GROUP A ELEMENTS: PROJECT MANAGEMENT

Element 1. Title and Approval Sheets

QUALITY ASSURANCE PROJECT PLAN

Long-term Monitoring of Bass Lakes and Reservoirs in California

Bioaccumulation Monitoring Program

Surface Water Ambient Monitoring Program

Version 3

October 2021

Program Title SWAMP Bioaccumulation Monitoring Program Long-term

Monitoring of Bass Lakes and Reservoirs in California

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- **Effective Date** This Quality Assurance Project Plan (QAPP) is effective from April 2021 to May 2030 unless otherwise revised, approved and distributed accordingly at an earlier date.
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QAPP Preface

This Quality Assurance Project Plan (QAPP) document defines procedures and criteria that will be used for this project, conducted by the Surface Water Ambient Monitoring Program (SWAMP) Bioaccumulation Monitoring Program (Program) in association with the Moss Landing Marine Labs Marine Pollution Studies Laboratory (MPSL), the San Francisco Estuary Institute (SFEI), and Babcock Laboratories (Babcock) and their subcontractors. Included are criteria for data quality acceptability, procedures for sampling, testing (including deviations) and calibration, as well as preventative and corrective measures. The responsibilities of MPSL, Babcock, SGS-Axys, and SFEI also are contained within. The Program selects the sampling sites, the types and size of tissue samples, and the number of analyses to be conducted. This QAPP meets the SWAMP Statewide Project Planning requirements within the <u>2017 SWAMP Quality</u> <u>Assurance Program Plan</u> (2017 SWAMP QAPrP).

This work is funded through the US EPA F106 SWAMP Bioaccumulation funding.

Approvals

The approvals below were submitted via DocuSign or separately, preventing their inclusion in this signature block. They can be viewed online via the <u>signature page</u>. Originals are kept on file by Autumn Bonnema of MPSL.

Jay Davis

Lead Scientist

	Date	
Andrew Hamilton SWRCB Quality Assurance Officer		
	Date	
Tessa Fojut SWAMP Program Quality Assurance Officer		
	Date	
Autumn Bonnema Project Manager/ MPSL Quality Assurance Officer		
	Date	
Stacey Fry Babcock Laboratories Quality Assurance Manager		
	Date	
Sean Campbell SGS-Axys Quality Assurance Manager		
	Date	

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Element 3. Distribution List and Contact Information

A copy of this Quality Assurance Project Plan (QAPP), in hardcopy or electronic format, is to be received and retained by at least one person from each participating entity. At least one person from each participating entity shall be responsible for receiving, retaining and distributing the QAPP to their respective staff within their own organization. Contact information for the primary contact person (listed first) for each participating organization also is provided below in Table 1.

Table 1. Contact Information

Name, agency, address, phone number (where applicable) and email address for primary contact from each participating agency. When two names are listed, the first is the primary contact. The person responsible for receiving, retaining and distributing the QAPP to their respective staff within their own organization are designated with an asterisk (*).

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		Sidney BC V8L 5X2	
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	Control Board	Sacramento CA 95814	
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Tessa Fojut*	State Water Resources	1001 I Street, 19 th Floor	
	Control Board	Sacramento CA 95814	
		tessa.fojut@waterboards.ca.gov	

Element 4. Project Organization

The lines of communication between the participating entities, project organization and responsibilities are outlined in Table 2 and Figure 1.

Table 2. Positions and duties

Responsibilities of individuals directly involved in the program.

Position	Name	Responsibilities
Region 9 EPA Surface Water Standards Coordinator	Terry Fleming (USEPA)	Oversees SWAMP federal funding and Program outputs.
State Board Management	Greg Gearhart (SWRCB) Melissa Morris (SWRCB) Ali Dunn (OIMA)	Program planning and oversight; project budget allocation and reconciliation with program objectives
Contract Manager	Chad Fearing (OIMA)	Approves invoices
Program Coordinator	Anna Holder (OIMA)	Communication and coordination liaison with the Lead Scientist and Project Manager and SWRCB/OIMA, Review contract deliverables, General Program and Safe to Eat Workgroup coordination and support
Lead Scientist	Jay Davis (SFEI-ASC)	Advisory role; data reporting; oversee development and submission of contract deliverables in coordination with the Project Manager (e.g. monitoring plans, QAPP, reports); coordination with the technical Safe to Eat Workgroup
Project Manager	Autumn Bonnema (MPSL)	Generation and maintenance of project QAPP; project coordination; ensures all activities are completed within proper timeframes; oversees project deliverables in coordination with the Lead Scientist, entry of field and laboratory generated data into SWAMP formats

Position	Name	Responsibilities
State Board QA Officer	Andrew Hamilton (SWRCB)	Approves QAPP; reports to EPA and SWRCB management
Program QA Officer, Database Manager, SWAMP IQ	Tessa Fojut (OIMA)	Review and approve project QAPP; oversees Data Quality Managers; establishes program level quality objectives and requirements for project; reports to EPA and SWRCB management and coordinates with SWRCB QAO.
SWAMP IQ Data Quality Manager	Jennifer Salisbury (OIMA)	Reviews, verifies, validates and loads chemistry and composite data to SWAMP database; generates QA narrative; reports to Program QAO
Laboratory QA Officer	Autumn Bonnema (MPSL) Stacey Fry (Babcock) Sean Campbell (SGS-Axys)	Ensures that the laboratory quality assurance plan and quality assurance project plan criteria are met through routine monitoring and auditing of the systems; review and approve data prior to submission to SWAMP IQ; investigate and conduct laboratory corrective action.
Sample Collection Coordinator	Billy Jakl (MPSL)	Sampling coordination, operations, and implementing field-sampling procedures.
Laboratory Director	Wes Heim (MPSL) Caroline Sangari (Babcock) Sean Campbell (SGS-Axys)	Supervises laboratory staff; data validation, management and reporting
Sample Custodian	Autumn Bonnema (MPSL) Sean Campbell (SGS-Axys) Additional staff	Sample storage; not responsible for any deliverables; may oversee Technicians
Technicians	Technical Staff MPSL SGS-Axys	Conduct tissue dissection, digestion, and chemical analyses; verify field and lab datasheet entry; responsible for chemistry data submission to Laboratory QAO

4.1. Involved parties and roles

Chad Fearing of the Office of Information Management and Analysis (OIMA) is the Contract Manager (CM), who is responsible for approving invoices and ensuring the Contractors meet the contract terms.

Anna Holder of OIMA is the Program Coordinator and will (1) serve as communication and coordination liaison with the Lead Scientist and Project Manager, (2) serve as Water Boards internal communication liaison for the Program, (3) review contract deliverables in coordination with the Contract Manager, and (4) provide general Program and Safe to Eat Workgroup (Workgroup) coordination and support.

Jay Davis of San Francisco Estuary Institute (SFEI-ASC) is the Lead Scientist (LS) and primary contact of this project. The LS will (1) generate the Monitoring Plan, (2) approve the QAPP, and (3) provide the State Water Board with a final report on completion of this project, and present the results to the Workgroup.

Autumn Bonnema of MPSL will serve as the Project Manager (PM). The PM will (1) prepare the QAPP, (2) ensure all laboratory activities are completed within the required timelines, (3) review, evaluate and document project reports, and (4) verify the completeness of all tasks. In addition, the PM may assist field crew in preparation and logistics.

Billy Jakl of MPSL directs fish collection for this project. He will (1) oversee preparation for sampling, including vehicle and vessel maintenance and (2) oversee sample and field data collection, data entry and submission to the SWAMP Information Management and Quality Assurance Center (SWAMP IQ).

Sean Campbell is responsible for sample storage and custody at SGS-Axys. Autumn Bonnema will do the same for samples processed at MPSL, in addition to overseeing compositing of tissue samples.

Caroline Sangari will serve as the Laboratory Director (LD) for Babcock Labs. Her specific duties will be to (1) provide oversight for organics analyses on fish tissues to be done for this project, and (2) ensure that all Babcock and subcontracted activities are completed within the proper timelines. Her counterpart at subcontracted SGS-Axys is Sean Campbell.

Wes Heim will serve as the LD for the MPSL component of this project. His specific duties will be to (1) provide oversight for mercury analyses on fish tissues to be done for this project, and (2) ensure that all MPSL activities are completed within the proper timelines.

Members of the Workgroup provide input and advice on the Monitoring Plans and long-term strategy and are not responsible for any deliverables. The members are also the end users of the data generated by the Bioaccumulation Monitoring Program projects, with the primary objectives of the data used to answer Management Questions laid out in the original 2015 <u>Monitoring Plan</u> as well as the 2021 Monitoring Plan Update. Workgroup representatives include, but are not limited to, individuals from the following organizations: United States

Environmental Protection Agency (USEPA), Office of Environmental Health Hazard Assessment (OEHHA), <u>SWAMP Regional Coordinators</u>, and Statewide mercury Control Program.

A Peer Review Panel reviews Monitoring Plans as well as technical reports. This panel consists of Bruce Monson (Minnesota Pollution Control Agency, St. Paul, Minnesota), Chris Schmitt, (United States Geological Survey, Columbia, Missouri) and Harry Ohlendorf (CH2M HILL, Sacramento, California).

4.2. Quality Assurance Officer (QAO) Role

Autumn Bonnema is the MPSL Laboratory QAO (LQAO), Stacey Fry is the Babcock Laboratories LQAO, and Sean Campbell is LQAO at SGS-Axys. The role of the LQAO is to ensure that quality control for sample processing and data analysis procedures described in this QAPP are maintained throughout the project.

The LQAOs will review and approve all quality control data prior to submission. They will review and assess all procedures during the life of this project against QAPP requirements and assess whether the procedures were performed according to protocol. The LQAOs will report all findings (including qualified data) to the Program QAO and the PM, including all requests for corrective action. The Laboratory and Program QAOs have the authority to stop all actions if there are significant deviations from required procedures or evidence of a systematic failure.

SWAMP IQ serves as the project quality assurance and data management team. The SWAMP IQ Data Quality Manager (DQM) reviews, verifies, validates, and loads the composite and chemistry data to the SWAMP database. Deviations from the project QAPP are flagged and reported to the PM and Program QAO prior to loading. The DQMs are responsible for developing the project QA narrative report. The Program QAO (Tessa Fojut, SWAMP IQ) assesses the data for compliance with the project and SWAMP and ensures that the project meets USEPA requirements for projects receiving federal EPA funds. The Program QAO also works with the State Board QA Officer, Andrew Hamilton, to ensure that the project and data meets the requirements of the SWRCB's Quality Management Plan.

4.3. Persons responsible for QAPP update and maintenance

Revisions and updates to this QAPP will be carried out by Autumn Bonnema, with technical input from the Laboratory and Program QAOs. All changes will be considered draft until reviewed and approved by the PM, the Program QAO, and SWRCB QAO.

The QAPP must be reviewed at least annually and revised where necessary. It must meet USEPA, SWRCB and SWAMP quality system requirements to be approved.

Copies of this QAPP will be distributed to all parties involved in the project. Any future amended QAPPs will be held and distributed in the same fashion. All originals of these first and subsequent amended QAPPs will be held on site at SFEI-ASC, Babcock, SGS-Axys and MPSL.

4.4. Organizational chart and responsibilities

Figure 1. Organizational Chart



Element 5. Problem Definition

5.1. Problem statement

5.1.1. Addressing Multiple Monitoring Objectives and Assessment Questions for Beneficial Uses Related to Harvesting of Wild Fish for Consumption

The Workgroup has developed a set of monitoring objectives and assessment questions for a statewide program evaluating the impacts of bioaccumulation on beneficial uses related to harvesting of wild fish for consumption. There are currently two statewide beneficial uses that apply to the harvesting of wild-caught species for consumption – "commercial and sport fishing" (COMM), and "shellfish harvesting" (SHELL). Two additional beneficial uses relating to harvesting fish have been established by the North Coast Regional Water Board: "Native American Culture" (CUL) and "Subsistence Fishing" (FISH). These North Coast Region beneficial uses have also prompted the creation of statewide uses of a similar nature that were adopted by the State Board in May 2017 (Resolution 2017-0027): "Tribal Tradition and Culture" (CUL), "Tribal Subsistence Fishing" (T-SUB), and "Subsistence Fishing" (SUB). SWAMP Bioaccumulation Monitoring Program (Program) data will be used to evaluate the status of all beneficial uses related to harvesting of wild fish (i.e., COMM, CUL, T-SUB, SUB, FISH, and any new uses that are adopted). Since the adoption of Resolution 2017-0027, each region has started the process of adopting the new tribal beneficial use (TBU) definitions into their respective Basin Plans, and undergoing the beneficial use designation process which varies from region to region. The Water Boards' Tribal Affairs Unit tracks and publishes the status of TBUs within Regional Basin <u>Plans</u> on a quarterly basis.

The Workgroup assessment framework is consistent with frameworks developed for other components of SWAMP (Bernstein 2010) and is intended to guide the Program over the long-term. The four objectives can be summarized as (1) status; (2) trends; (3) sources and pathways; and (4) effectiveness of management actions.

Over the long-term, the primary emphasis of the statewide Program will be on evaluating status and trends. Monitoring status and trends in bioaccumulation will provide some information on sources and pathways and effectiveness of management actions at a broader geographic scale. However, other types of monitoring (i.e., water and sediment monitoring) and other programs (regional Total Maximum Daily Load [TMDL] programs) are also needed for addressing sources and pathways and effectiveness of management actions.

5.2. Decisions or outcomes

Two primary management questions have been articulated to guide the design of this longterm monitoring effort. In addition, two secondary management questions have been identified to guide interpretation of the results of the monitoring.

5.2.1. Management Questions

5.2.1.1. Management Question 1 (MQ1)

What are the recent average concentrations of contaminants of concern in each priority bass lake or reservoir?

Answering this question will address the critical need of managers and the public for timely, high-quality information on the status of contaminant bioaccumulation in priority water bodies. As mentioned above, this information will be useful to the state and regional boards in impairment assessments and 303(d) list updates. A list of priority bass lakes to include in this monitoring has been developed with input from the regional boards.

Mercury is the contaminant of greatest concern in most bass lakes and will be the primary focus of this monitoring. However, PCBs and organochlorine pesticides also reach levels of concern in a small subset of these lakes and will be monitored in those situations.

