

**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**

The Bioaccumulation Oversight Group (BOG)

Surface Water Ambient Monitoring Program

**Version 2  
September 2017**

<b>Program Title</b>	SWAMP Bioaccumulation Oversight Group Long-term Monitoring of Bass Lakes and Reservoirs in California
<b>Lead Organization</b>	SJSURF Marine Pollution Studies Lab 7544 Sandholdt Road Moss Landing, CA 95039 Autumn Bonnema, Project Coordinator
<b>Primary Contact</b>	Jay Davis, Lead Scientist San Francisco Estuary Institute 4911 Central Avenue Richmond, CA 94804 (510) 746-7368  Autumn Bonnema, Project Manager SJSURF Marine Pollution Studies Lab 7544 Sandholdt Road Moss Landing, CA 95039 (831) 771-4175
<b>Effective Date</b>	This Quality Assurance Project Plan (QAPP) is effective from April 2017 to May 2030 unless otherwise revised, approved and distributed accordingly at an earlier date.
<b>Citation for QAPP</b>	Bonnema, A. 2017. Quality Assurance Project Plan: Long-term Monitoring of Bass Lakes and Reservoirs in California. Moss Landing Marine Labs. Prepared for SWAMP BOG, 48 pages plus appendices and attachments.

## QAPP Preface

This Quality Assurance Project Plan (QAPP) document defines procedures and criteria that will be used for this project, conducted by the Bioaccumulation Oversight Group (BOG) in association with the Moss Landing Marine Labs Marine Pollution Studies Laboratory (MPSL-DFG), California Department of Fish and Wildlife Water Pollution Control Laboratory (DFG-WPCL), and the San Francisco Estuary Institute (SFEI). 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-DFG, Caltest, and SFEI also are contained within. The BOG 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 [2017 SWAMP Quality Assurance Program Plan](#) (2017 SWAMP QAPrP).

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

## Approvals

The approvals below were submitted separately, preventing their inclusion in this signature block. Instead, they appear in Appendix V of this document. Originals are kept on file by Autumn Bonnema of MPSL-DFG.

**Jennifer Salisbury**  
**Contract Contact**

\_\_\_\_\_ Date \_\_\_\_\_

**Jay Davis**  
**Lead Scientist**

\_\_\_\_\_ Date \_\_\_\_\_

**Renee Spears**  
**SWRCB Quality Assurance Officer**

\_\_\_\_\_ Date \_\_\_\_\_

**Melissa Morris**  
**SWAMP Program Quality Assurance Officer**

\_\_\_\_\_ Date \_\_\_\_\_

**Autumn Bonnema**  
**Project Manager/ MPSL-DFG Quality Assurance Officer**

\_\_\_\_\_ Date \_\_\_\_\_

**Gail Cho**  
**DFG-WPCL Quality Assurance Officer**

\_\_\_\_\_ Date \_\_\_\_\_

**Emily Volkmar**  
**Caltest Quality Assurance Officer**

\_\_\_\_\_ Date \_\_\_\_\_

## Element 2. Table of Contents

Group A Elements: Project Management .....	1
Element 1. Title and Approval Sheets .....	1
Element 2. Table of Contents.....	4
Element 3. Distribution List and Contact Information .....	7
Element 4. Project Organization .....	7
Element 5. Problem Definition .....	11
Element 6. Project Description .....	14
Element 7. Quality Objectives, Indicators and Acceptability Criteria for Measurement Data	20
Element 8. Special Training Requirements/Safety .....	29
Element 9. Documentation and Records.....	31
Group B Elements. Data Generation and Acquisition.....	32
Element 10. Sample Process Design.....	32
Element 11. Sampling Methods.....	32
Element 12. Sample Handling and Custody .....	34
Element 13. Analytical Methods.....	35
Element 14. Quality Control .....	39
Element 15. Instrument/Equipment Testing, Inspection and Maintenance .....	39
Element 16. Instrument/Equipment Calibration and Frequency.....	40
Element 17. Inspection/Acceptance of Supplies and Consumables .....	41
Element 18. Non-Direct Measures.....	42
Element 19. Data Management.....	42
Group C Elements: Assessment and Oversight .....	43
Element 20. Assessments and Response Actions .....	43
Element 21. Reports to Management.....	44
Group D Elements: Data Validation and Usability.....	44
Element 22. Data Review, Verification and Validation Requirements .....	44
Element 23. Verification and Validation Methods .....	45
Element 24. Reconciliation with User Requirements .....	45
References.....	46

## LIST OF TABLES

Table 1. Contact information .....	7
Table 2. Positions and duties .....	8
Table 3. Constituents to be analyzed – fish attributes .....	15
Table 4. Constituents to be analyzed - metals and metalloids in tissue.....	16
Table 5. Constituents to be Analyzed - polychlorinated byphenyls (PCB) in tissue.....	17
Table 6. Constituents to be Analyzed - organochlorine (OC) pesticides in tissue .....	18
Table 7. Project schedule timeline .....	19
Table 8. Sport fish assessment thresholds.....	21
Table 9. Measurement Quality Indicators for laboratory measurements in tissue .....	21
Table 10. Measurement Quality Objectives - inorganic analytes in tissue.....	23
Table 11. Measurement Quality Objectives1 - synthetic organic compounds in tissue2 .....	25
Table 12. Field collection corrective actions .....	33
Table 13. Sample handling and holding times for tissue .....	34
Table 14. Methods for laboratory analyses.....	35
Table 15. Trace metal analytical parameters, reporting units and reporting limits (RL) in tissue	35
Table 16. Polychlorinated biphenyl analytical parameters, reporting units, and reporting limits (RL) for tissue .....	37
Table 17. Organochlorine pesticide analytical parameters, reporting units, and reporting limits (RL) for tissue .....	38
Table 18. Equipment maintenance and calibration frequency.....	40
Table 19. Inspection/acceptance testing requirements for consumables and supplies .....	42

## LIST OF FIGURES

Figure 1. Organizational Chart .....	11
--------------------------------------	----

## LIST OF APPENDICES

Appendix I. List of Associated QAPs.....	49
Appendix II. Monitoring Plan.....	50
Appendix III. MPSL-DFG SOPs .....	99
Appendix III A. MPSL-101 Sample Container Preparation for Organics and Trace Metals, Including Mercury and Methylmercury.....	100
Appendix III B. MPSL-102a Sampling Marine and Freshwater Bivalves, Fish and Crabs for Trace Metal and Synthetic Organic Analysis .....	119
Appendix III C. MPSL-104 Sample Receipt and Check-In .....	130
Appendix III D. MPSL-105 Laboratory Preparation of Trace Metal and Synthetic Organic Samples of Tissues in Marine and Freshwater Bivalves and Fish.....	134
Appendix III E. Modifications to EPA 3052 .....	144
Appendix IV. SWAMP SOPs.....	145
Appendix IV A. SWAMP SOP Chemistry Data Verification v1.1 .....	146
Appendix IV B. BOG Data Validation SOP.....	147
Appendix V. Signature Pages .....	157

## LIST OF ATTACHMENTS

Attachment 1. Chain of Custody Form Example.....	158
Attachment 2. Field Data Sheet Example.....	160
Attachment 3. Analysis Authorization Form Example.....	162
Attachment 4. Laboratory Data Sheet Example .....	163

### 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 (names shown with asterisk\*) 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**

<u>Name</u>	<u>Agency, Company or Organization</u>
<b><u>SAN FRANCISCO ESTUARY INSTITUTE</u></b>	
Jay Davis*	4911 Central Avenue Richmond, CA 94804 Phone: 510-746-7368 Email: <a href="mailto:jay@sfei.org">jay@sfei.org</a>
<b><u>MARINE POLLUTION STUDIES LAB</u></b>	
<b><u>CALIFORNIA DEPARTMENT OF FISH AND WILDLIFE</u></b>	
Autumn Bonnema* Billy Jakl	7544 Sandholdt Road Moss Landing, CA 95039 Phone: 831-771-4175  Email: <a href="mailto:bonnema@mlml.calstate.edu">bonnema@mlml.calstate.edu</a>
<b><u>WATER POLLUTION CONTROL LAB</u></b>	
<b><u>CALIFORNIA DEPARTMENT OF FISH AND WILDLIFE</u></b>	
Pete Ode Gail Cho*	2005 Nimbus Road Rancho Cordova, CA 95670 Phone: 916-358-0316 Email: <a href="mailto:peter.ode@wildlife.ca.gov">peter.ode@wildlife.ca.gov</a>
<b><u>CALTEST ANALYTICAL LABORATORY</u></b>	
Shawna Rees Emily Volkmar*	1885 North Kelly Road Napa, CA 94558 Phone: 707-258-4000 x20 Email: <a href="mailto:shawna_rees@caltest.com">shawna_rees@caltest.com</a>
<b><u>STATE WATER RESOURCES CONTROL BOARD</u></b>	
Renee Spears*	1001 I Street, 19 <sup>th</sup> Floor Sacramento, CA 95814 Phone: 916-341-5583 Email: <a href="mailto:renee.spears@waterboards.ca.gov">renee.spears@waterboards.ca.gov</a>
<b><u>OFFICE OF INFORMATION MANAGEMENT</u></b>	
<b><u>SWAMP INFORMATION MANAGEMENT AND QUALITY ASSURANCE</u></b>	
Melissa Morris*	1001 I Street, 19 <sup>th</sup> Floor Sacramento, CA 95814 Phone: 916-41-5868 Email: <a href="mailto:melissa.morris@waterboards.ca.gov">melissa.morris@waterboards.ca.gov</a>

\* Indicates person responsible for receiving, retaining, and distributing the final QAPP to staff within their organization

### Element 4. Project Organization

The lines of communication between the participating entities, project organization and responsibilities are outlined in Table 2 and Figure 1. Please note DFG-WPCL has subcontracted

analyses to Caltest for samples collected in 2017, and are therefore responsible for ensuring Caltest meets the guidelines in this Quality Assurance document.

**Table 2. Positions and duties**

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) Rich Bruer (SWRCB) Lori Webber (SWRCB)	Program planning and oversight; project budget allocation and reconciliation with program objectives
Contract Manager	Chad Fearing (OIMA)	Approves invoices
Contract Contact	Jennifer Salisbury (OIMA)	Reviews deliverables and invoices, and submits recommendations for invoice approval to contract manager
Lead Scientist	Jay Davis (SFEI)	Advisory roll; data reporting; development of Monitoring Plan; coordination with BOG technical workgroup
Project Manager	Autumn Bonnema (MPSL-DFG)	Generation and maintenance of project QAPP; project coordination; ensures all activities are completed within proper timeframes; oversees project deliverables, entry of field and laboratory generated data into SWAMP formats
State Board QA Officer	Renee Spears (SWRCB)	Approves QAPP; reports to EPA and SWRCB management
Program QA Officer, Database Manager, SWAMP IQ	Melissa Morris (SWRCB)	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 Managers	Kimberly Pham (SWRCB) Brian Ogg (SWRCB)	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-DFG) Gail Cho (DFG-WPCL) Emily Volkmar (Caltest)	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-DFG) Gary Ichikawa (MPSL-DFG)	Sampling coordination, operations, and implementing field-sampling procedures.
Laboratory Director	Wes Heim (MPSL-DFG) Pete Ode (DFG-WPCL) Shawna Rees (Caltest)	Supervises laboratory staff; data validation, management and reporting
Sample Custodian	Stephen Martenuk (MPSL-DFG) Melinda Kelley and Greg Martindale (Caltest) additional staff	Sample storage; not responsible for any deliverables; may oversee Technicians
Technicians	Technical staff MPSL-DFG Caltest	Conduct tissue dissection, digestion, and chemical analyses; verify field and lab datasheet entry; responsible for chemistry data submission to LQAO

#### 4.1. Involved parties and roles

Jennifer Salisbury of the Office of Information Management and Analysis (OIMA) will be the Contract Contact (CC) for this project. The CC will review reports and invoices, and submit recommendations for approval of invoice for payment to Chad Fearing, the Contract Manager (CM).

Jay Davis of San Francisco Estuary Institute (SFEI) 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 BOG with a final report on completion of this project.

Autumn Bonnema of MPSL-DFG will serve as the Project Manager (PM). The PM will 1) prepare the QAPP, 2) ensure all laboratory activities are completed within the proper 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 and Gary Ichikawa of MPSL-DFG share the responsibility of directing fish collection for this project. Together they will 1) oversee preparation for sampling, including vehicle and vessel maintenance and 2) oversee sample and field data collection, data entry and submission to SWAMP IQ.

Melinda Kelley and Greg Martindale are responsible for sample storage and custody at Caltest. Stephen Martenuk will do the same for samples processed at MPSL, in addition to overseeing compositing of tissue samples.

Shawna Rees will serve as the Laboratory Director (LD) for the Caltest component of this project. 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 Caltest activities are completed within the proper timelines. Pete Ode, LD for DFG-WPCL, will assist with these responsibilities as necessary during the subcontract period.

Wes Heim will serve as the LD for the MPSL-DFG 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-DFG activities are completed within the proper timelines.

Additional members of the BOG 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 BOG projects, with the primary objectives of the data used to answer Management Questions laid out in the Monitoring Plan (Appendix II). These members are: Terry Fleming (United States Environmental Protection Agency (USEPA)), Susan Klasing, Wesley Smith, and Shannon Murphy (Office of Environmental Health Hazard Assessment (OEHHA)), Rich Fadness (Regional Water Quality Control Board 1(RWQCB1), Kevin Lunde and Kristina Yoshida, (RWQCB2), Karen Worcester (RWQCB3), Michael Lyons (RWQCB4), Lauren Smitherman, Carrie Austin and Patrick Morris (RWQCB5), Carly Nilson (RWQCB6), Jeff Geraci (RWQCB7), Heather Boyd (RWQCB8), Chad Loflen (RWQCB9), and Jennifer Salisbury (State Water Resources Control Board (SWRCB)).

A Peer Review Panel consisting of experts reviews Monitoring Plans as well as technical reports. This panel consists of Jim Wiener (University of Wisconsin, La Crosse (retired)), 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-DFG LQAO (LQAO), Gail Cho is the DFG-WPCL LQAO and Emily Volkmar is the Caltest LQAO. 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 and assurance 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 are 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.

The SWAMP IQ serves as the project quality assurance control team. The SWAMP IQ Data Quality Managers (DQM) review, verify, validate, and load 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 (Melissa Morris, SWAMP Information Management and Quality Assurance [SWAMP IQ]) assesses the data for compliance with the project and SWAMP program 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, Renee Spears, to ensure that the project and data meets the requirements of the SWRBC's Quality Assurance Program Plan.

#### **4.3. Persons responsible for QAPP update and maintenance**

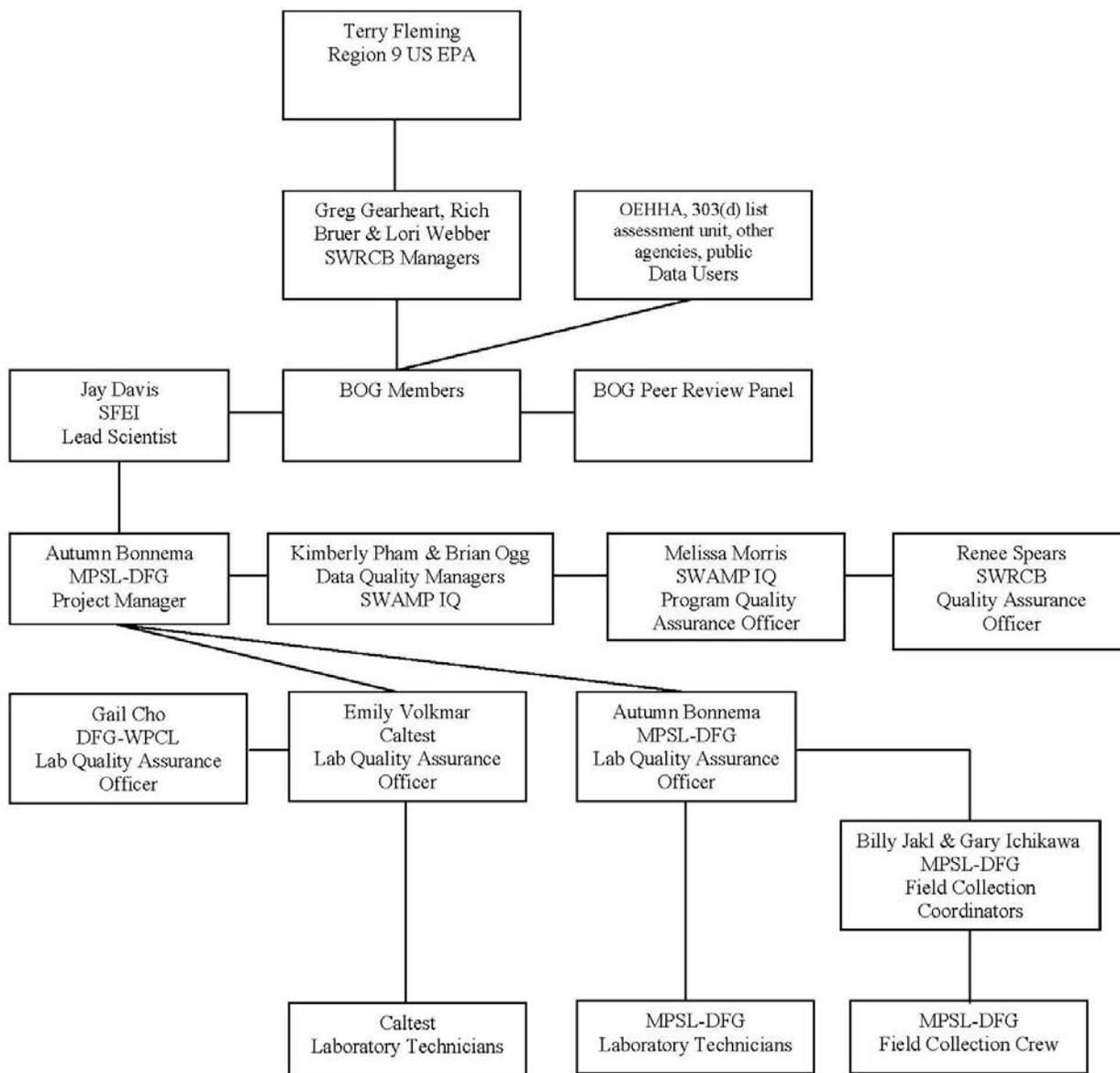
Revisions and updates to this QAPP will be carried out by Autumn Bonnema, with technical input from the Laboratory and Program QAOs. All changes will be considered draft until reviewed and approved by the PM, the Program QAO, and SWRCB QAO.

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, Caltest and MPSL-DFG.

#### **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 BOG 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 two statewide uses of a similar nature that are slated to be adopted in 2017: “Tribal Cultural Use” and “Tribal Fish Use” (State Water Resources Control Board Resolution 2016-0011). SWAMP sport fish monitoring data will be used to evaluate the status of all beneficial uses related to harvesting of wild fish (i.e., COMM, CUL, and FISH, and any new uses that are adopted).

The BOG assessment framework is consistent with frameworks developed for other components of SWAMP (Bernstein 2010), and is intended to guide the bioaccumulation monitoring 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 bioaccumulation monitoring 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 long-term 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 of organic contaminants of concern (PCBs and legacy pesticides).

The State Water Board has an established policy for placing water bodies on the 303(d) list. The listing policy can be found here:  
[http://www.waterboards.ca.gov/water\\_issues/programs/tmdl/303d\\_listing.shtml](http://www.waterboards.ca.gov/water_issues/programs/tmdl/303d_listing.shtml).

### **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 control program for mercury is being developed by the State Water Resources Control Board: ([http://www.waterboards.ca.gov/water\\_issues/programs/mercury/](http://www.waterboards.ca.gov/water_issues/programs/mercury/)). 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 BOG is coordinating with other efforts 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 (Appendix II). A technical report will summarize the data generated.

## 6.2. Constituents to be analyzed and measurement techniques.

A detailed Monitoring Plan is in Appendix II. Chemistry analytical methods are summarized in Section G. 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 have calculated PCB as Aroclors for comparison with older data sets and health thresholds, BOG agrees that these calculations are not as valuable as individual congener data, and has therefore ceased reporting these calculated values. OEHHA no longer intends to use calculated data; however, these values can be calculated at a later time using the provided congener data.

In the SWAMP Lakes Study (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.

**Table 3. Constituents to be analyzed – fish attributes**

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

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

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

<b>Analyte</b>	<b>Matrix Type</b>	<b>Analytical Method</b>
Total Mercury	Whole Body Small Fish and Sport Fish filet muscle	EPA 7473 (USEPA 1998)
Total Selenium	Whole Body Small Fish and Sport Fish filet muscle	EPA 3052M (Appendix III E) EPA 200.8 (USEPA 1994a)

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

<b>Polychlorinated Biphenyl (PCB) Congeners (by USEPA Method 8082A, USEPA 2007)</b>	
PCB 001 <sup>+</sup>	PCB 128
PCB 005 <sup>+</sup>	PCB 132 <sup>+</sup>
PCB 008	PCB 137
PCB 018	PCB 138
PCB 027	PCB 141
PCB 028	PCB 146
PCB 029	PCB 149 <sup>+</sup>
PCB 031	PCB 151
PCB 033	PCB 153
PCB 037 <sup>+</sup>	PCB 156
PCB 044	PCB 157
PCB 049	PCB 158
PCB 052	PCB 158
PCB 056	PCB 166 <sup>+</sup>
PCB 060	PCB 167 <sup>+</sup>
PCB 064	PCB 168 <sup>+</sup>
PCB 066	PCB 169
PCB 070	PCB 170
PCB 074	PCB 174
PCB 077	PCB 177
PCB 080 <sup>+</sup>	PCB 180
PCB 087	PCB 183 <sup>+</sup>
PCB 090 <sup>+</sup>	PCB 184 <sup>+</sup>
PCB 095	PCB 187
PCB 097	PCB 189
PCB 099	PCB 194
PCB 101	PCB 195
PCB 105	PCB 198
PCB 110	PCB 199
PCB 114	PCB 200
PCB 118	PCB 201
PCB 119 <sup>+</sup>	PCB 203
PCB 123 <sup>+</sup>	PCB 206
PCB 126	PCB 209

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

<b>Organochlorine Pesticides (by EPA 8081A, USEPA 1996b)</b>	
<b>Group</b>	<b>Parameter</b>
Chlordanes	Chlordane, cis- Chlordane, trans- Heptachlor Heptachlor epoxide Nonachlor, cis-* Nonachlor, trans-* Oxychlordane*
DDTs	DDD(o,p') DDD(p,p') DDE(o,p') DDE(p,p') DDMU(p,p')* DDT(o,p') DDT(p,p')
Cyclodienes	Aldrin Dieldrin Endrin
HCHs	HCH, alpha HCH, beta
Others	Dacthal* Endosulfan I Hexachlorobenzene* Methoxychlor Mirex Oxadiazon*

\* Not available from Caltest, but BOG 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.

**Table 7. Project schedule timeline**

<b>Item</b>	<b>Activity and/or Deliverable</b>	<b>Deliverable Due Date</b>
<b>1</b>	<b>Quality Assurance Project Plan &amp; Monitoring Plan</b>	
1.1	Draft Quality Assurance Project Plan	July 2017
1.2	Final Quality Assurance Project Plan	September 2017
<b>2</b>	<b>Sample Collection</b>	October of each sampling year
<b>3</b>	<b>Sample Selection and Chemical Analysis</b>	
3.1	Selection of Tissue for Analysis	October of each sampling year
3.2	Creation of Sample Composites	November of each sampling year
3.3	Chemical Analysis	February following each sampling year
3.4	Data Reported to SWAMP	March following each sampling year
<b>4</b>	<b>Data Quality Assessment and Narrative</b>	May following each sampling year
<b>5</b>	<b>Interpretive Report</b>	
5.1	Draft Report	September following each sampling year
5.2	Final Report	December following each sampling year

### 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 California Environmental Data Exchange Network (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 during this study between April and October 2016. 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 SWAMP QAPrP.

“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 waterbody;
- 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 Advisory Tissue Levels developed by OEHHA (Klasing and Brodberg, 2008) (Table 8). 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 Safe to Eat Portal (<http://www.mywaterquality.ca.gov/>) and via reports and fact sheets.

The Measurement Quality Objectives (MQOs, Tables 10 and 11) 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.

BOG 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 which may be unduly influenced by analytical blank contamination, poor accuracy or poor precision based on the Measurement Quality Indicators (MQIs) as compared with the MQOs. Validation and data rejection points can be found in Appendix IV B.

**Table 8. Sport fish assessment thresholds**

**Thresholds for concern based on an assessment of human health risk from these pollutants by OEHHA (Klasing and Brodberg, 2008). All values given in ng/g (ppb). The lowest available threshold for each pollutant is in bold font. One serving is defined as 8 ounces (227 g) prior to cooking. The FCG and ATLS for mercury are for the most sensitive population (i.e., women aged 18 to 45 years and children aged 1 to 17 years).**

<b>Pollutant</b>	<b>Fish Contaminant Goal</b>	<b>Advisory Tissue Level (3 servings/week)</b>	<b>Advisory Tissue Level (2 servings/week)</b>	<b>Advisory Tissue Level (No Consumption)</b>
Chlordanes	<b>5.6</b>	190	280	560
DDTs	<b>21</b>	520	1000	2100
Dieldrin	<b>0.46</b>	15	23	46
Mercury	220	<b>70</b>	150	440
PCBs	<b>3.6</b>	21	42	120
Selenium	7400	2500	4900	15000

Data quality indicators for all sample collection and laboratory analyses will include representativeness, accuracy (bias), precision, completeness, comparability and sensitivity, where applicable. Measurement Quality Indicators for analytical measurements in tissue are in Table 9.

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.

**Table 9. Measurement Quality Indicators for laboratory measurements in tissue**

<b>Parameter</b>	<b>Accuracy</b>	<b>Precision</b>	<b>Recovery</b>	<b>Completeness</b>	<b>Sensitivity</b>
Trace metals (including mercury)	CRM 75% - 125%	Duplicate RPD <25%; n/a if concentration of either sample <RL  Matrix Spike Duplicate RPD <25%	Matrix Spike 75% - 125%	90%	See Table 15

Synthetic Organics (including PCBs, and pesticides)	CRM, PT within 70-130% of the certified 95% CI stated by provider of material. If not certified then within 50-150% of reference value.	Duplicate RPD <25%; n/a if concentration of either sample <RL  Matrix Spike Duplicate RPD <25%	Matrix spike 50% - 150% or control limits based on 3x the standard deviation of laboratory's actual method recoveries	90%	See Tables 16-17
---	---	--	---	-----	------------------

## 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.

$$\% \text{ recovery} = \frac{V_{\text{analyzed}}}{V_{\text{certified}}} \times 100$$

Where:

$V_{\text{analyzed}}$ : the analyzed concentration of the reference material

$V_{\text{certified}}$ : the certified concentration of the reference material

The acceptance criteria for reference materials are listed in Tables 10-11.

### 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.

$$\% \text{ recovery} = \frac{(V_{\text{MS}} - V_{\text{ambient}})}{V_{\text{spike}}} \times 100$$

Where:

$V_{\text{MS}}$ : the concentration of the spiked sample

$V_{\text{ambient}}$ : the concentration of the original (unspiked) sample

$V_{\text{spike}}$ : 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.

$$\text{RPD} = \left| \frac{(V_{\text{MS}} - V_{\text{MSD}})}{\text{mean}} \right| \times 100$$

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,

$V_{\text{MS}}$ : the concentration for the matrix spike

$V_{\text{MSD}}$ : 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,

$V_{\text{MS}}$ : the recovery associated with the matrix spike

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

The MQO for the RPD between the MS and MSD is the same regardless of the method of calculation; detailed in Tables 10-11.

### **Table 10. Measurement Quality Objectives - inorganic analytes in tissue**

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

\*Unless method specifies more stringent requirements.

MDL = Method Detection Limit

RL = Reporting Limit

n/a = not applicable

**Table 11. Measurement Quality Objectives<sup>1</sup> - synthetic organic compounds in tissue<sup>2</sup>**

Laboratory Quality Control	Frequency of Analysis	Measurement Quality Objective
<b>Tuning<sup>3</sup></b>	Per analytical method	Per analytical method
<b>Calibration Standard</b>	Initial method setup or when the calibration verification fails	<ul style="list-style-type: none"> <li>Correlation coefficient (<math>r^2 &gt; 0.990</math>) for linear and non-linear curves</li> <li>If <math>RSD &lt; 15\%</math> average RF may be used to quantitate; otherwise use equation of the curve.</li> <li>First- or second-order curves only (not forced through the origin)</li> <li>Refer to SW-846 methods for SPCC and CCC criteria<sup>3</sup></li> <li>Minimum of 5 points per curve (one of them at or below RL)</li> </ul>
<b>Continuing Calibration Verification</b>	Per 12 hours	<ul style="list-style-type: none"> <li>Expected response or expected concentration <math>\pm 20\%</math></li> <li>RF for SPCCs=initial calibration<sup>3</sup></li> </ul>
<b>Laboratory Blank</b>	Per 20 samples or per analytical batch, whichever is more frequent	$<RL$ for target analytes
<b>Reference Material</b>	Per 20 samples or per analytical batch (preferably blind)	70-130% recovery if certified, otherwise 50-150% recovery
<b>Matrix Spike</b>	Per 20 samples or per analytical batch, whichever is more frequent	50-150% or based on historical laboratory control limits (average $\pm 3SD$ )
<b>Matrix Spike Duplicate</b>	Per 20 samples or per analytical batch, whichever is more frequent	50-150% or based on historical laboratory control limits (average $\pm 3SD$ ); RPD $< 25\%$
<b>Laboratory Duplicate</b>	Per 20 samples or per batch, whichever is more frequent	RPD $< 25\%$ ; n/a if concentration of either sample $< RL$
<b>Surrogate</b>	Included in all samples and all QC samples	Based on historical laboratory control limits (50-150% or better)
<b>Internal Standard</b>	Included in all samples and all QC samples (as available)	Per laboratory procedure

<sup>1</sup>Unless method specifies more stringent requirements.

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

<sup>3</sup>Mass spectrometry only

MDL = method detection limit (to be determined according to the SWAMP QA Management Plan)

RL = Reporting Limit

n/a = not applicable

## 7.2. 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).

$$RPD = \left| \frac{(V_{\text{sample}} - V_{\text{duplicate}})}{\text{mean}} \right| \times 100$$

Where:

$V_{\text{sample}}$ : the concentration of the original sample digest

$V_{\text{duplicate}}$ : 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 10-11.

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  $\leq 25\%$  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:

$$RSD = \frac{\text{Stdev}(v_1, v_2, \dots, v_n)}{\text{mean}} \times 100$$

Where:

$\text{Stdev}(v_1, v_2, \dots, v_n)$ : the standard deviation of the values (concentrations) of the replicate analyses.

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

### 7.3. Contamination Assessment – Method blanks

Laboratory method blanks (also called extraction blanks, procedural blanks, or preparation blanks) are used to assess 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 it 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.

#### **7.4. Routine Monitoring of Method Performance for Organic Analysis – Surrogates**

Surrogates are compounds chosen to simulate the analytes of interest in organic analyses. Surrogates will be used to estimate analyte 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 50-150%.

