Group A Elements: Project Management

Element 1. Title and Approval Sheets

QUALITY ASSURANCE PROJECT PLAN

A SECOND STATEWIDE SURVEY OF BIOACCUMULATION ON THE CALIFORNIA COAST

Bioaccumulation Oversight Group (BOG)

Surface Water Ambient Monitoring Program

Version 1
December 2018

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Program Title	SWAMP Bioaccumulation Oversight Group A Second Statewide Survey of Bioaccumulation on the California Coast
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Effective Date	This Quality Assurance Project Plan (QAPP) is effective from April 2018 to May 2021 unless otherwise revised, approved and distributed accordingly at an earlier date.
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Approvals

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

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Contract Contact

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QAPP Preface

This Quality Assurance Project Plan (QAPP) document defines procedures and criteria that will be used for this project, conducted by the Bioaccumulation Oversight Group (BOG) in association with the Moss Landing Marine Labs Marine Pollution Studies Laboratory (MPSL-DFW), Southern California Bight (Bight '18), Southern California Coastal Water Research Project (SCCWRP), 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 each agency or laboratory also are contained within. 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, with coordination from BIGHT '18, Regional Water Quality Control Board 4 (RWQCB4), Regional Water Quality Control Board 8 (RWQCB8) and the Regional Monitoring Program in the San Francisco Estuary (RMP).

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Element 3. Distribution List and Contact Information

A copy of this Quality Assurance Project Plan (QAPP), in hardcopy or electronic format, is to be received and retained by at least one person from each participating entity. At least one person from each participating entity (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

Name	Agency, Company or Organization
SAN FRANCISCO ESTUARY INSTITUTE	
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SOUTHERN CALIFORNIA COASTAL WATER RESEARCH PROJECT	
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MARINE POLLUTION STUDIES LAB	
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DELTA ENVIRONMENTAL LABORATORIES	
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OFFICE OF INFORMATION MANAGEMENT **SWAMP INFORMATION MANAGEMENT AND QUALITY ASSURANCE**	
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* Indicates person responsible for receiving, retaining, and distributing the final QAPP to staff within their organization

Element 4. Project Organization

The lines of communication among the participating entities, project organization and responsibilities are outlined in Table 2 (below) and Figure 1 (Section 4.4).

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.
SWRCB Management	Greg Gearhart	Program planning and oversight; project budget allocation and reconciliation with program objectives
SWRCB QA Officer	Renee Spears (SWRCB)	Approves QAPP; reports
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) Ken Schiff (SCCWRP)	Advisory role; data reporting; development of Monitoring Plan; coordination with BOG technical workgroup
Project Manager	Autumn Bonnema (MPSL-DFW)	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
Acting 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
SWAMP IQ Data Quality Managers	Kimberly Pham (SWRCB) Brian Ogg (SWRCB)	Reviews, verifies, validates and loads chemistry and composite data to SWAMP database; reports to Program QAO
Laboratory QA Officer	Autumn Bonnema (MPSL-DFW) Timea Majoros (DeltaEnv) Various, Bight ’18 Laboratories	Ensures that the laboratory quality assurance plan and quality assurance project plan criteria are met through routine monitoring and auditing of the systems; reviews and approves data prior to submission to SWAMP IQ; investigates and conducts laboratory corrective action.
Sample Collection Coordinator	Billy Jakl (MPSL-DFW) Gary Ichikawa (MPSL-DFW)	Sampling coordination, operations, and implementing field-sampling procedures.
Laboratory Director	Wes Heim (MPSL-DFW) Timea Majoros (DeltaEnv)	Supervises laboratory staff; data validation, management and reporting
Sample Custodian	April Guimarães (MPSL-DFW) Timea Majoros (DeltaEnv) Various, Bight ’18 Laboratories	Sample storage; not responsible for any deliverables; may oversee Technicians
Technicians	Technical staff MPSL-DFW DeltaEnv Various, Bight ’18 Laboratories	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 SFEI and Ken Schiff of SCCWRP are the Lead Scientists (LS) and primary contacts of this project. Ken Schiff is coordinating efforts with Bight '18. 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-DFW 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-DFW 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.

Timea Majoros is responsible for sample storage and custody at DeltaEnv. April Guimarães will do the same for samples processed at MPSL, in addition to overseeing compositing of tissue samples.

Timea Majoros will serve as the Laboratory Director (LD) for the DeltaEnv 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 DeltaEnv activities are completed within the proper timelines.

Wes Heim will serve as the LD for the MPSL-DFW 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-DFW 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, Shannon Murphy and Lori Chumney (Office of Environmental Health Hazard Assessment (OEHHA)), Rich Fadness (Regional Water Quality Control Board 1(RWQCB1), Carrie Austin (RWQCB2), Mary Hamilton and Melissa Daugherty (RWQCB3), Jun Zhu (RWQCB4), Lauren Smitherman, Jennifer Fuller and Patrick Morris (RWQCB5), Kelly Huck, Carly Nilson and Mary Fiore-Wagner (RWCQB6), Jeff Geraci (RWCB7), Heather Boyd and Terri Reeder (RWQCB8), Chad Loflen (RWQCB9), and Jennifer Salisbury, Dawit Tadesse, and Ali Dunn (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 Bruce Monson (Minnesota Pollution Control Agency, St. Paul, Minnesota), Chris Schmitt, (United States Geological Survey, Columbia, Missouri) and Harry Ohlendorf (CH2M HILL, Sacramento, California).

4.2. Quality Assurance Officer (QAO) Role

Autumn Bonnema is the MPSL-DFW LQAO (LQAO) and Timea Majoros is the DeltaEnv 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 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.