The data needed to answer this question are average concentrations of contaminants of concern in the species with a tendency to accumulate high concentrations. For mercury, top predators such as black bass tend to accumulate relatively high concentrations. Furthermore, black bass have been established as an excellent quantitative mercury bioaccumulation indicator for California because they are amenable to size-standardization. High-lipid, bottom-feeding species such as catfish, carp, and sucker have a tendency to accumulate relatively high concentrations.

The State Water Board has an established policy for placing water bodies on the 303(d) list.

5.2.1.2. Management Question 2 (MQ2)

What is the trend in statewide average bass mercury concentrations in fish in priority bass lakes and reservoirs?

A statewide <u>control program for mercury</u> is being developed by the State Water Resources Control Board. Mercury TMDLs also have been developed for other water bodies, including the Delta, San Francisco Bay, and some lakes and reservoirs. For all of the mercury control plans in the state, it is critically important to know whether food web mercury concentrations are trending up or down on a regional or statewide scale. A statewide increasing trend could obscure the beneficial effects of management actions to reduce mercury bioaccumulation. In the absence of awareness of such a trend, false conclusions could be drawn that actions are not having the desired effect. On the other hand, the existence of a general declining trend could give the impression that actions are more effective than they actually are.

It is plausible to hypothesize that food web mercury could be increasing across the state, either due to increasing atmospheric mercury emissions in Asia (Chen et al. 2012, Drevnick et al. 2015) or due to global warming (Schneider et al. 2009). Several recent studies have reported evidence of regional increases in food web mercury in north-central North America (e.g., Monson 2009, Monson et al. 2011, Gandhi et al. 2014), although the most recent data from Minnesota suggest a return to a long-term pattern of decline (Bruce Monson, personal communication).

Hypothesized causes of these regional trends include global atmospheric emissions, climate change, invasive species, and changes in food web structure.

The data needed to answer this question are measurements of statewide average concentrations that are repeated over time. The large number and wide distribution of bass lakes that have been identified as priorities for sampling provide a population of water bodies that can be sampled to assess statewide and regional trends in food web mercury over time. Repeated rounds of sampling of randomly selected subsets of these lakes would yield a time series of representative, average statewide concentrations. These statewide averages would be based on concentrations in black bass, which have been demonstrated to be indicator species that are representative of conditions in the water body where they are collected and that yield data that are comparable across water bodies and over time.

5.2.1.3. Secondary Management Questions

5.2.1.3.1. What fractions of the lakes show decreases, increases, or no change in mercury concentration in fish?

Monitoring of mercury in clusters of lakes in other regions of North America have shown that temporal trends in fish mercury levels commonly vary among lakes, with some lakes showing decreases, some showing increases, and some showing no change. Examination of fish mercury levels from the small number of California lakes that have been sampled twice (first in 2007-2008 and again in 2012 or 2013) suggest that this outcome can be expected in California as well.

5.2.1.3.2. What factors appear to be driving changes in mercury concentrations in fish?

Environmental managers will want to know what causal factors of processes are contributing to such variability in temporal trends among lakes. The monitoring data obtained in this program will be used to develop hypotheses regarding factors and processes causing observed trends. The development of hypotheses may stimulate focused investigations by scientists in academic, state, and federal sectors.

5.2.2. Overall Approach

The overall approach to be taken to answer these questions will be to establish a long-term cycle for sampling the 187 priority bass lakes and reservoirs that have been identified by the regional boards. Sampling of the entire group of lakes and reservoirs will occur in five biennial rounds of sampling over a 10-year period. The cycle will then be repeated. This effort will ensure that each of these lakes is sampled once every 10 years to provide updated information on concentrations of priority contaminants. By creating five randomly selected subsets (or "rotating panels") of the overall population, each round of sampling will yield a representative estimate of the statewide average mercury concentration that will add to a long-term time series to allow evaluation of the statewide trend in food web mercury.

5.2.3. Coordination

The Program coordinates with other efforts through the Safe to Eat Workgroup to leverage the SWAMP statewide monitoring funds available for this survey.

The Regional Boards will be contacted prior to each round of sampling to explore opportunities for coordinated sampling, in-kind support, or direct funding of this sampling program.

Element 6. Project Description

6.1. Work statement and produced products

Sport and prey-sized fish will be collected from lakes around California, as laid out below and in the Monitoring Plan. A technical report will summarize the data generated.

6.2. Constituents to be analyzed and measurement techniques.

Chemistry analytical methods are summarized in Section G of the Monitoring Plan. Constituents to be analyzed are summarized in Tables 3-6, below. All tissue chemistry data will be reported on a wet weight basis. Analytical methods are listed in each table as appropriate.

Though previous studies calculated PCBs as Aroclors for comparison with older data sets and health thresholds, the Program has ceased reporting these calculated values because the Workgroup and data users provided input that these calculations are not as valuable as individual congener data. OEHHA no longer intends to use calculated data; however, these values can be calculated as needed using the reported congener data if they are of interest to a data user.

In the <u>SWAMP Lakes Study</u> (conducted in 2007 and 2008), PBDE data were provided at a screening level only as a free service from the analytical lab. These compounds are important emerging contaminants however they are cost prohibitive and not part of our current analyte list. Archives of each sample will be retained for potential future analysis.

Fish Attributes
Total length (mm)
Fork Length (mm)
Standard Length (mm; small fish only)
Weight (g)
Sex (sport fish only)
Moisture (%)
Lipid (%; only when organics are analyzed)
Collection Location (UTMs)

Table 3. Constituents to be analyzed – fish attributes

EPA 3052M*

EPA 200.8M*

Fish attributes are physical measurements or observations. These are not covered in analytical methods.

	•	
Analyte	Matrix Type	Analytical Method
Total Mercury	Whole Body Small Fish and Sport Fish	EPA 7473 (USEPA 2007a)

 Table 4. Constituents to be analyzed - metals and metalloids in tissue

*Contact LQAO for method modifications

Total Selenium

filet muscle

filet muscle

Table 5. Constituents to be Analyzed - polychlorinated biphenyls (PCB) in tissue

Whole Body Small Fish and Sport Fish

Analyte	Matrix Type	Analytical Method
CL1-PCB-1 ⁺	Sport Fish filet muscle	EPA 1668A (MLA-010*)
CL1-PCB-2 [‡]	Sport Fish filet muscle	EPA 1668A (MLA-010*)
CL1-PCB-3 [‡]	Sport Fish filet muscle	EPA 1668A (MLA-010*)
CL2-PCB-4 [‡]	Sport Fish filet muscle	EPA 1668A (MLA-010*)
CL2-PCB-5 ⁺	Sport Fish filet muscle	EPA 1668A (MLA-010*)
CL2-PCB-6 [‡]	Sport Fish filet muscle	EPA 1668A (MLA-010*)
CL2-PCB-7 [‡]	Sport Fish filet muscle	EPA 1668A (MLA-010*)
CL2-PCB-8	Sport Fish filet muscle	EPA 1668A (MLA-010*)
CL2-PCB-9 [‡]	Sport Fish filet muscle	EPA 1668A (MLA-010*)
CL2-PCB-10 [‡]	Sport Fish filet muscle	EPA 1668A (MLA-010*)
CL2-PCB-11	Sport Fish filet muscle	EPA 1668A (MLA-010*)
CL2-PCB-12/13 [‡]	Sport Fish filet muscle	EPA 1668A (MLA-010*)
CL2-PCB-14 [‡]	Sport Fish filet muscle	EPA 1668A (MLA-010*)
CL2-PCB-15 [‡]	Sport Fish filet muscle	EPA 1668A (MLA-010*)
CL3-PCB-16 [‡]	Sport Fish filet muscle	EPA 1668A (MLA-010*)
CL3-PCB-17 [‡]	Sport Fish filet muscle	EPA 1668A (MLA-010*)
CL3-PCB-19 [‡]	Sport Fish filet muscle	EPA 1668A (MLA-010*)
CL3-PCB-21/33	Sport Fish filet muscle	EPA 1668A (MLA-010*)
CL3-PCB-22 [‡]	Sport Fish filet muscle	EPA 1668A (MLA-010*)
CL3-PCB-23 [‡]	Sport Fish filet muscle	EPA 1668A (MLA-010*)
CL3-PCB-24 [‡]	Sport Fish filet muscle	EPA 1668A (MLA-010*)
CL3-PCB-25 [‡]	Sport Fish filet muscle	EPA 1668A (MLA-010*)
CL3-PCB-26/29	Sport Fish filet muscle	EPA 1668A (MLA-010*)
CL3-PCB-27	Sport Fish filet muscle	EPA 1668A (MLA-010*)

Analyte	Matrix Type	Analytical Method
CL3-PCB-28/20	Sport Fish filet muscle	EPA 1668A (MLA-010*)
CL3-PCB-30/18	Sport Fish filet muscle	EPA 1668A (MLA-010*)
CL3-PCB-31	Sport Fish filet muscle	EPA 1668A (MLA-010*)
CL3-PCB-32 [‡]	Sport Fish filet muscle	EPA 1668A (MLA-010*)
CL3-PCB-34 [‡]	Sport Fish filet muscle	EPA 1668A (MLA-010*)
CL3-PCB-35 [‡]	Sport Fish filet muscle	EPA 1668A (MLA-010*)
CL3-PCB-36 [‡]	Sport Fish filet muscle	EPA 1668A (MLA-010*)
CL3-PCB-37 ⁺	Sport Fish filet muscle	EPA 1668A (MLA-010*)
CL3-PCB-38 [‡]	Sport Fish filet muscle	EPA 1668A (MLA-010*)
CL3-PCB-39 [‡]	Sport Fish filet muscle	EPA 1668A (MLA-010*)
CL4-PCB-41 [‡] /40 [‡] /71 [‡]	Sport Fish filet muscle	EPA 1668A (MLA-010*)
CL4-PCB-42 [‡]	Sport Fish filet muscle	EPA 1668A (MLA-010*)
CL4-PCB-43 [‡]	Sport Fish filet muscle	EPA 1668A (MLA-010*)
CL4-PCB-44/47 [‡] /65 [‡]	Sport Fish filet muscle	EPA 1668A (MLA-010*)
CL4-PCB-45 [‡] /51 [‡]	Sport Fish filet muscle	EPA 1668A (MLA-010*)
CL4-PCB-46 [‡]	Sport Fish filet muscle	EPA 1668A (MLA-010*)
CL4-PCB-48 [‡]	Sport Fish filet muscle	EPA 1668A (MLA-010*)
CL4-PCB-50 [‡] /53 [‡]	Sport Fish filet muscle	EPA 1668A (MLA-010*)
CL4-PCB-52	Sport Fish filet muscle	EPA 1668A (MLA-010*)
CL4-PCB-54 [‡]	Sport Fish filet muscle	EPA 1668A (MLA-010*)
CL4-PCB-55 [‡]	Sport Fish filet muscle	EPA 1668A (MLA-010*)
CL4-PCB-56	Sport Fish filet muscle	EPA 1668A (MLA-010*)
CL4-PCB-57 [‡]	Sport Fish filet muscle	EPA 1668A (MLA-010*)
CL4-PCB-58 [‡]	Sport Fish filet muscle	EPA 1668A (MLA-010*)
CL4-PCB-59 [‡] /62 [‡] /75 [‡]	Sport Fish filet muscle	EPA 1668A (MLA-010*)
CL4-PCB-60	Sport Fish filet muscle	EPA 1668A (MLA-010*)
CL4-PCB-61 [‡] /70/74/76 [‡]	Sport Fish filet muscle	EPA 1668A (MLA-010*)
CL4-PCB-63 [‡]	Sport Fish filet muscle	EPA 1668A (MLA-010*)
CL4-PCB-64	Sport Fish filet muscle	EPA 1668A (MLA-010*)
CL4-PCB-66	Sport Fish filet muscle	EPA 1668A (MLA-010*)
CL4-PCB-67 [‡]	Sport Fish filet muscle	EPA 1668A (MLA-010*)
CL4-PCB-68 [‡]	Sport Fish filet muscle	EPA 1668A (MLA-010*)
CL4-PCB-69 */49	Sport Fish filet muscle	EPA 1668A (MLA-010*)
CL4-PCB-72 [‡]	Sport Fish filet muscle	EPA 1668A (MLA-010*)
CL4-PCB-73 [‡]	Sport Fish filet muscle	EPA 1668A (MLA-010*)