The reported concentration of each analyte is adjusted to correct for the recovery of the surrogate compound by dividing the measured sample concentration by the surrogate percent recovery. The exception is when surrogate recovery cannot be calculated due to matrix or dilution, the results are reported uncorrected and flagged appropriately.

#### **7.5. Internal Standards**

For Gas Chromatography Mass Spectrometry (GC-MS) analysis, internal standards (i.e., injection internal standards) will be added to each sample extract just prior to injection to enable optimal quantification, particularly of complex extracts subject to retention time shifts relative to the analysis of standards. Internal standards are essential if the actual recovery of the surrogates added prior to extraction is to be calculated. The internal standards can also be used to detect and correct for problems in the GC injection port or other parts of the instrument. The compounds used as internal standards will be different from those already used as surrogates. The analyst(s) will monitor internal standard retention times and recoveries to determine if instrument maintenance or repair, or changes in analytical procedures, are indicated. Corrective action will be initiated based on the judgment of the analyst(s). Instrument problems that may have affected the data or resulted in the reanalysis of the sample will be documented properly in logbooks and internal data reports and used by the laboratory personnel to take appropriate corrective action.

#### **7.6. Dual-column Confirmation**

Dual-column chromatography is required for analyses using Gas Chromatography Electron Capture Detector (GC-ECD) due to the high probability of false positives arising from single-column analyses.

#### **7.7. 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 (Appendix II). 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.

## **7.8. 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 (Appendix II, Tables 4-5) 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% (Table 9). 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.

## **7.9. Sensitivity**

SWRCB is in the process of developing a statewide tissue objective for mercury that is anticipated to be 0.2 ppm wet weight (all concentrations mentioned in this document are presented on a wet weight basis). This threshold will be used for the next round of 303(d) listing. Through BOG discussion, the 0.2 ppm objective and listing threshold was selected as the criterion for classifying lakes as having relatively low concentrations of mercury. To be confident that a lake truly has fish mercury concentrations below 0.2 ppm, it is desirable to have measured concentrations in species such as black bass that are known to accumulate high concentrations. The analytical reporting limit for mercury (Table 9) is 16 times less than the objective; well within usability criteria (Group D Elements).

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. FCGs are being used by the Water Boards in the latest round of 303(d) listing determinations. BOG 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 (Table 11) are sufficiently low to assess summed data for 303(d) listing determination, however those for Chlordanes and Dieldrin are slightly higher than the FCGs. 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. The sum of PCB congener RLs (34.8 ppb) in Table 10 is greater than the ATL. RLs for Chlordanes, Dieldrin and DDTs are sufficiently low enough to compare data to the ATL for each.

## **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 10 and 11) 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 Bioaccumulation 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, 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 Caltest and MPSL-DFG. 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 Caltest and MPSL-DFG 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 Caltest 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 contract contact in electronic format)
- Monitoring Plan (submitted to contract contact in and electronic format)
- Archived Sample Sheets (internal documentation available on request)
- Chain-of-Custody Forms (exchanged for signatures with chemistry lab, and kept on file)
- Analysis Authorization Forms (accompany external analytical COCs generated by PM, submitted to SWAMP IQ and contract contact per the conditions of the contract)
- 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 SWAMPIQ in electronic format)
- Draft Interpretive Report (produced in electronic format)
- Final Interpretive Report (in electronic format)
- Data Appendix (submitted to CM in paper and 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-DFG. 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 database, 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, Section III F, pp. 11-15 (Appendix II). 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 MPSL-102a (Appendix III B). 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 Section III G-H, pp 15-18 of the Monitoring Plan (Appendix II), 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 (Appendix II) and MPSL-DFG SOPs (Appendix III).

#### **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 (Appendix II) 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 7.4 (Appendix III B) 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 data sheets (Attachment 2).

Details on targeted fish species, number of individuals and size ranges can be found in the Monitoring Plan (Appendix II, Tables 4-5).

The following adaptation to MPSL-102a, Section 7.4.5 (Appendix III B) 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 clean aluminum foil; fork and total length are recorded. Weight is recorded. Large fish such as carp will then be placed on the cutting board covered with a foil 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 cleaver and cutting board are 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 10% bleach solution is sprayed on all boat 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 12 describes action to take in the event of a collection failure.

**Table 12. Field collection corrective actions**

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.

## 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 (COC, Attachment 1) 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-DFG will be logged in according to MPSL-104 (Appendix III C).

Authorization forms will be provided to each dissecting laboratory detailing the dissection and analysis to be performed (Attachment 3). Samples will be dissected according to MPSL-105 (Appendix III D) and data retained on the lab data sheets in Attachment 4.

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 Coordinators (s) will be notified. Affected data will be flagged appropriately in the final results submitted to SWAMP.

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 13.

**Table 13. Sample handling and holding times for tissue**

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

## Element 13. Analytical Methods

Methods and equipment for laboratory analyses are listed in Table 14. USEPA methods can be downloaded from [www.nemi.gov](http://www.nemi.gov). USEPA method numbers followed by “M” indicate modifications have been made. Modifications and non-USEPA SOPs can be found in Appendix III and IV. Method validation data for modifications and SOPs can be obtained by contacting the analytical laboratory (Table 1.)

**Table 14. Methods for laboratory analyses**

Parameter	Method	Instrument
Mercury	EPA 7473 (USEPA 1998)	Milestone DMA 80
Selenium	EPA 3052M (USEPA 1996a, Appendix III E) EPA 200.8 (USEPA 1994a)	CEM MARSXpress Digester Perkin-Elmer Elan 9000 ICP-MS
Organochlorine Pesticides	EPA 3541 (USEPA 1994c) EPA 8081A (USEPA 1996b)	Aglient 6890N GC with Aglient 5975 MSD and Micro ECD
Polychlorinated Biphenyls	EPA 3546 (USEPA 2007a) EPA 8082A (USEPA 2007b)	Agilent 7890A Dual ECD Detectors with Agilent 7683B autosampler

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, 1998) 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  $\pm 20\%$  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 15 and MQOs in Section 7, Table 10.

Selenium sport and small fish composites will be digested according to EPA 3052M, “Microwave Assisted Acid Digestion of Siliceous and Organically Based Matrices” (USEPA, 1996a), modified (Appendix III E), and will be analyzed according to EPA 200.8, “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  $\pm 20\%$  of the true value, or the previous 10 samples must be reanalyzed. Two blanks, a certified reference material (NIST 2976, NRCC DORM-3 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 15 and MQOs in Section 7, Table 10.

**Table 15. Trace metal analytical parameters, reporting units and reporting limits (RL) in tissue**

<b>Parameter</b>	<b>Method</b>	<b>RL</b>
Mercury	EPA 7473 (USEPA 1998)	0.012 µg/g wet wt
Selenium	EPA 3052M (USEPA 1996a, Appendix III E) EPA 200.8 (USEPA 1994a)	0.40 µg/g wet wt

Organochlorine and PCB compounds will be extracted following EPA Methods 3541, and 3546, respectively. (USEPA 1994c, 2007a) Organochlorine pesticides will be analyzed according to EPA 8081BM, “Organochlorine Pesticides by Gas Chromatography” (USEPA 1996b). PCBs will be analyzed according to EPA 8082A, “Polychlorinated Biphenyls (PCBs) by Gas Chromatography” (USEPA 1996e). 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 ±25% of the true value, or the previous 10 samples must be reanalyzed. One blank, a laboratory control spike (LCS), a method duplicate and an MS pair will be run with each set of samples. RLs can be found in Tables 16 and 17, and MQOs in Section 7, Table 11.

**Table 16. Polychlorinated biphenyl analytical parameters, reporting units, and reporting limits (RL) for tissue**

<b>Polychlorinated Biphenyl congeners (by USEPA Method 8082A, USEPA 2007)</b>			
<b>PCB</b>	<b>RL ppb (ng/g wet wt)</b>	<b>PCB</b>	<b>RL ppb (ng/g wet wt)</b>
PCB 001 <sup>+</sup>	5	PCB 128	0.5
PCB 005 <sup>+</sup>	0.5	PCB 132 <sup>+</sup>	0.5
PCB 008	0.5	PCB 137	*
PCB 018	0.5	PCB 138	0.5
PCB 027	*	PCB 141	0.5
PCB 028	0.5	PCB 146	*
PCB 029	*	PCB 149 <sup>+</sup>	0.5
PCB 031	0.5	PCB 151	0.5
PCB 033	0.5	PCB 153	0.5
PCB 037 <sup>+</sup>	0.5	PCB 156	1
PCB 044	0.5	PCB 157	0.5
PCB 049	0.5	PCB 158	0.5
PCB 052	0.5	PCB 158	0.5
PCB 056	0.5	PCB 166 <sup>+</sup>	0.5
PCB 060	0.5	PCB 167 <sup>+</sup>	0.5
PCB 064	*	PCB 168 <sup>+</sup>	0.5
PCB 066	1	PCB 169	0.5
PCB 070	0.5	PCB 170	0.5
PCB 074	0.5	PCB 174	0.5
PCB 077	0.5	PCB 177	0.5
PCB 080 <sup>+</sup>	0.5	PCB 180	0.5
PCB 087	0.5	PCB 183 <sup>+</sup>	0.5
PCB 090 <sup>+</sup>	0.5	PCB 184 <sup>+</sup>	0.5
PCB 095	0.5	PCB 187	0.5
PCB 097	0.5	PCB 189	0.5
PCB 099	0.5	PCB 194	0.5
PCB 101	0.5	PCB 195	0.5
PCB 105	0.5	PCB 198	*
PCB 110	0.5	PCB 199	*
PCB 114	0.5	PCB 200	*
PCB 118	0.5	PCB 201	0.5
PCB 119 <sup>+</sup>	0.5	PCB 203	0.5
PCB 123 <sup>+</sup>	0.5	PCB 206	0.5
PCB 126	0.5	PCB 209	0.5

<sup>+</sup>New to 2017 analyte list

\* Not available from Caltest, but BOG is still interested in analysis for future projects

**Table 17. Organochlorine pesticide analytical parameters, reporting units, and reporting limits (RL) for tissue**

<b>Organochlorine Pesticides (by USEPA 8081A, USEPA 1996b)</b>		
<b>Group</b>	<b>Parameter</b>	<b>RL (ng/g wet wt)</b>
Chlordanes	Chlordane, cis-	2
	Chlordane, trans-	2
	Heptachlor	2
	Heptachlor epoxide	2
	Nonachlor, cis-	*
	Nonachlor, trans-	*
	Oxychlordane	*
DDTs	DDD(o,p')	2
	DDD(p,p')	2
	DDE(o,p')	2
	DDE(p,p')	2
	DDMU(p,p')	*
	DDT(o,p')	2
	DDT(p,p')	2
Cyclodienes	Aldrin	2
	Dieldrin	2
	Endrin	2
HCHs	HCH, alpha	2
	HCH, beta	2
	HCH, gamma	2
Others	Dacthal	*
	Endosulfan I	2
	Hexachlorobenzene	*
	Methoxychlor	2
	Mirex	20
	Oxadiazon	*

\* Not available from Caltest, but BOG 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 or method specifications. 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 13). 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-DFG and Caltest 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 9-11 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 18). 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. Caltest SOPs are available upon request from the Laboratory Director by email: [shawna.rees@caltest.com](mailto:shawna.rees@caltest.com). Likewise, MPSL-DFG SOPs are available upon request from the LQAO by email: [bonnema@mlml.calstate.edu](mailto:bonnema@mlml.calstate.edu).

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 18) are calibrated, standardized and maintained according to procedures detailed in laboratory QAPs (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.

**Table 18. Equipment maintenance and calibration frequency**

Instrument	Inspection/Maintenance Frequency	Calibration Frequency
Aglient 6890N GC with Aglient 5975 MSD and Micro ECD (Caltest)	As needed	At least once a week or prior to each batch
Agilent 7890A Dual ECD Detectors with Agilent 7683B autosampler	As needed	At least once prior to each batch
Milestone DMA-80 Direct Mercury Analyzer (MPSL-DFG)	As needed	At least once every 2 weeks
Perkin-Elmer Elan 9000 Inductively Coupled Plasma - Mass Spectrometer (MPSL-DFG)	As needed	At least once prior to each batch

### 16.1. Analytical Instrumentation

#### 16.1.1. Instrument calibration

Upon initiation of an analytical run, after each major equipment disruption, and whenever on-going 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  $R^2$  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, National Research Council Canada, 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 10-11. 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 verification solution that failed the MQOs will be reanalyzed following the recalibration. Only the re-analysis 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, L QAO 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 19 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.

**Table 19. Inspection/acceptance testing requirements for consumables and supplies**

<b>Project-Related Supplies (source)</b>	<b>Inspection / Testing Specifications</b>	<b>Acceptance Criteria</b>	<b>Frequency</b>	<b>Responsible Individual</b>
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-DFG or Caltest 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-DFG or Caltest 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-DFG or Caltest personnel

## **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 log book, and copies of the COCs (Attachment 2) will be kept by each receiving laboratory. SWAMP Authorization forms will also accompany samples sent to each laboratory (Attachment 3).

All data generated by Caltest will be maintained as described in Caltest SOPs and the Caltest Quality Assurance Manual (listed in Appendix I). The Caltest QAO will be responsible for oversight of the collection of all organic chemical analysis data and submission of QA-checked data into the SWAMP database.

Likewise, all MPSL-DFG data will be generated and maintained according to the Marine Pollution Studies Laboratory Quality Assurance Plan (Appendix I). The MPSL-DFG QAO will be responsible for oversight of the collection of all dissection and metals analysis data and submission of QA-checked data into the SWAMP database.

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 the PC 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 or SWAMP Tissue Database 2.5. 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 10-11.
- 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 BOG participants will also be notified of progress.

The LS will provide a regular updates to SWRCB Managers and the Region 9 US EPA representative, usually during SWAMP Round Table conference calls, other meetings, or providing Technical Memos or brief articles for the SWAMP Newsletter, when requested. Findings or highlights from the project will be included in the [SWAMP Annual Water quality Status Report](#), written in coordination with the Program Oversight Staff. In addition, a draft final [SWAMP Statewide Project Report](#) will be distributed the Scientific Review Panel, BOG Members, SWRCB Managers 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 the SWAMP IQ.

Data generated by project activities will be reviewed against the MQOs in Tables 10-11. 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 through the SWAMP database or shell database. 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 the SWAMP IQ for verification, validation, and inclusion in the SWAMP Database version 2.5. The SWAMP IQ will follow SWAMP SOP Chemistry Data Verification V1.1 (Appendix V A). 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 BOG Data Validation (Appendix V B), outlined below. 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 BOG 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 CEDEN website (<http://www.ceden.us/AdvancedQueryTool>).

## **Element 24. Reconciliation with User Requirements**

Data will be reported in the SWAMP Database. Data that do not meet with the MQOs in Tables 10-11 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 (Appendix V B). 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 (Appendix II). 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 SFEI, with input as needed from the RWQCBs and OEHHA.

MQ1 will be assessed by comparing the concentrations of the lakewide composites, as well as any location composites analyzed, to the BOG 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.

California Department of Fish and Wildlife. 2013. Aquatic Invasive Species Decontamination Protocol.

Davis, J.A., J. L. Grenier, A.R. Melwani, S. Bezalel, E. Letteney, and E. Zhang. 2007. Bioaccumulation of pollutants in California waters: a review of historic data and assessment of impacts on fishing and aquatic life. Prepared for the Surface Water Ambient Monitoring Program, California Water Resources Control Board, Sacramento, CA.

Klasing, S. and R. Brodberg. 2008. Development of Fish Contaminant Goals and Advisory Tissue Levels for Common Contaminants in California Sport Fish: Chlordane, DDTs, Dieldrin, Methylmercury, PCBs, Selenium, and Toxaphene. California Office of Environmental Health Hazard Assessment, Sacramento, CA.

Stanley, T. W., and S. S. Verner. 1985. The U. S. Environmental Protection Agency's quality assurance program. pp 12-19 In: J. K. Taylor and T. W. Stanley (eds.). Quality Assurance for Environmental Measurements, ASTM STP 867. American Society for Testing and Materials, Philadelphia, Pennsylvania.

State Water Resources Control Board (SWRCB). 2003. 2002 California 305(b) Report on Water Quality [prepared as required by Federal Clean Water Act section 305(b)]. Sacramento, CA: State Water Resources Control Board.

Surface Water Ambient Monitoring Program Quality Assurance Team (SWAMPQAT). 2008. The Surface Water Ambient Monitoring Program Quality Assurance Program Plan, Version 1.0. Prepared for the Surface Water Ambient Monitoring Program, California Water Resources Control Board, Sacramento, CA.

US Environmental Protection Agency. 1994a. Method 200.8. Determination of Trace Elements in Waters and Wastes by Inductively Coupled Plasma – Mass Spectrometry. Revision 5.4. US Environmental Protection Agency, Washington, DC.

US Environmental Protection Agency. 1994b. Method 3460A. Gel-Permeation Cleanup. Revision 1. US Environmental Protection Agency, Washington, DC.

US Environmental Protection Agency, 1994c. Method 3541 (SW-846): Automated Soxhlet Extraction. Revision 0. US Environmental Protection Agency, Washington, DC.

US Environmental Protection Agency. 1996a. Method 3052. Microwave Assisted Acid Digestion of Siliceous and Organically Based Matrices. Revision 0. US Environmental Protection Agency, Washington, DC.

US Environmental Protection Agency. 1996b. Method 8081A. Organochlorine Pesticides by Gas Chromatography. Revision 1. US Environmental Protection Agency, Washington, DC.

US Environmental Protection Agency. 1998. Method 7473. Mercury in Solids and Solutions by Thermal Decomposition, Amalgamation, and Atomic Absorption Spectrophotometry. Revision 0. US Environmental Protection Agency, Washington, DC.

US Environmental Protection Agency. 2000. Guidance for Assessing Chemical Contaminant Data for Use in Fish Advisories: Volume 1, Fish Sampling and Analysis, Third Edition. EPA 823-R-93-002B-00-007. US Environmental Protection Agency, Office of Water, Washington, DC.

US Environmental Protection Agency. 2002. Guidance for Quality Assurance Project Plans. US Environmental Protection Agency, Office of Water, Washington, DC.

US Environmental Protection Agency. 2007a. Method 3546 (SW-846). Microwave Extraction. Version 0. US Environmental Protection Agency, Office of Water, Washington, DC.

US Environmental Protection Agency. 2007b. Method 8082A. Polychlorinated Biphenyls (PCBs) by Gas Chromatography. Revision 1. US Environmental Protection Agency, Washington, DC.

## **Appendix I. List of Associated QAPs**

MPSL-DFG Laboratory QAP, Revision 7. November 2016

Caltest Laboratory QAPP, Revision 22. September 2017

## **Appendix II. Monitoring Plan**

# **Bioaccumulation Monitoring Plan for Lakes and Reservoirs in California: 2016**

The Bioaccumulation Oversight Group (BOG)  
Surface Water Ambient Monitoring Program

Revised August 2016

## **ACKNOWLEDGEMENTS**

This Sampling Plan was prepared by the San Francisco Estuary Institute (SFEI) and Moss Landing Marine Laboratory (MLML) on behalf of the Bioaccumulation Oversight Group (BOG) and the Surface Water Ambient Monitoring Program (SWAMP). Substantial input to the plan was received from the BOG and the BOG Peer Review Panel.

### **Bioaccumulation Oversight Group (BOG) Members**

Terry Fleming, USEPA  
Rich Fadness, Region 1 Water Board  
Karen Taberski, Region 2 Water Board  
Carrie Austin, Region 2 Water Board  
Karen Worcester, Region 3 Water Board  
Michael Lyons, Region 4 Water Board  
Laura McLellan, Region 5 Water Board  
Michelle Wood, Region 5 Water Board  
Tom Suk, Region 6 Water Board  
Carly Nilson, Region 6 Water Board  
Jeff Geraci, Region 7 Water Board  
Heather Boyd, Region 8 Water Board  
Chad Loflen, Region 9 Water Board  
Lori Webber, State Water Board  
Jennifer Salisbury, State Water Board  
Jon Marshack, State Water Board  
Susan Klasing, OEHHA  
Wesley Smith, OEHHA  
Shannon Murphy, OEHHA  
Tom Maurer, USFWS  
Jay Davis, SFEI  
Autumn Bonnema, MLML  
Gary Ichikawa, CDFW  
Stacey Swenson, MLML  
Gail Cho, CDFW  
Steven Martenuk, MLML  
Marco Sigala, MLML

### **SWAMP Bioaccumulation Peer Review Panel**

Jim Wiener, University of Wisconsin, La Crosse (retired)  
Chris Schmitt, USGS, Columbia, Missouri  
Harry Ohlendorf, CH2M HILL, Sacramento, CA

## I. INTRODUCTION

This document presents a plan for sampling and analysis of sport fish in California lakes and reservoirs (collectively referred to as “lakes” in this document). This work will be performed as part of the State Water Resources Control Board's Surface Water Ambient Monitoring Program (SWAMP). The SWAMP mission is to provide resource managers, decision makers, and the public with timely, high-quality information to evaluate the condition of all waters throughout California.

Oversight for this Project is being provided by the SWAMP Roundtable. The Roundtable is composed of State and Regional Water Board staff and representatives from other agencies and organizations including USEPA, the California Department of Fish and Wildlife, the California Office of Environmental Health Hazard Assessment (OEHHA), and the University of California. Interested parties, including members of other agencies, consultants, and other stakeholders are also welcome to participate.

A subcommittee of the Roundtable, the Bioaccumulation Oversight Group (BOG), focuses on bioaccumulation monitoring. The BOG is composed of State and Regional Water Board staff and representatives from other agencies and organizations including USEPA, the Department of Fish and Wildlife, the Office of Environmental Health Hazard Assessment, and the San Francisco Estuary Institute. The members of the BOG individually and collectively possess extensive experience with bioaccumulation monitoring.

The BOG has also convened a Bioaccumulation Peer Review Panel that provides programmatic evaluation and review of specific deliverables emanating from the Project, including this Sampling and Analysis Plan. The members of the Panel are internationally recognized authorities on bioaccumulation monitoring.

The BOG was formed and began developing a strategy for designing and implementing a statewide bioaccumulation monitoring program in September 2006. To date the efforts of the BOG to monitoring bioaccumulation in California water bodies have included:

- a two-year screening survey of bioaccumulation in sport fish in lakes (2007 and 2008),
- a two-year screening survey of the coast (2009 and 2010),
- a one-year survey of rivers and streams (2011),
- a two-year study of mercury accumulation in grebes on lakes (2012-2013),
- a one-year study (2014) of lakes with relatively low concentrations of contaminants in sport fish, and
- initiating a long-term program for monitoring bass lakes (2015).

Final reports on the sport fish surveys and the grebe study are available (Davis et al. 2010; Davis et al. 2012; Davis et al. 2013; Ackerman et al. 2015;

[http://www.mywaterquality.ca.gov/monitoring\\_council/bioaccumulation\\_oversight\\_group/#mpr](http://www.mywaterquality.ca.gov/monitoring_council/bioaccumulation_oversight_group/#mpr)).

## **II. GENERAL ASPECTS OF THE SWAMP BIOACCUMULATION MONITORING PROGRAM**

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

The BOG 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 development of two statewide uses of a similar nature that are slated to be adopted in 2017: “Tribal Cultural Use” and “Tribal Fish Use” (State Water Resources Control Board Resolution 2016-0011). SWAMP sport fish monitoring data will be used to evaluate the status of all beneficial uses related to harvesting of wild fish (i.e., COMM, CUL, and FISH, and any new uses that are adopted).

The BOG assessment framework is consistent with frameworks developed for other components of SWAMP (Bernstein 2010), and is intended to guide the bioaccumulation monitoring 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 bioaccumulation monitoring program will be on evaluating status and trends. Bioaccumulation monitoring is a very effective and essential tool for evaluating status, and is the most cost-effective tool for evaluating trends for many contaminants. Monitoring status and trends in bioaccumulation will provide information useful for identifying sources and pathways and for evaluating the effectiveness of management actions at a broader geographic scale. However, other types of monitoring (i.e., water and sediment monitoring) and other programs (regional TMDL programs) are also needed for addressing sources and pathways, and effectiveness of management actions.

SWAMP sport fish surveys have done a great deal to document the status of bioaccumulation impacts on beneficial uses in California, through sampling of 272 lakes, 68 coastal locations, 63 river and stream locations. Mercury has been shown to be a particular concern across all water body types, and this has triggered the development of a statewide TMDL for mercury in reservoirs. In 2015, SWAMP initiated a long-term plan to provide updated information on status and to track statewide trends, with a

particular emphasis on lakes where black bass are present. That long-term plan calls for revisiting 187 bass lakes throughout the state on a 10-year cycle with a probabilistic design, combined with revisits of other water body types on cycles ranging from 10 to 20 years (Bioaccumulation Oversight Group 2015).

The long-term plan covers much, but not all, of the highest priority bioaccumulation monitoring that is needed. This 2016 sampling plan addresses two types of monitoring that are needed to supplement the long-term plan. First, although many of the most important water bodies that support the fishing beneficial use have been sampled, they represent only a subset of the total number present in the state. This information gap is greatest for the state's 9,000 lakes. Second, in some cases the initial rounds of sampling did not generate sufficient data to support impairment determinations under Clean Water Act Section 303(d) or to support development of consumption advisories. In many of these cases, there is a need to obtain additional data more quickly than the long-term plan would provide.

This monitoring plan therefore addresses these two general categories of information needs for lakes across the state: 1) sampling of lakes that have not previously been sampled, and 2) filling data gaps for lakes that have been previously sampled.

### **III. SAMPLING DESIGN**

#### **A. Management Questions for this Study**

Different management questions apply to the two categories of sampling included in this sampling plan.

##### **1. Management Questions for New Lakes**

The management questions for new lakes are the same as those articulated for the lakes survey in 2007-2008, which sampled 272 unsampled lakes in those years.

##### **Management Question 1 (MQ1)**

*Should a specific lake be considered impaired and placed on the 303(d) list due to bioaccumulation of contaminants in sport fish?*

Answering this question is critical to determining the need for cleanup actions to reduce contaminant exposure in specific water bodies. SWAMP bioaccumulation data are used in making 303(d) listing determinations. TMDLs are required for water bodies placed on the 303(d) list. This is the principal regulatory mechanism being used by the State Water Board, the Regional Water Boards, and USEPA to establish priorities for management actions. The State Water Board is developing a statewide mercury control program for reservoirs that are included on the 303(d) list for mercury in fish. These listings were primarily made based on SWAMP bioaccumulation data.

The State Water Board has an established policy for placing water bodies on the 303(d) list. The information needed to make a listing determination is concentrations from two independent samples from the water body that exceed the relevant threshold of concern. The more representative the samples are of the water body, the better.

**Management Question 2 (MQ2)**

*Should additional sampling of bioaccumulation in sport fish (e.g., more species or larger sample size) in a lake be conducted for the purpose of developing comprehensive consumption guidelines?*

Consumption guidelines provide a mechanism for reducing human exposure in the short-term. The information requirements for consumption guidelines are more extensive than for 303(d) listing. The California Office of Environmental Health Hazard Assessment (OEHHA), the agency responsible for issuing consumption guidelines, needs samples representing 9 or more fish from a variety of species abundant in a water body in order to issue guidance. It is valuable to have information not only on the species with high concentrations, but also the species with low concentrations so anglers can be encouraged to target the low species. It is also valuable to have information from repeated sampling to confirm the results of initial rounds of sampling. SWAMP sampling of “new” water bodies is typically less intensive than what OEHHA needs for development of consumption advice, but the SWAMP results provide an indication of the need and urgency of gathering more data for this purpose.

**2. Management Questions for Addressing Data Gaps**

Management questions 1 and 2 also apply to the lakes that will be sampled to address data gaps. One additional question applies to the data gap sampling. This question was the primary driver of the Clean Lakes Study in 2014. A few of the lakes sampled in the Clean Lakes Study showed indications of low concentrations, but did not yield the complete dataset required to be classified as “clean”. These select lakes will be revisited in this effort to obtain complete datasets that allow for a definitive classification.

**Management Question 3 (MQ3)**

*Which popular lakes in California can be confirmed to have relatively low concentrations of contaminants in sport fish?*

Answering this question will address the critical need of managers and the public to know which water bodies can be considered relatively clean. With this information, the fishing public can be directed to water bodies where they can enjoy the benefits of fishing and fish consumption and have reduced exposure to contaminants.

The data needed to answer this question are repeated observations of low concentrations of all contaminants of concern (including methylmercury, PCBs, legacy pesticides, and selenium) in the species with the greatest tendency to accumulate high

concentrations. For methylmercury, top predators such as black bass tend to accumulate relatively high concentrations. High-lipid, bottom-feeding species such as catfish, carp, and sucker have the greatest tendency to accumulate relatively high concentrations of organic contaminants of concern (PCBs and legacy pesticides). Selenium also biomagnifies primarily through accumulation in muscle, but past monitoring in the San Joaquin Valley (Beckon et al. 2010) suggests that bottom-feeders accumulate slightly higher concentrations. Measuring low concentrations of contaminants in both of these types of indicator species provides compelling evidence that a water body has a low overall degree of contamination. Given the variance associated with contaminant concentrations, the evidence becomes even more compelling if the low concentrations are observed on more than one occasion. This higher level of confidence obtained through repeated observation of low concentrations in both types of indicator species is desirable to be assured of providing reliable information to the public to guide their decisions on where to fish.

In some water bodies, it is not feasible to obtain both types of indicator species because they are not present in high enough abundance. Lakes at higher elevations with colder water where trout species predominate are a common example. For these lakes, repeated observation of the species that do occur there and are most likely to have high concentrations is the best basis that can be obtained for characterizing a lake as one with relatively low concentrations.

## **B. Overall Approach**

The overall approach to be taken to answer these questions will be to sample a set of lakes identified by the Regional Water Boards as high priorities for data collection. The Regional Boards have selected a combination of unsampled lakes and lakes with data gaps to consider for inclusion in the 2016 sampling. The lakes will be sampled with a similar approach to that used in prior rounds of lake sampling (e.g., Bioaccumulation Oversight Group 2007), with two principal enhancements: 1) inclusion of prey fish, and 2) increased effort at trout lakes to get resident fish if they are present.

## **C. Coordination**

The BOG is coordinating with other efforts to leverage the SWAMP statewide monitoring funds available for this survey.

Region 5 is adding \$35,000 to the budget for this study to allow for inclusion of additional lakes in their region.

## **D. Selection of Lakes to Be Sampled**

Lakes were selected for this targeted sampling effort based on Regional Board priorities. Two general categories of lakes were included: 1) unsampled lakes and 2) lakes that have been sampled but that still have data gaps. In some cases, candidate lakes were excluded because they were sampled in 2015 or are scheduled for sampling in 2017 or 2019 as part of the long-term bass lake plan.

A list of candidate lakes was assembled based on input from the Regional Boards (Table 2). A final list of lakes to sample in 2016 (Figure 1, Table 3) was developed based on priorities of the regions and consideration of sampling that is planned as part of the long-term bass lake monitoring.

## **1. Unsampld Lakes**

Unsampled lakes were identified for inclusion (Table 2) based on various sources of information. Regional Board knowledge of the value and importance of each lake in supporting the fishing beneficial use was the primary source. Another supplemental source was Stienstra (2012), the statewide fishing guidebook that was also used in selection of lakes for the 2007-2008 survey. The Stienstra guide rates fishing spots on a 10 point scale (10 being the best rating) according to three criteria: number of fish, size of fish, and scenic beauty. The ratings are considered another index of the value of each water body for supporting the fishing beneficial use, and are useful because they are fairly comprehensive and quantitative. The 2007-2008 survey covered many of the highest-rated lakes in Stienstra. For the present study, we selected candidates from the next tier of the most highly-rated lakes and the Regional Boards selected lakes of interest from this pool. The Stienstra ratings for lakes included in this study ranged from 2-8 (Table 2).