4.3. Persons responsible for QAPP update and maintenance

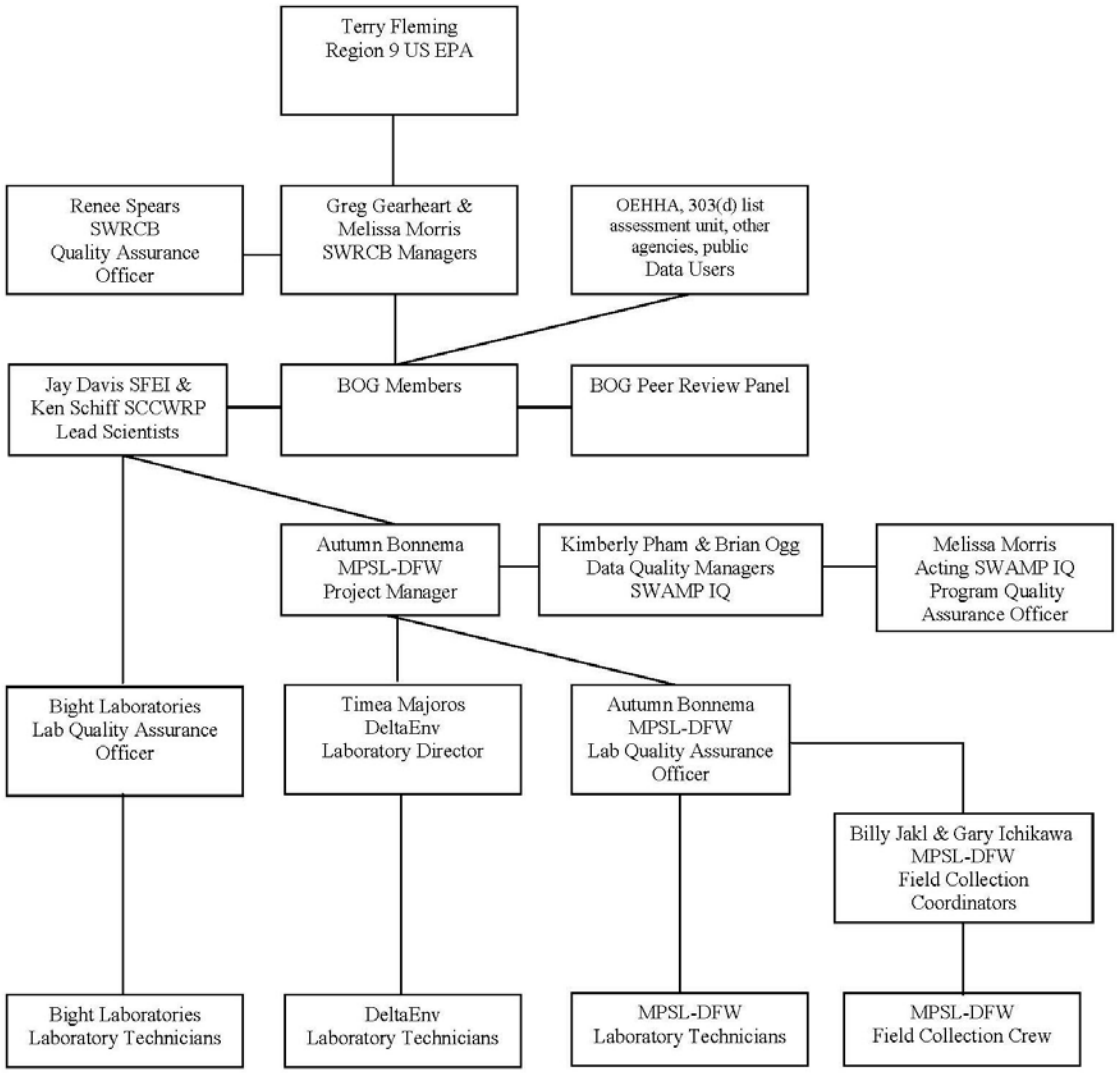
Revisions and updates to this QAPP will be carried out by Autumn Bonnema, with technical input from the Laboratory and Program QAOs. All changes will be considered draft until reviewed and approved by the PM, the Program QAO, and SWRCB QAO.

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, SCCWRP, DeltaEnv and MPSL-DFW.

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). Additional beneficial uses relating to harvesting

fish have been established by the SWRCB: Water Contact Recreation (REC-1), Native American Culture (CUL), Tribal Subsitence Fishing (T-SUB) and Subsistence Fishing (SUB) (State Water Resources Control Board Resolution 2017-0027). 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, REC-1, T-SUB and SUB, 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

The following information is excerpted from the Monitoring Plan for a Second Statewide Survey of Bioaccumulation on the California Coast (Monitoring Plan, Appendix II):

Four management questions have been articulated to guide this second SWAMP survey of the status of bioaccumulation in sport fish on the California coast. These management questions are essentially the same as the questions that guided the initial screening effort, with a revised wording of the first management question.

5.2.1. Management Questions

5.2.1.1.Management Question 1 (MQ1)

Status: What is the status of contaminants in representative fish species in popular fishing areas?

Answering this question is critical to determining the degree of impairment of the fishing beneficial uses (COMM, REC-1, CUL, T-SUB, SUB, etc.) across the state due to bioaccumulation. This question places emphasis on characterizing the status of the fishing beneficial use through monitoring of the predominant pathways of exposure – representative fish species and popular fishing areas. This focus will provide information on the resources that water quality managers and people care most about.

The data needed to answer this question are average concentrations in representative fish species from popular coastal fishing locations. Inclusion of as many species as possible is important to understanding the nature of impairment in any areas with concentrations above thresholds. In some areas, some fish may be safe for consumption while others are not, and this

is valuable information for anglers. Monitoring species that accumulate high concentrations of contaminants ("indicator species") is valuable in answering this question: if concentrations in these species are below thresholds, this is a strong indication that an area has low concentrations.

5.2.1.2.Management Question 2 (MQ2)

Regional Distribution: What is the distribution of contaminant concentrations in fish within regions?

Answering this question will provide information that is valuable in formulating management strategies for observed contamination problems. This information will allow managers to prioritize their efforts and focus attention on the areas with the most severe problems. Data on regional distribution will also provide information on contaminant sources and fate that will be useful to managers.

This question can be answered with different levels of certainty. For a higher and quantified level of certainty, a statistical approach with replicate observations in the spatial units to be compared is needed. In some cases, managers can attain an adequate level of understanding for their needs with a non-statistical, non-replicated approach. With either approach, reliable estimates of average concentrations within each spatial unit are needed.

5.2.1.3.Management Question 3 (MQ3)

Trends: What are the trends in contaminant concentrations in representative fish species in popular fishing areas?