Analyte	Matrix Type	Analytical Method
CL4-PCB-77	Sport Fish filet muscle	EPA 1668A (MLA-010*)
CL4-PCB-78 [‡]	Sport Fish filet muscle	EPA 1668A (MLA-010*)
CL4-PCB-79 [‡]	Sport Fish filet muscle	EPA 1668A (MLA-010*)
CL4-PCB-80 ⁺	Sport Fish filet muscle	EPA 1668A (MLA-010*)
CL4-PCB-81 [‡]	Sport Fish filet muscle	EPA 1668A (MLA-010*)
CL5-PCB-82 [‡]	Sport Fish filet muscle	EPA 1668A (MLA-010*)
CL5-PCB-83 [‡] /99	Sport Fish filet muscle	EPA 1668A (MLA-010*)
CL5-PCB-84 [‡]	Sport Fish filet muscle	EPA 1668A (MLA-010*)
CL5-PCB-88 [‡] /91 [‡]	Sport Fish filet muscle	EPA 1668A (MLA-010*)
CL5-PCB-89 [‡]	Sport Fish filet muscle	EPA 1668A (MLA-010*)
CL5-PCB-92 [‡]	Sport Fish filet muscle	EPA 1668A (MLA-010*)
CL5-PCB-94 [‡]	Sport Fish filet muscle	EPA 1668A (MLA-010*)
CL5-PCB-95 [‡] /100 [‡] /93 [‡] /102 [‡] /98 [‡]	Sport Fish filet muscle	EPA 1668A (MLA-010*)
CL5-PCB-96 [‡]	Sport Fish filet muscle	EPA 1668A (MLA-010*)
CL5-PCB-103 [‡]	Sport Fish filet muscle	EPA 1668A (MLA-010*)
CL5-PCB-104 [‡]	Sport Fish filet muscle	EPA 1668A (MLA-010*)
CL5-PCB-105	Sport Fish filet muscle	EPA 1668A (MLA-010*)
CL5-PCB-106 [‡]	Sport Fish filet muscle	EPA 1668A (MLA-010*)
CL5-PCB-107 [‡] /124 [‡]	Sport Fish filet muscle	EPA 1668A (MLA-010*)
CL5-PCB-108 [‡] /119 ⁺ /86 [‡] /97/125 [‡] /87	Sport Fish filet muscle	EPA 1668A (MLA-010*)
CL5-PCB-109 [‡]	Sport Fish filet muscle	EPA 1668A (MLA-010*)
CL5-PCB-110/115 [‡]	Sport Fish filet muscle	EPA 1668A (MLA-010*)
CL5-PCB-111 [‡]	Sport Fish filet muscle	EPA 1668A (MLA-010*)
CL5-PCB-112 [‡]	Sport Fish filet muscle	EPA 1668A (MLA-010*)
CL5-PCB-113 [‡] /90 ⁺ /101	Sport Fish filet muscle	EPA 1668A (MLA-010*)
CL5-PCB-114	Sport Fish filet muscle	EPA 1668A (MLA-010*)
CL5-PCB-117 [‡] /116 [‡] /85 [‡]	Sport Fish filet muscle	EPA 1668A (MLA-010*)
CL5-PCB-118	Sport Fish filet muscle	EPA 1668A (MLA-010*)
CL5-PCB-120 [‡]	Sport Fish filet muscle	EPA 1668A (MLA-010*)
CL5-PCB-121 [‡]	Sport Fish filet muscle	EPA 1668A (MLA-010*)
CL5-PCB-122 [‡]	Sport Fish filet muscle	EPA 1668A (MLA-010*)
CL5-PCB-123 ⁺	Sport Fish filet muscle	EPA 1668A (MLA-010*)
CL5-PCB-126	Sport Fish filet muscle	EPA 1668A (MLA-010*)
CL5-PCB-127 [‡]	Sport Fish filet muscle	EPA 1668A (MLA-010*)
CL6-PCB-128/166 +	Sport Fish filet muscle	EPA 1668A (MLA-010*)

Analyte	Matrix Type	Analytical Method
CL6-PCB-130 [‡]	Sport Fish filet muscle	EPA 1668A (MLA-010*)
CL6-PCB-131 [‡]	Sport Fish filet muscle	EPA 1668A (MLA-010*)
CL6-PCB-132 +	Sport Fish filet muscle	EPA 1668A (MLA-010*)
CL6-PCB-133 [‡]	Sport Fish filet muscle	EPA 1668A (MLA-010*)
CL6-PCB-134 [‡] /143 [‡]	Sport Fish filet muscle	EPA 1668A (MLA-010*)
CL6-PCB-136 [‡]	Sport Fish filet muscle	EPA 1668A (MLA-010*)
CL6-PCB-137	Sport Fish filet muscle	EPA 1668A (MLA-010*)
CL6-PCB-138/163 */129 */160 *	Sport Fish filet muscle	EPA 1668A (MLA-010*)
CL6-PCB-139 [‡] /140 [‡]	Sport Fish filet muscle	EPA 1668A (MLA-010*)
CL6-PCB-141	Sport Fish filet muscle	EPA 1668A (MLA-010*)
CL6-PCB-142 [‡]	Sport Fish filet muscle	EPA 1668A (MLA-010*)
CL6-PCB-144 [‡]	Sport Fish filet muscle	EPA 1668A (MLA-010*)
CL6-PCB-145 [‡]	Sport Fish filet muscle	EPA 1668A (MLA-010*)
CL6-PCB-146	Sport Fish filet muscle	EPA 1668A (MLA-010*)
CL6-PCB-147 [‡] /149	Sport Fish filet muscle	EPA 1668A (MLA-010*)
CL6-PCB-148 [‡]	Sport Fish filet muscle	EPA 1668A (MLA-010*)
CL6-PCB-150 [‡]	Sport Fish filet muscle	EPA 1668A (MLA-010*)
CL6-PCB-151/135 [‡] /154 [‡]	Sport Fish filet muscle	EPA 1668A (MLA-010*)
CL6-PCB-152 [‡]	Sport Fish filet muscle	EPA 1668A (MLA-010*)
CL6-PCB-153/168 +	Sport Fish filet muscle	EPA 1668A (MLA-010*)
CL6-PCB-155 [‡]	Sport Fish filet muscle	EPA 1668A (MLA-010*)
CL6-PCB-156 [‡] /157	Sport Fish filet muscle	EPA 1668A (MLA-010*)
CL6-PCB-158	Sport Fish filet muscle	EPA 1668A (MLA-010*)
CL6-PCB-159 [‡]	Sport Fish filet muscle	EPA 1668A (MLA-010*)
CL6-PCB-161 [‡]	Sport Fish filet muscle	EPA 1668A (MLA-010*)
CL6-PCB-162 [‡]	Sport Fish filet muscle	EPA 1668A (MLA-010*)
CL6-PCB-164 [‡]	Sport Fish filet muscle	EPA 1668A (MLA-010*)
CL6-PCB-165 [‡]	Sport Fish filet muscle	EPA 1668A (MLA-010*)
CL6-PCB-167 ⁺	Sport Fish filet muscle	EPA 1668A (MLA-010*)
CL6-PCB-169	Sport Fish filet muscle	EPA 1668A (MLA-010*)
CL7-PCB-170	Sport Fish filet muscle	EPA 1668A (MLA-010*)
CL7-PCB-171 [‡] /173 [‡]	Sport Fish filet muscle	EPA 1668A (MLA-010*)
CL7-PCB-172 [‡]	Sport Fish filet muscle	EPA 1668A (MLA-010*)
CL7-PCB-174	Sport Fish filet muscle	EPA 1668A (MLA-010*)
CL7-PCB-175 [‡]	Sport Fish filet muscle	EPA 1668A (MLA-010*)

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Analyte	Matrix Type	Analytical Method
CL7-PCB-176 [‡]	Sport Fish filet muscle	EPA 1668A (MLA-010*)
CL7-PCB-177	Sport Fish filet muscle	EPA 1668A (MLA-010*)
CL7-PCB-178 [‡]	Sport Fish filet muscle	EPA 1668A (MLA-010*)
CL7-PCB-179 [‡]	Sport Fish filet muscle	EPA 1668A (MLA-010*)
CL7-PCB-180/193 [‡]	Sport Fish filet muscle	EPA 1668A (MLA-010*)
CL7-PCB-181 [‡]	Sport Fish filet muscle	EPA 1668A (MLA-010*)
CL7-PCB-182 [‡]	Sport Fish filet muscle	EPA 1668A (MLA-010*)
CL7-PCB-183/185 [‡]	Sport Fish filet muscle	EPA 1668A (MLA-010*)
CL7-PCB-184 ⁺	Sport Fish filet muscle	EPA 1668A (MLA-010*)
CL7-PCB-186 [‡]	Sport Fish filet muscle	EPA 1668A (MLA-010*)
CL7-PCB-187	Sport Fish filet muscle	EPA 1668A (MLA-010*)
CL7-PCB-188 [‡]	Sport Fish filet muscle	EPA 1668A (MLA-010*)
CL7-PCB-189	Sport Fish filet muscle	EPA 1668A (MLA-010*)
CL7-PCB-190 [‡]	Sport Fish filet muscle	EPA 1668A (MLA-010*)
CL7-PCB-191 [‡]	Sport Fish filet muscle	EPA 1668A (MLA-010*)
CL7-PCB-192 [‡]	Sport Fish filet muscle	EPA 1668A (MLA-010*)
CL8-PCB-194	Sport Fish filet muscle	EPA 1668A (MLA-010*)
CL8-PCB-195	Sport Fish filet muscle	EPA 1668A (MLA-010*)
CL8-PCB-196 [‡]	Sport Fish filet muscle	EPA 1668A (MLA-010*)
CL8-PCB-197 [‡] /200	Sport Fish filet muscle	EPA 1668A (MLA-010*)
CL8-PCB-198/199	Sport Fish filet muscle	EPA 1668A (MLA-010*)
CL8-PCB-201	Sport Fish filet muscle	EPA 1668A (MLA-010*)
CL8-PCB-202 [‡]	Sport Fish filet muscle	EPA 1668A (MLA-010*)
CL8-PCB-203	Sport Fish filet muscle	EPA 1668A (MLA-010*)
CL8-PCB-204 [‡]	Sport Fish filet muscle	EPA 1668A (MLA-010*)
CL8-PCB-205 [‡]	Sport Fish filet muscle	EPA 1668A (MLA-010*)
CL9-PCB-206	Sport Fish filet muscle	EPA 1668A (MLA-010*)
CL9-PCB-207 [‡]	Sport Fish filet muscle	EPA 1668A (MLA-010*)
CL9-PCB-208 [‡]	Sport Fish filet muscle	EPA 1668A (MLA-010*)
CL10-PCB-209	Sport Fish filet muscle	EPA 1668A (MLA-010*)

⁺ New to analyte list in 2017

[‡] New to analyte list in 2021

*Contact LQAO for method modifications

Analyte Group	Analyte	Matrix Type	Analytical Method
Chlordanes	Chlordane, cis-	Sport Fish filet muscle	EPA 1699 (MLA-028)
	Chlordane, trans-	Sport Fish filet muscle	EPA 1699 (MLA-028)
	Heptachlor	Sport Fish filet muscle	EPA 1699 (MLA-028)
	Heptachlor epoxide	Sport Fish filet muscle	EPA 1699 (MLA-028)
	Nonachlor, cis-	Sport Fish filet muscle	EPA 1699 (MLA-028)
	Nonachlor, trans-	Sport Fish filet muscle	EPA 1699 (MLA-028)
	Oxychlordane	Sport Fish filet muscle	EPA 1699 (MLA-028)
DDTs	DDD(o,p')	Sport Fish filet muscle	EPA 1699 (MLA-028)
	DDD(p,p')	Sport Fish filet muscle	EPA 1699 (MLA-028)
	DDE(o,p')	Sport Fish filet muscle	EPA 1699 (MLA-028)
	DDE(p,p')	Sport Fish filet muscle	EPA 1699 (MLA-028)
	DDMU(p,p') *	Sport Fish filet muscle	Not currently available
	DDT(o,p')	Sport Fish filet muscle	EPA 1699 (MLA-028)
	DDT(p,p')	Sport Fish filet muscle	EPA 1699 (MLA-028)
Cyclodienes	Aldrin	Sport Fish filet muscle	EPA 1699 (MLA-028)
	Dieldrin	Sport Fish filet muscle	EPA 1699 (MLA-028)
	Endrin	Sport Fish filet muscle	EPA 1699 (MLA-028)
HCHs	HCH, alpha	Sport Fish filet muscle	EPA 1699 (MLA-028)
	HCH, beta	Sport Fish filet muscle	EPA 1699 (MLA-028)
Others	Dacthal	Sport Fish filet muscle	EPA 1699 (MLA-028)
	Endosulfan I	Sport Fish filet muscle	EPA 1699 (MLA-028)
	Hexachlorobenzene	Sport Fish filet muscle	EPA 1699 (MLA-028)
	Methoxychlor	Sport Fish filet muscle	EPA 1699 (MLA-028)
	Mirex	Sport Fish filet muscle	EPA 1699 (MLA-028)
	Oxadiazon	Sport Fish filet muscle	MLA-028

Table 6. Constituents to be Analyzed - organochlorine (OC) pesticides in tissue

* Not available from SGS-Axys but the Workgroup is still interested in analysis for future projects

6.3. Project schedule and number of samples to be analyzed.

Key tasks in the project and their expected due dates are outlined in Table 7.

Item	Activity and/or Deliverable	Deliverable Due Date				
1	Quality Assurance Project Plan & Monito	Quality Assurance Project Plan & Monitoring Plan				
1.1	Draft Quality Assurance Project Plan	March 2021				
1.2	Final Quality Assurance Project Plan	May 2021				
2	Sample Collection	October of each sampling year				
3	Sample Selection and Chemical Analysis					
3.1	Selection of Tissue for Analysis	November of each sampling year				
3.2	Creation of Sample Composites	December of each sampling year				
3.3	Chemical Analysis	February following each sampling year				
3.4	Data Reported to SWAMP	March following each sampling year				
4	Data Quality Assessment and Narrative	May following each sampling year				
5	Interpretive Report					
5.1	Draft Report	September following each sampling year				
5.2	Final Report	December following each sampling year				

Table 7. Project schedule timeline

6.4. Geographical setting and sample sites

The pool of lakes considered for sampling consisted primarily of those included in the 2007-2008 SWAMP lakes survey, with the addition of others sampled from 2002-2012 for which data were placed in the <u>California Environmental Data Exchange Network</u> (CEDEN), a centralized repository of data on California's water bodies, including streams, lakes, rivers, and the coastal ocean. Each lake will be sampled once on a ten-year rotation, in 5 panels. Panel 4 will be sampled in 2021. Precise dates for collection at each lake are not known and will be scheduled with cooperation from lake managers.

6.5. Constraints

All sampling must be completed by the end of the current year's sampling season in order to meet analysis and reporting deadlines set forth in Table 7.

Element 7. Quality Objectives, Indicators and Acceptability Criteria for Measurement Data

The data collection for this project is intended to support the management questions detailed in Element 5 as well as to assist in the development of fish consumption advisories by OEHHA. Therefore this project is categorized under the Public Health; Fish Consumption Advisories, Intended Data Use Category of the 2017 <u>SWAMP QAPrP</u>.

"Due to the importance of protecting human health, data collected under this category should be timely and of a level of quality sufficient to accurately assess human health risks. The sensitivity, amount of data collected, and timeliness of the data release should meet the unique requirements necessary to make a decision to post warnings or advisories that are protective of human health for that beneficial use" (2017 SWAMP QAPrP).

The tissue data collected by this project will follow, to the best of its ability, similar fish sampling and analysis protocols to ensure that data collected are useful in the development of advisories. The data collected will attempt to mirror the OEHHA protocols for selecting:

- target species and number of species representative of what anglers are likely to catch in a given water body;
- number and type of samples;
- fish size;
- sample timing;
- collection method;
- sample preparation;
- and chemical analysis.

