	10-145
$^{13}C_{12}$-2,3,3',4,5,5'-HxCB	159L	50-145	40-145	10-145
$^{13}C_{12}$-2,3',4,4',5,5'-HxCB	167L	50-145	40-145	10-145
$^{13}C_{12}$-3,3',4,4',5,5'-HxCB	169L	50-145	40-145	10-145
$^{13}C_{12}$-2,2',3,3',4,4',5-HpCB	170L	50-145	40-145	10-145
$^{13}C_{12}$-2,2',3,4,4',5,5'-HpCB	180L	50-145	40-145	10-145
$^{13}C_{12}$-2,2',3,4',5,6,6'-HpCB	188L	50-145	40-145	10-145
$^{13}C_{12}$-2,3,3',4,4',5,5'-HpCB	189L	50-145	40-145	10-145
$^{13}C_{12}$-2,2',3,3',4,4',5,5'-OcCB	194L	50-145	40-145	10-145
$^{13}C_{12}$-2,2',3,3',5,5',6,6'-OcCB	202L	50-145	40-145	10-145
$^{13}C_{12}$-2,2',3,3',4,4',5,5',6-NoCB	206L	50-145	40-145	10-145
$^{13}C_{12}$-2,2',3,3',4,5,5',6,6'-NoCB	208L	50-145	40-145	10-145
$^{13}C_{12}$-DeCB	209L	50-145	40-145	10-145
Cleanup Standards				
$^{13}C_{12}$-3,3',4,5'-TeCB	79L	50-145	40-145	10-145
$^{13}C_{12}$-2,2'3,3'5,5'6-HpCB	178L	50-145	40-145	10-145

[1]VER = Calibration verification
[2]OPR = Ongoing precision and recovery
[3]The suffix "L" in a congener number indicates an isotopically labeled compound.