Information on trends is essential to effective management of contaminants that bioaccumulate in sport fish. It is critically important to know whether the problem is getting better or worse; in other words, whether food web mercury concentrations are trending up or down on a local, regional, or statewide scale. A statewide increasing trend could obscure the beneficial effects of management actions to reduce bioaccumulation. On the other hand, evidence of a general declining trend could give the impression that actions are more effective than they actually are.

The data needed to answer this question are measurements that are repeated over time to derive average concentrations for indicator species in popular fishing areas. Striving for consistency in the sampling design (e.g., species and locations within zones) over time will maximize the utility of the data for long-term trend analysis. With a 10-year cycle for statewide sampling, this approach will establish a foundation for and gradually build a long-term time series for trend evaluation.

5.2.1.4.Management Question 4 (MQ4)

Need for Further Sampling: Should additional sampling of bioaccumulation in sport fish (e.g., more species or larger sample size) in an area be conducted for the purpose of developing more comprehensive consumption guidelines?

Consumption guidelines provide a mechanism for reducing human exposure to bioaccumulated contaminants in the short-term. Based largely on the data generated in the SWAMP coastal survey of 2009-2010, OEHHA issued a statewide consumption advisory for the entire coast in 2016 (Smith et al. 2016). In developing consumption advice, 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 them. The diversity of species on the coast demands a relatively large effort to characterize interspecific variation. The present round of coastal sampling will address data gaps identified by OEHHA in the process of developing the statewide coastal advisory. After the results of this round are reviewed, OEHHA will be able to further refine the list of data gaps related to advisory development.

5.2.2. Overall Approach

The overall approach to be taken to answer these questions will be to perform a statewide survey of bioaccumulation in sport fish on the California coast. This will update the assessment decision-makers need to understand the scope of the bioaccumulation problem, as well as provide regulators with information they need to establish priorities for cleanup actions and further development of consumption guidelines.

5.2.3. Coordination

Through coordination with other programs, SWAMP funds for this survey are going to be highly leveraged to achieve a much more thorough statewide assessment than could be achieved by SWAMP alone. Details on coordination with the Bay RMP, the Bight '18, RWQCB4, RWQCB8, and RWQCB9 can be found in the MP (Appendix II, pp 9-10).

It is important to note that Bight '18 laboratories will be held to the QA standards set forth in the Bight '18 QAP (Appendix V). Some of these standards overlap with this document. Differences are detailed in Tables 11 and 12.

A laboratory intercalibration is being conducted under the Bight '18 program, and all laboratories referenced herein are participating. Results will be compiled prior to the analysis of any BOG samples to ensure analytical labs are comparable.

Element 6. Project Description

6.1. Work statement and produced products

The study is being phased to facilitate coordination and continuing demonstration of successful monitoring by placing a priority on generating information that is of maximum value to regulators and the public. As in 2009-2010, this survey will be conducted over two years to allow thorough coverage of the entire coast with available funds. However, this survey will be spread over three calendar years, with BOG crews sampling in 2018 and 2020, and the coordinated effort of Bay RMP occurring in 2019.

Year 1 sampling (2018) will focus on the Bight '18 (Water Board Regions 4, 8, and 9, and some of Region 3 (Appendix II, Figure 1). This will allow for coordination with Bight '18, and will provide a basis for a report on year 1 that describes the bioaccumulation in the most populated and heavily fished areas near Los Angeles.

RMP will conduct sampling and analysis in 2019. A stand-alone report will be generated for this effort.

Year 2 sampling (2020) will cover the other coastal regions (1 and 3) and any other remaining areas not covered in 2018 or 2019. A comprehensive assessment of the entire data set, including the Bay RMP data, will be presented in a final report.

6.2. Constituents to be analyzed and measurement techniques.

A detailed Monitoring Plan is in Appendix II. Chemistry analytical methods are summarized in Section F. Constituents to be analyzed are summarized in Tables 3-7, below. All tissue chemistry data will be reported on a wet weight basis.

MPSL-DFW and DeltaEnv will be the primary analytical laboratories for the BOG effort. MPSL-DFW will analyze mercury and selenium in all samples that satisfy the BOG monitoring plan. DeltaEnv will analyze all organic (PCBs, and some OCHs and PBDEs) in samples from North and Central coast zones, but only those samples from species that are not part of the Bight '18 monitoring plan in the Southern zones. Bight consortium laboratories will analyze metals/metalloids (mercury, selenium and arsenic) and organics (PCBs, DDTs, chlordanes and dieldrin) in samples from Bight zones (Appendix V, Table 6-1). Bight labs will be held to QA standards set forth in the Bight '18 Sampling Plan and Analytical methods are listed in Tables 15, 17-18 as appropriate.

Regional Water Board 1 requested Dioxin analysis be analyzed in samples from Humboldt Bay (Zone 64). Dioxins will also be analyzed in Bay RMP samples collected in 2019, therefore this zone may be sampled in 2019 and analyzed by the laboratory identified in the Bay RMP sampling plan and Quality Assurance Plan. At this time, neither document is available to reference, though past plans may be found at http://www.sfei.org/documents/2014-quality-assurance-program-plan-regional-monitoring-program-water-quality-san.

Though previous studies have calculated PCBs 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. For this study, all congeners analyzed will be summed.

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. PBDEs were a concern in San Francisco Bay in the early 2000s, but by 2014 were no longer of concern in RMP samples as they were well below the lowest OEHHA Advisory Tissue Level for seven servings per week (Sun et al. 2017; Klasing and Brodberg 2008). Bay RMP will analyze these compounds in 2019;

however they are not part of the current BOG standard analyte list. However, PBDEs are still of concern in Zone 1: Tijuana to North Island. Region 9 has specifically requested analysis of these compounds from all species analyzed in this zone.