Data collected for this project will be as sensitive as possible to be evaluated against the SWRCB statewide water quality objectives for mercury, and Advisory Tissue Levels and Fish Contaminant Goals developed by OEHHA (Klasing and Brodberg, 2008) (Tables 8a-c). The data will be assessed against these levels within the data analysis and reporting portions of the project.

Advisory Tissue Levels (ATLs) consider both the toxicity of contaminants and the health benefits of fish consumption. They are used to develop sport fish consumption advice for the public. They will also be used to communicate results of the study to the public via the <u>Safe to Eat</u> Portal and via reports and fact sheets.

The Measurement Quality Objectives (MQOs, Tables 9 and 10) that will be used for this study are existing limits that have been used for the study historically and will be continued for comparability purposes. The error limits and reporting levels presented represent realistic performance-based objectives for the methodologies employed by the study.

Program data undergo a further step of validation to determine usability of the data (Element 22) prior to assessment for human health concerns or 303(d) listing. It is particularly important to identify and remove data that may be unduly influenced by analytical blank contamination,

poor accuracy or poor precision based on the Data Quality Indicators (DQIs) as compared with the MQOs following <u>Bioaccumulation Monitoring Program Data Validation Standard Operating</u> <u>Procedures</u>.

Tables 8 a-c. Fish assessment thresholds

Thresholds for assessment of pollutants in fish from the SWRCB and OEHHA (Klasing and Brodberg, 2008). All values given in ng/g (ppb) on a wet weight basis. One serving is defined as 8 ounces (227 g) prior to cooking. The Fish Contaminant Goals (FCG) and Advisory Tissue Levels (ATLs) for mercury are for the most sensitive population (i.e., women aged 18 to 49 years and children aged 1 to 17 years).

Table 8 a	. SWRCB Statewide	Mercury	Objectives	(ppb)
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Pollutant	SWRCB Statewide Sport Fish Water Quality Objective	SWRCB Statewide Tribal Subsistence Fishing Water Quality Objective	SWRCB Statewide Prey Fish Water Quality Objective
Mercury	200	40	50

Table 8 b. OEHHA Advisory Tissue Levels (ppb)

	Advisory Tissue Level							
Pollutant	7 servings per week	6 servings per week	5 servings per week	4 servings per week	3 servings per week	2 servings per week	1 serving per week	No Consumption
Mercury	≤31	>31-36	>36-44	>44-55	>55-70	>70-150	>150- 440	>440
PCBs	≤9	>9-10	>10-13	>13-16	>16-21	>21-42	>42-120	>120
Seleniu m	≤1000	>1000- 1200	>1200- 1400	>1400- 1800	>1800- 2500	>2500- 4900	>4900- 15000	>15000
PBDEs	≤45	>45-52	>52-63	>63-78	>78-100	>100- 210	>210- 630	>630

Table 8 c. OEHHA Fish Contaminant Goals (ppb)

Pollutant	Fish Contaminant Goals
Chlordanes	5.6
DDTs	21
Dieldrin	0.46
Mercury	220
PCBs	3.6
Selenium	7400

Data quality indicators for all sample collection and laboratory analyses will include representativeness, accuracy (bias), precision, completeness, comparability and sensitivity, where applicable.

Field duplicates, field blanks and travel blanks are not collected in this study for any analytes. True field duplicates cannot be collected due to the disparate nature of individual fish, but analytical duplicates are conducted. Field and/or travel blanks are not collected because only the unexposed filet tissue of each fish is utilized, eliminating contamination from field sources.

Previously collected data will not be utilized in this study.

7.1. Accuracy and Bias

7.1.1. Accuracy

Accuracy is a measure of the agreement of a measurement to a known value, and includes both random error (precision) and systematic error (bias) of analytical operations (EPA QA/G-5, 2002).

Evaluation of the accuracy of laboratory procedures is achieved through the preparation and analysis of reference materials with each analytical batch. Ideally, the reference materials selected are similar in matrix and concentration range to the samples being prepared and analyzed. The accuracy of the results is assessed through the calculation of a percent recovery.

% recovery =
$$\left(\frac{V_{analyzed}}{V_{certified}}\right) x100$$

Where:

V analyzed: the analyzed concentration of the reference material

V certified: the certified concentration of the reference material

The acceptance criteria for reference materials are listed in Tables 9-10.

7.1.2. Bias

Bias is the systematic or persistent distortion of a measurement process that skews data in one direction. Certified Reference Materials (CRM) and Matrix Spike (MS) samples are used to determine the analyte-specific bias associated with each analytical laboratory. CRMs are used to determine analytical bias, and MS are used to determine the bias associated with the tissue matrix.

An MS will be prepared by adding a known concentration of the target analyte to a field sample, which is then subjected to the entire analytical procedure. If the ambient concentration of the field sample is known, the amount of spike added is within a specified range of that concentration. Matrix spikes will be analyzed in order to assess the magnitude of matrix interference and bias present. Because matrix spikes are analyzed in pairs, the second spike is called the matrix spike duplicate (MSD). The MSD provides information regarding the precision of the matrix effects. Both the MS and MSD are split from the same original field sample.

The success or failure of the matrix spikes will be evaluated by calculating the percent recovery.

% recovery =
$$\left(\frac{V_{MS} - V_{ambient}}{V_{spike}}\right) x100$$

Where:

V $_{MS}$: the concentration of the spiked sample V $_{ambient}$: the concentration of the original (unspiked) sample V $_{spike}$: the concentration of the spike added

In order to properly assess the degree of matrix interference and potential bias, the spiking level will be approximately 2-5 times the ambient concentration of the spiked sample but at least 3 times the reporting limit. If the MS or MSD is spiked too high or too low relative to the ambient concentration, the calculated recoveries are no longer an acceptable assessment of analytical bias. In order to establish spiking levels prior to analysis of samples, the laboratories will review any relevant historical data. In many instances, the laboratory will be spiking the samples blind and will not meet a spiking level of 2-5 times the ambient concentration. However, the results of affected samples will not be automatically rejected and will be reviewed on a case-by-case basis to determine if a different matrix spike will need to be performed.

In addition to the recoveries, the Relative Percent Difference (RPD) between the MS and MSD will be calculated to evaluate how matrix affects precision.

$$RPD = \left|\frac{V_{MS} - V_{MSD}}{mean}\right| x100$$

There are two different ways to calculate this RPD, depending on how the samples are spiked.

1) The samples are spiked with the same amount of analyte. In this case,

V $_{MS}$: the concentration for the matrix spike V $_{MSD}$: the concentration of the matrix spike duplicate mean: the mean of the two concentrations (MS + MSD)

2) The samples are spiked with different amounts of analyte. In this case,

V $_{MS}$: the recovery associated with the matrix spike V $_{MSD}$: the recovery associated with matrix spike duplicate mean: the mean of the two recoveries (recovery $_{MS}$ + recovery $_{MSD}$)

The MQO for the RPD between the MS and MSD is the same regardless of the method of calculation; detailed in Tables 9-10.

Laboratory Quality Control	Frequency of Analysis	Measurement Quality Objective
Calibration Standard	Per analytical method or manufacturer's specifications	Per analytical method or manufacturer's specifications
Continuing Calibration Verification	Per 10 analytical runs	80-120% recovery
Laboratory Blank	Per 20 samples or per batch, whichever is more frequent	<rl analyte<="" for="" target="" td=""></rl>
Reference Material	Per 20 samples or per batch, whichever is more frequent	75-125% recovery
Matrix Spike	Per 20 samples or per batch, whichever is more frequent	75-125% recovery
Matrix Spike Duplicate	Per 20 samples or per batch, whichever is more frequent	75-125% recovery, RPD ≤25%
Laboratory Duplicate	Per 20 samples or per batch, whichever is more frequent	RPD <25%; n/a if concentration of either sample <rl< td=""></rl<>
Internal Standard	Accompanying every analytical run when method appropriate	60-125% recovery

Table 9 Measurement Quality	v Ohiectives .	- inorganic analy	vtes in tissue
Tuble 5. Medsarement Quant	y objectives	mor Sume unun	yees in closue

*Unless method specifies more stringent requirements.

MDL = Method Detection Limit

RL = Reporting Limit

n/a = not applicable

Laboratory Quality Control	Frequency of Analysis	Measurement Quality Objective
Tuning ³	Per analytical method	Per analytical method
Calibration Standard	Initial method setup or when the calibration verification fails	PCBs For 6- or 7-point calibration, a relative standard deviation of the RRF's ≤20% for all compounds. Ion ratios for all congeners must be within ±15% of theoretical for CS-0.5. Minimum S:N ratio 10:1 for all calibration standards. For CS-0.5, S:N ratio may be as low as 3:1 for di-PCBs and nona-PCBs. Pesticides For opening and closing Cal Vers concentrations of native compounds must be within ±20% of expected values for targets with a labeled analog present, and within ±35% for targets with no labeled analog present. For opening Cal Vers concentrations of labeled compounds must be within ±35%
		of expected values.
Continuing Calibration Verification	Per 12 hours	Expected response or expected concentration ±20% RF for SPCCs=initial calibration ³

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Table 10. Measurement Quality	γ Οbjectiv	es ⁺ - synthetic	organic com	pounds in tissue ²

Laboratory Quality Control	Frequency of Analysis	Measurement Quality Objective
Laboratory Blank	Per 20 samples or per	PCB:
	analytical batch, whichever is more frequent	Congeners 77, 81, 114, 123, 126, 169: 2 pg/sample
		Congeners 156, 157, 167 and 189: 10 pg/sample
		Congener PCB 11: 150 pg/sample All other congeners: 50 pg/sample
		Total PCB* (sum of 209 congeners) 1100pg/sample Higher levels are acceptable where sample concentrations exceed 10 times the blank levels. Pesticides: Acceptance criteria is analyte specific. Allowable limits are
		between 1 ng/sample and 0.1 ng/sample or <10% of analyte value.
Reference Material	Per 20 samples or per analytical batch (preferably blind)	70-130% recovery if certified, otherwise 50-150% recovery
Laboratory Duplicate	Per 20 samples or per batch, whichever is more frequent	RPD <25%; n/a if concentration of either sample <rl< td=""></rl<>
Surrogate	Included in all samples and all QC samples	Based on laboratory control limits
Internal Standard	Included in all samples and all QC samples (as available)	Per laboratory procedure

¹ Unless method specifies more stringent requirements.

² All detected analytes must be confirmed with a second column, second technique, or mass spectrometry

³ Mass spectrometry only

MDL = method detection limit (to be determined according to the SWAMP QA Management Plan); RL = Reporting Limit; n/a = not applicable

7.1.2.1. Contamination Assessment – Method blanks

Laboratory method blanks (also called extraction blanks, procedural blanks, or preparation blanks) are used to assess bias from laboratory contamination during all stages of sample preparation and analysis. At least one laboratory method blank will be run in every sample batch of 20 or fewer field samples. The method blanks will be processed through the entire analytical procedure in a manner identical to the samples. The QC criterion for method blank analysis states that the blanks must be less than the Reporting Limit (<RL) for target analytes. If blank values exceed the RL, the sources of the contamination are determined and corrected, and in the case of method blanks, the previous samples associated with the blank are re-analyzed. All blank analysis results will be reported. If is not possible to eliminate the contamination source, all impacted analytes in the analytical batch will be flagged. In addition, a detailed description of the contamination sources and the steps taken to eliminate/minimize the contaminants will be included in interim and final reports.

7.1.2.2. Routine Monitoring of Method Performance for Organic Analysis – Surrogates

Surrogates are compounds chosen to simulate the analytes of interest in organic analyses to assess accuracy and bias. Surrogates are used to correct analyte concentrations for losses during the extraction and clean-up process, and must be added to each sample, including QC samples, prior to extraction. The surrogate recovery data will be carefully monitored. If possible, isotopically-labeled analogs of the analytes will be used as surrogates. Surrogate recoveries for each sample will be reported with the target analyte data. The surrogate is considered acceptable if the percent recovery is within method acceptance criteria.

7.2. Precision

Precision is the degree of agreement among repeated measurements of the same property under identical conditions (EPA QA/G-5, 2002). In order to evaluate the precision of an analytical process, a field sample will be selected and digested or extracted in duplicate. Following analysis, the results from the duplicate samples are evaluated by calculating the Relative Percent Difference (RPD).

$$RPD = \left| \frac{V_{sample} - V_{duplicate}}{mean} \right| x100$$

Where:

V sample: the concentration of the original sample digest

 $V_{\mbox{ duplicate}}$: the concentration of the duplicate sample digest mean: the mean concentration of both sample digests

The acceptance criteria for laboratory duplicates are specified in Tables 9-10.

A minimum of one duplicate per analytical batch will be analyzed. If the analytical precision is unacceptable, calculations and instruments will be checked. A repeat analysis may be required to confirm the results.

Duplicate precision is considered acceptable if the resulting RPD is < 25% for analyte concentrations that are greater than the Reporting Limit (RL).

7.2.1. Replicate Analysis

Replicate analyses are distinguished from duplicate analyses based simply on the number of involved analyses. Duplicate analyses refer to two sample digests, while replicate analyses refer to three or more. Analysis of replicate samples is not explicitly required; however, it is important to establish a consistent method of evaluating these analyses. The method of evaluating replicate analysis is by calculation of the relative standard deviation (RSD). Expressed as a percentage, the RSD is calculated as follows:

$$RSD = \left(\frac{Stdev(v_1, v_2, \dots v_n)}{mean}\right) x100$$

Where:

Stdev (v $_1$, v $_2$,..., v $_n$): the standard deviation of the values (concentrations) of the replicate analyses.

mean: the mean of the values (concentrations) of the replicate analyses.

7.5. Representativeness

The representativeness of the data is mainly dependent on the sampling locations and the sampling procedures adequately representing the true condition of the sample site. Requirements for selecting sample sites are discussed in more detail in the Monitoring Plan. Sample site selection, sampling of relevant media (water, sediment and biota), and use of only approved/documented analytical methods will determine that the measurement data does represent the conditions at the investigation site, to the extent possible.

7.6. Completeness

Completeness is defined as "a measure of the amount of data collected from a measurement process compared to the amount that was expected to be obtained under the conditions of measurement" (Stanley and Verner, 1985).