## **2. Lakes with Data Gaps**

The other lakes included (Table 2) have been sampled previously but the data that have been obtained are not sufficient for 303(d) impairment determination or for development of consumption guidelines. A specific category related to development of consumption guidelines was lakes that were included in the 2014 Clean Lakes but where conclusive data were not obtained. In a few cases, monitoring related to specific management actions taking place in the watersheds was also considered.

## **E. Overall Design of the Sampling Effort**

A simple targeted approach will be followed for the 2016 sampling.

## **F. Sampling Design At Each Lake**

The sampling design within each lake will be similar to past SWAMP surveys, but with two important enhancements: monitoring of prey fish and an intensified effort to obtain other, less abundant sport fish species in trout lakes. The design for each lake will also be targeted toward addressing specific data gaps remaining after past sampling efforts.

**1. Sport Fish**

**a. Targeted Species**

This monitoring will focus, where possible, on species that have been established as robust indicators in past sampling: bass species for mercury and high-lipid bottom-feeders for organics.

Methylmercury biomagnifies primarily through its accumulation in muscle tissue, so top predators such as largemouth bass tend to have the highest concentrations. Past sampling has demonstrated that measurement of mercury in individual largemouth bass yields data (size-standardized concentrations) that provide a reliable index of the degree of food web contamination in each lake, and that can be compared across lakes and within lakes over time.

In contrast, although the organic contaminants of concern biomagnify, they do so primarily through accumulation in lipid. Concentrations of organics are therefore influenced by the lipid content of the species, with species that are higher in lipid having higher concentrations. Bottom-feeding species such as common carp and channel catfish tend to have the highest lipid concentrations in their muscle tissue, and therefore usually have the highest concentrations of organics.

Prior sampling for these mercury and organics indicator species has established a baseline against which future data can be compared to allow assessment of long-term trends.

Since some of the lakes included in this survey have been sampled before, the Regional Boards and OEHHHA have identified specific data needs for specific lakes. The species targeted for each lake in each region are listed in Appendix 1.

If the target species are not available, other potential targets will be considered (Tables 4 and 5). Fish species are distributed unevenly across the State, with different assemblages in different regions (e.g., high Sierra Nevada, Sierra Nevada foothills, and Central Valley) and a variable distribution within each region. To cope with these differences in species availability, the sampling crew will have a prioritized menu of potential target species. Primary target species will be given the highest priority. If primary targets are not available in sufficient numbers, secondary targets will be collected.

Some lakes, particularly high elevation lakes, have only one abundant top trophic level species (e.g., rainbow trout, and frequently these are stocked fish). In these cases in the 2007-2008 survey, the one species present was often sampled as an indicator of all the target analytes. Rainbow trout in the 2007-2008 survey generally had low concentrations of methylmercury, with a statewide average of 0.05 ppm. Concentrations of organics in trout were also generally low. To some degree, this was due to lower concentrations of

contaminants in these lakes, but other factors also likely played a role. Trout generally occupy a lower trophic position and accumulate lower concentrations of methylmercury and other pollutants than black bass. However, a factor that probably contributed to lower observed concentrations in trout is that, in many lakes, recently planted hatchery fish are part of the catch. A previous study found that trout from California hatcheries consistently had very low concentrations of methylmercury (rainbow trout from four hatcheries all had less than 0.023 ppm - Grenier et al. 2007). With the level of effort that could be expended in the 2007-2008 survey it is possible that other resident species with a potential to have higher concentrations were missed, such as resident populations of trout or small populations of warmwater predators like black bass or bottom feeders like sucker. A significant outstanding question therefore is whether the 2007-2008 survey accurately characterized the status of lakes in which only rainbow trout were collected.

To attempt to address, and avoid perpetuating, this data gap, a greater effort (more hours spent fishing per lake) will be made to collect non-trout, primary indicator sport fish species at lakes where trout are abundant. The added effort will allow more definitive conclusions about the impact of contaminants on the fishing beneficial use at these water bodies.

Other species will also be observed in the process of electroshocking. This “bycatch” will not be collected, but the sampling crew will record estimates of the numbers of each species observed. This information may be useful if additional follow-up studies are needed at any of the sampled lakes.

#### **b. Sampling Locations Within Each Lake**

##### **Unsampled Lakes**

Lakes and reservoirs in California vary tremendously in size, from hundreds of small ponds less than 10 ha to Lake Tahoe at 50,000 ha. As lakes increase in size it becomes necessary to sample more than one location to obtain a representative characterization of the water body.

In sport fish sampling using an electroshocking boat, it is frequently necessary to sample over a linear course of 0.5 – 1 mi to obtain an adequate number of fish. A sampling location in this study can therefore be thought of as a circle with a diameter of 1 mile. For small lakes less than 500 ha in size, one sampling location covers a significant fraction of the surface area of the lake. Therefore, for lakes less than 500 ha, one location will be sampled. For lakes of medium size (500 – 1000 ha), two locations will generally be sampled. For lakes in the large (1000 – 5000 ha) and very large categories (>5000 ha), two to four locations will be sampled, with a goal of three locations for large lakes and four locations for very large lakes. Since the primary goal of the study is to characterize human exposure, locations will be established near centers of fishing activity.

Decisions regarding the number and placement of locations will be made in consultation with Regional Board staff with local knowledge of the lakes. Criteria to be

considered in determining the placement of sampling locations will include the existence of discrete centers of fishing activity, known patterns of spatial variation in contamination or other factors influencing bioaccumulation, road or boat ramp access, and possibly other factors.

#### Lakes With Data Gaps

As much as possible, the same sampling locations visited in previous sampling will be visited again for this survey.

#### c. Fish Size Ranges and Composite Preparation

Chemical analysis of mercury is relatively inexpensive, and SWAMP partners would like to be able to have statistical power to quantitatively answer questions related to mercury trends over time and differences among lakes. Consequently, the sampling design for the mercury indicator species includes analysis of mercury in individual fish. For the mercury indicator species, an analysis of covariance approach will be employed where possible, in which the size:mercury or age:mercury relationship will be established for each location and an ANCOVA will be performed. The ANCOVA will allow evaluation of differences in slope of the regression relation among the locations and comparison of mean concentrations and confidence intervals at a standardized total length, following the approach of Tremblay (1998). Experience applying this approach in past sampling indicates that to provide robust regressions, 11 fish spanning a broad range in size are needed (Davis et al. 2003, Melwani et al. 2007). A power analysis conducted to guide the long-term bass lake sampling design (Appendix 2 in Bioaccumulation Oversight Group 2015) indicated that increasing the number of fish per lake had little effect on power. The target number per lake was therefore kept at 11.

Chemical analysis of trace organics is relatively expensive, and the management questions established for this study can be adequately addressed with good information on average concentrations. Therefore a compositing strategy will be employed for these chemicals (Figures 2 and 3).

Specific size ranges to be targeted for each species are listed in Table 5. Black bass (including largemouth, smallmouth, and spotted bass) and Sacramento pikeminnow (included in Group 1) are the key mercury indicators. These species have a high trophic position and a strong size:mercury relationship. These species will be analyzed for mercury only (unless a bottom-feeding species is not present), and will be analyzed individually. The numbers and sizes indicated for these species will provide the size range needed to support ANCOVA. In addition, the size range for black bass takes the legal limit for these species (305 mm, or 12 inches) into account. The goal for black bass is to have a size distribution that encompasses the standardized total length (350 mm) to be used in statistical comparisons. This length is near the center of the distribution of legal-sized fish encountered in past studies (Davis et al. 2003, Melwani et al. 2007).

Catfish, carp, bullhead, and sucker are the primary targets for high-lipid bottom-feeders. These species will be analyzed for organics in selected lakes, based on Regional Board information needs. Organics are expected to be highest in these species based on past monitoring in the Toxic Substances Monitoring Program and other studies (Davis et al. 2007). Samples for these species will be analyzed as composites.

Secondary targets have been identified (Table 6) for collection if the primary targets are not available. These species would be processed for potential analysis of mercury and organics. The samples would be analyzed as composites. The size ranges established for bottom-feeders are based on a combination of sizes prevalent in past sampling (Melwani et al. 2007) and the 75% rule recommended by USEPA (2000) for composite samples.

In addition to these general targets, the sampling will attempt to address very specific data gaps (species and contaminant combinations) that have been identified for some of the lakes that will be revisited (Table 3).

The sampling crew will be reporting their catch back to the BOG on a weekly basis to make sure that the appropriate samples are collected and to address any unanticipated complications.

#### **d. Compositing and Archiving**

Strategies for compositing and archiving will vary somewhat for lakes of different size. The overall strategy will be described first for small lakes, followed by a discussion of the differences for larger lakes.

##### *Small Lakes*

Figures 2a,b illustrate the approach to be taken for the predator and bottom-feeding species in small lakes (<500 ha). As described above, the predator species will be analyzed for mercury only and as individual fish. All samples of the predator species will be analyzed. Small lakes will be treated as one sampling location, so fish from anywhere in the lake will be counted toward meeting the targets for each size range listed in Table 6. For ANCOVA, one common regression line will be developed to describe the size:mercury relationship for the lake as a whole. Aliquots from these samples will be archived after they are analyzed in case of any problems or other circumstances calling for reanalysis at a later time.

The bottom-feeding species in selected lakes (Table 3) will be analyzed as composites for organics (Figures 2a,b). For the lakes that have not previously been sampled, the "supercompositing" approach employed in the 2007-2008 survey will be followed (Figures 2a). For the lakes that are being revisited, all composite samples will be processed and analyzed. Aliquots from all composites will be archived in case of any problems or other circumstances calling for analysis or reanalysis at a later time.

### *Larger Lakes*

For lakes in the medium, large, and very large categories the basic approach will be similar, with a two modifications. Figures 3a,b illustrate the approach using a medium lake as the example. The first difference from the small lake approach is that sampling locations will be treated discretely. For the predator species, this means that 11 fish spanning a wide range of sizes will be targeted for each location to support the development of a size:mercury regression and an estimated mean concentration at standardized total length for each location. From these location means a lake-wide mean can be calculated. Similarly, the design for large and very large lakes will treat each sampling location discretely, typically with three and four locations, respectively, in each lake.

For the bottom-feeder species, discrete composites will be prepared for each location. These composites will be homogenized, analyzed, and archived. The “supercompositing” approach employed in the 2007-2008 survey will be followed for the lakes that were previously unsampled (Figure 3a). For the lakes that are being revisited, the individual composites will be analyzed.

#### **2. Prey Fish**

Prey fish (25-100 mm) will be sampled using traps, seines, and dip nets from shoreline areas adjacent to the locations where sport fish are collected. Ten individuals each from the three most common prey fish species will be sampled from each lake. We will target the following primary prey fish target species at all lakes: inland silversides, young-of-the-year largemouth bass, young-of-the-year bluegill, and threadfin shad. Other species that are within the target size range may be collected if the primary targets are not available. Efforts will be made to sample the same species across all lakes, and when not possible fish that overlap in trophic guild will be sampled. At trout lakes, it may not be possible to collect three prey species and the prey species present may be different. Extra species of fish in the correct size ranges will be retained, and decisions on species to analyze for mercury will be made after all fish are collected each year.

Prey fish will be composited by species in each lake and analyzed for mercury and selenium.

### **G. Sample Processing and Analysis**

#### **1. Sport Fish**

Fish will be collected in accordance with MPSSL-102a, Section 7.4 (Appendix 3). Whenever possible an electro-fishing boat will be used; however, it may be necessary to employ another method also described in Section 7.4.

The following adaptation to MPSL-102a, Section 7.4.5 (Appendix 3) has been made for this study: at the dock, each fish collected will be placed on a measuring board covered with a clean plastic bag; fork and total length will be recorded. Weight will be measured with a digital spring scale and recorded. Large fish will be partially dissected in the field using the following protocol: fish will be placed on a cutting board covered with a clean plastic bag where the head, tail, and guts are removed using a clean (laboratory detergent, DI) cleaver. The cleaver and cutting board will be re-cleaned between fish species, per site if multiple stations are sampled.

Upon collection, each fish collected will be tagged with a unique ID. Each fish collected will be linked to the latitude/longitude where it was collected. Several parameters will be measured in the field, including total length (longest length from tip of tail fin to tip of nose/mouth), fork length (longest length from fork to tip of nose/mouth), and weight. Total length changes with freezing and thawing and is best noted in the field for greatest accuracy and because it is the measure used by fishers and wardens to determine whether a fish is legal size. Determining fork length at the same time simplifies matters, and might help with IDs later to sort out freezer mishaps. For large fish (e.g., carp, which can be greater than 40 lb) there will be times when it is necessary to process fish in the field.

Whole fish or field-processed fish will be wrapped in aluminum foil and placed in a clean, labeled zipper-style bag. All samples will be kept cold on ice until frozen in a freezer or on dry ice within 24 hours of collection. Samples will be stored at -20°C at the laboratory until dissection and homogenization. Homogenates will also be frozen until analysis is performed. Frozen tissue samples have a 12-month hold time from the date of collection (USEPA 2000); however, the BOG Review Panel has advised that samples kept frozen, with minimal thaw-freeze cycles, for several years have no appreciable degradation of organic contaminants.

All sport fish will be dissected "skin off". This is inconsistent with the guidance of USEPA (2000) that recommends that fish with scales should have the scales removed and be processed with skin on, and skin should only be removed from scale-less fish (e.g., catfish). The BOG is aware of this difference, but favors skin removal. Skin removal has been repeatedly used in past California monitoring. All fish (with limited exceptions) in Toxic Substances Monitoring Program, the Coastal Fish Contamination Program, and the Fish Mercury Project also have been analyzed skin-off. Processing fish with the skin on is very tedious and results in lower precision because the skin is virtually impossible to homogenize thoroughly and achieving a homogenous sample is difficult. Also, skin-on preparation actually dilutes the measured concentration of mercury because there is less mercury in skin than in muscle tissue. The most ubiquitous contaminant in fish in California that leads to most of our advisories is mercury. By doing all preparation skin off we will be getting more homogeneous samples, better precision for all chemicals, and definitely a better measure of mercury concentrations, which are our largest concern. The analysis of axial fillets without skin was also advised by a bi-national workgroup concerning the monitoring and analysis of mercury in fish (Wiener et al. 2007).

Fish are filleted to expose the flesh. It is important to maintain the cleanliness of the tissue for analysis; therefore any flesh that has been in direct contact with the skin, with instruments in contact with skin, or with any potential contaminant surface such as foil or a plastic bag, must be eliminated from the analyzed sample. The exposed edges of the fillet should be trimmed by 1/4 inch with a clean scalpel or fillet knife to remove this potentially contaminated tissue.

How a sample is dissected is greatly dependent on the types of analyses being conducted. Tissue from individual fish for mercury analysis only will be dissected from the fillet above the lateral line. When composites must be created, equal tissue weights are taken from 5 individual fish following the 75% size rule recommended by USEPA (2000) and homogenized into a Location Composite with a target weight of 200 g or greater. Tissue for composites will be taken from the fillet of each fish above the lateral line and from the belly to include areas of higher lipid content. Figures 2 and 3 diagram compositing strategies and target sizes for predator and bottom species.

Mercury will be analyzed by the Moss Landing Marine Laboratory Marine Pollution Studies Lab according to USEPA Method 7473, "Mercury in Solids and Solutions by Thermal Decomposition, Amalgamation, and Atomic Absorption Spectrophotometry" using a Direct Mercury Analyzer. 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  $\pm 20\%$  of the true value, or the previous 10 samples must be reanalyzed. Three blanks, a standard reference material (such as IAEA-407 or NRCC DORM-4), as well as a method duplicate and a matrix spike pair will be run with each set of samples.

Selenium will be analyzed by the Moss Landing Marine Laboratory Marine Pollution Studies Lab. Selenium will be digested according to EPA 3052M, "Microwave Assisted Acid Digestion of Siliceous and Organically Based Matrices", modified, and analyzed according to EPA 200.8, "Determination of Trace Elements in Waters and Wastes by Inductively Coupled Plasma-Mass Spectrometry". 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  $\pm 20\%$  of the true value, or the previous 10 samples must be reanalyzed. Two blanks, a standard reference material (2976 or NRCC DORM-3), as well as a method duplicate and a matrix spike pair will be run with each set of samples.

Organics analyses will be performed by the California Department of Fish and Wildlife Water Pollution Control Lab in Rancho Cordova, CA. Organochlorine pesticides and PCBs will be analyzed according to WPCL-GC-006 "Analysis of Extractable Synthetic Organic Compounds in Tissues and Sediment (including Organochlorine Pesticides, Polychlorinated Biphenyls (PCBs) and PBDEs) by GC/ECD

analyzed in all lakes because of the high proportion of lakes with elevated concentrations, and an interest in tracking whether the few lakes with low concentrations remain that way.

#### *Selenium*

Selenium concentrations in California sport fish are generally below thresholds of concern. However, because of the well-known interrelations of mercury and selenium in their bioaccumulation and in influencing the toxicity of mercury in avian and human consumers of fish (e.g., demethylation, sequestration), selenium analyses in sport fish and prey fish are included in this study. Although OEHHA does not have reference doses to consider relative molar concentrations of mercury and selenium when developing consumption advisories, these data are being collected so they are available for consideration relative to fish-eating birds and humans.

#### *PCBs*

PCBs are the contaminant of second-greatest concern with respect to bioaccumulation on a statewide basis (Davis et al. 2010). PCBs will be analyzed using a congener-specific method. A total of 55 congeners will be analyzed (Table 7). The species with the greatest expected concentrations (i.e., the organics indicator species where they are present) will be analyzed. PCBs will be measured in selected lakes to address data gaps identified by the Regional Boards (Table 3).

#### *Legacy Pesticides*

Legacy pesticides may be present at concentrations of concern in some locations. Individual compounds recommended by USEPA (2000) will be analyzed (Table 7). The species with the greatest expected concentrations (i.e., the organics indicator species where they are present) will be analyzed. Organochlorine pesticides will be measured in selected lakes to address data gaps identified by the Regional Boards (Table 3).

### **I. Quality Assurance**

This effort will adhere to quality assurance requirements established for the SWAMP. A QAPP that applies to this effort has been prepared (Bonnema 2016).

### **J. Archiving**

#### **1. Sport Fish**

Sample aliquots will be stored in short-term archives. Samples in the short-term archive are stored at -20 °C and are intended for use in the identification of short-term time trends (i.e., < 5-10 years), the investigation of yet-unidentified chemical contaminants, and addressing quality assurance issues that may arise during the routine

analyses of samples. These samples are intended for the analysis of chemicals that are not expected to degrade in five years of storage at -20 °C. The short-term archives will be located in an off-site freezer facility rented by Moss Landing Marine Laboratory. The facility is equipped with a backup generator in the event of a power outage.

A number of small-volume sub-samples, rather than one or two large-volume samples, will be prepared for archiving to avoid subjecting the samples to several freeze-thaw cycles. Each sub-sample will contain a sufficient amount of material for most chemical analysis, and when needed, can be removed from the freezer and sent to the appropriate laboratory without the need to sub-sample.

For each sampling location, up to three 40-50 g aliquots of each composite analyzed for organics will be archived. This will provide an integrative, representative sample for each location that can be reanalyzed in later years to confirm earlier analyses, look for new chemicals of concern, provide material for application of new analytical methods, provide material for other ecological research, and other purposes. Samples for the short-term archive will be stored in either glass jars with Teflon-lined lids for non-fluorinated organic chemical and trace metal analysis or in polyethylene or polypropylene for fluorinated chemical (i.e., PFCs) or trace metals analysis. Two of the three archive jars will be glass with a Teflon-lined lid (e.g., I-Chem 200 series glass jars). One separate aliquot will be kept in a polypropylene jar for potential analysis of perfluorinated compounds.

For storage of samples in the short-term archive, glass and plastic containers will be pre-cleaned using appropriate acids or solvents by MPSL-DFG or purchased pre-cleaned commercially (e.g., from Fisher or ESS Vial). For containers purchased 'pre-cleaned' from ESS Vial or other companies, a minimum of two per shipment will not be opened and kept in storage with the other samples in case container contamination issues arise.

## **K. Ancillary Data**

In addition to the primary and secondary target species, other species will be observed in the process of sample collection. This "bycatch" will not be collected, but the sampling crew will record estimates of the numbers of each species observed. This information may be useful if follow-up studies are needed in any of the sampled locations.

## **L. Timing**

The sampling will be conducted from May 2016 through November 2016. Seasonal variation in body condition and reproductive physiology, as well as limnological characteristics, are recognized as factors that could affect contaminant concentrations, so the period of sampling will be kept as narrow as possible.

## **M. Data Assessment**

Data from this study will generally be assessed relative to advisory tissue levels established by the California Office of Environmental Health Hazard Assessment (Klasing and Brodberg 2008) (Table 8). Advisory Tissue Levels (ATLs) consider both the toxicity of contaminants and the human 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 Safe to Eat Portal (<http://www.mywaterquality.ca.gov/>) and via SWAMP reports and fact sheets.

MQ1 will be assessed by the Regional Boards. The Regional Boards will compare the distributions of concentrations of priority contaminants in the lakes that are sampled to the listing thresholds developed by the Water Boards.

MQ2 will be assessed by OEHHA. OEHHA will evaluate the completeness of the datasets for each species in each lake, and whether the data for each lake are sufficient to support a specific advisory for that lake.

MQ3 will be assessed in coordination with OEHHA. OEHHA will evaluate the completeness of the datasets for each species in each lake, and whether the data for each lake are sufficient to support a specific advisory highlighting that lake as one with relatively low concentrations of contaminants.

## **N. Products and Timeline**

A data report on this 2016 sampling will be drafted by December 2017. A fact sheet and a final data report, incorporating revisions in response to reviewer comments, will be completed and released in May 2018. The data will be posted to the My Water Quality Portal in May 2018.

## REFERENCES

- Ackerman, J.T., C.A. Hartman, C.A. Eagles-Smith, M.P. Herzog, J.A. Davis, G. Ichikawa, and A. Bonnema. 2015. Estimating Mercury Exposure to Piscivorous Birds and Sport Fish in California Lakes Using Prey Fish Monitoring: A Tool for Managers. U.S. Geological Survey Open-File Report 2015-1106, 48 p., <http://dx.doi.org/10.3133/ofr20151106>.
- Beckon, W.N., M.C.S. Eacock, A. Gordus, and R. McNeal. 2010. Biological Effects of the Grassland Bypass Project. Chapter 7 in Grassland Bypass Project Annual Report 2008-2009. <http://www.sfei.org/gbp/reports/annual-reports>
- Bernstein, B. 2010. SWAMP Assessment Framework. Surface Water Ambient Monitoring Program. California State Water Resources Control Board, Sacramento, CA.
- Bioaccumulation Oversight Group. 2015. Sampling and Analysis Plan for Long-term Monitoring of Bass Lakes and Reservoirs in California. Surface Water Ambient Monitoring Program. California State Water Resources Control Board, Sacramento, CA.
- BOG (Bioaccumulation Oversight Group). 2007. Sampling and Analysis Plan for a Screening Study of Bioaccumulation in California Lakes and Reservoirs. Surface Water Ambient Monitoring Program. California State Water Resources Control Board, Sacramento, CA.
- Bonnema, A. 2016. Quality Assurance Project Plan: Monitoring of Lakes and Reservoirs in California: 2016. Moss Landing Marine Labs. Prepared for SWAMP BOG, 64 pages plus appendices and attachments
- Davis, J. A., B. K. Greenfield, G. Ichikawa, and M. Stephenson. 2003. Mercury in sport fish from the Delta region (Task 2A). An assessment of the ecological and human health impacts of mercury in the Bay-Delta watershed. CALFED Bay-Delta Mercury Project, Sacramento, CA.
- Davis, J.A., J. L. Grenier, A. R. Melwani, S. N. Bezalel, E. M. Letteney, E. J. Zhang, M. Odaya. 2007. Bioaccumulation of Pollutants in California Waters: A Review of Historic Data and Assessment of Impacts on Fishing and Aquatic Life. Prepared for the Surface Water Ambient Monitoring Program, California State Water Resources Control Board, Sacramento, CA.
- Davis, J.A., A.R. Melwani, S.N. Bezalel, J.A. Hunt, G. Ichikawa, A. Bonnema, W.A. Heim, D. Crane, S. Swenson, C. Lamerdin, and M. Stephenson. 2010. Contaminants in Fish from California Lakes and Reservoirs, 2007-2008: Summary Report on Two-Year Screening Survey. A Report of the Surface Water Ambient Monitoring Program (SWAMP). California State Water Resources Control Board, Sacramento, CA. [http://www.swrcb.ca.gov/water\\_issues/programs/swamp/lakes\\_study.shtml](http://www.swrcb.ca.gov/water_issues/programs/swamp/lakes_study.shtml)
- Davis, J.A., J.R.M. Ross, S.N. Bezalel, J.A. Hunt, A.R. Melwani, R.M. Allen, G. Ichikawa, A. Bonnema, W.A. Heim, D. Crane, S. Swenson, C. Lamerdin, M. Stephenson, and K. Schiff. 2012. Contaminants in Fish from the California Coast, 2009-2010: Summary Report on a Two-Year Screening Survey. A Report of the Surface Water Ambient Monitoring Program (SWAMP). California State Water Resources Control Board, Sacramento, CA.
- Davis, J.A., J.R.M. Ross, S.N. Bezalel, J.A. Hunt, G. Ichikawa, A. Bonnema, W.A. Heim, D. Crane, S. Swenson, and C. Lamerdin. 2013. Contaminants in Fish from California Rivers

- and Streams, 2011. A Report of the Surface Water Ambient Monitoring Program (SWAMP). California State Water Resources Control Board, Sacramento, CA.
- Grenier, J.L. et al. 2007. California Bay-Delta Authority Fish Mercury Project: Year 1 Annual Report - Sport Fish Sampling and Analysis. San Francisco Estuary Institute, Richmond, CA.
- Klasing, S. and R. Brodberg. 2008. Development of Fish Contaminant Goals and Advisory Tissue Levels for Common Contaminants in California Sport Fish: Chlordane, DDTs, Dieldrin, Methylmercury, PCBs, Selenium, and Toxaphene. California Office of Environmental Health Hazard Assessment, Sacramento, CA.
- Melwani, A.R., W. Heim, M. Gassel, A. Bonnema, S.N. Bezalel, J.A. Hunt, J.L. Grenier, J.A. Davis, G. Ichikawa, and B. Jakl. 2007. Fish Mercury Project Year 2 Annual Report Sport Fish Sampling and Analysis. Contribution #535. San Francisco Estuary Institute, Richmond, CA.
- Stienstra, T. 2012. Moon California Fishing: The Complete Guide to Fishing on Lakes, Streams, Rivers, and the Coast. Avalon Travel, Berkeley, CA.
- Tremblay, G., P. Legendre, J. Doyon, R. Verdon, and R. Schetagne. 1998. The use of polynomial regression analysis with indicator variables for interpretation of mercury in fish data. *Biogeochemistry* 40:189-201.
- USEPA. 2000. Guidance for Assessing Chemical Contaminant Data for Use in Fish Advisories: Volume 1, Fish Sampling and Analysis, Third Edition. EPA 823-R-93-002B-00-007. U.S. Environmental Protection Agency, Office of Water, Washington, D.C.
- Wiener, J.G., R.A. Bodaly, S.S. Brown, M. Lucotte, M.C. Newman, D.B. Porcella, R.J. Reash, and E.B. Swain. 2007. Monitoring and evaluating trends in methylmercury accumulation in aquatic biota. Chapter 4 in R. C. Harris, D. P. Krabbenhoft, R. P. Mason, M. W. Murray, R. J. Reash, and T. Saltman (editors) *Ecosystem responses to mercury contamination: indicators of change*. SETAC Press, Pensacola, Florida.

Figure 1. Map of sampling locations. Lake names are indicated in Table 3.



Figure 2a. Sampling design for a small lake that has not previously been sampled.

**Small Lake (0 – 500 ha)**  
**Previously Unsampled**

Analyze Orgs + Hg + Se	Analyze Hg
Archive Orgs + Hg + Se	Analyze Se

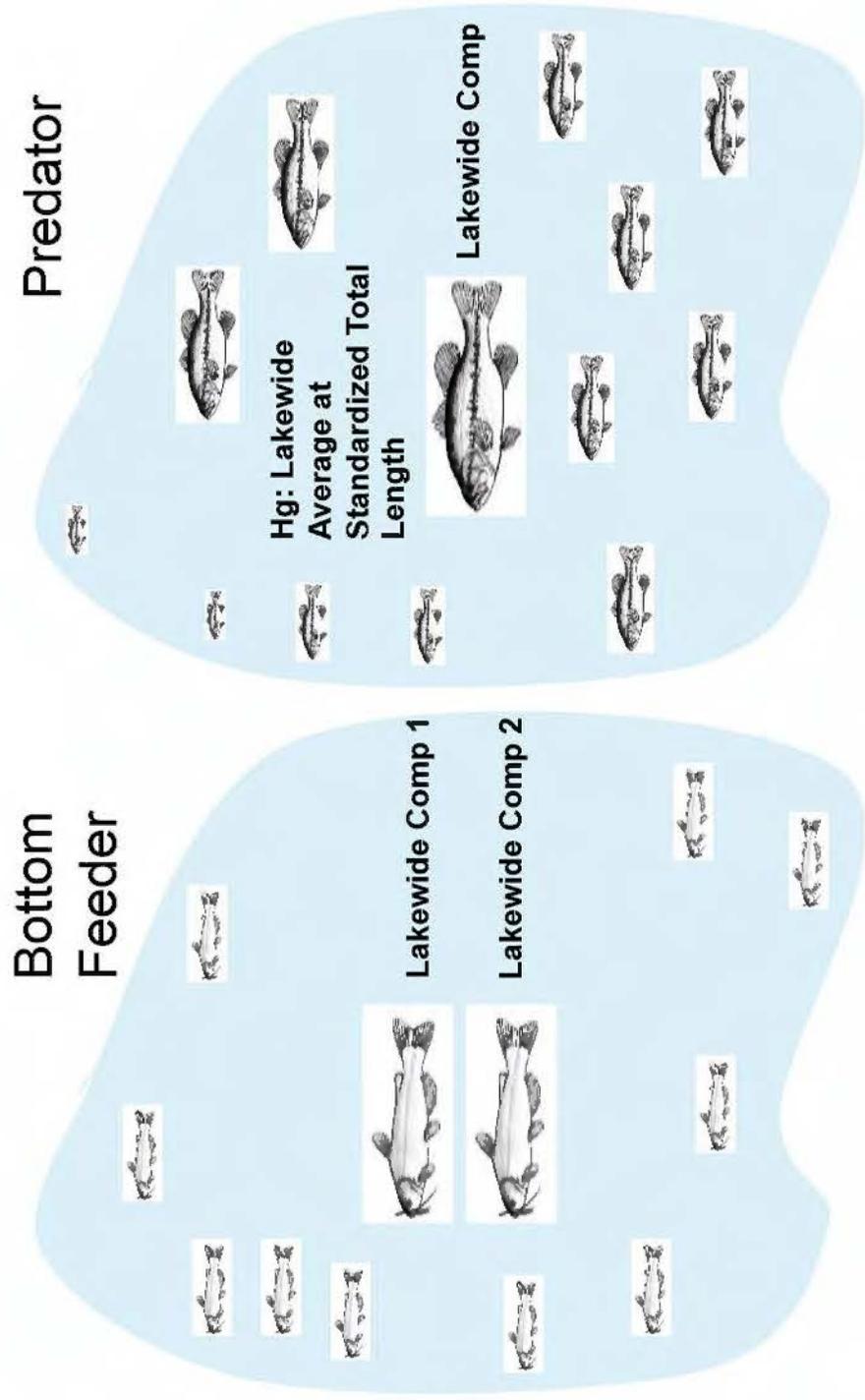


Figure 2b. Sampling design for a small lake that has previously been sampled.