Table 3. Constituents to be analyzed – fish attributes

Fish Attributes
Total Length (mm)
Fork Length (mm)
Weight (g)
Sex
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

Analyte	Matrix Type	Laboratory	Analytical Method
Total Mercury	Sport Fish filet muscle or Whole body, minus head, gut and tail	MPSL-DFW	EPA 7473 (USEPA 1998)
Total Mercury	Sport Fish filet muscle or Whole body, minus head, gut and tail	Bight Labs	Method chosen by each lab*
Total Selenium	Sport Fish filet muscle or Whole body, minus head, gut and tail	MPSL-DFW	EPA 3052M (Appendix III E) EPA 200.8 (USEPA 1994a)
Total Selenium	Sport Fish filet muscle or Whole body, minus head, gut and tail	Bight Labs	Method chosen by each lab*
Total Arsenic (Bight ’18 only)	Sport Fish filet muscle or Whole body, minus head, gut and tail	Bight Labs	Method chosen by each lab*

* See Appendix V for more details on Bight ’18

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

Polychlorinated Biphenyl (PCB) Congeners (by USEPA Method 8082A, USEPA 2007b)	
PCB 008	PCB 128*
PCB 018*	PCB 137
PCB 027	PCB 138*
PCB 028*	PCB 141
PCB 029	PCB 146
PCB 031	PCB 149*
PCB 033	PCB 151*
PCB 044*	PCB 153*
PCB 049*	PCB 156*
PCB 052*	PCB 157*
PCB 056	PCB 158*
PCB 060	PCB 169*
PCB 064	PCB 170*
PCB 066*	PCB 174
PCB 070*	PCB 177*
PCB 074*	PCB 180*
PCB 077*	PCB 183*
PCB 087*	PCB 187*
PCB 097	PCB 189*
PCB 099*	PCB 194*
PCB 101*	PCB 195
PCB 105*	PCB 200
PCB 110*	PCB 201*
PCB 114*	PCB 203
PCB 118*	PCB 206*
PCB 126*	PCB 209

* Part of Bight ’18 Analyte List; Bight ’18 list also includes PCBs 037, 081, 119, 123, 167, 168. See Appendix V for more details on Bight ’18

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

Organochlorine Pesticides (by EPA 8081B, USEPA 2007c)	
Group	**Parameter**
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

* Part of Bight ’18 Analyte List. See Appendix V for more details on Bight ’18

Table 7. Constituents to be Analyzed – Polybrominated Diphenyl Ethers (PBDE)

Polybrominated Diphenyl Ethers (PBDEs) (by EPA Method 8081BM)
PBDE 017
PBDE 028
PBDE 047
PBDE 066
PBDE 085
PBDE 099
PBDE 100
PBDE 138
PBDE 153
PBDE154
PBDE 183

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 8.

Table 8. Project schedule timeline

Item	Activity and/or Deliverable	Deliverable Due Date
1	**Quality Assurance Project Plan & Monitoring Plan**	
1.1	Draft Quality Assurance Project Plan	March 2018
1.2	Final Quality Assurance Project Plan	December 2018
2	**Sample Collection**	October of each sampling year
3	**Sample Selection and Chemical Analysis**	
3.1	Selection of Tissue for Analysis	November 2018 and 2020
3.2	Creation of Sample Composites	December 2018 and 2020
3.3	Chemical Analysis	February 2019 and 2021
3.4	Data Reported to SWAMP	March 2019 and 2021
4	**Data Quality Assessment and Narrative**	May 2019 and 2021
5	**Technical Report**	
5.1	Draft Report	2018 collection year: July 2019 2018 and 2020 collection year: December 2021
5.2	Final Report	2018 collection year: September 2019 2018 and 2020 collection year: March 2022

6.4. Geographical setting and sample sites

California has over 3000 miles of coastline that span a diversity of habitats and fish populations. Along the coast and bays there are dense human population centers with a multitude of popular fishing locations.

To sample this vast area, the coast was initially divided into 69 spatial units called "zones". Due to access issues and other sampling constraints, some zones were combined in the 2009-2010 effort, resulting in 65 zones to collect in this and subsequent Coastal studies. All zones will be sampled, making a probabilistic sampling design unnecessary.

Sampling will focus on nearshore areas, including bays and estuaries, in waters not exceeding 200m, and mostly less than 60m deep.

Details on the determination of zone boundaries can be found in Appendix II, pp. 10-11.

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 8.

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

The data collection for this project is intended to answer 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, insofar as possible, similar fish sampling and analysis protocols to ensure that data collected are useful in the development of advisories, and will follow OEHHAs recommendations for the following:

- target species and number of species representative of what anglers are likely to catch at a given waterbody or location;
- 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 (ATL) developed by OEHHA (Klasing and Brodberg, 2008) (Table 9). Less sensitive methods, as demonstrated by MDLs, will result in chemical summations that are not comparable to the lowest ATL. The data will be assessed against these levels within the data analysis and reporting portions of the project.

Advisory Tissue Levels 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) 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. Please note Bight '18 labs will follow the Bight '18 MQOs (Appendix V, Tables 6-2 and 6-3), which may differ from SWAMP MQOs.

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.

Table 9. Sport fish assessment thresholds

TABLE 2. ADVISORY TISSUE LEVELS (ATLS) FOR SELECTED FISH CONTAMINANTS BASED ON CANCER OR NON-CANCER RISK USING AN 8 OUNCE SERVING SIZE (PRIOR TO COOKING) (PPB, WET WEIGHT)

Contaminant	Consumption Frequency Categories (8-ounce servings/week)[a] and ATLs (in ppb)							
	7	6	5	4	3	2	1	0
Chlordanes[c]	≤ 80	>80-90	>90-110	>110-140	>140-190	>190-280	>280-560	>560
DDTs[**]	≤ 220	>220-260	>260-310	>310-390	>390-520	>520-1,000	>1,000-2,100	>2,100
Dieldrin[C]	≤ 7	>7-8	>8-9	>9-11	>11-15	>15-23	>23-46	>46
Mercury[nc] (Women 18-45 and children 1-17)	≤ 31	>31-36	>36-44	>44-55	>55-70	>70-150	>150-440	>440
Mercury[nc] (Women > 45 and men)	≤ 94	>94-109	>109-130	>130-160	>160-220	>220-440	>440-1,310	>1,310
PBDEs[nc]	≤ 45	>45-52	>52-63	>63-78	>78-100	>100-210	>210-630	>630
PCBs[nc]	≤ 9	>9-10	>10-13	>13-16	>16-21	>21-42	>42-120	>120
Selenium[nc]	≤ 1000	>1,000-1200	>1,200-1,400	>1,400-1,800	>1,800-2,500	>2,500-4,900	>4,900-15,000	>15,000
Toxaphene[c]	≤ 87	>87-100	>100-120	>120-150	>150-200	>200-300	>300-610	>610

[c]ATLs are based on cancer risk
[nc]ATLs are based on non-cancer risk
*Serving sizes are based on an average 160 pound person. Individuals weighing less than 160 pounds should eat proportionately smaller amounts (for example, individuals weighing 80 pounds should eat one 4-ounce serving a week when the table recommends eating one 8-ounce serving a week).
**ATLS for DDTs are based on non-cancer risk for two and three servings per week and cancer risk for one serving per week.