Field personnel will always strive to achieve or exceed the SWAMP completeness goals of 90% for fish samples when target species are present. Due to the variability and uncertainty of species availability in each zone, this level of completeness may not be attainable. If fish cannot be collected from a particular location, another location may be chosen to replace it. Additional locations will be chosen by the PI with input from Regional Board staff.

In the event field documentation is incomplete, datasheets will be returned to the collection crew for amendment.

Laboratories will strive for analytical completeness equal to or greater than 90%. In the event laboratory documentation is incomplete, datasheets will be returned to the dissector for amendment.

Occasionally digestates or extracts are rendered unusable for various reasons in the preparation process. If this occurs, the sample(s) affected will be re-processed.

7.7. Sensitivity

SWRCB adopted statewide tissue water quality objectives for methylmercury in fish in 2017. The objectives document states that "For any of the mercury fish tissue water quality objectives, measurements of total mercury concentrations in fish tissue may be substituted for methylmercury concentrations in fish tissue." Measurement of total mercury is more straightforward, so this is the approach used by SWAMP. A suite of objectives was adopted to protect different beneficial uses: a sport fish water quality objective of 0.2 ppm applicable to trophic level (TL) 3 or 4 fish; a tribal subsistence fishing water quality objective of 0.04 ppm for TL 3 or 4 fish; and a prey fish water quality objective of 0.05 ppm. SWAMP data should be usable for comparison to these objectives. The statewide tissue WQOs are being used by the Water Boards in the latest round of 303(d) listing determinations.

The analytical reporting limit for mercury (Table 14) is 0.03 ppm, lower than the lowest water quality objective (0.04 ppm).

OEHHA has established two sets of thresholds - fish contaminant goals (FCGs) and advisory tissue levels (ATLs) - that are relevant as selection criteria for lakes to be included in this study (Klasing and Brodberg [2008], Table 8). FCGs are health protective values for lifetime exposure and consider only the toxicity of the contaminants. They were developed by OEHHA to assist other agencies to establish fish tissue-based criteria for cleanup. For the two main chemicals of concern in this study, the FCGs are 0.22 ppm for mercury and 3.6 ppb for PCBs. The FCG for mercury (0.22 ppm) is of the same magnitude as the statewide tissue objective of 0.2 ppm, based only on toxicity and one serving per week of consumption. The Workgroup has opted to use the statewide tissue objective in lieu of FCGs for the current study, but it is important to be aware how similar these two numbers are. For organics, given their use in 303(d) listing determinations, the FCGs are a relevant benchmark to use in assessing the degree of contamination. To be confident that a lake truly has organics concentrations below FCGs, it is desirable to have measured concentrations in species such as catfish, carp, or sucker that are known to accumulate high concentrations. The RLs for DDTs and Chlordanes (Table 16) are sufficiently low to assess summed data for 303(d) listing determination; however, Dieldrin RL is slightly higher than the FCG. PCB RLs are not low enough to compare summed data to the relevant FCG. Limitations in analytical instrumentation and methods prevent lower RLs. Summation criteria are summarized in Element 24.

ATLs consider both the toxicity of contaminants and the health benefits of fish consumption. They are used to develop sport fish consumption advice for the public (MQ3). OEHHA has developed ATL ranges for one to seven servings per week. A comparison of the same

consumption frequency (one serving per week), shows that, for mercury, the low end of the ATL range (150 to 440 ppb) for the sensitive population (children and women of child-bearing age) encompasses the statewide tissue objective (200 ppb). For PCBs, the low end of the ATL range (21 ppb) for a 2 servings per week consumption rate was also considered as a lake selection criterion.

7.10. Comparability

Comparability expresses the measure of confidence that one dataset can be compared to and combined with another for a decision(s) to be made (US EPA QA/G-5, 2002). For this project, the methodologies for site selection, sample collection, analysis, data reporting, as well as the Measurement Quality Objectives (MQOs, Tables 9 and 10) have been used for the study historically and will be continued. This will ensure that the data collected by the project will be comparable to the data collected throughout the lifetime of the bioaccumulation program. Additionally, the Program coordinates with OEHHA to ensure that the project data can be combined with other sources of data to develop Fish Advisories.

Element 8. Special Training Requirements/Safety

8.1. Specialized training and safety requirements

Field and laboratory personnel are trained to conduct a wide variety of activities using standard protocols to ensure samples are collected and analyzed in a consistent manner. Training of each person includes the use of specialized field and/or laboratory equipment and conducting collection or analytical protocols, and other general processes including sample handling, glassware cleaning, sampling preparation and processing, hazardous materials handling, storage, and disposal. All staff must demonstrate proficiency in all the aforementioned and required laboratory activities that are conducted, as certified by the supervisor or LQAO. Training records are retained by individual supervisors or the LQAO as appropriate.

8.2. Training, safety and certification documentation

Staff and safety training is documented at Babcock, SGS-Axys and MPSL. Documentation consists of a record of the training date, instructor and signatures of completion. The LQAO will certify the proficiency of staff at chemical analyses. Certification and records are maintained and updated by the LQAO, or their designee, for all laboratory and field staff.

8.3. Training personnel

The Babcock, SGS-Axys, and MPSL Lab Director (LD) trains or appoints senior staff to train personnel within each lab. The LQAO ensures that training is given according to standard laboratory methods, maintains documentation and conducts performance audits to ensure that personnel have been trained properly.

8.3.1. Field Safety

Field personnel receive task specific safety training as needed by senior staff. Employees are required to review the safety program, and to have relevant safety equipment with them. This equipment may be related to vehicular, boating, or other work, and is task specific.

8.3.2. Laboratory Safety

New laboratory employees receive training in laboratory safety and chemical hygiene prior to performing any tasks in the laboratory. Employees are required to review the laboratory's safety program and chemical hygiene plan and acknowledge that they have read and understood the training. An experienced laboratory employee or the laboratory safety officer is assigned to the new employee to provide additional information and answer any questions related to safety that the new employee may have.

On-going safety training is provided by quarterly safety meetings conducted by the laboratory's safety officer or an annual laboratory safety class conducted by the Babcock Safety Officers and MLML Chemical Safety Officer.

8.3.3. Technical Training

New employees and employees required to learn new test methods are instructed to thoroughly review the appropriate standard operating procedure(s) (SOP) and are paired with a staff member who is experienced and qualified to teach those test methods and observe and evaluate performance. Employees learning new test methods work with experienced staff until they have demonstrated proficiency for the method both by observation and by obtaining acceptable results for QC samples. This demonstration of proficiency is documented and certified by the section leader, LQAO and the laboratory director prior to the person independently performing the test method. Training records are retained on file for each employee by their supervisor or QAO. On-going performance is monitored by reviewing QC sample results.

Element 9. Documentation and Records

The following documents, records, and electronic files will be produced:

- Quality Assurance Project Plan (submitted to Program Coordinator in electronic format)
- Monitoring Plan (submitted to Program Coordinator in electronic format)
- Archived Sample Sheets (internal documentation available on request)
- <u>Chain-of-Custody Forms</u> (exchanged for signatures with chemistry lab, and kept on file)
- Lab Sample Disposition Logs (internal documentation available on request)
- Refrigerator and Freezer Logs (internal documentation available on request)
- Quarterly Progress Reports (oral format to CM)
- Results in SWAMP format (submitted to SWAMP IQ in electronic format)
- Draft Interpretive Report (produced in electronic format)
- Final Interpretive Report (in electronic format)

- Data Appendix (submitted to Program Coordinator and CM electronic spreadsheet formats)
- Corrective Action Reports (submitted to Program QAO in electronic format upon request)

Copies of this QAPP will be distributed by the project manager to all parties directly involved in this project as well as uploaded the State Board website by SWAMP IQ. Any future amended QAPPs will be distributed in the same fashion. All originals of the first and subsequent amended QAPPs will be held at MPSL. Copies of versions, other than the most current, will be discarded to avoid confusion.

The final interpretive report will include summary data tables and an appendix that contains all project data in electronic SWAMP compatible spreadsheet format. All laboratory logs and data sheets will be maintained at the generating laboratory by the Laboratory Manager for five years following project completion and are available for review by the CM or designee during that time. Copies of reports will be maintained at SFEI for five years after project completion then discarded, except for the SWAMP database 2.5, which will be maintained without discarding. Laboratories will provide electronic copies of tabulated analytical data (including associated QA/QC information outlined below) in the SWAMP database format or a format agreed upon by the CM. All electronic data are stored on computer hard drives and electronic back-up files are created every two weeks or more frequently. Data will be made available to CEDEN by SWAMP IQ.

Laboratories will generate records for sample receipt and storage, analyses and reporting.

Laboratories maintain paper copies of all analytical data, field data forms and field notebooks, raw and condensed data for analysis performed on-site, and field instrument calibration notebooks.

The PM will be responsible for sending out the most current electronic copies of the approved QAPP to all appropriate persons listed in Table 1.

GROUP B ELEMENTS. DATA GENERATION AND ACQUISITION

Element 10. Sample Process Design

The project design is described in the Monitoring Plan. As much as possible, the same sampling locations visited in previous sampling will be visited again for this survey

Potential small fish and sport fish sampling equipment and methods can be found in <u>MPSL-102a</u>. Once samples have been identified for composite creation, they will be processed according to the timeline in Table 7.

All measurements and analyses to be performed in tissue are critical to address the objectives laid out in the Monitoring Plan, with the addition of selenium in composites of all species

analyzed for mercury. Fish weight, sex, age, and moisture content are not critical measurements. These parameters may be used to support other data gathered.

10.1. Variability

Due to potential variability of contaminant loads in individual tissue samples, samples will be analyzed in composites as outlined in the Monitoring Plan and MPSL SOPs.

10.2. Bias

Bias can be introduced by using fish of one particular species and/or total length for chemistry regressions and statistical analyses. The Monitoring Plan was reviewed by a Scientific Review Panel which approved of the inclusion of length ranges and multiple target species to reduce the associated bias.

Element 11. Sampling Methods

Fish will be collected in accordance with MPSL-102a, Section 13.4 except where noted here. Because habitats may vary greatly, field crews will evaluate each fishing site and species targeted to determine the correct method to be employed. Potential sampling methods include, but are not limited to: electroshocking, seining, gill netting, and hook and line. Field Crew will determine the appropriate collection method based on physical site parameters such as depth, width, flow, and accessibility. Field crew will indicate collection method on <u>field data</u> <u>sheets</u>.

Details on targeted fish species, number of individuals and size ranges can be found in the Monitoring Plan.

The following adaptation to MPSL-102a, Section 13.4.6 has been made: Collected fish may be partially dissected in the field. At the dock, the fish is placed on a measuring board covered with new clear 33-gallon trash bag; fork and total length are recorded. Weight is recorded. Large fish such as carp will then be placed on the covered cutting board where the head, tail, and guts are removed using a clean cleaver (scrubbed with Micro[™], rinsed with tap and deionized water). The fish cross section is tagged with a unique numbered ID, wrapped in aluminum foil, and placed in a clean labeled bag. When possible, parasites and body anomalies are noted. The cutting board is covered with a new bag, and the cleaver is re-cleaned with Micro[™], rinsed with tap and deionized water between fish species, per site if multiple stations are sampled.

Special care is being taken to prevent the potential contamination of invasive species from one location to another (CDFW 2013). A 0.2% Lavender-Quat solution is sprayed on all boat, collection, and personal gear components that come into contact with ambient water from each location. In addition, a visual inspection of the boat or equipment is conducted to ensure any algae or other organisms are not transferred between locations. Furthermore, boat bilges are verified to be dry before the boat is launched into a location.

Further details on sample collection and processing can be found in the Monitoring Plan (Appendix II).

11.1. Corrective Action

In the event samples cannot be collected, the Sample Collection Coordinator will determine if corrective actions are appropriate. Table 11 describes action to take in the event of a collection failure.

Collection Failure	Corrective Action
Target Species not present	Collect secondary target; it is advisable to consult with OEHHA prior to choosing secondary target species; document the occurrence.
No Fish present	Inform PM and move on to another location; document the occurrence; PM and Lead Scientist may replace with next lake on the alternate list.
Water body not able to be sampled	Replace with next lake on the alternate list.

Table 11. Field collection corrective actions

Element 12. Sample Handling and Custody

The field coordinator will be responsible for ensuring that each field sampling team adheres to proper custody and documentation procedures. A master sample logbook of field data sheets shall be maintained for all samples collected during each sampling event. A chain-of-custody form must be completed after sample collection, archive storage, and prior to sample release.

Fish samples will be wrapped in aluminum foil and frozen on dry ice for transportation to the storage freezer or laboratory, where they will be stored at -20°C until dissection and homogenization. Samples delivered to MPSL will be logged in according to <u>MPSL-104</u>.

Samples will be dissected according to <u>MPSL-105</u>, taking care to exclude any exposed flesh that may be contaminated for target analytes, and data retained on the <u>lab data sheets</u>.

Lab homogenates will be frozen until analysis is performed. Frozen tissue samples have a 12 month hold time from the date of collection. If a hold-time violation has occurred, the PM and Regional Coordinator(s) will be notified. Affected data will be flagged appropriately in the final results submitted to SWAMP IQ.

Organic compounds frequently have 40-day hold times between extraction and analysis. Please refer to the appropriate method for specific holding time requirements. Violations will be flagged appropriately in the final results, and the PM and Regional Coordinator(s) will be notified. This type of hold time is not applicable to metals and metalloids.

Holding times for each analyte can be found in Table 12.

Parameter	Container	Preservation	Holding Time
Mercury	Wrapped in foil, zip top bag; Polyethylene	Cool to ≤6°C within 24 hours, then freeze to ≤-20°C	1 year
Selenium	Wrapped in foil, zip top bag; Polyethylene	Cool to ≤6°C within 24 hours, then freeze to ≤-20°C	1 year
Polychlorinated Biphenyls	Wrapped in foil, zip top bag; Glass	Cool to ≤6°C within 24 hours, then freeze to ≤-20°C	1 year; samples must be extracted within 14 days of thawing and analyzed within 40 days of extraction
Organochlorine Pesticides	Wrapped in foil, zip top bag; Glass	Cool to ≤6°C within 24 hours, then freeze to ≤-20°C	1 year; samples must be extracted within 14 days of thawing and analyzed within 40 days of extraction

Table 12.	Sample	handling	and hol	lding time	s for tissue
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Element 13. Analytical Methods

Methods and equipment for laboratory analyses are listed in Table 13. USEPA methods can be downloaded from <u>www.nemi.gov</u>. USEPA method numbers followed by "M" or "MLA-XXX" indicate modifications have been made. Modifications and non-USEPA SOPs can be obtained through hyperlinks in this document or by contacting the LQAO (Table 2), as can method validation data for modifications and SOPs.