**Small Lake (0 – 500 ha)  
Previously Sampled**

Analyze Orgs\* + Hg + Se      Analyze Hg  
Analyze Se

\* Where specifically requested

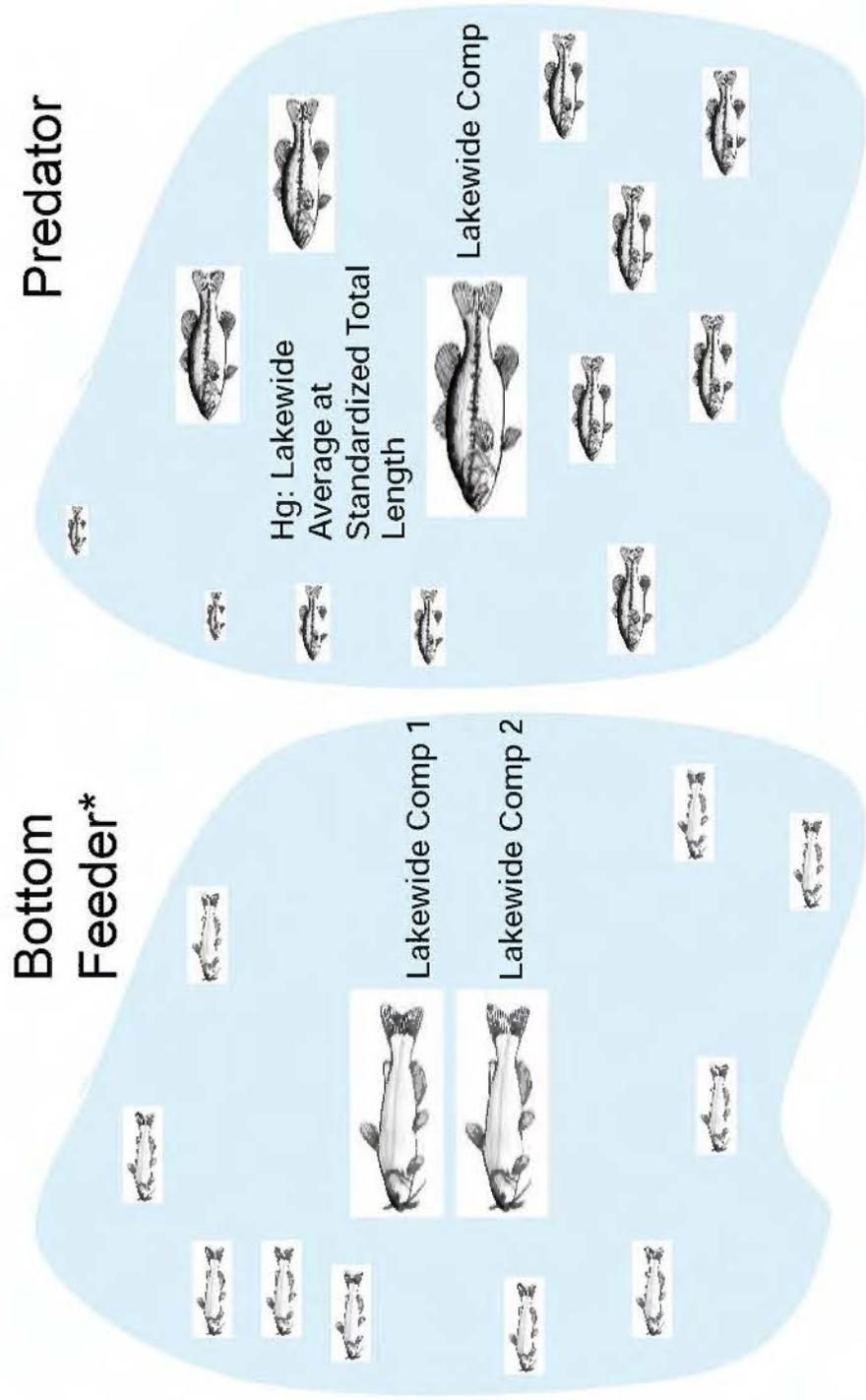


Figure 3a. Sampling design for a medium lake that has not previously been sampled.

**Medium Lake  
 (500 – 1000 ha)  
 Previously Unsampled**

Analyze Orgs + Hg + Se      Analyze Hg  
 Archive Orgs + Hg + Se      Analyze Se

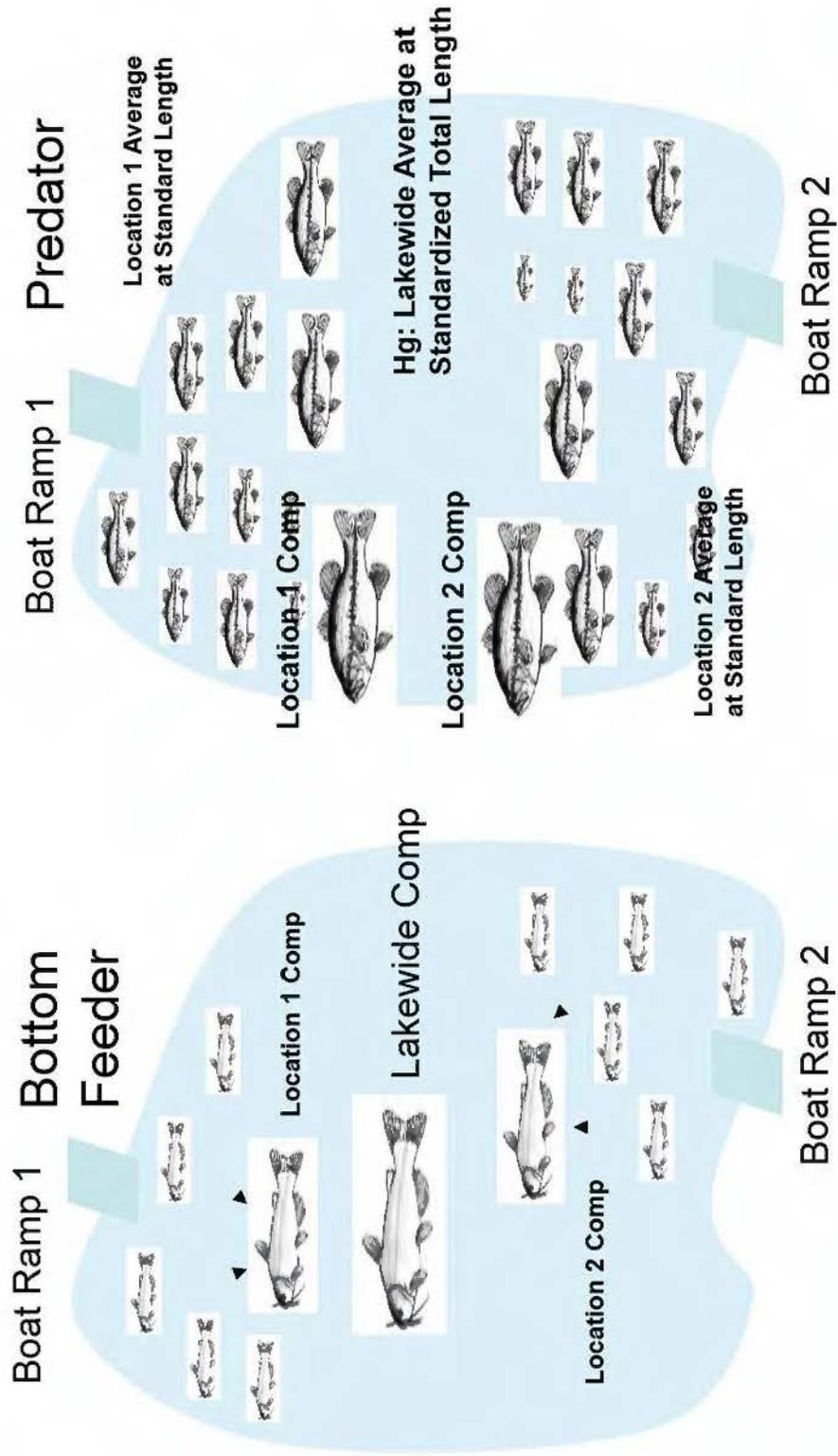


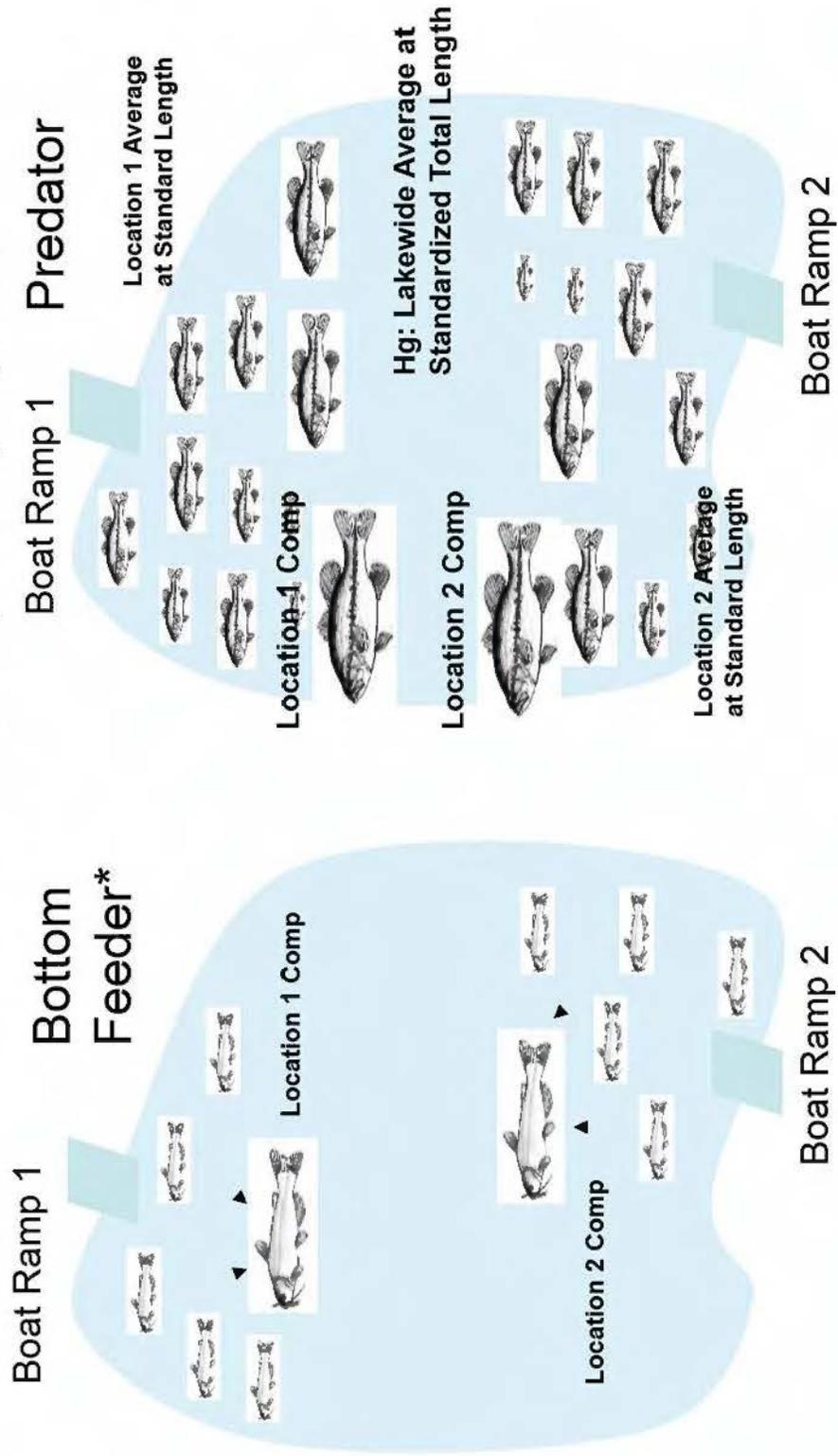
Figure 3b. Sampling design for a medium lake that has previously been sampled.

**Medium Lake  
 (500 – 1000 ha)  
 Previously Sampled**

Analyze Orgs\* + Hg + Se      Analyze Hg

Analyze Se

\* Where specifically requested



**Table 1. Bioaccumulation monitoring assessment framework for the fishing beneficial use.**

- D.1. Determine the status of the fishing beneficial use throughout the State with respect to bioaccumulation of toxic pollutants*
- D.1.1.1 What are the extent and location of water bodies with sufficient evidence to indicate that the fishing beneficial use is at risk due to pollutant bioaccumulation?
  - D.1.2 What are the extent and location of water bodies with some evidence indicating the fishing beneficial use is at risk due to pollutant bioaccumulation?
  - D.1.3 What are the extent and location of water bodies with no evidence indicating the fishing beneficial use is at risk due to pollutant bioaccumulation?
  - D.1.4 What are the proportions of water bodies in the State and each region falling within the three categories defined in questions D.1.1.1, D.1.2, and D.1.3?
- D.2. Assess trends in the impact of bioaccumulation on the fishing beneficial use throughout the State*
- D.2.1 Are water bodies improving or deteriorating with respect to the impact of bioaccumulation on the fishing beneficial use?
    - D.2.1.1 Have water bodies fully supporting the fishing beneficial use become impaired?
    - D.2.1.2 Has full support of the fishing beneficial use been restored for previously impaired water bodies?
  - D.2.2 What are the trends in proportions of water bodies falling within the three categories defined in questions D.1.1, D.1.2, and D.1.3 regionally and statewide?
- D.3. Evaluate sources and pathways of bioaccumulative pollutants impacting the fishing beneficial use*
- D.3.1 What are the magnitude and relative importance of pollutants that bioaccumulate and indirect causes of bioaccumulation throughout each Region and the state as a whole?
  - D.3.2 How is the relative importance of different sources and pathways of bioaccumulative pollutants that impact the fishing beneficial use changing over time on a regional and statewide basis?
- D.4. Provide the monitoring information needed to evaluate the effectiveness of management actions in reducing the impact of bioaccumulation on the fishing beneficial use*
- D.4.1 What are the management actions that are being employed to reduce the impact of bioaccumulation on the fishing beneficial use regionally and statewide?
  - D.4.2 How has the impact of bioaccumulation on the fishing beneficial use been affected by management actions regionally and statewide?

Table 2. Short list of candidate lakes.

Region	Map Lab	Lake	Stenstra Rating	Bass, Trout, Both	Previously Sampled	Bass Pan	Regional Priority for 2016	Potential for Followup Based on Clean Lakes	Short List for 2016	Final List for 2016	Include PCBS	Include OC Pesticides	Reason For Not Making Final Lis
1	1	Freshwater Lagoon	7	Trout	-	-	1		X	X	X		
1	2	Ewing Reservoir	4	Trout	-	-	2		X	X	X		
1	3	Plaskett Lake	5	Neither (ha	2008	-	3		X	X	X		
2	4	Alpine Lake	3	Bass	-	-	3		X	X	X		
2	5	Kent Lake	3	Bass	-	-	4		X	X	X		
2	6	Lake Temescal	6	Bass	2013	-	1		X	X	X		
2	7	Stafford Lake	6	Bass	-	-	2		X	X	X		
2	2	Lexington Reservoir	8	Both	2004, 2005	-	xx		X	X	X	X	Scheduled for
2	2	Stevens Creek Reservoir	5	Bass	2007	-	xx		X	X	X	X	Scheduled for
3		San Felipe Lake	-	Bass	-	-	1		X	X	X		Scheduled for
3	8	Coyote Lake	-	Bass	2008	-	2		X	X	X		San Felipe La
3	9	Pacheco Lake	-	Both	-	-	4		X	X	X		
3	10	Whale Rock Reservoir	2	Trout, othe	-	-	3		X	X	X		
5	11	Spaulding Lake	-	Trout	2008	-	1		X	X	X		
5	12	Union Valley Reservoir	Both	Both	2008	2021	2		X	X	X		
5	13	Little Grass Valley Reservoir	Trout, Bullt	Trout, Bullt	2008	-	3		X	X	X		
5	14	Siy Creek Reservoir	Trout	Trout	2007	-	4		X	X	X		
5	15	Wishon Reservoir	Trout	Trout	2007	-	5		X	X	X		
5	16	Bethany Reservoir	4	Bass	-	2023	6		X	X	X		
5	17	Rancho Seco Lake	6	Bass	-	-	7		X	X	X		
5	18	Lake Clementine	4	SMB, RBT	-	-	8		X	X	X		
5	5	Fordyce Lake	Trout	Trout	-	-	-		X	X	X		quite hard to
6		Crater Lake	Trout	Trout	2007	-	5-6		X	X	X		
6		South Lake	Trout	Trout	-	-	5-6		X	X	X		
6	19	Lower Echo Lake - El Dorado County	Trout	Trout	-	-	4		X	X	X		
6	20	Red Lake - Alpine County	Trout	Trout	-	-	1-3		X	X	X		
6	21	Diaz Lake - Lone Pine	5	Bass	-	-	1-3		X	X	X		
6	22	Hesperia Lake - Hesperia	-	Bass	-	-	1-3		X	X	X		
7	23	Salton Sea	Tiapha	Tiapha	2007	-	1		X	X	X		
7	24	Finney Lake	Bass	Bass	2014	-	4		X	X	X		
7	25	Squaw Lake	Bass	Bass	2014	-	2		X	X	X		
7		Senator Wash Reservoir	Bass	Bass	2007, 2014	-	??		X	X	X		
7	26	Taylor Lake	Bass	Bass	2014	-	3		X	X	X		
7		Wiest Lake	Bass	Bass	2004, 2007, 201	-	??		X	X	X		Scheduled for
8	27	Big Bear Lake	Bass	Bass	2004, 2005, 200	2021	1		X	X	X	X	
8	28	Irvine Lake	Bass	Bass	2007	2023	2		X	X	X		
8	29	Lee Lake	Bass	Bass	2008	2021	4		X	X	X		
8	29	Perris Reservoir	Bass	Bass	2006, 2013	2021	5		X	X	X		Low water lev
8	30	Lake Hemet	Trout	Trout	2008	2019	3		X	X	X		
9	31	Diamond Valley Lake	Bass	Bass	-	-	1		X	X	X	X	
9	32	Lake Murray (Murray Reservoir)	Bass	Bass	-	2019	2		X	X	X	X	
9	33	Dixon Lake	Bass	Bass	2006, 2014	-	3		X	X	X	X	

Table 3. List of lakes selected for inclusion.

Region	Map Label	Lake	Stienstra Ratio	Bass, Trout, Both	Previously Sampled	Bass Pan	Regional Priority for 2016	Potential for Followup Based on Clean Lakes	Short List for 2016	Final List for 2016	Include PCBs	Include Pesticides
1	1	Freshwater Lagoon	7	Trout	-	-	1		X	X	X	
1	2	Ewing Reservoir	4	Trout	-	-	2		X	X	X	
1	3	Plaskett Lake	5	Neither (ha)	2008	-	3		X	X		
2	4	Alpine Lake	3	Bass	-	-	3		X	X	X	
2	5	Kent Lake	3	Bass	-	-	4		X	X	X	
2	6	Lake Temescal	6	Bass	2013	-	1		X	X	X	
2	7	Stafford Lake	6	Bass	-	-	2		X	X	X	
3	8	Coyote Lake	-	Bass	2008	-	2		X	X	X	
3	9	Pacheco Lake	-	Both	-	-	4		X	X	X	
3	10	Whale Rock Reservoir	2	Trout, othe	-	-	3		X	X	X	
5	11	Spaulding, Lake	-	Trout	2008	-	1		X	X	X	
5	12	Union Valley Reservoir	-	Both	2008	2021	2		X	X	X	
5	13	Little Grass Valley Reservoir	-	Trout, Bullr	2008	-	3		X	X	X	
5	14	Sly Creek Reservoir	-	Trout	-	-	4		X	X	X	
5	15	Wishon Reservoir	-	Trout	2007	-	5		X	X	X	
5	16	Bethany Reservoir	4	Bass	-	2023	6		X	X	X	
5	17	Rancho Seco Lake	6	Bass	-	-	7		X	X	X	
5	18	Lake Clementine	4	SMB, RBT	-	-	8		X	X	X	
6	19	Lower Echo Lake - El Dorado County	-	Trout	-	-	4		X	X	X	
6	20	Red Lake - Alpine County	-	Trout	-	-	1-3		X	X	X	
6	21	Diaz Lake - Lone Pine	5	Bass	-	-	1-3		X	X	X	
6	22	Hesperia Lake - Hesperia	-	Bass	-	-	1-3		X	X	X	
7	23	Salton Sea	-	Tilapia	2007	-	1		X	X	X	
7	24	Finney Lake	-	Bass	2014	-	4		X	X	X	X
7	25	Squaw Lake	-	Bass	2014	-	2		X	X	X	
7	26	Taylor Lake	-	Bass	2014	-	3		X	X	X	
8	27	Big Bear Lake	-	Bass	2004, 2005, 200	2021	1		X	X	X	X
8	28	Irvine Lake	-	Bass	2007	2023	2		X	X	X	
8	29	Perris Reservoir	-	Bass	2008, 2013	2021	5		X	X	X	
8	30	Lake Hemet	-	Trout	2008	2019	3		X	X	X	
9	31	Diamond Valley Lake	-	Bass	-	2019	1		X	X	X	X
9	32	Lake Murray (Murray Reservoir)	-	Bass	-	2023	2		X	X	X	X
9	33	Dixon Lake	-	Bass	2008, 2014	-	3	X	X	X	X	X

Table 4. Target species and their characteristics.

Species	Foraging Type		Trophic Level	Distribution			Priority for Collection
	Water column	Bottom feeder		Low Elevation	Foothills	High Elevation	
Largemouth bass	X		4	X	X		A
Smallmouth bass	X		4	x	X		A
Spotted bass	X		4	x	X		A
Sacramento pikeminnow	X		4	x	x		B
White catfish		X	4	x	x		A
Brown bullhead		X	3	x			B
Channel catfish		X	4	X	X		A
Carp		X	3	X	X		A
Sacramento sucker		X	3	x	x		B
Tilapia		X	3				B
Bluegill	X		3	X	X		B
Green sunfish	X		3	X	X		B
Crappie	X		3/4	x	x		B
Redear sunfish	X		3	X	X		B
Rainbow trout	X		3/4	x	x	X	A
Brown trout	X		3/4		x	x	A
Brook trout	X		3		x	x	A
Kokanee	X		3	?	x	x	B

Trophic levels are the hierarchical strata of a food web characterized by organisms that are the same number of steps removed from the primary producers. The USEPA's 1997 Mercury Study Report to Congress used the following criteria to designate trophic levels based on an organism's feeding habits:

- Trophic level 1: Phytoplankton.
  - Trophic level 2: Zooplankton and benthic invertebrates.
  - Trophic level 3: Organisms that consume zooplankton, benthic invertebrates, and TL2 organisms.
  - Trophic level 4: Organisms that consume trophic level 3 organisms.
- X widely abundant x less widely abundant "A" primary target for collection "B" secondary target for collection

Table 5. Target species, size ranges, and processing instructions. I - process as individuals. C - process as composites.

	Process for Mercury	Process for Organics and Selenium	Numbers and Size Ranges (mm)
<b>Primary Targets: stay on location until one of these targets from both Group 1 and 2 is obtained, or collect secondary targets if primary targets are not available</b>			
<b>Group 1) Predator</b>			
Black bass	I		2X(200-249), 2X(250-304), 6X(305-407), 2X(>407)
Sacramento pikeminnow	I		3X(200-300), 5X(300-400), 3X(400-500)
Brown trout	I	C	3X(200-300), 5X(300-400), 3X(400-500)
Rainbow trout	C	C	5X(300-400)
Brook trout	C	C	5X(300-400)
<b>Group 2) Bottom feeder</b>			
White catfish	C	C	5X(229-305)
Channel catfish	C	C	5X(375-500)
Common carp	C	C	5X(450-600)
Brown bullhead	C		5X(262-350)
Sacramento sucker	C	C	5X(375-500)
<b>Secondary Targets: collect these if primary targets are not available</b>			
Bluegill	C	C	5X(127-170)
Redear sunfish	C	C	5X(165-220)
Black crappie	C	C	5X(187-250)
Tilapia	C	C	5X(235-314)
Green sunfish	C	C	Xx
Kokanee	C	C	5X(300-400)

Table 6. Summary of sport fish analytes included in the study.

<b>Analyte</b>	<b>Included in Study?</b>
Methylmercury <sup>1</sup>	Some individuals, all composites
PCBs	Selected composites
DDTs	Selected composites
Dieldrin	Selected composites
Aldrin	Selected composites
Chlordanes	Selected composites
Selenium	All composites
Microcystins	Not included
PBDEs	Not included
Dioxins	Not included
Perfluorinated chemicals	Not included
Omega-3 fatty acids	Not included

<sup>1</sup> Measured as total mercury, which provides a direct estimate of methylmercury in fish muscle.

Table 7. Parameters to be measured in sport fish.

**FISH ATTRIBUTES**

1. Total length
2. Fork length
3. Standard length (small fish only)
4. Weight
5. Sex
6. Moisture
7. Lipid content
8. Estimated age (for black bass)

**METALS AND METALLOIDS**

1. Total mercury
2. Selenium

**PESTICIDES**

**Chlordanes**

1. Chlordane, cis-
2. Chlordane, trans-
3. Heptachlor
4. Heptachlor epoxide
5. Nonachlor, cis-
6. Nonachlor, trans-
7. Oxychlordane

**DDTs**

1. DDD(o,p')
2. DDD(p,p')
3. DDE(o,p')
4. DDE(p,p')
5. DDMU (p,p')
6. DDT(o,p')
7. DDT(p,p')

**Cyclodienes**

1. Aldrin
2. Dieldrin
3. Endrin

**HCHs**

1. HCH, alpha
2. HCH, beta

**Others**

1. Dacthal
2. Endosulfan I
3. Hexachlorobenzene
4. Methoxychlor
5. Mirex
6. Oxadiazon

**PCBs**

1. PCB 008
2. PCB 011
3. PCB 018
4. PCB 027
5. PCB 028
6. PCB 029
7. PCB 031
8. PCB 033
9. PCB 044
10. PCB 049
11. PCB 052
12. PCB 056
13. PCB 060
14. PCB 064
15. PCB 066
16. PCB 070
17. PCB 074
18. PCB 077
19. PCB 087
20. PCB 095
21. PCB 097
22. PCB 099
23. PCB 101
24. PCB 105
25. PCB 110
26. PCB 114
27. PCB 118
28. PCB 126
29. PCB 128
30. PCB 137
31. PCB 138
32. PCB 141
33. PCB 146
34. PCB 149
35. PCB 151
36. PCB 153
37. PCB 156

- 38. PCB 157
- 39. PCB 158
- 40. PCB 169
- 41. PCB 170
- 42. PCB 174
- 43. PCB 177
- 44. PCB 180
- 45. PCB 183
- 46. PCB 187
- 47. PCB 189
- 48. PCB 194
- 49. PCB 195
- 50. PCB 198/199
- 51. PCB 200
- 52. PCB 201
- 53. PCB 203
- 54. PCB 206
- 55. PCB 209

## APPENDIX 1

### Species Targets for Each Lake

## Region 1

### Freshwater Lagoon

Size category: small (one sampling location)

Lake type: Trout lake

Not previously sampled

Target species and analyses:

- Sport fish: Target 4 species, one of each category
  - Trout: mercury, Se, PCBs (if bass or a bottom-feeder not available)
  - Bass (if possible): mercury, Se, PCBs (if a bottom-feeder not available)
  - Bottom-feeder (if possible): mercury, Se, PCBs
  - Sunfish species (if possible): mercury, Se
- Prey fish: Targeting 3 species; mercury, Se

### Ewing Reservoir

Size category: small (one sampling location)

Lake type: Trout lake

Not previously sampled

Target species and analyses:

- Sport fish: Target 4 species, one of each category
  - Trout: mercury, Se, PCBs (if bass or a bottom-feeder not available)
  - Bass (if possible): mercury, Se, PCBs (if a bottom-feeder not available)
  - Bottom-feeder (if possible): mercury, Se, PCBs
  - Sunfish species (if possible): mercury, Se
- Prey fish: Targeting 3 species; mercury, Se

### Plaskett Lake

Size category: small (one sampling location)

Lake type: Trout

Previously sampled: BOG 2008

Target species and analyses (note: PCBs are not being measured):

- Sport fish: Target 4 species, one of each category
  - Trout: mercury, Se
  - Bass (if possible): mercury, Se
  - Hardhead\* or other bottom-feeder (if possible): mercury, Se
  - Sunfish species (if possible): mercury, Se
- Prey fish: Targeting 3 species; mercury, Se

\* indicates species needed by OEHHHA

## Region 2

### Lake Temescal

Size category: small (one sampling location)

Lake type: Bass and trout lake

Previously sampled: 2013

Target species and analyses (note: organics are not being measured):

- Sport fish: Target 4 species, one of each category
  - Trout\*: mercury, Se
  - Bass: mercury, Se
  - Carp\* or other bottom-feeder: mercury, Se
  - Sunfish species (if possible): mercury, Se
- Prey fish: Targeting 3 species; mercury, Se

\* indicates species needed by OEHHIA

### Stafford Lake

Size category: small (one sampling location)

Lake type: Bass lake

Not previously sampled

Target species and analyses:

- Sport fish: Target 3 species, one of each category
  - Bass: mercury, Se, PCBs (if a bottom-feeder not available)
  - Bottom-feeder: mercury, Se, PCBs
  - Sunfish species: mercury, Se
- Prey fish: Targeting 3 species; mercury, Se

### Alpine Lake

Size category: small (one sampling location)

Lake type: Bass lake

Not previously sampled

Target species and analyses:

- Sport fish: Target 3 species, one of each category
  - Bass: mercury, Se, PCBs (if a bottom-feeder not available)
  - Bottom-feeder: mercury, Se, PCBs
  - Sunfish species: mercury, Se
- Prey fish: Targeting 3 species; mercury, Se

### Kent Lake

Size category: small (one sampling location)

Lake type: Bass lake

Not previously sampled

Target species and analyses:

- Sport fish: Target 3 species, one of each category
  - Bass: mercury, Se, PCBs (if a bottom-feeder not available)
  - Bottom-feeder: mercury, Se, PCBs
  - Sunfish species: mercury, Se
- Prey fish: Targeting 3 species; mercury, Se

### Region 3

#### San Felipe Lake

Size category: small (one sampling location)

Lake type: Bass lake

Not previously sampled

Target species and analyses:

- Sport fish: Target 3 species, one of each category
  - Bass: mercury, Se, PCBs (if a bottom-feeder not available)
  - Bottom-feeder: mercury, Se, PCBs
  - Sunfish species: mercury, Se
- Prey fish: Targeting 3 species; mercury, Se

#### Coyote Lake

Size category: small (one sampling location)

Lake type: Bass lake

Previously sampled: 2008

Target species and analyses (note: organics are not being measured):

- Sport fish: Target 3 species, one of each category
  - Bass: mercury, Se
  - Carp\* or other bottom-feeder: mercury, Se
  - Sunfish\* species: mercury, Se
- Prey fish: Targeting 3 species; mercury, Se

\* indicates species needed by OEHHA

#### Whale Rock Reservoir

Size category: small (one sampling location)

Lake type: Trout lake (steelhead)

Not previously sampled

Target species and analyses:

- Sport fish: Target 4 species, one of each category
  - Trout (steelhead): mercury, Se, PCBs (if bass or a bottom-feeder not available)
  - Bass (if possible, thought to not be present): mercury, Se, PCBs (if a bottom-feeder not available)
  - Bottom-feeder (probably sucker): mercury, Se, PCBs
  - Sunfish species: mercury, Se
- Prey fish: Targeting 3 species; mercury, Se

#### Pacheco Lake

Size category: small (one sampling location)

Lake type: Trout and bass (?)