Source: Klasing and Brodberg (2008)

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 10.

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 will not be collected because only the unexposed filet tissue of each fish is utilized, eliminating contamination from field sources.

Data from this round of collections will be compared with data collected in 2008-2009.

Table 10. Measurement Quality Indicators for laboratory measurements in tissue

Parameter	Accuracy	Precision	Recovery	Completeness	Sensitivity
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 16
Synthetic Organics (including PCBs, pesticides, and PBDEs)	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 17-19

* See Appendix V for more details on Bight '18 QA requirements as Bight '18 MQOs may differ from SWAMP MQOs.

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 (USEPA, 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.

$$\% \text{ recovery} = \frac{V_{analyzed}}{V_{certified}} x100$$

Where:

$V_{analyzed}$: the analyzed concentration of the reference material
$V_{certified}$: the certified concentration of the reference material

The acceptance criteria for reference materials are listed in Tables 11 and 12.

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 samples 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 spike samples 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.

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

Where:

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

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. To establish spiking levels prior to analysis of samples, the laboratories will review any relevant historical data. In some instances, the laboratory will be spiking the samples blind. Blind spiking will occur when insufficient material is available to perform a "spike scan" (preliminary digestion or extraction of a particular sample to obtain native concentrations). It is likely blind-spiked samples will not meet a spiking level of 2-5 times the ambient concentration. However, the results of affected samples will not be automatically rejected and will be reviewed on a case-by-case basis to determine if a different matrix spike will need to be performed.

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

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

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

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

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

2) The samples are spiked with different amounts of analyte. In this case,
V_{MS}: the recovery associated with the matrix spike
v_{MSD}: the recovery associated with matrix spike duplicate mean: the mean of the two recoveries ($recovery_{MS}$ + $recovery_{MSD}$)

The MQO for the RPD between the MS and MSD is the same regardless of the method of calculation; detailed in Tables 11 and 12.

Table 11. Measurement Quality Objectives[1] - inorganic analytes in tissue

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

* See Appendix V for more details on Bight '18 QA requirements as Bight '18 MQOs may differ from SWAMP MQOs.
[1]Unless method specifies more stringent requirements.
MDL = Method Detection Limit
RL = Reporting Limit
n/a = not applicable

Table 12. Measurement Quality Objectives[1] - synthetic organic compounds in tissue[2]

Laboratory Quality Control	Frequency of Analysis	Measurement Quality Objective
Tuning[3]	Per analytical method	Per analytical method
Calibration Standard	Initial method setup or when the calibration verification fails	• Correlation coefficient (r^2> 0.990) for linear and non-linear curves • If RSD<15% average RF may be used to quantitate; otherwise use equation of the curve. • First- or second-order curves only (not forced through the origin) • Refer to SW-846 methods for SPCC and CCC criteria[3] • Minimum of 5 points per curve (one of them at or below RL)
Continuing Calibration Verification	Per 12 hours	• Expected response or expected concentration ±20% • RF for SPCCs=initial calibration[3]
Laboratory Blank	Per 20 samples or per analytical batch, whichever is more frequent	<RL for target analytes
Reference Material	Per 20 samples or per analytical batch (preferably blind)	70-130% recovery if certified, otherwise 50-150% recovery
Matrix Spike	Per 20 samples or per analytical batch, whichever is more frequent	50-150% or based on historical laboratory control limits (average±3SD)
Matrix Spike Duplicate	Per 20 samples or per analytical batch, whichever is more frequent	50-150% or based on historical laboratory control limits (average±3SD); 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	Included in all samples and all QC samples	Based on historical laboratory control limits (50-150% or better)
Internal Standard	Included in all samples and all QC samples (as available)	Per laboratory procedure

* See Appendix V for more details on Bight '18 QA requirements as Bight '18 MQOs may differ from SWAMP MQOs.

[1]Unless method specifies more stringent requirements.
[2]All detected analytes must be confirmed with a second column, second technique, or mass spectrometry
[3]Mass spectrometry only
MDL = method detection limit (to be determined according to the SWAMP QA Management Plan)
RL = Reporting Limit
n/a = not applicable

7.2. Precision

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

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

Where:

V_{sample}: the concentration of the original sample digest
$V_{duplicate}$: 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 11 and 12.

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 Analyses

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 analyses is by calculation of the relative standard deviation (RSD). Expressed as a percentage, the RSD is calculated as follows:

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

Where:

Stdev(v_1,v_2,…,v_n): the standard deviation of the values (concentrations) of the replicate analyses.
mean: the mean of the values (concentrations) of the replicate analyses.

7.3. 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 fish, and use of only approved/documented analytical methods will determine that the measurement data 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 6) 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 within a particular zone, 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 10). 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 adopted statewide tissue objectives for mercury on 2 May 2017 (https://www.waterboards.ca.gov/water_issues/programs/mercury/docs/hg_prov_final.pdf). Through BOG discussion, the 0.2mg/kg wet weight (equal to 0.2µg/g wet wt) objective and listing threshold was selected as the criterion for classifying zones as having relatively low concentrations of mercury. To be confident that a zone truly has fish mercury concentrations below 0.2 ppm, it is desirable to have measured concentrations in predator species such as sharks

that are known to accumulate high concentrations. The analytical reporting limit for mercury (Table 10) is 16 times less than the objective; well within usability criteria (Group D Elements).