Parameter	Method	Instrument
Mercury	EPA 7473 (USEPA 2007a)	Milestone DMA 80
Selenium	EPA 3052M (USEPA 1996a*)	CEM MARSXpress Digester
	EPA 200.8M (USEPA 1994a*)	Perkin-Elmer NexION 1000 ICP-MS
Polychlorinated Biphenyls	EPA 1668A (MLA- 010) (USEPA 2003*)	Micromass Ultima high resolution mass spectrometer equipped with an HP 6890 gas chromatograph using an SPB- Octyl column (30 m, 0.25 mm I.D., 0.25 μm film thickness)
Organochlorine Pesticides	EPA 1699 (MLA-028) (USEPA 2007b*)	Micromass Ultima high resolution mass spectrometer equipped with an HP 6890 gas chromatograph using a DB-5 column (60 m, 0.25 mm i.d., 0.10 µm film thickness)

 Table 13. Methods for laboratory analyses

*Contact LQAO for method modifications

Mercury in fish tissues will be analyzed according to EPA 7473, "Mercury in Solids and Solutions by Thermal Decomposition, Amalgamation, and Atomic Absorption Spectrophotometry" (USEPA, 2007) using a Direct Mercury Analyzer (DMA 80). Samples, blanks, and standards will be prepared using clean techniques. ASTM Type II water and analytical grade chemicals will be used for all standard preparations. A continuing calibration verification (CCV) will be performed after every 10 samples. Initial and continuing calibration verification values must be within ±20% of the true value, or the previous 10 samples must be reanalyzed. Three blanks, a CRM (DORM-4 or similar), a method duplicate, and an MS pair will be run with each analytical batch of samples. RLs can be found in Table 14 and MQOs in Section 7, Table 9.

Selenium sport and small fish composites will be digested according to <u>EPA 3052M</u>, "Microwave Assisted Acid Digestion of Siliceous and Organically Based Matrices" (USEPA, 1996a), modified (Appendix III E), and will be analyzed according to <u>EPA 200.8M</u>, "Determination of Trace Elements in Waters and Wastes by Inductively Coupled Plasma-Mass Spectrometry" (USEPA, 1994a). Samples, blanks, and standards will be prepared using clean techniques. ASTM Type II water and analytical grade chemicals will be used for all standard preparations. A CCV will be performed after every 10 samples. Initial and continuing calibration verification values must be within ±20% of the true value, or the previous 10 samples must be reanalyzed. Two blanks, a certified reference material (NIST 2976, NRCC DORM-4 or similar), as well as a method duplicate, and a matrix spike pair will be run with each set of samples. RLs can be found in Table 14 and MQOs in Section 7, Table 9.

Table 14. Trace metal analytical parameters, reporting units and target reporting limits (RL) in tissue

Parameter	Method	Target RL
Mercury	EPA 7473 (USEPA 2007a)	0.030 μg/g wet wt
Selenium	EPA 200.8M* (USEPA 1994a)	0.70 μg/g wet wt

*Contact LQAO for method modifications

Organochlorine and PCB compounds will be extracted following EPA Methods 1699 (MLA-028), and 1668 (MLA-010), respectively (USEPA 2007b, 2003). Organochlorine pesticides will be analyzed according to EPA 1699, "Pesticides in Water, Soil, Sediment, Biosolids, and Tissue by HRGC/HRMS" (USEPA 1996b. PCBs will be analyzed according to EPA 1668A, "Chlorinated Biphenyl Congeners in Water, Soil, Sediment, Biosolids, and Tissues by HRGC/HRMS. Revision A (USEPA 2003). Samples, blanks, and standards will be prepared using clean techniques. ASTM Type II water and analytical grade chemicals will be used for all standard preparations. A CCV will be performed after every 12 hours. Initial and continuing calibration verification values must be within ±30% of the true value, or the previous samples must be reanalyzed. One blank, a laboratory control spike (LCS), and a method duplicate will be run with each set of samples. RLs can be found in Tables 15 and 16, and MQOs in Section 7, Table 10.

Table 15. Polychlorinated biphenyl analytical parameters, reporting units, and target
reporting limits (RL) for tissue

Analyte	Analytical Method	Target RL
CL1-PCB-1 ⁺	EPA 1668A (MLA-010*)	3.0 pg/g wet wt
CL1-PCB-2 [‡]	EPA 1668A (MLA-010*)	3.0 pg/g wet wt
CL1-PCB-3 [‡]	EPA 1668A (MLA-010*)	3.0 pg/g wet wt
CL2-PCB-4 [‡]	EPA 1668A (MLA-010*)	3.0 pg/g wet wt
CL2-PCB-5 ⁺	EPA 1668A (MLA-010*)	3.0 pg/g wet wt
CL2-PCB-6 [‡]	EPA 1668A (MLA-010*)	3.0 pg/g wet wt
CL2-PCB-7 [‡]	EPA 1668A (MLA-010*)	3.0 pg/g wet wt
CL2-PCB-8	EPA 1668A (MLA-010*)	3.0 pg/g wet wt
CL2-PCB-9 [‡]	EPA 1668A (MLA-010*)	3.0 pg/g wet wt
CL2-PCB-10 [‡]	EPA 1668A (MLA-010*)	3.0 pg/g wet wt
CL2-PCB-11	EPA 1668A (MLA-010*)	6.2 pg/g wet wt
CL2-PCB-12/13 [‡]	EPA 1668A (MLA-010*)	3.0 pg/g wet wt
CL2-PCB-14 [‡]	EPA 1668A (MLA-010*)	3.0 pg/g wet wt
CL2-PCB-15 [‡]	EPA 1668A (MLA-010*)	3.0 pg/g wet wt
CL3-PCB-16 [‡]	EPA 1668A (MLA-010*)	3.0 pg/g wet wt
CL3-PCB-17 [‡]	EPA 1668A (MLA-010*)	3.0 pg/g wet wt
CL3-PCB-19 [‡]	EPA 1668A (MLA-010*)	3.0 pg/g wet wt

Analyte	Analytical Method	Target RL
CL3-PCB-21/33	EPA 1668A (MLA-010*)	3.0 pg/g wet wt
CL3-PCB-22 [‡]	EPA 1668A (MLA-010*)	3.0 pg/g wet wt
CL3-PCB-23 [‡]	EPA 1668A (MLA-010*)	3.0 pg/g wet wt
CL3-PCB-24 [‡]	EPA 1668A (MLA-010*)	3.0 pg/g wet wt
CL3-PCB-25 [‡]	EPA 1668A (MLA-010*)	3.0 pg/g wet wt
CL3-PCB-26/29	EPA 1668A (MLA-010*)	3.0 pg/g wet wt
CL3-PCB-27	EPA 1668A (MLA-010*)	3.0 pg/g wet wt
CL3-PCB-28/20	EPA 1668A (MLA-010*)	3.0 pg/g wet wt
CL3-PCB-30/18	EPA 1668A (MLA-010*)	3.0 pg/g wet wt
CL3-PCB-31	EPA 1668A (MLA-010*)	3.0 pg/g wet wt
CL3-PCB-32 [‡]	EPA 1668A (MLA-010*)	3.0 pg/g wet wt
CL3-PCB-34 [‡]	EPA 1668A (MLA-010*)	3.0 pg/g wet wt
CL3-PCB-35 [‡]	EPA 1668A (MLA-010*)	3.0 pg/g wet wt
CL3-PCB-36 [‡]	EPA 1668A (MLA-010*)	3.0 pg/g wet wt
CL3-PCB-37 ⁺	EPA 1668A (MLA-010*)	3.0 pg/g wet wt
CL3-PCB-38 [‡]	EPA 1668A (MLA-010*)	3.0 pg/g wet wt
CL3-PCB-39 [‡]	EPA 1668A (MLA-010*)	3.0 pg/g wet wt
CL4-PCB-41 [‡] /40 [‡] /71 [‡]	EPA 1668A (MLA-010*)	3.0 pg/g wet wt
CL4-PCB-42 [‡]	EPA 1668A (MLA-010*)	3.0 pg/g wet wt
CL4-PCB-43 [‡]	EPA 1668A (MLA-010*)	3.0 pg/g wet wt
CL4-PCB-44/47 [‡] /65 [‡]	EPA 1668A (MLA-010*)	3.0 pg/g wet wt
CL4-PCB-45 [‡] /51 [‡]	EPA 1668A (MLA-010*)	3.0 pg/g wet wt
CL4-PCB-46 [‡]	EPA 1668A (MLA-010*)	3.0 pg/g wet wt
CL4-PCB-48 [‡]	EPA 1668A (MLA-010*)	3.0 pg/g wet wt
CL4-PCB-50 [‡] /53 [‡]	EPA 1668A (MLA-010*)	3.0 pg/g wet wt
CL4-PCB-52	EPA 1668A (MLA-010*)	3.0 pg/g wet wt
CL4-PCB-54 [‡]	EPA 1668A (MLA-010*)	3.0 pg/g wet wt
CL4-PCB-55 [‡]	EPA 1668A (MLA-010*)	3.0 pg/g wet wt
CL4-PCB-56	EPA 1668A (MLA-010*)	3.0 pg/g wet wt
CL4-PCB-57 [‡]	EPA 1668A (MLA-010*)	3.0 pg/g wet wt
CL4-PCB-58 [‡]	EPA 1668A (MLA-010*)	3.0 pg/g wet wt
CL4-PCB-59 */62 */75 *	EPA 1668A (MLA-010*)	3.0 pg/g wet wt
CL4-PCB-60	EPA 1668A (MLA-010*)	3.0 pg/g wet wt
CL4-PCB-61 [‡] /70/74/76 [‡]	EPA 1668A (MLA-010*)	3.0 pg/g wet wt
CL4-PCB-63 [‡]	EPA 1668A (MLA-010*)	3.0 pg/g wet wt

Analyte	Analytical Method	Target RL
CL4-PCB-64	EPA 1668A (MLA-010*)	3.0 pg/g wet wt
CL4-PCB-66	EPA 1668A (MLA-010*)	3.0 pg/g wet wt
CL4-PCB-67 [‡]	EPA 1668A (MLA-010*)	3.0 pg/g wet wt
CL4-PCB-68 [‡]	EPA 1668A (MLA-010*)	3.0 pg/g wet wt
CL4-PCB-69 [‡] /49	EPA 1668A (MLA-010*)	3.0 pg/g wet wt
CL4-PCB-72 [‡]	EPA 1668A (MLA-010*)	3.0 pg/g wet wt
CL4-PCB-73 [‡]	EPA 1668A (MLA-010*)	3.0 pg/g wet wt
CL4-PCB-77	EPA 1668A (MLA-010*)	3.0 pg/g wet wt
CL4-PCB-78 [‡]	EPA 1668A (MLA-010*)	3.0 pg/g wet wt
CL4-PCB-79 [‡]	EPA 1668A (MLA-010*)	3.0 pg/g wet wt
CL4-PCB-80 ⁺	EPA 1668A (MLA-010*)	3.0 pg/g wet wt
CL4-PCB-81 [‡]	EPA 1668A (MLA-010*)	3.0 pg/g wet wt
CL5-PCB-82 [‡]	EPA 1668A (MLA-010*)	3.0 pg/g wet wt
CL5-PCB-83 [‡] /99	EPA 1668A (MLA-010*)	3.0 pg/g wet wt
CL5-PCB-84 [‡]	EPA 1668A (MLA-010*)	3.0 pg/g wet wt
CL5-PCB-88 [‡] /91 [‡]	EPA 1668A (MLA-010*)	3.0 pg/g wet wt
CL5-PCB-89 [‡]	EPA 1668A (MLA-010*)	3.0 pg/g wet wt
CL5-PCB-92 [‡]	EPA 1668A (MLA-010*)	3.0 pg/g wet wt
CL5-PCB-94 [‡]	EPA 1668A (MLA-010*)	3.0 pg/g wet wt
CL5-PCB-95 [‡] /100 [‡] /93 [‡] /102 [‡] /98 [‡]	EPA 1668A (MLA-010*)	3.0 pg/g wet wt
CL5-PCB-96 [‡]	EPA 1668A (MLA-010*)	3.0 pg/g wet wt
CL5-PCB-103 [‡]	EPA 1668A (MLA-010*)	3.0 pg/g wet wt
CL5-PCB-104 [‡]	EPA 1668A (MLA-010*)	3.0 pg/g wet wt
CL5-PCB-105	EPA 1668A (MLA-010*)	3.0 pg/g wet wt
CL5-PCB-106 [‡]	EPA 1668A (MLA-010*)	3.0 pg/g wet wt
CL5-PCB-107 [‡] /124 [‡]	EPA 1668A (MLA-010*)	3.0 pg/g wet wt
CL5-PCB-108 [‡] /119 ⁺ /86 [‡] /97/125 [‡] /87	EPA 1668A (MLA-010*)	3.0 pg/g wet wt
CL5-PCB-109 [‡]	EPA 1668A (MLA-010*)	3.0 pg/g wet wt
CL5-PCB-110/115 [‡]	EPA 1668A (MLA-010*)	3.0 pg/g wet wt
CL5-PCB-111 [‡]	EPA 1668A (MLA-010*)	3.0 pg/g wet wt
CL5-PCB-112 [‡]	EPA 1668A (MLA-010*)	3.0 pg/g wet wt
CL5-PCB-113 [‡] /90 ⁺ /101	EPA 1668A (MLA-010*)	3.0 pg/g wet wt
CL5-PCB-114	EPA 1668A (MLA-010*)	3.0 pg/g wet wt
CL5-PCB-117 [‡] /116 [‡] /85 [‡]	EPA 1668A (MLA-010*)	3.0 pg/g wet wt
CL5-PCB-118	EPA 1668A (MLA-010*)	3.0 pg/g wet wt