Not previously sampled

Target species and analyses:

- Sport fish: Target 4 species, one of each category
  - Trout: mercury, Se, PCBs (if bass or a bottom-feeder not available)
  - Bass: mercury, Se, PCBs (if a bottom-feeder not available)

- Bottom-feeder: mercury, Se, PCBs
- Sunfish species: mercury, Se
- Prey fish: Targeting 3 species; mercury, Se

## Region 5

### Spaulding, Lake

Size category: small (one sampling location)

Lake type: Trout lake

Previously sampled: BOG 2008

Target species and analyses (note: organics will not be analyzed):

- Sport fish: Target 5 species
  - Brown trout#: mercury, Se
  - Rainbow trout\*: mercury, Se
  - Bass (if possible): mercury, Se
  - Bottom-feeder: mercury, Se
  - Sunfish species: mercury, Se
- Prey fish: Targeting 3 species; mercury, Se

# indicates species needed by Region 5

\* indicates species needed by OEHHHA

### Union Valley Reservoir

Size category: medium (two sampling locations)

Lake type: Trout lake

Previously sampled: BOG 2008

Target species and analyses (note: organics will not be analyzed):

- Sport fish: Target 4 species, one of each category
  - Rainbow trout#: mercury, Se
  - Smallmouth bass# (if possible): mercury, Se
  - Bottom-feeder: mercury, Se
  - Sunfish species: mercury, Se
- Prey fish: Targeting 3 species; mercury, Se

# indicates species needed by Region 5

### Little Grass Valley Reservoir

Size category: medium (two sampling locations)

Lake type: Trout lake

Previously sampled: BOG 2008

Target species and analyses (note: organics will not be analyzed):

- Sport fish: Target 5 species
  - Brown trout#: mercury, Se
  - Rainbow trout\*: mercury, Se
  - Bass (if possible): mercury, Se
  - Bullhead\* or other bottom-feeder: mercury, Se
  - Sunfish species: mercury, Se
- Prey fish: Targeting 3 species; mercury, Se

# indicates species needed by Region 5

\* indicates species needed by OEHHHA

### **Sly Creek Reservoir**

Size category: small (one sampling location)

Lake type: Trout lake

Not previously sampled

Target species and analyses:

- Sport fish: Target 4 species, one of each category
  - Brown trout#: mercury, Se, PCBs (if bass or a bottom-feeder not available)
  - Bass (if possible): mercury, Se, PCBs (if a bottom-feeder not available)
  - Bottom-feeder: mercury, Se, PCBs
  - Sunfish species: mercury, Se
- Prey fish: Targeting 3 species; mercury, Se

# indicates species needed by Region 5

### **Wishon Reservoir**

Size category: small (one sampling location)

Lake type: Trout lake

Previously sampled: BOG 2007

Target species and analyses (note: organics will not be analyzed):

- Sport fish: Target 4 species, one of each category
  - Brown trout#: mercury, Se
  - Bass (if possible): mercury, Se
  - Bottom-feeder: mercury, Se
  - Sunfish species: mercury, Se
- Prey fish: Targeting 3 species; mercury, Se

# indicates species needed by Region 5

\* indicates species needed by OEHHHA

### **Bethany Reservoir**

Size category: small (one sampling location)

Lake type: Bass lake

Not previously sampled

Target species and analyses:

- Sport fish: Target 3 species, one of each category
  - Bass: mercury, Se, PCBs (if a bottom-feeder not available)
  - Bottom-feeder: mercury, Se, PCBs
  - Sunfish species: mercury, Se
- Prey fish: Targeting 3 species; mercury, Se

### **Rancho Seco Lake**

Size category: small (one sampling location)

Lake type: Bass lake

**Not previously sampled**

**Target species and analyses:**

- Sport fish: Target 3 species, one of each category
  - Bass: mercury, Se, PCBs (if a bottom-feeder not available)
  - Bottom-feeder: mercury, Se, PCBs
  - Sunfish species: mercury, Se
- Prey fish: Targeting 3 species; mercury, Se

**Lake Clementine**

Size category: small (one sampling location)

Lake type: Bass and trout lake

Not previously sampled

**Target species and analyses:**

- Sport fish: Target 4 species, one of each category
  - Rainbow or brown trout: mercury, Se, PCBs (if bass or a bottom-feeder not available)
  - Bass (if possible): mercury, Se, PCBs (if a bottom-feeder not available)
  - Bottom-feeder: mercury, Se, PCBs
  - Sunfish species: mercury, Se
- Prey fish: Targeting 3 species; mercury, Se

## Region 6

### Red Lake - Alpine County

Size category: small (one sampling location)

Lake type: Trout lake

Not previously sampled

Target species and analyses:

- Sport fish: Target 4 species, one of each category
  - Trout: mercury, Se, PCBs (if bass or a bottom-feeder not available)
  - Bass (if possible): mercury, Se, PCBs (if a bottom-feeder not available)
  - Bottom-feeder: mercury, Se, PCBs
  - Sunfish species: mercury, Se

### Diaz Lake - Lone Pine

Size category: small (one sampling location)

Lake type: Bass lake

Not previously sampled

Target species and analyses:

- Sport fish: Target 3 species, one of each category
  - Bass: mercury, Se, PCBs (if a bottom-feeder not available)
  - Bottom-feeder: mercury, Se, PCBs
  - Sunfish species: mercury, Se
- Prey fish: Targeting 3 species; mercury, Se

### Hesperia Lake - Hesperia

Size category: small (one sampling location)

Lake type: Bass lake

Not previously sampled

Target species and analyses:

- Sport fish: Target 3 species, one of each category
  - Bass: mercury, Se, PCBs (if a bottom-feeder not available)
  - Bottom-feeder: mercury, Se, PCBs
  - Sunfish species: mercury, Se
- Prey fish: Targeting 3 species; mercury, Se

### Lower Echo Lake - El Dorado County

Size category: small (one sampling location)

Lake type: Trout lake

Not previously sampled

Target species and analyses:

- Sport fish: Target 4 species, one of each category
  - Trout: mercury, Se, PCBs (if bass or a bottom-feeder not available)
  - Bass (if possible): mercury, Se, PCBs (if a bottom-feeder not available)
  - Bottom-feeder: mercury, Se, PCBs
  - Sunfish species: mercury, Se

## Region 7

### Salton Sea

Size category: very large (four sampling locations, if possible). Sites recommended by Region7: USGS 2, 3,5,9 & 10

Lake type: Tilapia lake

Previously sampled: BOG 2007

Target species and analyses (note: organics will not be analyzed):

- Sport fish: Target 3 species
  - Tilapia: mercury, Se
  - Whatever else is there: mercury, Se
- Prey fish: Targeting 3 species; mercury, Se

### Finney Lake

Size category: small (one sampling location)

Lake type: Bass lake

Previously sampled: 2014

Target species and analyses:

- Sport fish: Target 3 species, one of each category
  - Largemouth bass#: mercury, Se, OC pesticides (if a bottom-feeder not available)
  - Bottom-feeder: mercury, Se, OC pesticides
  - Black crappie#: mercury, Se
- Prey fish: Targeting 3 species; mercury, Se

# indicates species needed by Region 7

\* indicates species needed by OEHHA

### Squaw Lake

Size category: small (one sampling location)

Lake type: Bass lake

Previously sampled: 2014

Target species and analyses (note: organics will not be analyzed):

- Sport fish: Target 3 species, one of each category
  - Bass: mercury, Se
  - Bottom-feeder (R7 and OEHHA looking for 5 flathead cats, or 7 channel cats, or 5 carp): mercury, Se
  - Sunfish: mercury, Se
- Prey fish: Targeting 3 species; mercury, Se

### Taylor Lake

Size category: small (one sampling location)

Lake type: Bass lake

Previously sampled: 2014

Target species and analyses (note: organics will not be analyzed):

- Sport fish: Target 3 species, one of each category

- Bass: mercury, Se
- Brown bullhead#\*: mercury, Se
- Bluegill or redear#\*: mercury, Se
- Prey fish: Targeting 3 species; mercury, Se

# indicates species needed by Region 7

\* indicates species needed by OEHHA

## Region 8

### Big Bear Lake

Size category: large (three sampling locations)

Lake type: Bass lake

Previously sampled: 2007

Target species and analyses:

- Sport fish: Target 3 species, one of each category
  - Bass: mercury, Se, PCBs & OC pesticides (if a bottom-feeder not available)
  - Bottom-feeder: mercury, Se, PCBs & OC pesticides
  - Sunfish: mercury, Se
- Prey fish: Targeting 3 species; mercury, Se

### Irvine Lake

Size category: small (one sampling location)

Lake type: Bass lake

Previously sampled: 2007

Target species and analyses:

- Sport fish: Target 3 species, one of each category
  - Bass: mercury, Se, PCBs (if a bottom-feeder not available)
  - Carp\*: mercury, Se, PCBs
  - Sunfish\*: mercury, Se
- Prey fish: Targeting 3 species; mercury, Se

\* indicates species needed by OEHHA

### Lake Hemet

Size category: small (one sampling location)

Lake type: Trout lake

Previously sampled: BOG 2008

Target species and analyses (note: organics will not be analyzed):

- Sport fish: Target 4 species, one of each category
  - Rainbow trout\*: mercury, Se
  - Bass (if possible): mercury, Se
  - Bottom-feeder: mercury, Se
  - Sunfish species: mercury, Se
- Prey fish: Targeting 3 species; mercury, Se

\* indicates species needed by OEHHA

### Perris Reservoir

Size category: small (one sampling location)

Lake type: Bass lake

Previously sampled: 2007

Target species and analyses:

- Sport fish: Target 3 species, one of each category
  - Bass: mercury, Se, PCBs (if a bottom-feeder not available)
  - Bottom feeder\*: mercury, Se, PCBs
  - Sunfish: mercury, Se, PCBs\*
- Prey fish: Targeting 3 species; mercury, Se

\* indicates needed by OEHHA

## Region 9

### Diamond Valley Lake

Size category: large (three sampling locations)

Lake type: Bass lake

Not previously sampled

Target species and analyses:

- Sport fish: Target 4 species
  - Striped bass: mercury, Se, PCBs & OC pesticides (if a bottom-feeder and largemouth bass not available)
  - Largemouth bass: mercury, Se, PCBs & OC pesticides (if a bottom-feeder not available)
  - Bottom-feeder: mercury, Se, PCBs & OC pesticides
  - Sunfish species: mercury, Se
- Prey fish: Targeting 3 species; mercury, Se

### Lake Murray (Murray Reservoir)

Size category: small (one sampling location)

Lake type: Bass lake

Not previously sampled

Target species and analyses:

- Sport fish: Target 3 species, one of each category
  - Bass: mercury, Se, PCBs & OC pesticides (if a bottom-feeder not available)
  - Bottom-feeder: mercury, Se, PCBs & OC pesticides
  - Sunfish species: mercury, Se
- Prey fish: Targeting 3 species; mercury, Se

### Dixon Lake

Size category: small (one sampling location)

Lake type: Bass lake

Previously sampled: 2014

Target species and analyses (note: organics will not be analyzed):

- Sport fish: Target 3 species, one of each category
  - Bass: mercury, Se
  - Channel catfish\*: mercury, Se
  - Sunfish\*: mercury, Se
- Prey fish: Targeting 3 species; mercury, Se

\* indicates species needed by OEHA

### Appendix III. MPSL-DFG SOPs

<b>MPSL-DFG EPA Modifications and Laboratory Procedures</b>			
Page	Procedure/Equipment	SOP Number	Revision Date
A	Sample Container Preparation for Organics and Trace Metals, Including Mercury and Methylmercury	MPSL-101	July 2012
B	Sampling Marine and Freshwater Bivalves, Fish and Crabs for Trace Metal and Synthetic Organic Analysis	MPSL-102a Tis Collection	Mar 2007
C	Sample Receipt and Check-In	MPSL-104 Receipt and Check-in	Feb 2006
D	Protocol for Tissue Sample Preparation	MPSL-105 Tissue Preparation	July 2012
E	Modifications to EPA 3052		

## Appendix III A. MPSL-101 Sample Container Preparation for Organics and Trace Metals, Including Mercury and Methylmercury

Method # MPSL-101:  
Date: 10 July 2012  
Page 1 of 18

### Method # MPSL-101

#### SAMPLE CONTAINER PREPARATION FOR ORGANICS AND TRACE METALS, INCLUDING MERCURY AND METHYLMERCURY

##### 1.0 Scope and Application

- 1.1 This procedure describes the preparation of sample containers for the determination of synthetic organics and metals including but not limited to: aluminum (Al), arsenic (As), cadmium (Cd), chromium (Cr), copper (Cu), lead (Pb), manganese (Mn), mercury (Hg), nickel (Ni), selenium (Se), silver (Ag) and zinc (Zn) in tissue, sediment and water.

##### 2.0 Summary of Method

- 2.1 Teflon, polyethylene, glass containers, and collection implements are detergent and acid cleaned prior to contact with tissue, sediment or water samples. Pre-cleaned containers may be purchased from the manufacturer in some instances.

##### 3.0 Interferences

- 3.1 Special care must be used in selecting the acid(s) used for cleaning. Only reagent grade, or better, acids should be used. Prior to use, all acids should be checked for contamination.
- 3.2 If samples are to be analyzed for mercury, only Teflon or glass/quartz containers with Teflon-lined caps may be used. Use of other plastics, especially linear polyethylene, will result in Hg contamination through gas-phase diffusion through the container walls.
- 3.3 Colored plastics should be avoided, as they sometimes contain metal compounds as dyes (i.e., cadmium sulfide for yellow, ferric oxide for brown, etc.).

##### 4.0 Apparatus and Materials

- 4.1 Crew Wipers: Fisher Scientific Part # 06-666-12
- 4.2 Disposable Filter Units, 250 mL: Nalge Nunc Inc. Part # 157-0045
- 4.3 Garbage Bag, clear 30 gallon
- 4.4 Glass Bottle Class 100 Amber, 4 L: I-Chem Part # 145-4000
- 4.5 Glass Bottle Class 200 Environmentally Cleaned, 250 mL: I-Chem Part # 229-0250
- 4.6 Glass Bottle Trace Clean, 250 mL: VWR Part # 15900-130

Method # MPSSL-101:  
Date: 10 July 2012  
Page 2 of 18

- 4.7 Glass Jar Class 100, 125 mL: I-Chem Part # 120-0125 (for use only when class 200 or 300 are not available)
- 4.8 Glass Jar Class 100, 500 mL: I-Chem Part # 121-0500 (for use only when class 200 or 300 are not available)
- 4.9 Glass Jar Class 200 Environmentally Cleaned, 125 mL: I-Chem Part # 220-0125
- 4.10 Glass Jar Class 200 Environmentally Cleaned, 500 mL: I-Chem Part # 221-0500
- 4.11 Glass Jar Class 300 Environmentally Cleaned, 125 mL: I-Chem Part # 320-0125
- 4.12 Glass Jar Class 300 Environmentally Cleaned, 500 mL: I-Chem Part # 321-0500
- 4.13 Heavy Duty Aluminum Foil
- 4.14 Homogenization Jar: Büchi Analytical Part # 26441
- 4.15 Immersion Heater: VWR Part # 33897-208
- 4.16 Lab Coats
- 4.17 Non-metal Scrub Brush
- 4.18 Non-metal Bottle Brush
- 4.19 Nylon Cable Ties, 7/16" wide x 7" long
- 4.20 Masterflex C-flex Tubing: ColeParmer Part # 06424-24
- 4.21 Plastic Knife
- 4.22 Polyethylene Bin, 63 L
- 4.23 Polyethylene Bin with Lid, 14.5"x10.5"x3.25": Cole Parmer Part # 06013-80
- 4.24 Polyethylene Bucket with Lid, medium: ColeParmer Part # 63530-12 and 63530-53
- 4.25 Polyethylene Bucket with Lid, small: ColeParmer Part # 63530-08 and 63530-52
- 4.26 Polyethylene Caps, 38mm-430: VWR Part # 16219-122
- 4.27 Polyethylene Gloves: VWR Part # 32915-166, 32915-188, and 32915-202
- 4.28 Polyethylene (HDPE) Bottle, 30 mL: Nalgene-Nunc, Inc. Part # 2089-0001

- 4.29 Polyethylene (HDPE) Bottle, 60 mL: Nalgene-Nunc, Inc. Part # 2089-0002
- 4.30 Polyethylene (HDPE) Jar, 30 mL: Nalgene-Nunc, Inc. Part # 2118-0001
- 4.31 Polyethylene (HDPE) Jar, 125 mL: Nalgene-Nunc, Inc. Part # 2118-0004
- 4.32 Polyethylene Scoop: VWR Part # 56920-400
- 4.33 Polypropylene Centrifuge Tubes, 15 mL: Fisher Scientific Part # 05-521
- 4.34 Polypropylene Cutter Tool: Büchi Analytical Part #24225
- 4.35 Polypropylene Diaphragm Seal: Büchi Analytical Part # 26900
- 4.36 Polypropylene "Snap Seal" Containers, 45 mL: Corning Part # 1730 2C
- 4.37 Polypropylene Spacer: Büchi Analytical Part # 26909
- 4.38 Precision Wipes: Fisher Scientific Part # 19-063-099
- 4.39 Sapphire Thermowell: CEM Part # 326280
- 4.40 Shoe covers: Cellucap Franklin Part # 28033
- 4.41 Steel Cutting Blade, Bottom: Büchi Analytical Part # 26907
- 4.42 Steel Cutting Blade, Top: Büchi Analytical Part # 26908
- 4.43 Syringe, 50 ml Luer Slip Norm-Ject: Air-Tite Part # A50
- 4.44 Teflon Centrifuge Tube, 30 mL: Nalge Nunc, Inc. Part # 3114-0030
- 4.45 Teflon HP500+ Control Cover: CEM Part # 431255
- 4.46 Teflon HP500+ Cover: CEM Part # 431250
- 4.47 Teflon HP500+ Liner: CEM Part # 431110
- 4.48 Teflon Sheet, 0.002"x12"x1000': Laird Plastics Part # 112486
- 4.49 Teflon Tape (plumbing tape)
- 4.50 Teflon Thermowell Nut: CEM Part #325028

Method # MPSL-101:  
Date: 10 July 2012  
Page 4 of 18

- 4.51 Teflon Tubing, 0.0625" ID 0.125" OD: ColeParmer Part # 06406-62
- 4.52 Teflon Tubing, 0.1875" ID 0.25"OD: ColeParmer Part # 06406-66
- 4.53 Teflon Vial with cap, 60 mL: Savillex Part # 0202
- 4.54 Teflon Vial with cap, 180 mL: Savillex Part # 0103L-2-2- 1/8"
- 4.55 Teflon Wash Bottle, 500 mL
- 4.56 Teflon Vent Nut: CEM Part # 431313
- 4.57 Titanium Cutter Screw: Büchi Analytical Part # 34376
- 4.58 Titanium Cutting Blade, Bottom: Büchi Analytical Part # 34307 DISCONTINUED
- 4.59 Titanium Cutting Blade, Top: Büchi Analytical Part # 34306 DISCONTINUED
- 4.60 Titanium Displacement Disc: Büchi Analytical Part # 26471
- 4.61 Ventilation Hood
- 4.62 Zipper-closure Polyethylene Bags, 4milx4"x6": Packaging Store Part # z140406redline
- 4.63 Zipper-closure Polyethylene Bags, 4milx6"x8": Packaging Store Part # z140608redline
- 4.64 Zipper-closure Polyethylene Bags, 4milx9"x12": Packaging Store Part # z1400912redline
- 4.65 Zipper-closure Polyethylene Bags, 4milx12"x15": Packaging Store Part # z1401215redline
- 4.66 Zipper-closure Polyethylene Bags, 4milx13"x18": Packaging Store Part # z1401318redline

## 5.0 Reagents

Reagent grade chemicals shall be used in all cleaning procedures. Unless otherwise indicated, it is intended that all reagents shall conform to the specification of the Committee on Analytical Reagents of the American Chemical Society, where such specifications are available. Other grades may be used, provided it is first ascertained that the reagent is of sufficiently high purity to permit its use without lessening the accuracy of the determination.

- 5.1 Tap water (Tap)
- 5.2 Deionized water (DI)

Method # MPSTL-101:  
Date: 10 July 2012  
Page 5 of 18

- 5.3 Type II Water (MilliQ): Use for the preparation of all reagents and as dilution water. (reference ASTM D1193 for more on Type II water)
- 5.4 All-purpose Cleaner, 409™
- 5.5 Hydrochloric Acid (HCl), BAKER ANALYZED, 36.5-38.0% (12N): VWR Part # JT9535-3
- 5.6 Hydrochloric Acid (HCl), BAKER ANALYZED, 6N: VWR Part # JT5619-3
- 5.7 Hydrochloric Acid (HCl), 6N (50%): prepared by adding 1 part Baker 12N HCl to 1 part MilliQ
- 5.8 Hydrochloric Acid (HCl), 4N (33%): prepared by adding 1 part Baker 12N HCl to 2 parts MilliQ
- 5.9 Hydrochloric Acid (HCl), 1.2N (10%): prepared by adding 1 part Baker 12N HCl to 9 parts MilliQ
- 5.10 Hydrochloric Acid (HCl), 0.06N (0.5%): prepared by adding 1 part Baker 12N HCl to 99.5 parts MilliQ
- 5.11 Methanol: VWR Part # JT9263-3
- 5.12 Micro Detergent: ColeParmer Part # 18100-20
- 5.13 Nitric Acid (HNO<sub>3</sub>), concentrated redistilled: Seastar Chemicals Part # BA-01
- 5.14 Nitric Acid (HNO<sub>3</sub>), BAKER INSTRA-ANALYZED\*, 69.0-70.0% (15N): VWR Part # JT9598-34
- 5.15 Nitric Acid (HNO<sub>3</sub>), 7.5N (50%): prepared by adding 1 part Baker HNO<sub>3</sub> to 1 part MilliQ
- 5.16 Nitric Acid (HNO<sub>3</sub>), 6%: prepared by adding 1 part Seastar HNO<sub>3</sub> to 16.67 parts MilliQ
- 5.17 Nitric Acid (HNO<sub>3</sub>), 1%: prepared by adding 1 part Seastar HNO<sub>3</sub> to 99 part MilliQ
- 5.18 Petroleum Ether: VWR Part # JT9265-3

## 6.0 Sample Collection, Preservation and Handling

- 6.1 All samples must be collected using a sampling plan that addresses the considerations discussed in each analytical procedure.
- 6.2 All samples shall be collected and analyzed in a manner consistent with the sampling and analytical sections of this QA/QC document (MPSTL QAP Appendix E).

## 7.0 Procedures

All chemicals must be handled appropriately according to the Moss Landing Marine Laboratories Health and Safety Plan. Rinsings must be neutralized to pH 5-10 prior to disposal through the sewer system.

Two forms of acid baths are used throughout these procedures: Cold Bath and Hot Bath. All acid baths must be lidded and secondarily contained. Allow hot acid to cool completely before removing cleaned equipment.

A cold bath may be created in any clean polyethylene container of appropriate size. A hot bath is created using a clean polyethylene bucket and lid, two 63 L polyethylene bins and an immersion heater. The two bins are put together, the outer serving as secondary containment. The acid filled bucket is placed inside the inner bin and water is added to surround the bucket, creating a water bath. The immersion heater is placed outside the acid bucket, but within the water bath. The immersion heater MUST be set in a Teflon cap or other heat resistant item of appropriate size to disperse the heat source and eliminate melting of the two outer bins.

### 7.1 Trace Metal (including, but not limited to: Al, As, Cd, Cr, Cu, Pb, Mn, Hg, Ni, Se, Ag, Zn) Sample Containers

#### 7.1.1 Carboy

7.1.1.1 Fill completely with dilute Micro/Tap solution and soak for three days.

7.1.1.2 Rinse three times in Tap and three times in DI.

7.1.1.3 Fill completely with 50% HCl and soak for three days.

7.1.1.4 Remove acid and rinse three to five times in MilliQ.

7.1.1.5 Fill with 10% HNO<sub>3</sub> and soak for three days.

7.1.1.6 Remove acid and rinse three to five times in MilliQ.

7.1.1.7 If carboy is to be used immediately, fill with MilliQ and soak for 3 days. Collect solution in cleaned Trace Metal and Mercury water sample containers and test for contaminants.

7.1.1.8 If carboy is to be stored, fill with 0.5% HCl. Double bag in new garbage bags. Label the outer bag with "Acid Cleaned" and the date of completion.

#### 7.1.2 Carboy Spigots and Tubing

Method # MPSL-101:  
Date: 10 July 2012  
Page 7 of 18

- 7.1.2.1 Soak in dilute Micro/Tap solution overnight.
  - 7.1.2.2 Rinse three to five times in Tap and DI, making sure to work the spigot valve to rinse all surfaces.
  - 7.1.2.3 Submerge in 4N HCl cold bath for three days.
  - 7.1.2.4 Rinse three to five times in MilliQ, making sure to work the spigot valve to rinse all surfaces.
  - 7.1.2.5 Dry completely on crew wipers, then bag in new appropriately sized zipper-closure polyethylene bags. Label outer bag "Acid Cleaned" along with the date of completion.
- 7.1.3 Syringes for Field Filtration (not for Hg use)
- 7.1.3.1 Pull plungers out of syringes and place the outer tube in a 10% HCl bath. Swirl to ensure ink removal.
  - 7.1.3.2 Once ink is completely gone, rinse three times with each Tap and DI.
  - 7.1.3.3 Submerge all syringe parts in 4N HCl cold bath for three days.
  - 7.1.3.4 Rinse three to five times with MilliQ.
  - 7.1.3.5 Allow to completely dry on clean Crew Wipers.
  - 7.1.3.6 Reassemble dry syringes and double bag in new appropriately sized zipper-closure polyethylene bags. Label outer bag "Acid Cleaned" along with the date of completion and the number of syringes within.
- 7.1.4 Polyethylene Water Containers (not for Hg use)
- 7.1.4.1 Fill each new 60 mL bottle with a dilute Micro/Tap solution. Place in a clean dissection bin and soak for one day.
  - 7.1.4.2 Rinse three times in Tap, followed by three rinses in DI.
  - 7.1.4.3 Fill each bottle with 50% HCl, soak for three days. (Note: HCl may only be used up to 6 times before it must be appropriately discarded.)
  - 7.1.4.4 Pour out HCl and rinse each bottle and lid three to five times in MilliQ.
  - 7.1.4.5 Fill each bottle with 1% Seastar HNO<sub>3</sub>, cap. Allow outside of bottle to dry.

Method # MPSL-101:  
Date: 10 July 2012  
Page 8 of 18

7.1.4.6 Double bag each bottle in new appropriately sized zipper-closure polyethylene bags. Label each outer bag with the date.

#### 7.1.5 Polyethylene Tissue Dissection Containers

7.1.5.1 Fill each new 60 mL or 125 mL jar with a dilute Micro/Tap solution. Place in a clean dissection bin and soak for one day.

7.1.5.2 Rinse three times in tap water, followed by three rinses in DI.

7.1.5.3 Fill each jar with 10% HCl, soak for three days. (Note: HCl may only be used up to 6 times before it must be appropriately discarded.)

7.1.5.4 Pour out HCl and rinse each jar and lid three times in MilliQ.

7.1.5.5 Fill with MilliQ and soak for three days.

7.1.5.6 Remove MilliQ and place cleaned jars in a dissection bin lined with clean crew wipers to dry.

7.1.5.7 Once completely dry, pair lids and jars and place in a new appropriately sized zipper-closure polyethylene bag. Label bag "Acid Cleaned" along with the date of completion.

#### 7.1.6 Polyethylene Scoops

7.1.6.1 (Performed by field crew) Thoroughly scrub new and used scoops in dilute Micro/Tap to ensure no residue remains in nicks and scratches. If soil cannot be completely removed, discard scoop.

7.1.6.2 (Performed by field crew) Rinse three times in Tap. Dry.

7.1.6.3 (In the lab) Submerge in 4N HCl cold bath for 3 days.

7.1.6.4 Rinse three to five times with MilliQ.

7.1.6.5 Let dry completely and double bag in new appropriately sized zipper-closure polyethylene bags. Label each outer bag with the date and number of scoops within.

#### 7.1.7 Polypropylene Knives for Aliquoting

7.1.7.1 Scrub knives in dilute Mirco/Tap solution.

7.1.7.2 Rinse three times with Tap, followed by three rinses in DI.

Method # MPSSL-101:  
Date: 10 July 2012  
Page 9 of 18

7.1.7.3 Allow to completely dry on Precision Wipes. Roll in Precision Wipes, then place in new appropriately sized zipper-closure polyethylene bags. Label outer bag with "Micro Clean" and the date of completion.

#### 7.1.8 Teflon Digestion Vessel and Lids

7.1.8.1 Using a soft, sponge-like bottle brush, scrub each vessel and lid with a dilute Micro/Tap solution.

7.1.8.2 Rinse three times with Tap, followed by three rinses with DI.

7.1.8.3 Submerge in 6% Seastar HNO<sub>3</sub> bath, heated for a minimum of 8 hours in a hotbath.

7.1.8.4 Rinse three to five times in MilliQ.

7.1.8.5 Place on new Crew Wipers under fume hood to dry.

7.1.8.6 Once completely dry, place in clean appropriately sized zipper-closure polyethylene bag. Label bag with the date of completion. (Note: You may use bags that have formerly contained clean digestion vessels or lids.)

#### 7.1.9 Polyethylene Digestate Bottles

7.1.9.1 Fill each new 30 mL bottle with a dilute Micro/Tap solution. Place in a clean dissection bin and soak for one day.

7.1.9.2 Rinse three times in tap water, followed by three rinses in DI.

7.1.9.3 Fill each cup with 50% HCl, soak for three days. (Note: HCl may only be used up to 6 times before it must be appropriately discarded.)

7.1.9.4 Pour out HCl and rinse each bottle and lid three times in MilliQ.

7.1.9.5 Fill with MilliQ and soak for three days.

7.1.9.6 Remove MilliQ and place cleaned bottles and lids upside-down in a dissection bin lined with clean crew wipers to dry.