OEHHA has established advisory tissue levels ATLs that are relevant as selection criteria for zones to be included in this study (Klasing and Brodberg, 2008; Table 9). 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 (131 to 440 ppb) for the sensitive population (children and women of child-bearing age) encompasses the statewide tissue objective (200 ppb). The sum of PCB congener RLs (32.4 ppb) in Table 17 fall in the range of the 2 servings per week 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 (USEPA, 2002). For this project, the methods for site selection, sample collection, analysis, data reporting, as well as the MQOs (Tables 11 and 12), 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, and hazardous materials handling, storage, and disposal. All staff must demonstrate proficiency in all the aforementioned and required 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 DeltaEnv and MPSL-DFW. 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 DeltaEnv and MPSL-DFW 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 semi-annual safety meetings conducted by the laboratory's safety officer or an annual laboratory safety class conducted by the DeltaEnv Safety Officers and the Moss Landing Marine Laboratory 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 LD 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 (COC) 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 SWAMP IQ 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 SWRCB 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-DFW. 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 for 5 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 5 years after project completion and 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 D-E, pp. 11-17 (Appendix II). As much as possible, the same sampling locations visited in 2008-2009 will be visited again for this survey

Potential 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 8.

All measurements and analyses to be performed in tissue are critical to address the objectives laid out in Section III G, pp. 20-22 of the Monitoring Plan (Appendix II). 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, selenium, PCB and OC samples will be analyzed in composites as outlined in the Monitoring Plan (Appendix II) and MPSL-DFW SOPs (Appendix III). Mercury samples will be analyzed in individual fish for the mercury indicator species (such as kelp bass), but as composite samples for other species.

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 Peer 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 8.4 (Appendix III B). 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 1).

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

Collected fish may be partially dissected in the field (Appendix III B, section 8.4.5). The fish 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 is re-cleaned with Micro™, and rinsed with tap and deionized water between fish species, per site if multiple stations are sampled. Similarly, a new plastic bag is placed over the cutting board to prevent cross-contamination.

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 13 describes action to take in the event of a collection failure.

Table 13. Field collection corrective actions

Collection Failure	Corrective Action
Primary target species not present (Appendix II, Table 6)	Collect secondary target; it is advisable to consult with OEHHA prior to choosing secondary target species; document the occurrence
Secondary target species not present	Contact PM for further instructions. PM may approve a move to another location; document the occurrence; PM and Lead Scientist may authorize collection of species not on the target species list replace with next zone on the 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 2) 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-DFW will be logged in according to MPSL-104 (Appendix III C).

Samples will be dissected according to MPSL-105 (Appendix III D) and data retained on the lab data sheets in Attachment 3.

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 14.

Table 14. Sample handling and holding times for tissue

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

Element 13. Analytical Methods

Methods and equipment for laboratory analyses are listed in Table 15. USEPA methods can be downloaded from 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 15. Methods for laboratory analyses

Parameter	Analytical Laboratory	Method	Instrument
Mercury	MPSL-DFW	EPA 7473 (USEPA 1998)	Milestone DMA 80
Mercury	Bight '18 Labs	Method chosen by each lab*	Instrumentation chosen by each lab*
Selenium	MPSL-DFW	EPA 3052M (USEPA 1996a, Appendix III E) EPA 200.8 (USEPA 1994a)	CEM MARSXpress Digester Perkin-Elmer Elan 9000 ICP-MS
Selenium	Bight '18 Labs	Method chosen by each lab*	Instrumentation chosen by each lab*
Organochlorine Pesticides	DeltaEnv	EPA 3510/3535/3546/3550/lab specific EPA 8081 or EPA 8290	GC – ECD GC/MS/MS LC-MS/MS HR/GC/HRMS
Organochlorine Pesticides	Bight '18 Labs	Method chosen by each lab*	Instrumentation chosen by each lab*
Polychlorinated Biphenyls	DeltaEnv	EPA 3510/3535/3546/3550/lab specific EPA 8082 or EPA 8290	GC – ECD HR/GC/HRMS
Polychlorinated Biphenyls	Bight '18 Labs	Method chosen by each lab*	Instrumentation chosen by each lab*
Polybrominated Diphenyl Ethers	DeltaEnv	lab specific for extraction EPA 1614	GC-ECD or MS HRGC/HRMS
Polybrominated Diphenyl Ethers	Bight '18 Labs	Method chosen by each lab*	Instrumentation chosen by each lab*

* See Appendix V for more details on Bight '18 QA requirements

MPSL-DFW will analyze mercury in fish tissues 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 ±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 16, and MQOs in Section 7, Table 11.

Selenium 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 ±20% of the true value, or the previous 10 samples must be reanalyzed. Two blanks, a certified reference material (NIST 2976, NRCC DORM-4 or similar), as well as a method duplicate and an MS pair, will be run with each set of samples. RLs can be found in Table 16, and MQOs in Section 7, Table 11.

Bight '18 Labs may choose any method for mercury, arsenic, and or selenium so long as the method conforms to the Bight '18 QAP (Appendix V).

Table 16. Trace metal/metalloid analytical parameters, reporting units and reporting limits (RL) in tissue

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

* See Appendix V for details on Bight '18 QA

Organochlorine and PCB compounds will be extracted and analyzed following lab-specific EPA methods. 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 17-19, and MQOs in Section 7, Table 12.

Bight '18 Labs may choose any method for metals/metalloids, PCBs and OCs so long as the method conforms to the Bight '18 QAP (Appendix V).