Analyte	Analytical Method	Target RL
CL5-PCB-120 [‡]	EPA 1668A (MLA-010*)	3.0 pg/g wet wt
CL5-PCB-121 [‡]	EPA 1668A (MLA-010*)	3.0 pg/g wet wt
CL5-PCB-122 [‡]	EPA 1668A (MLA-010*)	3.0 pg/g wet wt
CL5-PCB-123 ⁺	EPA 1668A (MLA-010*)	3.0 pg/g wet wt
CL5-PCB-126	EPA 1668A (MLA-010*)	3.0 pg/g wet wt
CL5-PCB-127 [‡]	EPA 1668A (MLA-010*)	3.0 pg/g wet wt
CL6-PCB-128/166 +	EPA 1668A (MLA-010*)	3.0 pg/g wet wt
CL6-PCB-130 [‡]	EPA 1668A (MLA-010*)	3.0 pg/g wet wt
CL6-PCB-131 [‡]	EPA 1668A (MLA-010*)	3.0 pg/g wet wt
CL6-PCB-132 ⁺	EPA 1668A (MLA-010*)	3.0 pg/g wet wt
CL6-PCB-133 [‡]	EPA 1668A (MLA-010*)	3.0 pg/g wet wt
CL6-PCB-134 [‡] /143 [‡]	EPA 1668A (MLA-010*)	3.0 pg/g wet wt
CL6-PCB-136 [‡]	EPA 1668A (MLA-010*)	3.0 pg/g wet wt
CL6-PCB-137	EPA 1668A (MLA-010*)	3.0 pg/g wet wt
CL6-PCB-138/163 */129 */160 *	EPA 1668A (MLA-010*)	3.0 pg/g wet wt
CL6-PCB-139 [‡] /140 [‡]	EPA 1668A (MLA-010*)	3.0 pg/g wet wt
CL6-PCB-141	EPA 1668A (MLA-010*)	3.0 pg/g wet wt
CL6-PCB-142 [‡]	EPA 1668A (MLA-010*)	3.0 pg/g wet wt
CL6-PCB-144 [‡]	EPA 1668A (MLA-010*)	3.0 pg/g wet wt
CL6-PCB-145 [‡]	EPA 1668A (MLA-010*)	3.0 pg/g wet wt
CL6-PCB-146	EPA 1668A (MLA-010*)	3.0 pg/g wet wt
CL6-PCB-147 [‡] /149	EPA 1668A (MLA-010*)	3.0 pg/g wet wt
CL6-PCB-148 [‡]	EPA 1668A (MLA-010*)	3.0 pg/g wet wt
CL6-PCB-150 [‡]	EPA 1668A (MLA-010*)	3.0 pg/g wet wt
CL6-PCB-151/135 [‡] /154 [‡]	EPA 1668A (MLA-010*)	3.0 pg/g wet wt
CL6-PCB-152 [‡]	EPA 1668A (MLA-010*)	3.0 pg/g wet wt
CL6-PCB-153/168 +	EPA 1668A (MLA-010*)	3.0 pg/g wet wt
CL6-PCB-155 [‡]	EPA 1668A (MLA-010*)	3.0 pg/g wet wt
CL6-PCB-156 [‡] /157	EPA 1668A (MLA-010*)	3.0 pg/g wet wt
CL6-PCB-158	EPA 1668A (MLA-010*)	3.0 pg/g wet wt
CL6-PCB-159 [‡]	EPA 1668A (MLA-010*)	3.0 pg/g wet wt
CL6-PCB-161 [‡]	EPA 1668A (MLA-010*)	3.0 pg/g wet wt
CL6-PCB-162 [‡]	EPA 1668A (MLA-010*)	3.0 pg/g wet wt
CL6-PCB-164 [‡]	EPA 1668A (MLA-010*)	3.0 pg/g wet wt
CL6-PCB-165 [‡]	EPA 1668A (MLA-010*)	3.0 pg/g wet wt

Analyte	Analytical Method	Target RL
CL6-PCB-167 ⁺	EPA 1668A (MLA-010*)	3.0 pg/g wet wt
CL6-PCB-169	EPA 1668A (MLA-010*)	3.0 pg/g wet wt
CL7-PCB-170	EPA 1668A (MLA-010*)	3.0 pg/g wet wt
CL7-PCB-171 [‡] /173 [‡]	EPA 1668A (MLA-010*)	3.0 pg/g wet wt
CL7-PCB-172 [‡]	EPA 1668A (MLA-010*)	3.0 pg/g wet wt
CL7-PCB-174	EPA 1668A (MLA-010*)	3.0 pg/g wet wt
CL7-PCB-175 [‡]	EPA 1668A (MLA-010*)	3.0 pg/g wet wt
CL7-PCB-176 [‡]	EPA 1668A (MLA-010*)	3.0 pg/g wet wt
CL7-PCB-177	EPA 1668A (MLA-010*)	3.0 pg/g wet wt
CL7-PCB-178 [‡]	EPA 1668A (MLA-010*)	3.0 pg/g wet wt
CL7-PCB-179 [‡]	EPA 1668A (MLA-010*)	3.0 pg/g wet wt
CL7-PCB-180/193 [‡]	EPA 1668A (MLA-010*)	3.0 pg/g wet wt
CL7-PCB-181 [‡]	EPA 1668A (MLA-010*)	3.0 pg/g wet wt
CL7-PCB-182 [‡]	EPA 1668A (MLA-010*)	3.0 pg/g wet wt
CL7-PCB-183/185 [‡]	EPA 1668A (MLA-010*)	3.0 pg/g wet wt
CL7-PCB-184 ⁺	EPA 1668A (MLA-010*)	3.0 pg/g wet wt
CL7-PCB-186 [‡]	EPA 1668A (MLA-010*)	3.0 pg/g wet wt
CL7-PCB-187	EPA 1668A (MLA-010*)	3.0 pg/g wet wt
CL7-PCB-188 [‡]	EPA 1668A (MLA-010*)	3.0 pg/g wet wt
CL7-PCB-189	EPA 1668A (MLA-010*)	3.0 pg/g wet wt
CL7-PCB-190 [‡]	EPA 1668A (MLA-010*)	3.0 pg/g wet wt
CL7-PCB-191 [‡]	EPA 1668A (MLA-010*)	3.0 pg/g wet wt
CL7-PCB-192 [‡]	EPA 1668A (MLA-010*)	3.0 pg/g wet wt
CL8-PCB-194	EPA 1668A (MLA-010*)	3.0 pg/g wet wt
CL8-PCB-195	EPA 1668A (MLA-010*)	3.0 pg/g wet wt
CL8-PCB-196 [‡]	EPA 1668A (MLA-010*)	3.0 pg/g wet wt
CL8-PCB-197 [‡] /200	EPA 1668A (MLA-010*)	3.0 pg/g wet wt
CL8-PCB-198/199	EPA 1668A (MLA-010*)	3.0 pg/g wet wt
CL8-PCB-201	EPA 1668A (MLA-010*)	3.0 pg/g wet wt
CL8-PCB-202 [‡]	EPA 1668A (MLA-010*)	3.0 pg/g wet wt
CL8-PCB-203	EPA 1668A (MLA-010*)	3.0 pg/g wet wt
CL8-PCB-204 [‡]	EPA 1668A (MLA-010*)	3.0 pg/g wet wt
CL8-PCB-205 [‡]	EPA 1668A (MLA-010*)	3.0 pg/g wet wt
CL9-PCB-206	EPA 1668A (MLA-010*)	3.0 pg/g wet wt
CL9-PCB-207 [‡]	EPA 1668A (MLA-010*)	3.0 pg/g wet wt

Analyte	Analytical Method	Target RL
CL9-PCB-208 [‡]	EPA 1668A (MLA-010*)	3.0 pg/g wet wt
CL10-PCB-209	EPA 1668A (MLA-010*)	3.0 pg/g wet wt

⁺ New to analyte list in 2017
[‡] New to analyte list in 2021

*Contact LQAO for method modifications

Table 16. Organochlorine pesticide analytical parameters, reporting units, and target
reporting limits (RL) for tissue

Analyte Group	Analyte	Analytical Method	Target RL	
Chlordanes	Chlordane, cis-	EPA 1699 (MLA-028)	0.4 ng/g wet wt	
	Chlordane, trans-	EPA 1699 (MLA-028)	0.4 ng/g wet wt	
	Heptachlor	EPA 1699 (MLA-028)	0.2 ng/g wet wt	
	Heptachlor epoxide	EPA 1699 (MLA-028)	0.64 ng/g wet wt	
	Nonachlor, cis-	EPA 1699 (MLA-028)	0.4 ng/g wet wt	
	Nonachlor, trans-	EPA 1699 (MLA-028)	0.4 ng/g wet wt	
	Oxychlordane	EPA 1699 (MLA-028)	0.4 ng/g wet wt	
DDTs	DDD(o,p')	EPA 1699 (MLA-028)	0.2 ng/g wet wt	
	DDD(p,p')	EPA 1699 (MLA-028)	0.2 ng/g wet wt	
	DDE(o,p')	EPA 1699 (MLA-028)	0.2 ng/g wet wt	
	DDE(p,p')	EPA 1699 (MLA-028)	0.2 ng/g wet wt	
	DDMU(p,p') *	Not currently available	Not currently available	
	DDT(o,p')	EPA 1699 (MLA-028)	0.2 ng/g wet wt	
	DDT(p,p')	EPA 1699 (MLA-028)	0.2 ng/g wet wt	
Cyclodienes	Aldrin	EPA 1699 (MLA-028)	0.416 ng/g wet wt	
	Dieldrin	EPA 1699 (MLA-028)	0.324 ng/g wet wt	
	Endrin	EPA 1699 (MLA-028)	0.651 ng/g wet wt	
HCHs	HCH, alpha	EPA 1699 (MLA-028)	0.4 ng/g wet wt	
	HCH, beta	EPA 1699 (MLA-028)	0.4 ng/g wet wt	
Others	Dacthal *	EPA 1699 (MLA-028)	Not currently available	
	Endosulfan I	EPA 1699 (MLA-028)	0.643 ng/g wet wt	
	Hexachlorobenzene	EPA 1699 (MLA-028)	0.2 ng/g wet wt	
	Methoxychlor	EPA 1699 (MLA-028)	0.328 ng/g wet wt	
	Mirex	EPA 1699 (MLA-028)	0.2 ng/g wet wt	
	Oxadiazon	MLA-028	Not Applicable	

* Not available from SGS-Axys but the Workgroup is still interested in analysis for future projects

13.2.1. Corrective Action

It is the responsibility of each analyst to take corrective action upon instrument failure. Corrective action will be conducted according to manufacturer, method specifications, or SWAMP specifications (see <u>MQO documents</u>). Additional information on corrective actions can be found in Section 20.2.

13.2.2. Turn-around time

All analyses must be completed within holding time specific to each analyte (Table 12). In addition, results need to be reported according to the timeline outlined in Table 7.

13.3. Sample Disposal

The laboratories are responsible for complying with all Federal, State and local regulations governing waste management, particularly hazardous waste identification rules and land disposal restrictions. Chemicals must be appropriately neutralized prior to disposal or must be handled as hazardous waste.

Element 14. Quality Control

MPSL and SGS-Axys conduct quality control through several activities and methods. These methods of quality control are performed to identify possible contamination problem(s), matrix interference and the ability to duplicate/repeat results. When control limits are exceeded the LQAO will review with appropriate laboratory staff to ascertain the possible cause of the exceedance. A review of SOPs will be conducted, and any deficiencies will be identified, documented, and corrected. A written report of the corrective action(s) will be provided to the LS and PM via email. The PM will contact the Program QAO as needed.

Each aspect of laboratory quality control is listed in Tables 9-10 for frequency as well as MQOs for each.

Element 15. Instrument/Equipment Testing, Inspection and Maintenance

Field equipment such as boats, nets, traps, etc., are inspected prior to each sampling event and are maintained throughout the field season and prior to storage during the off-season.

Laboratory instruments are inspected and maintained in accordance with lab SOPs, which include those specified by the manufacturer and those specified by the method (Table 17). These SOPs have been reviewed by each respective LQAO and found to be in compliance with SWAMP criteria. Analysts are responsible for equipment testing, inspection, and maintenance. Appendices III and IV list the referenced SOPs. SGS-Axys SOPs are available upon request from

the Laboratory Director by email: <u>Sean.campbell@sgs.com</u>. Likewise, MPSL SOPS are available from the MPSL QA officer by email: <u>autumn.bonnema@sjsu.edu</u>.

Electronic laboratory equipment usually has recommended maintenance prescribed by the manufacturer. These instructions will be followed as a minimum requirement. Due to the cost of some laboratory equipment, back up capability may not be possible. But all commonly replaced parts will have spares available for rapid maintenance of failed equipment. Such parts include but are not limited to: batteries; tubes; light bulbs; tubing of all kinds; replacement specific ion electrodes; electrical conduits; glassware; pumps; etc.

The lead chemist, or designee, is responsible for the testing, inspection, and maintenance of equipment. Each instrument has its own logbook where the results of tests, inspections, maintenance and repairs are documented. When an instrument's test results fail to meet accuracy and/or precision criteria after the lead chemist has performed maintenance, the manufacturer will be contacted.

Element 16. Instrument/Equipment Calibration and Frequency

Laboratory instruments (listed in Table 17) are calibrated, standardized and maintained according to procedures detailed in the laboratory Quality Assurance Manuals (listed in Appendix I). Instrument manuals identify step-by-step calibration and maintenance procedures. If analytical instrumentation fails to meet performance requirements, the instrument(s) will be checked according to their respective SOP(s) and recalibrated. If the instrument(s) still does not meet specifications, it will be repaired and retested until performance criteria are achieved. The maintenance will be entered in the instrument log. If sample analytical information is in question due to instrument performance, the PM will be contacted regarding the proper course of action including reanalyzing the sample(s).

At a minimum all calibration procedures will meet the requirements specified in the US EPA approved methods of analysis. The means and frequency of calibration recommended by the manufacturer of the equipment or devices as well as any instruction given in an analytical method will be followed. When such information is not specified by the method, instrument calibration will be performed at least once daily and continuing calibration will be performed on a 10% basis thereafter except for analysis by GC/MS. It is also required that records of calibration be kept by the person performing the calibration and be accessible for verification during either a laboratory or field audit.