7.1.9.7 Once completely dry, pair lids and bottles and place in a new appropriately sized zipper-closure polyethylene bag. Label bag "Acid Cleaned" along with the date of completion.

#### 7.1.10 Polypropylene Centrifuge Tubes, 15 mL ("ICP Tubes")

7.1.10.1 Soak tubes in dilute Micro/Tap bath for three days.

Method # MPSSL-101:  
Date: 10 July 2012  
Page 10 of 18

- 7.1.10.2 Rinse three times in Tap, followed by three rinses in DI.
- 7.1.10.3 Submerge tubes and caps in 50% HCl cold bath for three days.
- 7.1.10.4 Rinse each tube and cap three times with MilliQ.
- 7.1.10.5 Place tubes and caps on clean crew wipers to dry.
- 7.1.10.6 Once completely dry, place in a new appropriately sized zipper-closure polyethylene bag. Label bag "Acid Cleaned" along with the date of completion.

## 7.2 Mercury Only Sample Containers

### 7.2.1 Water Composite Bottles, 4L

- 7.2.1.1 Caps do not get micro cleaned.
- 7.2.1.2 Scrub the outside of each bottle with a dilute Micro/Tap solution, rinse with Tap.
- 7.2.1.3 Place a small volume of the Micro/Tap solution inside the bottle. Shake vigorously to coat all surfaces.
- 7.2.1.4 Rinse with Tap until no more suds appear.
- 7.2.1.5 Rinse three times with DI.
- 7.2.1.6 Fill each bottle with 3N HCl. Cap and let stand on counter for three days. (Note: Acid may be used for a total of six cleaning cycles.)
- 7.2.1.7 Empty bottles and rinse three to four times with MilliQ, and fill.
- 7.2.1.8 Pipette in 20 mL HCl, BAKER ANALYZED, top off with MQ, replace caps and let dry.
- 7.2.1.9 Once completely dry, double bag in new appropriately sized zipper-closure polyethylene bags. Label outer bag with the date of completion.
- 7.2.1.10 Place in original boxes, labeled with date of completion. Bag entire box in a new garbage bag.

### 7.2.2 Tubing Sets

- 7.2.2.1 Cable Ties

- 7.2.2.1.1 Soak new cable ties in dilute Micro/Tap solution for three days.
- 7.2.2.1.2 Remove and rinse three times with Tap, followed by three rinses in DI and three rinses in MilliQ.
- 7.2.2.1.3 Allow to completely dry on Crew Wipers, then place in new appropriately sized zipper-closure polyethylene bags. Label outer bag with "Micro Clean" and the date of completion.

#### 7.2.2.2 Polyethylene Caps with Holes

- 7.2.2.2.1 Drill a hole slightly smaller than 0.25 inches in the top of each new cap.
- 7.2.2.2.2 Soak in dilute Micro/Tap solution for three days.
- 7.2.2.2.3 Rinse three times with Tap, followed by three rinses in DI.
- 7.2.2.2.4 Soak in 4N HCl for 3 days.
- 7.2.2.2.5 Rinse three to five times in MilliQ. Let dry on Crew Wipers.
- 7.2.2.2.6 Once completely dry, place in new appropriately sized zipper-closure polyethylene bags until assembly. Label outer bag with "Acid Clean" and the date of completion.

#### 7.2.2.3 Teflon Tubing

- 7.2.2.3.1 Using clean utility shears, cut one 3 foot and one 2 foot piece of tubing for each tubing set to be made.
- 7.2.2.3.2 Soak in dilute Micro/Tap solution for 3 days, ensuring that the tube is completely filled.  
  
Note: Use Teflon tape to bind the two ends of each piece of tubing together. This will increase safety throughout the procedure.
- 7.2.2.3.3 Rinse three times in Tap, followed by three rinses in DI.
- 7.2.2.3.4 Submerge in 50% HNO<sub>3</sub> hot bath for 8 hours, ensuring that tubing is completely filled.
- 7.2.2.3.5 Rinse cooled tubing three to four times in MilliQ and let dry on clean Crew Wipers.

Note: Drying time may be decreased significantly by blowing reagent grade argon through the tubing to remove the water.

- 7.2.2.3.6 Once completely dry, place in new appropriately sized zipper-closure polyethylene bags until assembly. Label outer bag with "Acid Clean" and the date of completion.

#### 7.2.2.4 C-Flex Tubing

- 7.2.2.4.1 Using clean utility shears, cut one 2 foot and one 4 inch piece of tubing for each tubing set to be made.
- 7.2.2.4.2 Soak in dilute Micro/Tap solution for one day, ensuring that the tube is completely filled.
- 7.2.2.4.3 Rinse three times in Tap, followed by three rinses in DI.
- 7.2.2.4.4 Submerge for three days in 12N HCl under a fume hood.
- 7.2.2.4.5 Rinse three to four times in MilliQ.
- 7.2.2.4.6 Submerge for three days in 0.5% HCl under a fume hood.
- 7.2.2.4.7 Rinse three to four times in MilliQ. Let dry completely on clean Crew Wipers.

Note: Drying time may be decreased significantly by blowing reagent grade argon through the tubing to remove the water.

- 7.2.2.4.8 Once completely dry, place in new appropriately sized zipper-closure polyethylene bags until assembly. Label outer bag with "Acid Clean" and the date of completion.

#### 7.2.2.5 Tubing Set Assembly (using cleaned parts described above)

- 7.2.2.5.1 Using two cable ties, attach 2 foot Teflon tubing to 2 foot C-flex.
- 7.2.2.5.2 Next attach 4 foot Teflon to the other end of the 2 foot C-flex, again with 2 cable ties.
- 7.2.2.5.3 Add the 4 inch C-flex to the open end of the 4 foot Teflon tubing with 2 cable ties.
- 7.2.2.5.4 Put a drilled Poly cap on the open end of the 2 foot Teflon.

Method # MPLS-101:  
Date: 10 July 2012  
Page 13 of 18

7.2.2.5.5 Coil the assembled tubing set, and double bag in new appropriately sized zipper-closure polyethylene bags. Label outer bag with "Acid Clean" and the date of completion.

#### 7.2.2.6 In-Lab Mercury Filters

7.2.2.6.1 Fill upper reservoir with 10% HCl. Cap and apply vacuum.

7.2.2.6.2 Detach filter apparatus from vacuum manifold. Place finger over the valve and shake the unit to clean all surfaces of the lower reservoir.

7.2.2.6.3 Repeat two more times. Acid can be used 6 times.

7.2.2.6.4 Repeat wash three times with MilliQ. Cap and apply vacuum.

7.2.2.6.5 Discard MilliQ after each rinse.

#### 7.2.3 Water Sample Bottles, 250 mL

7.2.3.1 Rinse new bottles in DI. Place the caps only in a MilliQ bath for the duration of the bottle cleaning.

7.2.3.2 Submerge in 50% Baker HNO<sub>3</sub> hot bath for 8 hours, ensuring that each bottle is completely filled.

7.2.3.3 Rinse cooled bottles three to four times in MilliQ, then fill each with MilliQ.

7.2.3.4 Pipette in 1.25 mL 100% HCl, replace caps and let dry completely.

7.2.3.5 Double bag in new appropriately sized zipper-closure polyethylene bags. Label outer bag with the date of completion.

7.2.3.6 Place in original boxes, labeled with date of completion.

#### 7.2.4 Polypropylene "Snap Seal" Containers, 45 mL ("Trikona Tubes")

7.2.4.1 Rinse new tubes in dilute Micro/Tap.

7.2.4.2 Rinse three times in Tap, followed by three times in DI.

7.2.4.3 Submerge in 50% HNO<sub>3</sub> hot bath for 8 hours, ensuring that each tube is completely filled.

7.2.4.4 Rinse cooled tubes three to four times in MilliQ.

7.2.4.5 Let dry completely on clean Crew Wipers.

7.2.4.6 Place dry tubes in new appropriately sized zipper-closure polyethylene bags. Label outer bag with "Acid Clean" and the date of completion.

### 7.3 Methylmercury Only Sample Containers

#### 7.3.1 Teflon Digestion or Distillation Vials

7.3.1.1 Scrub vials with 409™ to remove any organic residue. It may be necessary to also soak the vials in dilute Micro/Tap for 3 days.

7.3.1.2 Rinse three times in DI.

7.3.1.3 Submerge in 50% HCl bath. Heat overnight, or soak for 3 days in cold bath.

7.3.1.4 Rinse three to five times in MilliQ; dry completely on clean crew wipers.

7.3.1.5 Place dry tubes in new appropriately sized zipper-closure polyethylene bags. Label outer bag with "Acid Clean" and the date of completion.

#### 7.3.2 Teflon Distillation Caps and Tubing

7.3.2.1 Scrub caps and tubing with 409™ to remove any organic residue.

7.3.2.2 Rinse three times in DI.

7.3.2.3 Submerge in 10% HCl hotbath overnight. Use a Teflon squirt bottle to fill the tubing with acid.

7.3.2.4 Rinse three to five times in MilliQ; dry completely on clean crew wipers.

Note: Hang tubing over a clean hook against crew wipers to speed drying time.

7.3.2.5 Place in new appropriately sized zipper-closure polyethylene bags. Label outer bag with "Acid Clean" and the date of completion.

### 7.4 Organic Sample Containers

#### 7.4.1 Aluminum Foil Sheets

7.4.1.1 Using a clean scalpel, cut a 4 foot long section of aluminum foil.

7.4.1.2 Fold in half, with dull side out. (The bright side may contain oils from the manufacturing process.)

Method # MPSSL-101:  
Date: 10 July 2012  
Page 15 of 18

7.4.1.3 Under a fume hood, rinse both exposed sides of the folded foil three times with Petroleum Ether. Make sure all exposed surfaces are well rinsed.

7.4.1.4 Set against a clean surface under the fume hood to dry.

7.4.1.5 Once completely dry, fold the sheet in quarters, ensuring the un-rinsed shiny side does not come in contact with the now cleaned dull side.

7.4.1.6 Place into a new appropriately sized zipper-closure polyethylene bag. Label bag "PE Cleaned" along with the date of completion and the number of sheets within.

#### 7.4.2 Dissection Jars (125mL, 500mL Glass Jars)

NOTE: Clean 100 series jars as follows below. 200 and 300 series jars may be used as is from the manufacturer, with a clean Teflon square (section 7.5.2) over the threads.

7.4.2.1 Using a clean scalpel, cut three inch squares from a sheet of new Teflon.

7.4.2.2 Fit Teflon square to the jar and lid, ensuring that the threads are completely covered and no leaks will occur.

7.4.2.3 Under a fume hood, rinse each jar and lid three times with Petroleum Ether by putting a small amount in the jar, sealing it and then shaking the jar to coat all sides.

Note: It is easiest to clean four jars simultaneously. Use each volume of PE once in each of the jars; repeat. After cleaning the fourth jar, discard PE into evaporation bin under the hood, or into designated solvent waste container.

7.4.2.4 Set jars aside in the hood to dry.

7.4.2.5 When completely dry, match the lids to the jar and place back in the original box. Label box "PE Cleaned" along with the date of completion.

#### 7.5 "Split" Sample Containers (for metals and organics)

##### 7.5.1 Teflon sheets

7.5.1.1 Cut new Teflon to desired length (1 or 2 feet long depending on application)

7.5.1.2 Submerge crumpled sheets in a 10% Micro/Tap bath overnight.

7.5.1.3 Remove sheets from micro bath and flatten. Rinse all surfaces of each sheet three times in tap water, followed by three rinses in deionized water.

Method # MPSSL-101:  
Date: 10 July 2012  
Page 16 of 18

- 7.5.1.4 Crumple rinsed sheets and submerge in 10% HCl in a hot bath; heat at least 8 hours.
- 7.5.1.5 Remove sheets from acid bath and flatten. Rinse all surfaces of each sheet five times in MilliQ.
- 7.5.1.6 Layer rinsed Teflon sheets on new Crew Wipers, with new Precision Wipes between each sheet. Cover stack with new Precision Wipes. Let dry.
- 7.5.1.7 Once the sheets are completely dry, rinse each surface three times with Petroleum Ether.
- 7.5.1.8 Place on clean Crew Wipers and Precision Wipes, as before, under hood and let dry.
- 7.5.1.9 Once the sheets are completely dry, fold sheets and place into a new appropriately sized zipper-closure polyethylene bag. Label bag "PE Cleaned" along with the date of completion and the number of sheets within.

#### 7.5.2 Teflon Squares for Dissection Jars

- 7.5.2.1 Using a cutting board and scalpel, cut Teflon sheet into 3-inch squares.
- 7.5.2.2 Soak in 6% Seastar HNO<sub>3</sub> coldbath overnight.
- 7.5.2.3 Rinse three times with MilliQ.
- 7.5.2.4 Rinse three times with Methanol, followed by three rinses with Petroleum Ether.
- 7.5.2.5 Lay on clean crew wipers to dry.
- 7.5.2.6 Once the squares are completely dry, place into a new appropriately sized zipper-closure polyethylene bag. Label bag "PE Cleaned" along with the date of completion.

#### 7.5.3 Dissection Jars (125mL, 500mL Glass Jars)

NOTE: Clean 100 series jars as follows below. 200 and 300 series jars may be used as is from the manufacturer, with a clean Teflon square (section 7.5.2) over the threads.

- 7.5.3.1 Using a clean scalpel, cut three inch squares from a sheet of new Teflon.
- 7.5.3.2 Fit Teflon square to the jar and lid, ensuring that the threads are completely covered and no leaks will occur.
- 7.5.3.3 Under a fume hood, rinse each jar and lid three times with 6% HNO<sub>3</sub> by putting a small amount in the jar, sealing it and then shaking the jar to coat all sides.

Note: It is easiest to clean four jars simultaneously. Use each volume of each chemical once in each of the jars; repeat. After cleaning the fourth jar, discard into the appropriate evaporation bin under the hood or into designated waste container.

7.5.3.4 Rinse each jar three times in MilliQ.

7.5.3.5 Rinse each jar three times in Methanol, let dry completely.

7.5.3.6 Rinse each jar three times in Petroleum Ether; set aside in the hood to dry.

7.5.3.7 When completely dry, match the lids to the jar and place back in the original box. Label box "Split Cleaned" along with the date of completion.

7.5.4 Homogenization Parts (Büchi) including glass, polypropylene, titanium and stainless steel

7.5.4.1 Scrub with dilute Micro/Tap, followed by 3 rinses with DI.

7.5.4.2 Rinse 3 times with 6% Seastar HNO<sub>3</sub> using a Teflon squirt bottle.

7.5.4.3 Rinse 3 times with MilliQ.

7.5.4.4 Rinse 3 times with Methanol, followed by 3 times with Petroleum Ether.

7.5.4.5 Allow parts to dry completely before assembly and homogenization.

## 8.0 Analytical Procedure

8.1 Tissue Preparation procedures can be found in Method # MPSSL-105.

8.2 Trace Metal and Mercury Only digestion procedures can be found in EPA 3052, modified, and Method # MPSSL-106, respectively.

8.3 Trace Metals are analyzed with ICP-MS according to EPA 200.8.

8.4 Mercury samples are analyzed by FIMS according to Method # MPSSL-103 or by DMA and EPA 7473.

8.5 Methylmercury tissue samples are extracted and analyzed according to Method # MPSSL-109.

8.6 Methylmercury sediment samples are extracted and analyzed according to Method # MPSSL-110 and modified EPA 1630, respectively.

## 9.0 Quality Control

9.1 See individual methods.

Method # MPSTL-101:  
Date: 10 July 2012  
Page 18 of 18

## **10.0 Method Performance**

- 10.1 System blanks are performed on Mercury Sample 250 mL and 4 L bottles and tubing sets to guarantee thorough cleaning.
- 10.2 Carboys are tested for all metals after cleaning.

## **11.0 References**



## Appendix III B. MPSL-102a Sampling Marine and Freshwater Bivalves, Fish and Crabs for Trace Metal and Synthetic Organic Analysis

Method # MPSL-102a  
Date: 14 March 2007  
Page 1 of 11

### Method # MPSL-102a

#### SAMPLING MARINE AND FRESHWATER BIVALVES, FISH AND CRABS FOR TRACE METAL AND SYNTHETIC ORGANIC ANALYSIS

##### 1.0 Scope and Application

- 1.1 The following procedures describe techniques of sampling marine mussels and crabs, freshwater clams, marine and freshwater fish for trace metal (TM) and synthetic organic (SO) analyses.

##### 2.0 Summary of Method

- 2.1 Collect mussels, clams, crabs, or fish. Mussels or clams to be transplanted are placed in polypropylene mesh bags and deployed. Mussels and clams to be analyzed for metals are double-bagged in plastic zipper-closure bags. Bivalves to be analyzed for organics are wrapped in PE cleaned aluminum foil prior to placement in the zipper-closure bags. Fish are wrapped whole or proportioned where necessary in cleaned Teflon sheets or aluminum foil and subsequently placed into zipper-closure bags. Crabs for TM and/or SO are double-bagged in plastic zipper-closure bags.
- 2.2 Each sample should be labeled with Date, Station Name, and any other information available to help identify the sample once in the lab.
- 2.3 After collection, samples are transported back to the laboratory in coolers with ice or dry ice. If ice is used, care must be taken to ensure that ice melt does not come into direct contact with samples.

##### 3.0 Interferences

- 3.1 In the field, sources of contamination include sampling gear, grease from ship winches or cables, ship and truck engine exhaust, dust, and ice used for cooling. Efforts should be made to minimize handling and to avoid sources of contamination.
- 3.2 Solvents, reagents, glassware, and other sample processing hardware may yield artifacts and/or elevated baselines, causing inaccurate analytical results. All materials should be demonstrated to be free from interferences under the conditions of the analysis by running method blanks initially and with each sample lot.
- 3.3 Polypropylene and polyethylene surfaces are a potential source of contamination for SO specimens and should not be used whenever possible.

##### 4.0 Apparatus and Materials

Method # MPSSL-102a  
Date: 14 March 2007  
Page 2 of 11

Procedures for equipment preparation can be found in Method # MPSSL-101.

- 4.1 Anchor Chains
- 4.2 Backpack Shocker (electro-fishing)
- 4.3 Boats (electro-fishing and/or for setting nets)
- 4.4 Bone Saw
- 4.5 Camera, digital
- 4.6 Cast Nets (10' and 12')
- 4.7 Data Sheets (see MPSSL QAP Appendix E for example)
- 4.8 Daypacks
- 4.9 Depth Finder
- 4.10 Dip Nets
- 4.11 Dry Ice or Ice
- 4.12 Gill Nets (various sizes)
- 4.13 GPS
- 4.14 Heavy Duty Aluminum Foil, prepared
- 4.15 Heavy Duty plastic bags, Clear 30 gallon
- 4.16 Inflatable Buoy
- 4.17 Labels, gummed waterproof: Diversified Biotech Part #: LCRY-1258
- 4.18 Nylon Cable Ties, 7/16" wide x 7" long
- 4.19 Other (minnow traps, set lines, throw nets, etc)
- 4.20 Otter Trawl (various widths as appropriate)
- 4.21 Permanent Marking Pen

Method # MPSSL-102a  
Date: 14 March 2007  
Page 3 of 11

- 4.22 Plastic bucket, 30 gallon
- 4.23 Plastic Ice Chests
- 4.24 Polyethylene Gloves: VWR Part # 32915-166, 32915-188, and 32915-202
- 4.25 Polypropylene Mesh, 76mm wide with 13mm mesh
- 4.26 Polypropylene Mesh, 50mm wide with 7mm mesh
- 4.27 Polypropylene Line, 16mm
- 4.28 Rods and Reels
- 4.29 Screw in Earth Anchor, 4-6" diameter
- 4.30 Scuba Gear
- 4.31 Seines (various size mesh and lengths as appropriate)
- 4.32 Stainless Steel Dive Knives
- 4.33 Trap Nets (hoop or fyke nets)
- 4.34 Teflon Forceps
- 4.35 Teflon Sheet, prepared
- 4.36 Teflon Wash Bottle, 500 mL
- 4.37 Wading Gear
- 4.38 Zipper-closure Polyethylene Bags, 4milx13"x18": Packaging Store Part # z1401318redline

## **5.0 Reagents**

- 5.1 Tap water (Tap)
- 5.2 Deionized water (DI)
- 5.3 Type II water (ASTM D1193): Use Type II water, also known as MilliQ, for the preparation of all reagents and as dilution water.
- 5.4 Micro Detergent: ColeParmer Part # 18100-20

5.5 Methanol: VWR Part # JT9263-3

5.6 Petroleum Ether: VWR Part # JT9265-3

## 6.0 Sample Collection, Preservation and Handling

- 6.1 All sampling equipment will be made of non-contaminating materials and will be inspected prior to entering the field. Nets will be inspected for holes and repaired prior to being used. Boats (including the electroshocking boat) will be visually checked for safety equipment and damage prior to being taken into the field for sample collection.
- 6.2 To avoid cross-contamination, all equipment used in sample collection should be thoroughly cleaned before each sample is processed. Ideally, instruments are made of a material that can be easily cleaned (e.g. Stainless steel, anodized aluminum, or borosilicate glass). Before the next sample is processed, instruments should be washed with a detergent solution, rinsed with ambient water, rinsed with a high-purity solvent (methanol or petroleum ether), and finally rinsed with MilliQ. Waste detergent and solvent solutions must be collected and taken back to the laboratory.
- 6.3 Samples are handled with polyethylene-gloved hands only. The samples should be sealed in appropriate containers immediately.
- 6.4 Mussels and clams to be analyzed for metals are double-bagged in zipper-closure bags. Bivalves to be analyzed for organics are wrapped in prepared aluminum foil prior to placement in zipper-closure bags.
- 6.5 Fish are wrapped in part or whole in prepared Teflon sheets and subsequently placed into zipper-closure bags.
- 6.6 Crabs analyzed for metals and/or organics are double-bagged in plastic zipper-closure bags.
- 6.7 Data is recorded for each site samples are transplanted to or collected from. Data includes, but is not limited to station name, sample identification number, site location (GPS), date collected or transplanted, collectors names, water depth, photo number, ocean/atmospheric conditions (if appropriate), description of site, and drawing if necessary.
- 6.8 A chain of custody form (MPSL QAP Appendix E) will accompany all samples that are brought to the lab. All samples that are processed in the lab MUST be checked in according to Method # MPSL-104.
- 6.9 Samples are maintained at -20°C and extracted or digested as soon as possible.

## 7.0 Procedure

### 7.1 Sample collection - mussels and clams

- 7.1.1 The mussels to be transplanted (*Mytilus californianus*) are collected from Trinidad Head (Humboldt Bay Intensive Survey), Montana de Oro (Diablo Canyon Intensive Survey), and Bodega Head (all other statewide transplants). The freshwater clam (*Corbicula fluminea*) source is Lake Isabella or the Sacramento River. Analyze mussel and clam samples for background contaminants prior to transplanting.
- 7.1.2 Polyethylene gloves are worn while prying mussels off rocks with dive knives. Note: polyethylene gloves should always be worn when handling samples. Mussels of 55mm to 65mm in length are recommended. Fifty mussels are collected for each TM and each SO sample.
- 7.1.3 Collected mussels are carried out of collection site in zipper-closure bags placed in cleaned nylon daypacks. For the collection of resident samples where only one or two samples are being collected the mussels are double bagged directly into a labeled zipper-closure bag. Samples for SO are wrapped first in prepared aluminum foil.
- 7.1.4 Clams (*Corbicula fluminea*) measuring 20 to 30mm are collected by dragging the clam dredge along the bottom of the lake or river. The clams are poured out of the dredge into a 30 gallon plastic bag. Clams can also be collected by gloved hands in shallow waters and placed in labeled zipper-closure bags. 25-200 clams are collected depending on availability and necessity for analyses.
- 7.1.5 Data is recorded for each site samples are collected from. Data includes, but is not limited to station name, date collected, collectors names, water depth, GPS readings, photo, ocean/atmospheric conditions (if appropriate), description of site, and drawing if necessary.

### 7.2 Transplanted sample deployment

- 7.2.1 With polyethylene gloves, fifty transplant mussels are placed in each 76mm X 13mm polypropylene mesh bag. Each bag represents one TM or one SO sample. A knot is tied at each end of mesh bag and reinforced with a cable tie. On one end another cable tie is placed under the cable tie which will be used to secure the bag to the line for transplant deployment. The mussels in the mesh bag are divided into three groups of approximately equal size and sectioned with two more cable ties.

Method # MPSSL-102a  
Date: 14 March 2007  
Page 6 of 11

- 7.2.2 Once bagged, the mussels are placed in a 30 gallon plastic bag and stored in a cooler (cooled with ice) for no more than 48 hours. The ice is placed in zipper-closure bags to avoid contamination.
- 7.2.3 If marine samples are held for longer than 48 hours they are placed in holding tanks with running seawater at the lab. Control samples for both SO and TM are also held in the tank.
- 7.2.4 For freshwater clams: clams (25-200) are placed in 50mm X 7mm polypropylene mesh bags using identical procedures to those used with mussels (section 7.2.1). If clams need to be stored for more than 48 hours, the mesh bags are deployed either in a clean source or in holding tanks with running freshwater at the lab until actual sample deployment.
- 7.2.5 The mussels are attached to an open water transplant system that consists of a buoy system constructed with a heavy weight anchor (about 100lbs) or screw-in earth anchor, 13mm polypropylene line, and a 30cm diameter subsurface buoy. The sample bags are attached with cable ties to the buoy line about 15 feet below the water surface. In some cases the sample is hung on suspended polypropylene lines about 15 feet below the water surface between pier pilings or other surface structures. Creosote-coated wooden piers are avoided because they are a potential source of contamination. In some cases the mussels are hung below a floating dock. In shallow waters a wooden or PVC stake is hammered into the substrate and the mussel bags are attached by cable ties to the stake.
- 7.2.6 The clams are deployed by attaching the mesh bag with cable ties to wooden or PVC stakes hammered into substrate or screw in earth anchors. The bags containing clams are typically deployed 15cm or more off the bottom. In areas of swift water, polypropylene line is also attached to the staked bags and a permanent object (piling, tree or rock).
- 7.2.7 Transplants are usually deployed for 1-4 months. Ideally mussels are transplanted in early September and retrieved in late December and early January. Clams are usually transplanted in March or April and retrieved in May or June.
- 7.2.8 Data is recorded for each site samples are transplanted to or collected from. Data includes, but is not limited to station name, date collected or transplanted, collectors names, water depth, GPS readings, photo, ocean/atmospheric conditions (if appropriate), description of site, and drawing if necessary.

### 7.3 Sample Retrieval

- 7.3.1 The transplanted or resident and control mussels analyzed for TM are double bagged in appropriately sized and labeled zipper-closure bags.

Method # MPSSL-102a  
Date: 14 March 2007  
Page 7 of 11

- 7.3.2 All mussels to be analyzed for SO are wrapped in prepared aluminum foil (Method # DFG 101). The foil packet is double bagged in appropriately sized and labeled zipper-closure bags. Note: samples should only contact the dull side of the foil.
- 7.3.3 The bags containing samples are clearly and uniquely identified using a water-proof marking pen or pre-made label. Information items include ID number, station name, depth (if from a multiple sample buoy), program identification, date of collection, species and type of analysis to be performed.
- 7.3.4 The samples are placed in non-metallic ice chests and frozen using dry ice or regular ice. (Dry ice is used when the collecting trip takes more than two days.) At the lab, samples should be stored at or below -20°C until processed.

#### 7.4 Sample Collection – Fish

- 7.4.1 Fish are collected using the appropriate gear for the desired species and existing water conditions.
  - 7.4.1.1 Electro-fisher boat- The electro-fisher boat is run by a trained operator, making sure that all on board follow appropriate safety rules. Once on site, adjustment of the voltage, amps, and pulse for the ambient water is made and recorded. The stainless steel fish well is rinsed with ambient water, drained and refilled. The shocked target fish are placed with a nylon net in the well with circulating ambient water. The nylon net is washed with a detergent and rinsed with ambient water prior to use. Electro-fishing will continue until the appropriate number and size of fish are collected.
  - 7.4.1.2 Backpack electro-fisher- The backpack shocker is operated by a trained person, making sure that all others helping follow appropriate safety rules. The backpack shocker is used in freshwater areas where an electro-fisher boat can not access. Once on site, adjustment of the voltage, amps, and pulse for the ambient water is made and recorded. The shocked target fish are captured with a nylon net and placed in a 30 gallon plastic bag. The nylon net is washed with a detergent and rinsed with ambient water prior to use. Electro-fishing will continue until the appropriate number and size of fish are collected.
  - 7.4.1.3 Fyke or hoop net- Six-36 inch diameter hoops connected with 1 inch square mesh net is used to collect fish, primarily catfish. The net is placed parallel to shore with the open hoop end facing downstream. The net is placed in areas of slow moving water. A partially opened can of cat food is placed in the upstream end of the net. Between 2-6 nets are placed at a site overnight. Upon retrieval a grappling hook is used to pull up the downstream anchor. The hoops and net are pulled together and placed on a 30

Method # MPSSL-102a  
Date: 14 March 2007  
Page 8 of 11

gallon plastic bag in the boat. With polyethylene gloves the desired fish are placed in a 30 gallon plastic bag and kept in an ice chest with ice until the appropriate number and size of fish are collected.

- 7.4.1.4 Otter-trawl- A 14 foot otter trawl with 24 inch wooden doors or a 20 foot otter trawl with 30 inch doors and 80 feet of line is towed behind a boat for water depths less than 25 feet. For water depths greater than 25 feet another 80 feet of line is added to capture fish on or near the substrate. Fifteen minute tows at 2-3 knots speed are made. The beginning and ending times are noted on data sheets. The trawl is pulled over the side of the boat to avoid engine exhaust. The captured fish are emptied into a 30 gallon plastic bag for sorting. Desired fish are placed with polyethylene gloves into another 30 gallon plastic bag and kept in an ice chest with ice.
- 7.4.1.5 Gill nets- A 100 yard monofilament gill net of the appropriate mesh size for the desired fish is set out over the bow of the boat parallel to shore. The net is retrieved after being set for 1-4 hours. The boat engine is turned off and the net is pulled over the side or bow of the boat. The net is retrieved starting from the down-current end. If the current is too strong to pull in by hand, then the boat is slowly motored forward and the net is pulled over the bow. Before the net is brought into the boat, the fish are picked out of the net and placed in a 30 gallon plastic bag and kept in an ice chest with ice.
- 7.4.1.6 Beach seines- In areas of shallow water, beach seines of the appropriate length, height, and mesh size are used. One sampler in a wetsuit or waders pulls the beach seine out from shore. The weighted side of the seine must drag on the bottom while the float side is on the surface. The offshore sampler pulls the seine out as far as necessary and then pulls the seine parallel to shore and then back to shore, forming a half circle. Another sampler is holding the other end on shore while this is occurring. When the offshore sampler reaches shore the two samplers come together with the seine. The seine is pulled onto shore making sure the weighted side drags the bottom. When the seine is completely pulled onshore, the target fish are collected with polyethylene gloves and placed in a 30 gallon plastic bag and kept in an ice chest with ice. The beach seine is rinsed off in the ambient water and placed in the rinsed 30 gallon plastic bucket.
- 7.4.1.7 Cast net- A 10 or 12 foot cast net is used to collect fish off a pier, boat, or shallow water. The cast net is rinsed in ambient water prior to use and stored in a covered plastic bucket. The target fish are sampled with polyethylene gloves and placed in a 30 gallon plastic bag and kept in an ice chest with ice.
- 7.4.1.8 Hook and line- Fish are caught off a pier, boat, or shore by hook and line. Hooked fish are taken off with polyethylene gloves and placed in a Ziploc™ bag or a 30 gallon plastic bag and kept in an ice chest with ice.