Table 17. Polychlorinated biphenyl analytical parameters, reporting units, and reporting limits (RL) for tissue

Polychlorinated Biphenyl congeners (by USEPA 8082 or 8290)			
PCB	**RL (ng/g wet wt)**	**PCB**	**RL (ng/g wet wt)**
PCB 008	0.6	PCB 128*	0.6
PCB 018*	0.6	PCB 137	0.6
PCB 027	0.6	PCB 138*	0.6
PCB 028*	0.6	PCB 141	0.6
PCB 029	0.6	PCB 146	0.6
PCB 031	0.6	PCB 149*	0.6
PCB 033	0.6	PCB 151*	0.6
PCB 044*	0.6	PCB 153*	0.6
PCB 049*	0.6	PCB 156*	0.6
PCB 052*	0.6	PCB 157*	0.6
PCB 056	0.6	PCB 158*	0.6
PCB 060	0.6	PCB 169*	0.6
PCB 064	0.6	PCB 170*	0.6
PCB 066*	0.6	PCB 174	0.6
PCB 070*	0.9	PCB 177*	0.6
PCB 074*	0.6	PCB 180*	0.6
PCB 077*	0.6	PCB 183*	0.6
PCB 087*	0.6	PCB 187*	0.6
PCB 097	0.6	PCB 189*	0.6
PCB 099*	0.6	PCB 194*	0.6
PCB 101*	0.9	PCB 195	0.6
PCB 105*	0.6	PCB 200	0.6
PCB 110*	0.9	PCB 201*	0.6
PCB 114*	0.6	PCB 203	0.6
PCB 118*	0.9	PCB 206*	0.6
PCB 126*	0.6	PCB 209	0.6

* Part of Bight ’18 Analyte List; Bight ’18 list also includes PCBs 037, 081, 119, 123, 167, 168. See Appendix V for more details.

Table 18. Organochlorine pesticide analytical parameters, reporting units, and reporting limits (RL) for tissue

Organochlorine Pesticides (by USEPA 8081 or 8290)		
Group	**Parameter**	**RL (ng/g wet wt)**
Chlordanes	Chlordane, cis-	1.0
	Chlordane, trans-	1.0
	Heptachlor	1.0
	Heptachlor epoxide	0.5
	Nonachlor, cis-	1.0
	Nonachlor, trans-	1.0
	Oxychlordane	1.0
DDTs	DDD(o,p')	0.5
	DDD(p,p')	0.5
	DDE(o,p')	0.5
	DDE(p,p')	1.0
	DDMU(p,p')	1.0
	DDT(o,p')	1.0
	DDT(p,p')	1.0
Cyclodienes	Aldrin	1.0
	Dieldrin	0.5
	Endrin	1.0
HCHs	HCH, alpha	0.5
	HCH, beta	1.0
	HCH, gamma	0.5
Others	Dacthal	0.5
	Endosulfan I	1.0
	Hexachlorobenzene	0.7
	Methoxychlor	1.0
	Mirex	1.0
	Oxadiazon	1.0

* Part of Bight ’18 Analyte List. See Appendix V for more details.

Table 19. Polybrominated Diphenyl Ether analytical parameters, reporting units, and reporting limits (RL) for tissue samples

Polybrominated Diphenyl Ethers (by USEPA 1614)	
PBDE	**RL ppb (ng/g wet wt)**
PBDE 017	2
PBDE 028	2
PBDE 047	5
PBDE 066	5
PBDE 085	5
PBDE 099	5
PBDE 100	5
PBDE 138	5
PBDE 153	5
PBDE154	5
PBDE 183	10

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 the holding time specific to each analyte (Table 14). In addition, results need to be reported according to the timeline outlined in Table 8.

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-DFW and DeltaEnv 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 10-12 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 20). 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. DeltaEnv SOPs are available upon request from the LD by email: timea.majoros@deltalabmail.com. Likewise, MPSL-DFW SOPs are available upon request from the LQAO by email: 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 20) 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 20. Equipment maintenance and calibration frequency

Instrument	Inspection/Maintenance Frequency	Calibration Frequency
AA (DeltaEnv)	As needed	Every batch
ICP-OES (DeltaEnv)	As needed	Calibration checks with every batch, when deviation is larger, recalibrate equipment
GC/MS or GC-ECD (DeltaEnv)	As needed	Calibration checks with every batch, when deviation is larger, recalibrate equipment
Milestone DMA-80 Direct Mercury Analyzer (MPSL-DFW)	As needed	At least once every 2 weeks
Perkin-Elmer NexION Inductively Coupled Plasma - Mass Spectrometer (MPSL-DFW)	As needed	At least once prior to each batch

16.1. Analytical Instrumentation

The following parameters are required, but the results are not submitted along with the data. It is the responsibility of each analyst to insure the instrument is in control throughout analysis.

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 3 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 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 11 and 12. 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, the LQAO will contact the PM to determine proceedings, and will flag the data and note the issue in interim and final reports.

Element 17. Inspection/Acceptance of Supplies and Consumables

All supplies will be examined for damage as they are received. Laboratory ordering personnel will review all supplies as they arrive to ensure the shipment is complete and intact. All chemicals are logged in to the appropriate logbook and dated upon receipt. All supplies are stored appropriately and are discarded upon expiration date. Table 21 indicates items that are considered for acceptance. If these items are not in compliance with the acceptance criteria, they will be returned to the manufacturer.

Table 21. Inspection/acceptance testing requirements for consumables and supplies

Project-Related Supplies (source)	Inspection / Testing Specifications	Acceptance Criteria	Frequency	Responsible Individual
Nitrile Gloves (Fisher Scientific or similar)	Carton seal is visually inspected for damage or tampering	Carton is intact and gloves within are clean and intact	At receipt date of shipment	MSPL-DFW, DeltaEnv or Bight '18 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-DFW, DeltaEnv or Bight '18 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-DFW, DeltaEnv or Bight '18 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.

All data generated by *DeltaEnv* will be maintained as described in *DeltaEnv* SOPs and the *DeltaEnv* Quality Assurance Manual (listed in Appendix I). The *DeltaEnv* 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-DFW data will be generated and maintained according to the Marine Pollution Studies Laboratory Quality Assurance Plan (Appendix I). The MPSL-DFW 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 generated by Bight '18 labs will conform to the Bight '18 QAP (Appendix V) as well as their own Quality Assurance Manuals. Each laboratory's QAO will be responsible for oversight of the metals and/or organic chemical analyses. Results will be reported to Ken Schiff or his designee at SCCWRP. He and the PM will work together to ensure results are formatted appropriately and submitted to the SWAMP Database.

All data will be entered into electronic spreadsheets that are SWAMP-compatible. Each data element is checked at a minimum by the technician who entered the data and verified by the technician's signature on the raw data sheet. 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

Preliminary reviews of QA data will be made by each LQAO prior to submission of each batch to the PM or SWAMP 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 do 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 (COC) 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 11 and 12.
- 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 are received by the LQAO in accordance with the timeline in Table 8.