Instrument	Inspection/Maintenance Frequency	Calibration Frequency
Milestone DMA-80 Direct Mercury Analyzer (MPSL)	As needed	At least once every 2 weeks
Perkin-Elmer NexION 1000 Inductively Coupled Plasma - Mass Spectrometer (MPSL)	As needed	At least once daily
Micromass Ultima high resolution mass spectrometer equipped with an HP 6890 gas chromatograph using an SPB- Octyl column (30 m, 0.25 mm I.D., 0.25 µm film thickness	As needed	If the 12-hour calibration verification test does not meet specification and this cannot be corrected by performing Minor Instrumental Maintenance Procedures.
Micromass Ultima high resolution mass spectrometer equipped with an HP 6890 gas chromatograph using a DB-5 column (60 m, 0.25 mm i.d., 0.10 µm film thickness)	As needed	If the 12-hour calibration verification test does not meet specification and this cannot be corrected by performing Minor Instrumental Maintenance Procedures. After all Major Instrumental Maintenance Procedures. If more than 180 days have elapsed since the last verified Initial Calibration

Table 17. Equipment maintenance and calibration frequency

16.1. Analytical Instrumentation

16.1.1. Instrument calibration

Upon initiation of an analytical run, after each major equipment disruption, and whenever ongoing calibration checks do not meet recommended MQOs, the system will be calibrated with a full range of analytical standards. Immediately after this procedure, the initial calibration must be verified through the analysis of a standard obtained from a different source than the standards used to calibrate the instrumentation, prepared in an independent manner, and ideally having certified concentrations of target analytes of a CRM or certified solution. Frequently, calibration standards (CCVs) are included as part of an analytical run, interspersed with actual samples. However, this practice does not document the stability of the calibration and is incapable of detecting degradation of individual components, particularly pesticides, in

standard solutions used to calibrate the instrument. The calibration curve is acceptable if it has an R² of 0.990 or greater for all analytes present in the calibration mixtures. If not, the calibration standards, as well as all the samples in the batch are re-analyzed. All calibration standards will be traceable to a recognized organization for the preparation and certification of QC materials (e.g., National Institute of Standards and Technology [NIST], National Research Council Canada [NRCC], US EPA, etc.).

Calibration curves will be established for each analyte and batch analysis from a calibration blank and a minimum of three analytical standards of increasing concentration, covering the range of expected sample concentrations. Only data which result from quantification within the demonstrated working calibration range may be reported (i.e., quantification based on extrapolation is not acceptable). Alternatively, if the instrumentation is linear over the concentration ranges to be measured in the samples, the use of a calibration blank and one single standard that is higher in concentration than the samples may be appropriate. Samples outside the calibration range will be diluted or concentrated, as appropriate, and reanalyzed.

16.1.2. Continuing calibration verification (CCV)

Calibration verification solutions traceable to a recognized organization are inserted as part of the sample stream. The sources of the calibration verification solutions are independent from the standards used for the calibration. Calibration verification solutions used for the CCV will contain all the analytes of interest. The frequency of these verifications is dependent on the type of instrumentation used and, therefore, requires considerable professional judgment. The required frequencies for this project are listed in Tables 9-10. All analyses are bracketed by acceptable calibration verification; all samples not bracketed by an in control CCV should be reanalyzed. If the control limits for analysis of the calibration verification solution are not met, the initial calibration will be repeated. All samples analyzed before the calibration. Only the reanalysis results will be reported. If it is not possible or feasible to perform reanalysis of samples, all earlier data (i.e., since the last successful calibration control verification) are suspect. In this case, the LQAO will contact the PM to determine proceedings, and will flag the data and note the issue in interim and final reports.

Element 17. Inspection/Acceptance of Supplies and Consumables

All supplies will be examined for damage as they are received. Laboratory ordering personnel will review all supplies as they arrive to ensure the shipment is complete and intact. All chemicals are logged in to the appropriate logbook and dated upon receipt. All supplies are stored appropriately and are discarded upon expiration date. Table 18 indicates items that are considered for accuracy, precision, and contamination. If these items are not found to be in compliance with the acceptance criteria, they will be returned to the manufacturer.

Project-Related Supplies (source)	Inspection / Testing Specifications	Acceptance Criteria	Frequency	Responsible Individual
Nitrile Gloves (Fisher Scientific or similar)	Carton seal is visually inspected for damage or tampering	Carton is intact and gloves within are clean and intact	At receipt date of shipment	MSPL or SGS-Axys personnel
Polyethylene Gloves (Fisher Scientific or similar)	Carton seal is visually inspected for damage or tampering	Carton is intact and gloves within are clean and intact	At receipt date of shipment	MSPL or SGS-Axys personnel
Polyethylene Jars (Nalgene or similar)	Carton seal is visually inspected for damage or tampering	Carton is intact and gloves within are clean and intact	At receipt date of shipment	MSPL or SGS-Axys personnel
Glass Jars (IChem, Qorpak or similar)	Carton seal is visually inspected for damage or tampering	Carton is intact and gloves within are clean and intact	At receipt date of shipment	MSPL or SGS-Axys personnel
Analytical Standards (Perkin- Elmer, VWR, Fisher Scientific or similar)	Solution bottles are inspected to verify factory seal	Manufacturer's seal intact	At receipt date of shipment	MSPL or SGS-Axys personnel
Certified Reference Materials (NIST, NRCC or similar)	Bottles are inspected to verify factory seal	Manufacturer's seal intact	At receipt date of shipment	MSPL or SGS-Axys personnel

Table 18. Inspection/acceptance testing requirements for consumables and supplies

Element 18. Non-Direct Measures

Data will not be used from non-direct measures in this study.

Element 19. Data Management

Field data will be entered into the SWAMP Database version 2.5 upon return to the lab. Original field sheets (Attachment 1) will be retained in a logbook, and copies of the COCs (Attachment 2) will be kept by each receiving laboratory.

All data generated by SGS-Axys will be maintained as described in SGS-Axys SOP titled SAD-022 Record Management and the SGS-Axys Quality Assurance Manual titled QDO-001 QAQC Policies and Procedures. The Babcock QAO will be responsible for oversight of the collection of all organic chemical analysis data and submission of QA-checked data to SWAMP IQ.

Likewise, all MPSL data will be generated and maintained according to the Marine Pollution Studies Laboratory Quality Manual (2021). The MPSL QAO will be responsible for oversight of the collection of all dissection and metals analysis data and submission of QA-checked data to SWAMP IQ.

All data collected will be entered into electronic spreadsheets that are SWAMP compatible. Each data element is checked at a minimum by the technician that entered the data and verified by the technician's signature on the data sheet. Tissue data will be provided to SWAMP IQ in Microsoft Excel spreadsheets. Data will be reviewed to ensure they are consistent with the format of the database and other data records.

All raw and statistical analysis data are subject to a 100% check for accuracy by the PM and LQAOs. Data are analyzed and proofread for accuracy, and then verified and validated against the QAPP and SWAMP criteria before being loaded into the SWAMP database by SWAMP IQ (Element 22). Original hard copies of the data are filed in a secure cabinet until requested by the PM and/or inclusion into the Final Report. Electronic copies are stored and backed up by each analyst and respective laboratory internal project manager.

Hardware and software will be updated as recommended by the manufacturer or as needed. Testing of each component is not required on a regular basis aside from day to day functionality. Each entity is responsible for the necessary updates or upgrades, whether provided regularly through an Information Technology department or otherwise.

Data management checklists are not required. Analytical completeness will be tracked through the SWAMP Database version 2.5.

GROUP C ELEMENTS: ASSESSMENT AND OVERSIGHT

Element 20. Assessments and Response Actions

20.1. Audits

All reviews of QA data will be made by the QAO of each laboratory (LQAO) prior to submission of each batch to the PM and SWAMP IQ. Reviews of the sampling procedures will be made by the Field Collection Coordinator and the Project Coordinator in case problems occur. As SOPs are updated and refined, additional reviews will be made. Each data technician is responsible for flagging all data that does not meet established QA/QC criteria.

Project data review established for this project will be conducted once all data sets have been received, and includes the following:

• Initial review of analytical and field data for complete and accurate documentation, chain of custody procedures, compliance with analytical holding times, and required frequency of laboratory QA samples.

- Comparison of all spike and duplicate results with the MQOs in Tables 9-10.
- Assigning data qualifier flags to the data as necessary to reflect limitations identified by the process.

If a review discovers any discrepancy, the LQAO will discuss it with the personnel responsible for the activity. The discussion will include the accuracy of the information, potential cause(s) leading to the deviation, how the deviation might impact data quality and the corrective actions that might be considered. If the discrepancy is not resolved, the LQAO will issue a stop work order until the problem is fixed.

Assessments by the LQAO will be oral; if no discrepancies are noted and corrective action is not required, additional records are not required. If discrepancies are observed, the details of the discrepancy and any corrective action will be reported and appended to the report.

All assessments will be conducted as data is received by the LQAO in accordance with the timeline in Table 7.

20.2. Deviations and corrective actions

Analyses are conducted according to procedures and conditions recommended by the US EPA and described in laboratory SOPs (Appendices III and IV), with the exception of those reported herein. Beyond those identified, deviations from these recommended conditions are reported to the LQAO. The PM and Program QAO will be notified within 48 hours of these deviations.

In the event of a SOP/QAPP deviation or corrective action, a Corrective and Preventative Action Report will be prepared, completed, signed and the PM and Program QAO notified. Best professional judgment will be used in interpretation of results obtained when deviations in the test conditions have occurred. All deviations and associated interpretations will be reported in interim and final reports. Protocol amendments will be submitted to the LQAO, Program QAO and PM. Upon approval, protocol amendments will be employed.

This study strives for 90% analytical data completeness. If this goal cannot be achieved, various corrective actions can be undertaken as described in Section D24.

Element 21. Reports to Management

Each LD shall regularly brief the LS and PM on the progress of all on-going chemical analyses in emails or conference calls. When deemed necessary for decision making, other Workgroup participants will also be notified of progress.

The LS will provide regular updates to the Program Coordinator, State Water Board Management, and the Region 9 US EPA representative, usually during SWAMP Round Table conference calls, other meetings, or providing Technical Memos, when requested. In addition, a draft final SWAMP Statewide Project Report will be distributed the Scientific Review Panel, Workgroup Members, the Program Coordinator, State Water Board Management and Region 9 US EPA representative for comment. The final report, once agreed upon by all participants, will be made available to the public by inclusion on the State Board website. These documents will be generated and released in accordance with the dates listed in Table 7.

GROUP D ELEMENTS: DATA VALIDATION AND USABILITY

Element 22. Data Review, Verification and Validation Requirements

All data reported for this project will be subject to a 100% check for errors in transcription, calculation and computer input by the laboratory internal project manager and/or LQAO. Additionally, the LQAO will review sample logs and data forms to ensure that requirements for sample preservation, sample integrity, data quality assessments and equipment calibration have been met. At the discretion of the LD, data that do not meet these requirements will either not be reported or will be reported with qualifiers which serve as an explanation of any necessary considerations.

Reconciliation and correction will be decided upon by LQAO and LD. The LQAO will be responsible for informing data users of the problematic issues that were discussed, along with the associated reconciliations and corrections, prior to submission to SWAMP IQ.

Data generated by project activities will be reviewed against the MQOs in Tables 9-10. Furthermore, the final dataset as a whole will be scrutinized for usability to answer the three Management Questions.

Element 23. Verification and Validation Methods

Field Data will be submitted electronically to the SWAMP database using either SWAMP field data templates or data entry shell databases. Field crews, after data entry, will check 100% of the data entered for typos and errors. DQMs will verify the data to ensure proper flagging for equipment failures and note obvious typos or impossible values. Discrepancies will be communicated to the PM and field crew coordinator before finalizing the records.

Laboratory data will be reported electronically to SWAMP IQ for verification, validation, and inclusion in the SWAMP Database version 2.5. SWAMP IQ will follow <u>SWAMP SOP-Chemistry</u> <u>Data Verification</u>. Discrepancies in laboratory data flagging noted during data verification will be communicated to the Program QAO, LQAO and PM prior to loading

All tissue data will be validated according to the Workgroup <u>Data Validation SOP</u>. Please refer to the appended document for complete descriptions and validation steps, as well as examples of potential QC failures.

QA narratives will be produced and incorporated in the Workgroup Lakes and Reservoirs Report. This narrative will summarize the data set from a QA standpoint. Validated data will be made available to users via the SWRCB <u>CEDEN Advanced Query Tool</u>.

Element 24. Reconciliation with User Requirements

Data will be reported in the SWAMP Database and will be publicly available via CEDEN. Data that do not meet with the MQOs in Tables 9-10 will be flagged accordingly as discussed in Section D23. Rejected data will not be included in data analyses, while data flagged as qualified will be evaluated for inclusion on a case-by-case basis in conjunction with the associated QA data and program objectives.

As stated earlier, PCBs will be summed for comparison with threshold values in Table 8. It is possible that some of the parameters that comprise each summation may be flagged as rejected through the Validation process. When this occurs, the censored results will not be included in the summation used for comparison. However, the difference between summations with and without rejected values will be compared to each other. If the rejected values comprise more than 30% of the total sum for a sample, and the concentration prior to censoring was above the threshold level in Table 3, then the sample will be designated for reanalysis. Samples with censoring of more than 30% but with uncensored sums below the threshold level will not be designated for reanalysis.

The project needs sufficient data, as represented by the completeness objective (Table 9), to address the management questions laid out in the Monitoring Plan. A failure to achieve the number of data points cited could mean an inability to answer these questions.

All management questions will be assessed by the LS, with input as needed from the Workgroup.

MQ1 will be assessed by comparing the concentrations of the lakewide composites, as well as any location composites analyzed, to the Workgroup adopted thresholds listed in Table 8.

MQ2 will be assessed by establishing time-series of representative, average statewide concentrations. These time series will be assessed for a) decreases, increases, or no changes in mercury concentration in fish and b) factors that appear to be driving changes (if any) in mercury concentration in fish.

REFERENCES

Bernstein, B. 2010. SWAMP Assessment Framework. Surface Water Ambient Monitoring Program. California State Water Resources Control Board, Sacramento, CA.

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