Method # MPSSL-102a  
Date: 14 March 2007  
Page 9 of 11

- 7.4.1.9 Spear fishing- Certain species of fish are captured more easily by SCUBA divers spearing the fish. Only appropriately trained divers following the dive safety program guidelines are used for this method of collection. Generally, fish in the kelp beds are more easily captured by spearing. The fish are shot in the head area to prevent the fillets from being damaged or contaminated. Spear tips are washed with a detergent and rinsed with ambient water prior to use.
- 7.4.2 As a general rule, five fish of medium size or three fish of larger size are collected as composites for analysis. The smallest fish length cannot be any smaller than 75% of the largest fish length. Five fish usually provides sufficient quantities of tissue for the dissection of 150 grams of fish flesh for organic and inorganic analysis. The medium size is more desirable to enable similar samples to be collected in succeeding collections.
- 7.4.3 When only small fish are available, sufficient numbers are collected to provide 150 grams of fish flesh for analysis. If the fish are too small to excise flesh, the whole fish, minus the head, tail, and guts are analyzed as composites.
- 7.4.4 Species of fish collected are chosen for their importance as indicator species, availability or the type of analysis desired. For example, livers are generally analyzed for heavy metals. Fish without well-defined livers, such as carp or goldfish, are not collected when heavy metal analyses are desired.
- 7.4.5 Fish collected, too large to fit in clean bags (>500 mm) are initially dissected in the field. At the dock, the fish are laid out on a clean plastic bag and a large cross section from behind the pectoral fins to the gut is cut with a cleaned bone saw or meat cleaver. The bone saw is cleaned (micro, DI, methanol) between fish and a new plastic bag is used. The internal organs are not cut into, to prevent contamination. For bat rays, a section of the wing is cut and saved. These sections are wrapped in prepared Teflon sheets, double bagged and packed in dry ice before transfer to the freezer. During lab dissection, a subsection of the cross section is removed, discarding any tissue exposed by field dissection.
- 7.4.6 Field data (MPSSL QAP Appendix E) recorded include, but are not limited to site name, sample identification number, site location (GPS), date of collection, time of collection, names of collectors, method of collection, type of sample, water depth, water and atmospheric conditions, fish total lengths (fork lengths where appropriate), photo number and a note of other fish caught.
- 7.4.7 The fish are then wrapped in aluminum foil or Teflon sheets if thylates are analyzed. The wrapped fish are then double-bagged in zipper-closure bags with the inner bag labeled.

Method # MPSSL-102a  
Date: 14 March 2007  
Page 10 of 11

The fish are put on dry ice and transported to the laboratory where they are kept frozen until they are processed for chemical analysis.

#### 7.5 Sample Collection- Crabs

- 7.5.1 Crab/lobster traps- Polyethylene traps are baited to collect crabs or lobsters. Traps are left for 1-2 hours. The crabs are placed in a zipper-closure bag or a 30 gallon plastic bag and kept in an ice chest with ice.

#### 8.0 Analytical Procedure

- 8.1 Tissue Preparation procedures can be found in Method # MPSSL-105.
- 8.2 Trace Metal and Mercury Only digestion procedures can be found in EPA 3052, modified, and Method # MPSSL-106, respectively.
- 8.3 Trace Metals are analyzed with ICP-MS according to EPA 200.8.
- 8.4 Mercury samples are analyzed by FIMS according to Method # MPSSL-103 or by DMA and EPA 7473.
- 8.5 Methylmercury tissue samples are extracted and analyzed according to Method # MPSSL-109.

#### 9.0 Quality Control

- 9.1 Field Replicates: project specific requirements are referenced for field replication.
- 9.2 A record of sample transport, receipt and storage is maintained and available for easy reference.

#### 10.0 References

- 10.1 Flegal, R.A. 1982. In: Wastes in the Ocean, Vol VI: Near Shore Waste Disposal. B.H. Ketchum (ed.). John Wiley and Sons Inc. Publishers, New York, 1982.
- 10.2 Goldberg, E.D., ed. 1980. The International Mussel Watch. National Academy of Sciences Publ., Washington, D.C.
- 10.3 Gordon, R.M., G.A. Knauer and J.H. Martin. 1980a. *Mytilus californianus* as a bioindicator of trace metal pollution: variability and statistical considerations. Mar. Poll. Bull. 9:195-198.

Method # MP5L-102a  
Date: 14 March 2007  
Page 11 of 11

- 10.4 Hayes, S. P. and P. T. Phillips. 1986. California State Mussel Watch: Marine water quality monitoring program 1984-85. State Water Resources Control Board Water Quality Monitoring Report No. 86-3WQ.
- 10.5 EPA. 1995. Guidance for Assessing Chemical Contaminant Data for Use in Fish Advisories. Volume 1: Fish Sampling and Analysis. EPA 823-R-95-007.

## Appendix III C. MPSL-104 Sample Receipt and Check-In

Method #: MPSL-104  
Date: February 2006  
Page 1 of 4

### Method # MPSL-104

#### SAMPLE RECEIPT AND CHECK-IN

##### 1.0 Scope and Application

- 1.1 This method describes the cataloging and handling of samples as they arrive at the laboratory for processing and analysis

##### 2.0 Summary of Method

- 2.1 A record of sample transport, receipt and storage is maintained and available for easy reference.
- 2.2 Each sample is assigned a unique lab identification number. The number is recorded in a logbook as well as on the sample itself.
- 2.3 Each sample is preserved according to the applicable analytical method and is stored accordingly. The preservation and storage is recorded in the logbook.

##### 3.0 Interferences

- 3.1 Not Applicable

##### 4.0 Apparatus and Materials

- 4.1 Bound logbook with numbered pages
- 4.2 Permanent Pen
- 4.3 Permanent Marker (i.e. Sharpie)
- 4.4 Digital Probe thermometer: Fisher Part # 15-077-32
- 4.5 3-Ring Binder
- 4.6 Copy Machine

##### 5.0 Reagents

- 5.1 Not Applicable

##### 6.0 Sample Collection

Method #: MPSL-104  
Date: February 2006  
Page 2 of 4

- 6.1 Water Samples are collected according to EPA 1669, modified, according to analytical or project specific methods.
- 6.2 Tissue samples are collected according to Method MPSL-102a, or according to analytical or project specific methods.
- 6.3 Sediment samples are collected according to Method MPSL-102b, or according to analytical or project specific methods.

## 7.0 Procedure

- 7.1 Samples accompanied by a Chain of Custody Record (COC) are delivered to the laboratory from the field crew. Samples may be hand delivered or shipped via FedEx or another overnight shipping service provided the samples maintain the appropriate temperatures during shipment.
- 7.2 Cooler temperature is measured prior to the removal of any sample. The probe of the digital thermometer is placed amongst the samples. Temperature is allowed to equilibrate prior to recording on the COC and logbook. It is noted when samples were delivered by the field crew and placed directly into the refrigerator or freezer, rendering a cooler temperature unobtainable.
- 7.3 The COC is reviewed for preservation and requested handling of the samples.
- 7.4 A new page in the log book is used for each COC. Entries MUST include the following:
  - 7.4.1 Date of entry.
  - 7.4.2 Project Name and Number
  - 7.4.3 Unique 9-digit Lab Number
    - 7.4.3.1 The first four digits are the year in which the sample was received.
    - 7.4.3.2 The second four digits are sequential numbers beginning with 0001. Each successive sample receives the next number.
    - 7.4.3.3 A single letter is appended to each Lab Number to indicate the matrix type (-w = water, -s = sediment, -t = tissue, -c = chlorophyll a).
  - 7.4.4 Date and time (if provided) of sample collection. Time shall be recorded using a 24-hour clock.
  - 7.4.5 Sample Identification; station information taken directly from the COC

Method #: MPSSL-104  
Date: February 2006  
Page 3 of 4

- 7.4.6 Analyte of suite of analytes requested for each sample.
- 7.4.7 At the end of the entry, the following are recorded:
  - 7.4.7.1 Type- Specify the matrix of the samples. List all that apply.
  - 7.4.7.2 Preservation/Storage- List for each matrix/analyte combination.
  - 7.4.7.3 From- the name of the person last in possession of the samples (signed the COC)
  - 7.4.7.4 Received by- the name of the person at the lab who first received the samples
  - 7.4.7.5 Date and Time of sample receipt as well as cooler temperature upon arrival.
  - 7.4.7.6 Checked by- the name of the person that verified the contents of the cooler with the COC and assigned the lab numbers.
  - 7.4.7.7 Any comments pertaining to the samples (special instructions, anomalies, etc.).
- 7.5 Water samples are preserved according to the specific analytical methods (EPA 1630, 1631E and 1638). Preserved samples are given to the analysts along with copies of the COC and log-book entry.
- 7.6 Tissue, sediment and chlorophyll a samples are stored in a walk-in freezer at -20°C until dissection and/or digestion can occur.
- 7.7 At least one copy is made of each COC and log book entry. One copy MUST be kept in the COC binder. Other copies may be stored with the samples themselves, or given to the analyst.
- 7.8 All entries are entered and maintained in a MS Access database.

## **8.0 Analytical Procedure**

- 8.1 Trace Metal tissue and sediment digestions are performed according to EPA 3052M, modified.
- 8.2 Mercury Only tissue and sediment digestion procedures can be found in Method # MPSSL-106 and Method # MPSSL-107, respectively.
- 8.3 Trace Metals are analyzed with ICP-MS according to EPA 200.8 (tissues and sediments) and EPA 1638, modified (waters).
- 8.4 Mercury tissue and sediment samples are analyzed by FIMS according to Method # MPSSL-103 or by DMA and EPA 7473.

Method #: MPLS-104  
Date: February 2006  
Page 4 of 4

- 8.5 Mercury water samples are analyzed according to EPA 1631E, modified.
- 8.6 Methylmercury tissue samples are extracted and analyzed according to SOP-CALFED.D03.
- 8.7 Methylmercury water samples are analyzed according to EPA 1630, modified.

**9.0 Quality Control**

- 9.1 MS Access database does not allow duplicate Lab Numbers
- 9.2 Each COC, along with a copy of the pertinent portion of the logbook, is retained for reference.

## Appendix III D. MPSL-105 Laboratory Preparation of Trace Metal and Synthetic Organic Samples of Tissues in Marine and Freshwater Bivalves and Fish

Method #: MPSL-105  
Date: 10 July 2012  
Page 1 of 10

### Method # MPSL-105

#### LABORATORY PREPARATION OF TRACE METAL AND SYNTHETIC ORGANIC SAMPLES OF TISSUES IN MARINE AND FRESHWATER BIVALVES AND FISH

##### 1.0 Scope and Application

- 1.1 The following procedures describe techniques for the laboratory preparation of marine and freshwater tissues for trace metal (TM) and synthetic organic (SO) analysis.

##### 2.0 Summary of Method

- 2.1 Laboratory processing is carried out under “clean room” conditions, with a positive pressure filtered air supply, non-contaminating laboratory surfaces, and a supply of deionized (DI) and Type II water (MilliQ).
- 2.2 All tools that come in contact with the sample are washed with Micro and water, rinsed with tap water and then DI. It is important to use tap water because DI alone will not remove Micro detergent.
- 2.3 Dissection information (initial jar weight, total weight, and tissue weight) is recorded in individual log books as well as project specific dissection sheets. Other information specific to each type of dissection is also recorded.
- 2.4 Personnel MUST wear polyethylene gloves at all times when handling samples and prepared dissection equipment.
- 2.5 All samples are dissected and placed in prepared containers appropriate for the analyses requested.
- 2.6 Any anomalies (parasites, injuries, etc) are recorded in all cases.
- 2.7 Dissected samples are homogenized to obtain a uniform sample. Aliquots of homogenate are distributed according to analyte and are acid-digested or solvent-extracted.

##### 3.0 Interferences

- 3.1 Solvents, reagents, glassware, and other sample processing hardware may yield artifacts and/or elevated baselines, causing inaccurate analytical results. All materials should be demonstrated to be free from interferences under the conditions of the analysis by running method blanks initially and with each sample lot.
- 3.2 Polypropylene and polyethylene surfaces are a potential source of contamination for SO specimens and should not be used whenever possible.

- 3.3 TO MINIMIZE CONTAMINATION, ALL SAMPLES ARE PROCESSED UNDER "CLEAN ROOM" CONDITIONS. Criteria enumerated in Flegal (1982) are recommended. Shoe covers and lab coats are worn in the laboratory to minimize transport of contaminants into the laboratory. The trace metal laboratory has no metallic surfaces, with bench tops, sinks and fume hoods constructed of acid resistant plastic to avoid metal contamination. A filtered air supply (class 100) which provides a positive pressure clean air environment is an important feature for reducing contamination from particulates.

#### 4.0 Apparatus and Materials

Procedures for equipment preparation can be found in Method # MPSSL-101.

- 4.1 Brinkmann Polytron model PT 10-35
- 4.2 Büchi Mixer B-400
- 4.3 Disposable Scalpel, #10: Fisher Scientific Part # 08-927-5A
- 4.4 Ear Protection
- 4.5 Fillet knives
- 4.6 Glass Jar Class 100, 500 mL, prepared
- 4.7 Glass Jar Class 200, 500 mL, prepared
- 4.8 Glass Jar Class 300, 500 mL, prepared
- 4.9 Glass Jar Class 100, 125 mL, prepared
- 4.10 Glass Jar Class 200, 125 mL, prepared
- 4.11 Glass Jar Class 300, 125 mL, prepared
- 4.12 Glass Jar Class 200, 60 mL: I-Chem Part # 220-0060
- 4.13 Glass Jar Class 300, 60 mL: I-Chem Part # 320-0060
- 4.14 Heavy Duty Beakers, 1000 mL
- 4.15 Heavy Duty Beakers, 400 mL
- 4.16 Garbage Bags, Clear 30 gallon

Method #: MP5L-105  
Date: 10 July 2012  
Page 3 of 10

- 4.17 Lab Coats
- 4.18 Plastic Knives, prepared
- 4.19 Polyethylene Gloves: VWR Part # 32915-166, 32915-188, and 32915-202
- 4.20 Polyethylene (HDPE) jar, 30 mL, prepared
- 4.21 Polyethylene (HDPE) jar, 125 mL, prepared
- 4.22 Shoe Covers: Cellucap Franklin Part # 28033
- 4.23 Teflon Forceps, prepared
- 4.24 Titanium Bars
- 4.25 Titanium Generator: Brinkmann Part # PTA 20

## 5.0 Reagents

- 5.1 Tap water (Tap)
- 5.2 Deionized water (DI)
- 5.3 Type II water (ASTM D1193): Use Type II water, also known as MilliQ, for the preparation of all reagents and as dilution water.
- 5.4 Micro Detergent: ColeParmer Part # 18100-20
- 5.5 Methanol: VWR Part # JT9263-3
- 5.6 Petroleum Ether: VWR Part # JT9265-3
- 5.7 Hydrochloric Acid (HCl), BAKER ANALYZED, 36.5-38.0%: VWR Part # JT9535-3
- 5.8 Hydrochloric Acid (HCl), 50%: prepared by adding 1 part Baker HCl to 1 part MilliQ
- 5.9 Nitric Acid (HNO<sub>3</sub>), BAKER INSTRA-ANALYZED\*\*, 69.0-70.0%: VWR Part # JT9598-34
- 5.10 Nitric Acid (HNO<sub>3</sub>), 50%: prepared by adding 1 part Baker HNO<sub>3</sub> to 1 part MilliQ

## 6.0 Sample Collection, Preservation and Handling

- 6.1 Samples should be collected according to Method # MSPL-102a, # MPSSL-102b, and EPA 1669, modified.
- 6.2 All dissection equipment and containers must be prepared according to Method # MPSSL-101.
- 6.3 Tissue dissections should be carried out by or under the supervision of a competent biologist. Each organism should be rinsed free of dirt with deionized water and handled with prepared stainless steel, quartz, or Teflon instruments. Fish or other samples processed as “whole body” must only come in contact with MilliQ water to reduce contamination. The SO specimens should come in contact with prepared glass, aluminum foil or Teflon surfaces only (Method # MPSSL-101).
- 6.4 Samples should be maintained at -20°C and extracted or digested as soon as possible.

## 7.0 Procedure

### 7.1 Dissection

#### 7.1.1 Bivalve Dissection

7.1.1.1 For both TM and SO: Frozen mussels are thawed, removed from the bags, and cleaned of epiphytic organisms, byssal threads and debris under running DI. Dissections are conducted on cleaned Teflon cutting boards.

7.1.1.2 The gametogenic condition of each sample is recorded in the logbook and dissection sheet a “ripe”, “partial” or “not ripe”.

7.1.1.3 For both TM and SO: The first 15 shell lengths are recorded. Lengths are measured across the longest part of each shell.

#### 7.1.1.4 TM Bivalve Dissection

7.1.1.4.1 Forty-five mussels are dissected per sample. These are divided into 3 groups of 15. Each group of 15 creates A, B, and C replicates. If there are fewer than 45 mussels the mussels are divided into three equal samples. The total number of mussels in each jar is recorded.

7.1.1.4.2 The adductor muscle is severed with a scalpel and the shell is pried open with the plastic end of the scalpel. The gonads are then excised. The weight of the gonads from the first 15 mussels is recorded. These and all subsequent gonads can then be thrown away.

Note: Gonads are not removed from clams.

7.1.1.4.3 The remainder of the soft part is removed from shell and placed in a pre-weighed, prepared polypropylene 125mL jar. The final sample weight for each jar is recorded. All jars must be properly labeled on both the lid and the jar itself.

#### 7.1.1.5 SO Bivalve Dissection

7.1.1.5.1 The adductor muscle is severed and the shell is pried open with clean titanium blade. The entire body, including gonads, is placed in a pre-weighed, prepared glass jar. All forty-five individuals are placed in the same jar. All jars must be properly labeled on both the lid and the jar itself.

#### 7.1.1.6 "Split" Bivalve Dissection

7.1.1.6.1 Samples are dissected as TM samples with the following exceptions:

7.1.1.6.1.1 All gonads from each sample of 45 mussels are excised and retained in prepared 125mL glass jar. The combined weight of all 45 gonads is recorded.

7.1.1.6.1.2 The remainder of the tissue from each of the 3 replicates is dissected into prepared 125mL glass jars.

#### 7.1.2 Fish Dissection

7.1.2.1 Large fish requiring dissection are partially thawed, then washed with DI water. It may be necessary to rub more vigorously in order to remove mucous. Place the rinsed fish in a clean, foil lined bin.

7.1.2.2 Total fish length and fork length are measured to the nearest millimeter. The body is then placed on a clean foil sheet on the balance and weighed. All lengths and weights are recorded.

7.1.2.3 Scaly fish (Large Mouth Bass, Perch, etc.) are de-scaled from the tail to the operculum above the lateral line with the titanium rod, and are dissected "skin-on". The skin is removed from scale-less fish in the same section as above, and the fish are dissected "skin-off". (EPA Guidelines) If the contract requires aging, 10 scales are taken from the appropriate region of the fish and placed in labeled coin envelopes for later age determination.

7.1.2.4 Fish are filleted to expose the flesh. It is important to maintain the cleanliness of the tissue for analysis, therefore any "skin-off" flesh that has been in direct contact with the skin or with instruments in contact with skin must be eliminated from the sample.

Method #: MPSSL-105  
Date: 10 July 2012  
Page 6 of 10

Trim the edges of the fillet with a clean scalpel or fillet knife to remove this contaminated tissue.

- 7.1.2.5 Fillets are cut into small pieces, less than 1 square inch for homogenization purposes.
- 7.1.2.6 Record the individual fillet weight. For composite samples, equal fillet weights are taken from each individual.
- 7.1.2.7 As much flesh as possible should be removed for each sample to meet the requirements for each analysis as well as have tissue retained for archive. Generally, 150-200g total sample weight is ideal.
- 7.1.2.8 If possible, the sex of each individual is determined and recorded.
- 7.1.2.9 If the contract requires liver analysis, the livers are removed from the predator species by opening the body cavity with the incision scalpel. The liver is freed by cutting with a fresh dissection scalpel and removed with a clean forceps. The livers are rinsed with MilliQ and placed in a prepared, pre-weighed sample jar. Individual liver weights recorded.
- 7.1.2.10 At this time vertebrae may be taken from ictalurids for aging. The first unfused vertebra is removed and placed in a 25mL beaker, covered with water and placed in the refrigerator until the flesh has broken down enough to be cleaned away. The vertebrae are placed in a coin envelope and may later be used for age determination.
- 7.1.2.11 Sections of fish, rather than whole body, may be delivered from the sampling crew. The lengths and weight will have already been recorded by the collection team. Tissue is dissected as before, however any exposed flesh must be eliminated from the sample.
- 7.1.2.12 Whole-bodied fish are thawed under MilliQ. They may be stripped of mucous by using prepared forceps. At no time may the whole body fish touch any unclean surface or instrument.
- 7.1.2.13 Total length, fork length and weight are recorded.
- 7.1.2.14 The body is cut into pieces smaller than 1 square inch for homogenization. It may be necessary to use a prepared bone saw to cut through larger vertebrae.
- 7.1.2.15 All samples are refrozen after dissection and maintained at -20°C until homogenization and/or analysis. It may be possible to homogenize fish samples immediately after dissection, but is not necessary.

## 7.2 Homogenization

### 7.2.1 TM Bivalve Homogenization

7.2.1.1 Samples are homogenized in the original sample jar using the Polytron and Titanium Generator.

Note: Ear Protection should be worn when operating any homogenizer.

7.2.1.2 Clean the generator by running it in a dilute Micro/Tap Solution. Rinse by running the generator in a 2 separate Tap baths, followed by 3 DI baths and 1 MQ bath. Allow to dry. Extra rinses may be necessary if tissue can be seen in any of the baths. If tissue is found in the DI or MQ baths, begin again with Tap water.

7.2.1.3 The tissue is homogenized to a paste-like consistency. No chunks of clearly defined tissue should be left in homogenate.

Note: operate the Polytron at the lowest speed possible to avoid heating the sample or splattering tissue.

7.2.1.4 The generator is cleaned with new solution baths between reps as well as between stations.

7.2.1.5 Samples must be refrozen at -20°C until acid-digestion can take place.

### 7.2.2 SO Bivalve Homogenization

7.2.2.1 Samples are homogenized in the original sample jar using the Polytron and either Stainless Steel or Titanium Generator.

Note: Ear Protection should be worn when operating any homogenizer.

7.2.2.2 Clean the generator by running it in 3 separate DI baths and 1 MQ bath, followed by 3 wash bottle rinses each with Methanol and Petroleum Ether. Extra rinses may be necessary if tissue can be seen in any of the baths. If tissue is found in the MQ bath, begin again with DI water.

7.2.2.3 The tissue is homogenized to a paste-like consistency. No chunks of clearly defined tissue should be left in homogenate.

Note: operate the Polytron at the lowest speed possible to avoid heating the sample or splattering tissue.

7.2.2.4 The generator is cleaned with new solution baths between stations.

7.2.2.5 Samples must be refrozen at -20°C until transfer to analytical lab and solvent extraction can occur.

### 7.2.3 "Split" Bivalve (TM and SO) Homogenization

7.2.3.1 Samples are homogenized as TM with the following exceptions:

7.2.3.1.1 The TM cleaned titanium generator is washed 3 times with 6% HNO<sub>3</sub> prior to the 3 MQ rinses, and is further rinsed 3 times each with Methanol and Petroleum Ether.

7.2.3.1.2 The retained gonads are homogenized in addition to the 3 replicates.

7.2.3.2 Homogenized samples are aliquoted for SO, ensuring enough tissue remains for TM analysis. Equal portions of body tissue are taken from each of the 3 replicates. The ratio of gonad:body weight is calculated for the entire sample, and the ratio is applied to the SO aliquot body weight to determine the amount of gonad material to add back in. Once all tissue is present in the SO sample, it is homogenized by hand with a prepared titanium rod.

### 7.2.4 Fish

7.2.4.1 Fish samples are removed from the freezer and are allowed to thaw long enough to be transferred to split-clean Büchi sample jar.

7.2.4.2 Prior to and after homogenization the blades and drive shaft of the Buchi are scrubbed with Micro, and rinsed 3 times each in tap and DL.

7.2.4.3 To TM clean the titanium blades, rinse 3 times in MilliQ.

7.2.4.4 To SO clean the steel blades, rinse 3 times in MilliQ, followed by 3 rinses each in methanol and PE. Air dry.

7.2.4.5 To split clean titanium blades, rinse 3 times in 6% HNO<sub>3</sub>, followed by 3 rinses in MilliQ. Follow up with 3 rinses each in methanol and PE. Air dry.

7.2.4.6 Assemble the homogenizer according to manufacturer specifications.

7.2.4.7 Place sample jar on tray; close and lock the homogenizer door.

7.2.4.8 Raise the sample jar into position with the on/off toggle. When the jar reaches the appropriate height, the blades will begin rotation and come in contact with the sample.

Method #: MPLS-105  
Date: 10 July 2012  
Page 9 of 10

7.2.4.9 It is important to PULSE the cutting unit in the sample by briefly releasing the toggle. This allows the entire sample to be homogenized, and not get pushed against the sides of the container, as well as keeping the friction to a minimum. It is imperative the sample not get hot.

7.2.4.10 Once the sample has fully homogenized, it may be aliquoted with a prepared titanium rod into the appropriate prepared sample containers for each analysis.

7.2.4.11 Samples are frozen at -20°C until acid-digestion or transfer to analytical lab and solvent extraction can occur.

## **8.0 Analytical Procedure**

- 8.1 Trace Metal and Mercury Only digestion procedures can be found in EPA 3052, modified, and Method # MPLS-106, respectively.
- 8.2 Trace Metals are analyzed with ICP-MS according to EPA 200.8.
- 8.3 Mercury samples are analyzed by FIMS according to Method # MPLS-103 or by DMA and EPA 7473.
- 8.4 Methylmercury tissue samples are extracted and analyzed according to Method # MPLS-109.

## **9.0 Quality Control**

- 9.1 Sample Archive: All remaining sample homogenates and extracts can be archived at -20°C for future analysis.
- 9.2 A record of sample transport, receipt and storage is maintained and available for easy reference.
- 9.3 All samples are prepared in a clean room to avoid airborne contamination.

## **10.0 Method Performance**

- 10.1 See individual analytical methods.

## **11.0 References**

- 11.1 Flegal, R.A. 1982. In: Wastes in the Ocean, Vol VI: Near Shore Waste Disposal. B.H. Ketchum (ed.). John Wiley and Sons Inc. Publishers, New York, 1982.
- 11.2 Goldberg, E.D., ed. 1980. The International Mussel Watch. National Academy of Sciences Publ., Washington, D.C.

Method #: MPSTL-105  
Date: 10 July 2012  
Page 10 of 10

- 11.3 Gordon, R.M., G.A. Knauer and J.H. Martin. 1980a. *Mytilus californianus* as a bioindicator of trace metal pollution: variability and statistical considerations. Mar. Poll. Bull. 9:195-198.
- 11.4 Hayes, S. P. and P. T. Phillips. 1986. California State Mussel Watch: Marine water quality monitoring program 1984-85. State Water Resources Control Board Water Quality Monitoring Report No. 86-3WQ

## Appendix III E. Modifications to EPA 3052

### Modification of EPA Method 3052

Autumn Bonnema, Lab Manager  
Marine Pollution Studies Lab  
7544 Sandholdt Road  
Moss Landing CA 95039  
831-771-4175

Mark Stephenson, Director  
Marine Pollution Studies Lab  
7544 Sandholdt Road  
Moss Landing CA 95039  
831-771-4177

Methods were modified from that described in EPA 3052 in order to reduce hazards to staff as well as more closely fit the requirements of the Microwave Assisted Reaction System (MARS) 5 unit.

It was determined through R&D that samples digested under the following conditions resulted in fully digested samples (modifications are listed according to section number):

- 7.2 All digestion vessels and vessel components are cleaned with hot 6% Double Distilled nitric acid for 8 hours, rinsed with reagent water and dried in a clean environment.
- 7.3.2 For tissue digestion, add 6 mL concentrated double distilled nitric acid to the vessel in a fume hood. For sediment digestion, add 5 ml concentrated double distilled nitric acid and 3 mL concentrated double distilled hydrofluoric acid to the vessel in a fume hood.
- 7.3.6 The following temperature and pressure settings are used for each matrix:
  - 15 minute ramp to 195°C and 250 psi (controlled by temperature)
  - 20 minute hold at temperature and pressureSediment samples (post boric addition):
  - 5 minute ramp to 195°C and 250 psi (controlled by temperature)
  - 15 minute hold at temperature and pressure
- 7.3.11 Transfer the sample into a pre-cleaned, pre-weighed 30 mL poly bottle. For tissues, bring the final solution weight to 20.00 ± 0.02 with reagent water. For sediments, record the solution volume.

## Appendix IV. SWAMP SOPs

<b>SWAMP IQ Procedures</b>			
Page	Procedure/Equipment	SOP Number	Revision Date
A	Verification of the Surface Water Ambient Monitoring Program Database		March 2011
B	BOG Data Validation SOP		May 2016

**Appendix IV A. SWAMP SOP Chemistry Data Verification v1.1**

## Appendix IV B. BOG Data Validation SOP

Tissue Validation Procedures  
rev. May 2016  
Page 1 of 10

### BOG Data Validation Standard Operating Procedure

#### Blank Contamination Check

Blank verification samples identify if the target analyte has contaminated field samples via lab contamination from any part of sample preparation and analysis. One method blank (laboratory derived) sample is run with each analytical batch ( $\leq 20$  samples). The method blanks will be processed through the entire analytical procedure in a manner identical to the field samples. The ideal scenario is that method blank samples are non-detects. If a field sample is contaminated from laboratory procedures and the analytical quantification of that field sample is low, then a high proportion of the field sample value could be from laboratory contamination which results in that value being uncertain and not usable. Laboratory blank contamination could result in a false positive when field sample results are low. There is less concern of blank contamination affecting a field sample if field samples are some multiple higher than the method blank result (in this case 3 times the method blank concentration).

In order to determine if field samples have been contaminated, the following data validation method is applied:

1. If there is more than 1 method blank in a batch, use the method blank with the highest concentration.
2. Second, compare the highest method blank concentration to the method blank MDL (Note: SWAMP has a method blank MQO of  $<$  Reporting Limit (RL) for all targeted analytes. If the method blank concentration is greater than the RL then corrective action needs to be taken by the lab prior to submitting data to the DMT. For the data validation exercise any quantitation of the method blank above the MDL is considered a detection and therefore the data validation exercise uses the MDL as the threshold for assessing blank contamination):
  - a. If the Method Blank concentration is less than ( $<$ ) the Method Blank MDL then there is no detection of that analyte in the blank sample. This suggests that there was no laboratory contamination of field samples and no further action for that analyte, in that batch, is required.
  - b. If the Method Blank concentration is greater than ( $>$ ) the Method Blank MDL then the method blank sample has been contaminated with the targeted analyte and there is possible contamination of associated field samples. For those cases where the method blank result is greater than the MDL, compare the field sample results to the highest Method Blank result for each batch. Be sure that the Method Blank results, MDLs, and field sample results are all in the same units and basis (wet weight or dry weight).
    - i. If the field result is less than the MDL, no further action is required. The compliance code is COM.
    - ii. If the *detected* ( $>$ MDL) field result is less than ( $<$ ) 3x highest Method Blank concentration then flag that field sample with a QACode of VRIP. This sample is considered a censored result (the blank contamination is likely too large a component of the field result to be differentiated, and may in fact show a false positive). The compliance code is REJ.
    - iii. If the field result is greater than ( $>$ ) 3x highest Method Blank, then the sample should be flagged with QACode VIP if not already IP flagged. The compliance code is QUAL.

### Accuracy check

Accuracy is the degree of agreement of a measurement with a known value and is utilized to assess the degree of closeness of field samples to their real value. Using the bull's-eye analogy (Figure 1), accuracy is the degree of closeness to the bull's-eye (which represents the true value). Over/under estimation of analytical quantification is important in this project. If the QA elements indicate overestimation of the field sample result than this could lead to false positives above particular human health consumption thresholds and potentially limit human consumption of particular sport fish species. If the QA elements indicate underestimated analytical quantification then low field sample values could falsely suggest that fish are below human health thresholds when they may actually be above the thresholds. Good accuracy in a data set increases the confidence and certainty that the field sample value is close to the true value. Accuracy is determined by such QC elements as: certified reference materials (CRM), laboratory control samples, blind spikes, matrix spikes, and performance samples. Tables 1-2 show the Measurement Quality Objectives (MQOs) for tissues.

Figure 1. Demonstration of target accuracy (black marks) to a known value (bull's-eye). The figure shows very good accuracy but poor precision.



Table 1. (Table 10, Bonnema 2016) shows BOG Measurement Quality Objectives for inorganic analytes in tissues

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 for target analyte
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
Internal Standard	Accompanying every analytical run when method appropriate	60-125% recovery

\*Unless method specifies more stringent requirements.  
 MDL = Method Detection Limit  
 RL = Reporting Limit  
 n/a = not applicable

Table 2. (Table 11, Bonnema 2016) shows BOG Measurement Quality Objectives for synthetic organic analytes in tissues

SWAMP Measurement Quality Objectives* - General		
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	75-125% recovery
Laboratory Blank	Per 20 samples or per batch, whichever is more frequent	<RL for target analytes
Reference Material	Method validation: as many as required to assess accuracy and precision of method before routine analysis of samples; routine accuracy assessment: per 20 samples or per batch (preferably blind)	70-130% of the certified 95% confidence interval stated by provider of material. If not certified then within 50-150% of reference value.
Matrix Spike	Per 20 samples or per batch, whichever is more frequent	50-150% recovery or control limits based on 3x the standard deviation of laboratory's actual method recoveries
Matrix Spike Duplicate	Per 20 samples or per batch, whichever is more frequent	50-150% 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
Surrogate or Internal Standard	As specified in method	50-150% recovery

\*Unless method specifies more stringent requirements.  
 MDL = method detection limit (to be determined according to the SWAMP QA Management Plan)  
 RL = Reporting Limit  
 n/a = not applicable

For the accuracy data validation, SWAMP follows a multiple failure rule. The possible QC elements for the accuracy check are:

CRM, Reference Material, LCS, Matrix Spike/Matrix Spike Duplicate<sup>1</sup>

Only samples in a quantitative range should be used for evaluation of accuracy, as non-quantitative results may be lucky passes or unlucky fails rather than true indications of the ability for the analysis to accurately determine concentrations

- For any of the accuracy QC samples, Expected Value must be at least 1xRL, otherwise it shouldn't be used.
- Additionally for MS/MSDs, the Matrix Spike Expected Value should be greater than or equal to 3x the Native Field Result.

**Data Validation for Accuracy:**

<sup>1</sup> Matrix Spike/Matrix Spike Duplicate, preferably, alone cannot be used to evaluate the precision and accuracy of individual samples. However, when exercising professional judgment, these QA elements should be used in conjunction with other available QC information.

If there are no valid QC elements available based on the quantitative range screening from above, then apply QACode "VQCA" to all of the related results in that batch.

For the remaining QC samples in a quantitative range, the following apply where there is more than one usable measure.

1. Following SWAMP MQOs, one QC element is allowed to be outside the MQO for accuracy (occurs when the QC element is less than or greater than the MQO target range (see Tables 1 and 2 above) but less than 2 times the MQO range (see method for determining this "2x" range in item 3 below) in a batch and still be compliant. If one QC element in a batch is outside the MQO, then the individual QC sample is given a QACode of (EUM, GBC, or GB). The compliance code for the associated field samples is COM.
2. When more than one QC element is outside of the MQO, each QC element is given a QACode (EUM, GBC, GB). The compliance code for the associated field samples is QUAL. In these cases, a QACode of "VIU" is applied to the field samples.
3. **Rejection Point:** The QACode "VRIU" is applied to the field samples when the % Recovery is more than 2 times outside the MQO target range (see Tables 1 and 2) or when the lower rejection limit is <10%, in 2 or more QC elements (CRM, Reference Material, LCS, MS/MSD). In these cases, the compliance code is changed to REJ. The QACode is applied to all field samples in the affected batch including those that are not quantifiable (flagged with ND (not detected) in ResQualCode). Below is the method for determining the upper and lower rejection limits:
  - Lower Rejection Limit =  $100 - (2 * (100 - \text{lower limit of the range}))$
  - Upper Rejection Limit =  $100 + (2 * (\text{upper limit of the range} - 100))$

As an example, the acceptable range for certified reference material for organics is percent recovery 70-130%. The lower rejection limit would be  $100 - (2 * (100 - 70)) = 40$  and the upper rejection limit would be  $100 + (2 * (130 - 100)) = 160$ . Recoveries less than 40% and greater than 160% are more than 2 times outside the MQO target Range which would result in a compliance code of REJ and a QACode of VRIU.

If there is only one usable QC sample for accuracy evaluation, the individual QC sample is flagged as appropriate, and the following applies to the batch:

4. In the case where there is only one QC element reported in the batch and the % Recovery is more than 1 time outside the MQO target range (see Tables 1 and 2) but less than 2 times the target range then the compliance code would be QUAL and a QACode VIU is applied to the field samples in that batch.
5. **Rejection Point:** In the case where there is only one QC element reported in the batch and the %Recovery was more than 2 times outside the MQO target range (see Tables 1 and 2) or when the lower rejection limit is <10%, then the compliance code would be REJ and the QACode VRIU is applied to the field samples in that batch.

Table 3 summarizes the application of QACodes for the accuracy check scenarios above.

Table 3. Accuracy Data Validation Rules – where there are more than 2 quantitative (usable) measures, A & B are the two quantitative measures with the worst performance for any given analyte

Measure A Range	Measure B Range	QA Code	Comment
>±2x range or when the lower rejection limit is <10%	>±2x range or when the lower rejection limit is <10%	VRIU	Both badly fail.
>±2x range or when the lower rejection limit is <10%	>±1x range - <±2x range	VIU	One badly, one marginally fail
>±2x range or when the lower rejection limit is <10%	Within range	None	One badly fail, remainder pass
>±2x range or when the lower rejection limit is <10%	Null	VRIU	One badly fail
>±1x range - <±2x range	>±1x range - <±2x range	VIU	Both marginally fail
>±1x range - <±2x range	Within range	None	One marginally fail, remainder pass
>±1x range - <±2x range	Null	VIU	One marginally fail
Within range	Within range	None	Both pass

**Precision check**

Precision is the degree to which repeated measurements under unchanged conditions show the same result (usually reported as a relative standard deviation [RSD] or relative percent difference [RPD]). The repeatability measure indicates the variability observed within a laboratory, over a short time, using a single operator, item of equipment, etc. These QA elements also show the reproducibility of an analytical measurement. Good precision provides confidence that the analytical process is consistently measuring the target analyte in a particular matrix.

The possible QC elements in the precision check are:

Lab duplicates, Matrix Spikes/Matrix Spike Duplicates, LCS/LCSD. See Tables 1 and 2 above for MQOs.

Similar to the case for evaluating accuracy, only results in a usable quantitative range should be used to calculate precision.

- Check for each sample (pair or set) analyzed in replicate that the average result is greater than (>) 1 times the RL. If the average result is greater than (>) 1 times the RL then include RPD or RSD in lab tests submission evaluation. Otherwise that set of sample replicates is not quantitative and thus not usable.

**Data Validation for Precision:**

If there are no valid precision QC elements available based on the quantitative range screening from above, then apply QA Code “VQCP” to all of the related results in that batch.

For the remaining QC samples in a quantitative range, the following apply where there is more than one set of replicates.

1. When one or more QC elements for precision (e.g. lab duplicate or MS/MSD) is greater than 1 time to less than 2 times the target (for organics and metals RPD or RSD greater than 25% to less than 50%, Tables 1 and 2 above) then the field samples within that batch are flagged with a QACode of VIL. The compliance code is QUAL.
2. If one QC element fails badly ( $> 50\%$  RPD), then consider the RPD/RSD of the other QC elements (e.g. MS/MSD, LCS/LCSD) for that analyte. If other QC elements pass ( $\leq 25\%$ ), or marginally fail ( $25\% < \text{RPD} < 50\%$ ), and there are no other indications of ongoing QA problems, then assign the samples within that batch, for that analyte, with a QACode of VIL. The compliance code is QUAL.
3. **Rejection Point:** If more than one QC element fails badly ( $> 50\%$  RPD), then assign a QACode of VRIL to the samples for that analyte in the batch and a compliance code of REJ.

If there is only one usable quantitative measure, the following apply:

4. If there is only one QC element reported in the batch and the RPD is greater than 1 time to less than 2 times the target (for organics and metals greater than 25% to less than 50%) then the field samples within that batch are flagged with a QACode of VIL. The compliance code is QUAL.
5. **Rejection Point :** If there is only one QC element reported in the batch and the RPD was more than 2 times outside the MQO target ( $> 50\%$ ) then the compliance code would be REJ and the QACode VRIL is applied to the associated field samples in that batch

Table 4 summarizes the application of QACodes for the precision check scenarios described above.

Table 4. Precision Data Validation Rules where there are more than two usable measures, use the two worst as A & B

Measure A	Measure B	QA Code	Comment
>50%	>50%	VRIL	Both bad fail.
>50%	>25%	VIL	One bad, one marginal fail
>50%	<25%	VIL	One bad fail, rest pass.
>50%	Null	VRIL	One usable, bad fail
>25%	>25%	VIL	Both marginal fail
>25%	<25%	VIL	One marginal fail, one pass
>25%	Null	VIL	One usable, marginal fail
<25%	<25%	None	Both good

(for analytes where RPD or RSD limits are not 25%, substitute 1x those limits for 25% and 2x those limits instead of 50%)

**Assumptions:**

Measure A and B can be either different types of elements (duplicates, MS/MSD) or pairs of the same type of measure. Each measure is treated separately and not averaged when there are multiple pairs of the same measure (e.g. do not average RPD if there are 2 sets of replicates).

## Glossary

**Calibration Standard:** Calibration standards are the measurement of an absolute value of a target analyte and in many cases, the standards are traceable back to standards at the National Institute for Standards and Technology. A **calibration curve** is a general method for determining the concentration of a substance in an unknown sample by comparing the unknown to a set of standard samples of known concentration. A calibration curve is one approach to the problem of instrument calibration.

**Certified Reference Material:** CRMs are similar in matrix and concentration range to the samples being prepared and analyzed. The accuracy of an analytical method can be assessed using CRMs only when certified values are provided for the target analytes.

**Continuing Calibration Verification:** 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.

**Expected Value:** the concentration of the analyte in a reference standard, laboratory control sample or matrix spike sample, or the value expected to be obtained from analysis of the QC sample. This consists of the native sample result concentration plus the spike amount.

**Internal (or Surrogate) Standard:** To optimize gas chromatography mass spectrometry (GC-MS) and Inductively Coupled Plasma Mass Spectrometry (ICP-MS) analyses, internal standards (also referred to as “injection internal standards”) may be added to field and QC sample extracts prior to injection. Use of internal standards is particularly important for analysis of complex extracts subject to retention time shifts relative to the analysis of standards. The internal standards can also be used to detect and correct for problems in the GC injection port or other parts of the instrument.

**Laboratory Control Sample:** An LCS is a specimen of known composition prepared using contaminant-free reagent water or an inert solid spiked with the target analyte at the midpoint of the calibration curve or at the level of concern. The LCS must be analyzed using the same preparation, reagents, and analytical methods employed for regular samples.

**Laboratory Duplicate:** In order to evaluate the precision of an analytical process, a field sample is selected and digested or extracted in duplicate and analyzed according to the method.

**Matrix Spike:** A matrix spike (MS) is prepared by adding a known concentration of the target analyte to a field sample (spike amount), 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 are analyzed in order to assess the magnitude of matrix interference. Because matrix spikes are analyzed in pairs, the second spike is called the matrix spike duplicate (MSD).

**Method Blank:** A laboratory blank prepared to represent the sample matrix as closely as possible and analyzed exactly like the calibration standards, samples, and quality control (QC) samples. Results of method blanks provide an estimate of the within-batch variability of the blank response.

**Method Detection Limit or Method Limit:** EPA defines the method detection limit as, "the minimum concentration of a substance that can be measured and reported with 99% confidence that the analyte concentration is greater than zero and is determined from analysis of a sample in a given matrix containing the analyte." Any sample that is not quantifiable is considered to be not detected and below the MDL.

**Measurement Quality Objectives:** Numerical acceptance criteria for the quality attributes measured by project data quality indicators. During project planning, measurement quality objectives are established as quantitative measures of performance against selected data quality indicators, such as precision, bias, representativeness, completeness, comparability, and sensitivity.

**Native Sample:** the original sample to which a known spike amount is added. The native sample plus spike becomes a Matrix Spike.

**Reference Material:** The distinction between a reference material and a certified reference material does not involve how the two are prepared, rather with the way that the reference values were established. Certified values are determined through replicate analyses using two independent measurement techniques for verification. The certifying agency may also provide "non-certified or "reference" values for other target analytes. Such values are determined using a single measurement technique that may introduce bias.

**Reporting Limit:** A reporting limit is the minimum value below which chemistry data are documented as detected but not quantified.

## References

Bonnema, A. 2016. Quality Assurance Project Plan: Monitoring Lakes and Reservoirs: 2016. Moss Landing Marine Labs. Prepared for SWAMP BOG.

Surface Water Ambient Monitoring Program Quality Assurance Team (SWAMPQAT). 2008. The Surface Water Ambient Monitoring Program Quality Assurance Program Plan, Version 1.0. Prepared for the Surface Water Ambient Monitoring Program, California Water Resources Control Board, Sacramento, CA.

## **Appendix V. Signature Pages**





## Attachment 2. Field Data Sheet Example

SWAMP Tissue Sampling - Non-Trawl (Event Type = TI) SWB_FishLk_2016				Entered in d-base (initial/date)		Pg of Pgs	
*StationCode: _____		*StationName: _____		*Purpose Failure Code: _____		Agency <b>MPSL-DFG</b>	
*Sampling Crew: _____		*Date (mm/dd/yyyy):    /    /		BEAUFORT SCALE (see attachment): _____		WIND DIRECTION (from): _____	
ArrivalTime: _____		DepartureTime: _____				PHOTOS (RB & LB assigned when facing downstream; RENAME to StationCode_yyyy_mm_dd_uniquecode):	
DOMINANTSUBSTRATE: Concrete,Cobble,Gravel,Sand,Mud,Other,unk		WATERCOLOR: Colorless, Green, Yellow, Brown		1: (RB / LB / BB / US / DS / ##)			
WATERCLARITY: Clear (see bottom), Cloudy (>4" vis), Murky (<4" vis)		OTHER PRESENCE: Vascular,Nonvascular,OilySheen,Foam,Trash,O		2: (RB / LB / BB / US / DS / ##)			
Comments:				3: (RB / LB / BB / US / DS / ##)			
<b>Tissue Collection</b>							
COLLECTION DEVICE: RV _____ Masta-Blasta, Big E, Lil E, Ghetto-Blasta, _____, Backpack Model _____, Net # _____							
Target:	Lat (dd.dxxxx)	Long (dd.dxxxx)		-			
GPS Model:		Datum: NAD83 WGS84 Other _____		*GPS / DGPS			
Location	*Depth (m):	Distance from Bank (m):		Accuracy (ft / m)	Latitude (dd.dxxxx)	Longitude (-ddd.dxxxx)	Depth (m)
COLLECTION METHOD:	E-boat, Backpack shocker, Fyke net, gill net, seine, hook & line		Start Time	Coord. 1			
SAMPLE LOCATION:	Location Shock/Net/Hook			Coord. 2			
HYDROMODIFICATION:	None, Bridge, Pipes, Concrete Channel, Grade Control, Culvert,		End Time	Coord. 3			
HYDROMODLOC(to sample):	US / DS / NA / W / Other _____	Geoshape: Line Poly Point		Coord. 4			
Location	*Depth (m):	Distance from Bank (m):			Latitude (dd.dxxxx)	Longitude (-ddd.dxxxx)	Depth (m)
COLLECTION METHOD:	E-boat, Backpack shocker, Fyke net, gill net, seine, hook & line		Start Time	Coord. 1			
SAMPLE LOCATION:	Location Shock/Net/Hook			Coord. 2			
HYDROMODIFICATION:	None, Bridge, Pipes, Concrete Channel, Grade Control, Culvert,		End Time	Coord. 3			
HYDROMODLOC(to sample):	US / DS / NA / W / Other _____	Geoshape: Line Poly Point		Coord. 4			
Location	*Depth (m):	Distance from Bank (m):			Latitude (dd.dxxxx)	Longitude (-ddd.dxxxx)	Depth (m)
COLLECTION METHOD:	E-boat, Backpack shocker, Fyke net, gill net, seine, hook & line		Start Time	Coord. 1			
SAMPLE LOCATION:	Location Shock/Net/Hook			Coord. 2			
HYDROMODIFICATION:	None, Bridge, Pipes, Concrete Channel, Grade Control, Culvert,		End Time	Coord. 3			
HYDROMODLOC(to sample):	US / DS / NA / W / Other _____	Geoshape: Line Poly Point		Coord. 4			
Failure Codes: Dry (no water), Instrument Failure, No Access, Non-sampleable, Pre-abandoned, Other							
Comments:							





## Attachment 4. Laboratory Data Sheet Example

SWAMP Lab Data Sheet - FISH		ProjectID: SWB_FishLk_2016		PrepPres: Skin OFF		LabID:		Pg: 1 of 3 Pgs				
StationCode: 203TEMLAK			Tissue: fillet			Entered d-base (initial/date)						
StationName: Lake Temescal			Homog. Method: BUCCHI POLYTRON OTHER			Staff: Diss. Homog.						
Species Name: Largemouth Bass			Date Diss. (mm/dd/yyyy): / /			Date Homog. (mm/dd/yyyy): / /						
#	Tissue/Bag ID	Fish #	Organism ID	Composite / Individual ID	FL (mm)	TL (mm)	Whole Fish Wt (g)	Part Wt (g)	Sex	Part	Anomaly	Body Location
1	BOG16_B1495_I	B1495	203TEMLAKBOG16LMB01-01	I_203TEMLAKBOG16LMB01-01	/	/	/	/	M / F / Unk	T / L / O		
2	BOG16_B1496_I	B1496	203TEMLAKBOG16LMB01-02	I_203TEMLAKBOG16LMB01-02	/	/	/	/	M / F / Unk	T / L / O		
3	BOG16_B1497_I	B1497	203TEMLAKBOG16LMB01-03	I_203TEMLAKBOG16LMB01-03	/	/	/	/	M / F / Unk	T / L / O		
4	BOG16_B1498_I	B1498	203TEMLAKBOG16LMB01-04	I_203TEMLAKBOG16LMB01-04	/	/	/	/	M / F / Unk	T / L / O		
5	BOG16_B1499_I	B1499	203TEMLAKBOG16LMB01-05	I_203TEMLAKBOG16LMB01-05	/	/	/	/	M / F / Unk	T / L / O		
6	BOG16_B1499_C	B1499	203TEMLAKBOG16LMB01-05	C1_203TEMLAKBOG16LMB	/	/	/	/	M / F / Unk	T / L / O		
7	BOG16_B1500_I	B1500	203TEMLAKBOG16LMB01-06	I_203TEMLAKBOG16LMB01-06	/	/	/	/	M / F / Unk	T / L / O		
8	BOG16_B1500_C	B1500	203TEMLAKBOG16LMB01-06	C1_203TEMLAKBOG16LMB	/	/	/	/	M / F / Unk	T / L / O		
9	BOG16_B1501_I	B1501	203TEMLAKBOG16LMB01-07	I_203TEMLAKBOG16LMB01-07	/	/	/	/	M / F / Unk	T / L / O		
10	BOG16_B1501_C	B1501	203TEMLAKBOG16LMB01-07	C1_203TEMLAKBOG16LMB	/	/	/	/	M / F / Unk	T / L / O		
11	BOG16_B1502_I	B1502	203TEMLAKBOG16LMB02-01	I_203TEMLAKBOG16LMB02-01	/	/	/	/	M / F / Unk	T / L / O		
12	BOG16_B1502_C	B1502	203TEMLAKBOG16LMB02-01	C1_203TEMLAKBOG16LMB	/	/	/	/	M / F / Unk	T / L / O		
13	BOG16_B1503_I	B1503	203TEMLAKBOG16LMB02-02	I_203TEMLAKBOG16LMB02-02	/	/	/	/	M / F / Unk	T / L / O		
14	BOG16_B1503_C	B1503	203TEMLAKBOG16LMB02-02	C1_203TEMLAKBOG16LMB	/	/	/	/	M / F / Unk	T / L / O		
15	BOG16_B1504_I	B1504	203TEMLAKBOG16LMB02-03	I_203TEMLAKBOG16LMB02-03	/	/	/	/	M / F / Unk	T / L / O		
16	BOG16_B1505_I	B1505	203TEMLAKBOG16LMB02-04	I_203TEMLAKBOG16LMB02-04	/	/	/	/	M / F / Unk	T / L / O		
17												
18												
19												
20												
21												
22												
23												
24												
25												

OrganismID: xxxxxxxxLLXX##YYZz-ZZ; unique code - StationCode (xxxxxxx), Location (LL), Project (XX), ProjectYear (##), OrganismCode (YYY), Bag # (zz), Fish # (ZZ); ex. 203SRF101L1SW04CAR01-01

TissueID: Differentiates different parts from same fish or differentiates composited vs. individual fish Part: Tissue (T), Liver (L), Other (O) - list in Comments

Comp/IndID: Unique code; include Agency code in the ID; e.g., 2003-1823-MLML or C031501-MLML

Anomalies: Ambicoloration (A), Albinism (B), Cloudiness (CL), Deformity-skeletal (D), Discoloration (DC), Depression (DS), Fin Erosion (F), Gill Erosion (T), Hemorrhage (H), Lesion (L), Parasite (P),

Body Locations: Branchial Chamber (BRC), Buccal Cavity (BC), Eyes (E), Musculoskeleton (M), Skin/Fins (SF) Popeye (PE), Tumor (T), Ulceration (U), White Spots (W), and any combination

Comments: Measure length to nearest 1 mm; Measure weight to nearest 0.01 g; Keep archive tissue if possible; if a duplicate is made, use DupID as identification for analysis

Modified 06/08/07

<b>SWAMP Lab Data Sheet - FISH</b>		ProjectID: SWB_FishLk_2016	PrepPres: Skin OFF	LabID:	Pg: 2 of 3 Pgs
StationCode: 203TEMLAK		Tissue: fillet		Entered d-base (initial/date)	
StationName: Lake Temescal		Homog. Method: BUCCHI POLYTRON OTHER		Staff. Diss.	Homog.
Species Name: Largemouth Bass		Date Diss. (mm/dd/yyyy): / /		Date Homog. (mm/dd/yyyy): / /	
<b>CHEMISTRY JARS</b>					
Individual ID: I_203TEMLAKBOG16LMB01-01		Individual ID: I_203TEMLAKBOG16LMB01-02		Individual ID: I_203TEMLAKBOG16LMB01-03	
Analysis: Mercury		Analysis: Mercury		Analysis: Mercury	
Jar Weight Full (g): _____		Jar Weight Full (g): _____		Jar Weight Full (g): _____	
Jar Weight Empty (g): _____		Jar Weight Empty (g): _____		Jar Weight Empty (g): _____	
Comp Tissue Wt (Jar Full - Empty; g): _____		Comp Tissue Wt (Jar Full - Empty; g): _____		Comp Tissue Wt (Jar Full - Empty; g): _____	
Individual ID: I_203TEMLAKBOG16LMB01-04		Individual ID: I_203TEMLAKBOG16LMB01-05		Individual ID: I_203TEMLAKBOG16LMB01-06	
Analysis: Mercury		Analysis: Mercury		Analysis: Mercury	
Jar Weight Full (g): _____		Jar Weight Full (g): _____		Jar Weight Full (g): _____	
Jar Weight Empty (g): _____		Jar Weight Empty (g): _____		Jar Weight Empty (g): _____	
Comp Tissue Wt (Jar Full - Empty; g): _____		Comp Tissue Wt (Jar Full - Empty; g): _____		Comp Tissue Wt (Jar Full - Empty; g): _____	
Individual ID: I_203TEMLAKBOG16LMB01-07		Individual ID: I_203TEMLAKBOG16LMB02-01		Individual ID: I_203TEMLAKBOG16LMB02-02	
Analysis: Mercury		Analysis: Mercury		Analysis: Mercury	
Jar Weight Full (g): _____		Jar Weight Full (g): _____		Jar Weight Full (g): _____	
Jar Weight Empty (g): _____		Jar Weight Empty (g): _____		Jar Weight Empty (g): _____	
Comp Tissue Wt (Jar Full - Empty; g): _____		Comp Tissue Wt (Jar Full - Empty; g): _____		Comp Tissue Wt (Jar Full - Empty; g): _____	
Individual ID: I_203TEMLAKBOG16LMB02-03		Individual ID: I_203TEMLAKBOG16LMB02-04			
Analysis: Mercury		Analysis: Mercury			
Jar Weight Full (g): _____		Jar Weight Full (g): _____			
Jar Weight Empty (g): _____		Jar Weight Empty (g): _____			
Comp Tissue Wt (Jar Full - Empty; g): _____		Comp Tissue Wt (Jar Full - Empty; g): _____			
Comments: Keep archive tissue if possible; if a duplicate is made, use Dup ID as identification for analysis					
Modified 06/08/07					



SWAMP Lab Data Sheet - FISH			ProjectID: SWB_FishLk_2016			PrepPres: Skin ON, Scales ON			LabID:			Pg: 1 of 2 Pgs					
StationCode: 203TEMLAK						Tissue: whole						Entered d-base (initial/date)					
StationName: Lake Temescal						Homog. Method: BUCCHI POLYTRON OTHER						Staff: Diss. Homog.					
Species Name: Bluegill prey						Date Diss. (mm/dd/yyyy): / /						Date Homog. (mm/dd/yyyy): / /					
#	Tissue/Bag ID	Fish #	Organism ID	Composite / Individual ID	FL (mm)	TL (mm)	Whole Fish Wt (g)	Part Wt (g)	Sex	Part	Anomaly	Body Location					
1	BOG16_TEMBGL01-01		203TEMLAKBOG16BGL01-01	C1_203TEMLAKBOG16BGL					M/F/Unk	T							
2	BOG16_TEMBGL01-02		203TEMLAKBOG16BGL01-02	C1_203TEMLAKBOG16BGL					M/F/Unk	T							
3	BOG16_TEMBGL01-03		203TEMLAKBOG16BGL01-03	C1_203TEMLAKBOG16BGL					M/F/Unk	T							
4	BOG16_TEMBGL01-04		203TEMLAKBOG16BGL01-04	C1_203TEMLAKBOG16BGL					M/F/Unk	T							
5	BOG16_TEMBGL01-05		203TEMLAKBOG16BGL01-05	C1_203TEMLAKBOG16BGL					M/F/Unk	T							
6	BOG16_TEMBGL01-06		203TEMLAKBOG16BGL01-06	C1_203TEMLAKBOG16BGL					M/F/Unk	T							
7	BOG16_TEMBGL01-07		203TEMLAKBOG16BGL01-07	C1_203TEMLAKBOG16BGL					M/F/Unk	T							
8	BOG16_TEMBGL01-08		203TEMLAKBOG16BGL01-08	C1_203TEMLAKBOG16BGL					M/F/Unk	T							
9	BOG16_TEMBGL01-09		203TEMLAKBOG16BGL01-09	C1_203TEMLAKBOG16BGL					M/F/Unk	T							
10	BOG16_TEMBGL01-10		203TEMLAKBOG16BGL01-10	C1_203TEMLAKBOG16BGL					M/F/Unk	T							
11																	
12																	
13																	
14																	
15																	
16																	
17																	
18																	
19																	
20																	
21																	
22																	
23																	
24																	
25																	
OrganismID: xxxxxxxxLLXX##YYzz-ZZ; unique code - StationCode (xxxxxxxx), Location (LL), Project (XX), ProjectYear (##), OrganismCode (YYY), Bag # (zz), Fish # (ZZ); ex. 203SRF101L1SW04CAR01-01																	
TissueID: Differentiates different parts from same fish or differentiates composited vs. individual fish								Part: Tissue (T), Liver (L), Other (O) - list in Comments									
Comp/IndID: Unique code; include Agency code in the ID; e.g., 2003-1823-MLML or C031501-MLML																	
Anomalies: Ambicoloration (A), Albinism (B), Cloudiness (CL), Deformity-skeletal (D), Discoloration (DC), Depression (DS), Fin Erosion (F), Gill Erosion (T), Hemorrhage (H), Lesion (L), Parasite (P),																	
Body Locations: Branchial Chamber (BRC), Buccal Cavity (BC), Eyes (E), Musculoskeleton (M), Skin/Fins (SF) Popeye (PE), Tumor (T), Ulceration (U), White Spots (W), and any combination																	
Comments: Measure length to nearest 1 mm; Measure weight to nearest 0.01 g; Keep archive tissue if possible; If a duplicate is made, use DupID as identification for analysis																	

<b>SWAMP Lab Data Sheet - FISH</b>		ProjectID: SWB_FishLk_2016	PrepPres: Skin ON, Scales ON	LabID:	Pg: 2 of 2 Pgs
StationCode: 203TEMLAK		Tissue: whole		Entered d-base (initial/date)	
StationName: Lake Temescal		Homog. Method: BUCCHI POLYTRON OTHER _____		Staff. Diss.	Homog.
Species Name: Bluegill prey		Date Diss. (mm/dd/yyyy): / /		Date Homog. (mm/dd/yyyy): / /	
<b>CHEMISTRY JARS</b>					
CompositeID: C1_203TEMLAKBOG16BGL		CompositeID: C1_203TEMLAKBOG16BGL			
Analysis: Mercury and Selenium		Analysis: Archive1			
Jar Weight Full (g): _____		Jar Weight Full (g): _____			
Jar Weight Empty (g): _____		Jar Weight Empty (g): _____			
Comp Tissue Wt (Jar Full - Empty; g): _____		Comp Tissue Wt (Jar Full - Empty; g): _____			
Glass 60mL					
<b>Comments:</b> Keep archive tissue if possible; If a duplicate is made, use Dup ID as identification for analysis					
Modified 06/08/07					