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, IV and V). 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 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 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 SWAMP Statewide Project Report will be distributed to the Peer 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 SWRCB website. These documents will be generated and released in accordance with the dates listed in Table 8.

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 11 and 12. Furthermore, the final dataset as a whole will be scrutinized for usability to answer the 4 Management Questions.

Element 23. Verification and Validation Methods

Field Data will be submitted electronically through the SWAMP database. After data entry, 100% of the data entered will be checked 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. Discrepancies in laboratory data flagging noted during data verification will be communicated to the Program QAO, LQAO, and PM before loading.

All tissue data will be validated according to BOG Data Validation (Appendix IV), outlined below. Please refer to the appended document for complete descriptions and validation steps, as well as examples of potential QC failures. 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 11 and 12 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 and OCs will be summed for comparison with threshold values in Table 9. PCB and OC results analyzed in Southern California samples may differ from those summed in other regions. Please refer to Table 16 and Appendix V for details on samples analyzed by Bight '18. It is possible that some of the parameters that comprise each summation may be flagged as rejected through the Validation process (Appendix IV 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 9, 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 10), 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 (MQ) will be assessed by SFEI, with input as needed from the RWQCBs and OEHHA.

MQ1 will be assessed by comparing the average concentrations of representative fish species in popular fishing locations to the BOG-adopted thresholds listed in Table 9.

MQ2 will be assessed by comparing average concentrations of fish species within zones. More in-depth statistical analyses may be made in zones with replicate observations.

MQ3 will be assessed by comparing average concentrations measure in this cycle to those from the previous cycle and other projects with data of high level of known quality.

MQ4 will be assessed by OEHHA to determine if further sampling is needed to minimize data gaps related to advisories.

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.

Klasing, S., and R. Brodberg. 2008 (Updated ATL Table November 2017). Development of Fish Contaminant Goals and Advisory Tissue Levels for Common Contaminants in California Sport

Fish: Chlordane, DDTs, Dieldrin, Methylmercury, PCBs, Selenium, and Toxaphene (Updated ATL Table). California Office of Environmental Health Hazard Assessment, Sacramento, CA.

Smith, W., H.T. Pham, S. Klasing, M. Gassel, and S. Murphy. 2016. Statewide Advisory for Eating Fish from California Coastal Locations Without Site-Specific Advice. 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, PA.

Sun, J., J.A. Davis, S. N. Bezalel, J.R.M.Ross, A.Wong, D. Yee, R. Fairey, A. Bonnema, D.B. Crane, R. Grace, R. Mayfield, and J. Hobbs. 2017. Contaminant Concentrations in Fish from San Francisco Bay, 2014. SFEI Contribution #806. Regional Monitoring Program for Water Quality in San Francisco Bay, Richmond, CA.

Surface Water Ambient Monitoring Program Information Management and Quality Assurance Center (SWAMP IQ). 2017. The Surface Water Ambient Monitoring Program Quality Assurance Program Plan. Prepared for the Surface Water Ambient Monitoring Program, California Water Resources Control Board, Sacramento, CA. https://www.waterboards.ca.gov/water_issues/programs/swamp/qapp/swamp_QAPrP_2017_Final.pdf

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, 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. 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. 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.

US Environmental Protection Agency. 2007c. Method 8081B. Organochlorine Pesticides by Gas Chromatography. Revision 2. US Environmental Protection Agency, Washington, DC.

Appendix I. List of Associated QAPs

MPSL-DFW Laboratory QAP, Revision 7. November 2016

Delta Environmental Laboratory QAP – contact Timea Majoros for details

Appendix II. Monitoring Plan

Final

Monitoring Plan for a Second Statewide Survey of Bioaccumulation on the California Coast

Bioaccumulation Oversight Group (BOG)

Surface Water Ambient Monitoring Program

November 2018

ACKNOWLEDGEMENTS

This Sampling Plan was prepared by SFEI, MLML, and SCCWRP on behalf of the Bioaccumulation Oversight Group and the SWAMP. Substantial input to the plan was received from the BOG and the BOG Peer Review Panel.

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I. INTRODUCTION

In 2009 and 2010, the State Water Resources Control Board's Surface Water Ambient Monitoring Program (SWAMP) conducted the first systematic statewide survey of bioaccumulation of contaminants in sport fish on the entire California coast (Davis et al. 2012). This document presents a plan for sampling and analysis of sport fish in a repeat of that systematic statewide survey. This work will be performed as part of the SWAMP Bioaccumulation Monitoring Program, which is providing comprehensive monitoring of contaminant bioaccumulation in California water bodies.

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

The Roundtable formed a subcommittee, the Bioaccumulation Oversight Group (BOG), that focuses on the Bioaccumulation Monitoring Program. The BOG is composed of State and Regional Board staff and representatives from other agencies and organizations including USEPA, CDFW, OEHHA, 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 Program, 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 the statewide Bioaccumulation Monitoring Program in September 2006. Since then, SWAMP has conducted statewide surveys of bioaccumulation in California's lakes, rivers, and coastal waters. Sampling and analysis plans, technical reports, fact sheets, and other documentation of this work are available from the BOG website.

II. GENERAL ASPECTS OF THE SWAMP BIOACCUMULATION MONITORING PROGRAM

A. Addressing Multiple Beneficial Uses

Bioaccumulation in California water bodies has an adverse impact on both fishing and aquatic life beneficial uses (Davis et al. 2007). Beneficial use for fishing is affected by human exposure to elevated concentrations of bioaccumulative contaminants through consumption of sport fish. Beneficial use for aquatic life is affected by exposure of fish

and wildlife to bioaccumulative contaminants, primarily piscivorous species exposed through consumption of small fish. Different indicators are used to monitor these different types of exposure. Monitoring of status and trends in human exposure is accomplished through sampling and analyzing sport fish. Monitoring of status and trends in wildlife exposure can be accomplished through sampling and analysis of wildlife prey (small fish, other prey species) or tissues of the species of concern (e.g., bird eggs or other tissues of juvenile or adults of the species at risk).

The BOG has focused primarily on sampling that addresses the issue of bioaccumulation in sport fish and impacts on human exposure and beneficial uses for fishing, which include Commercial and Sport Fishing (COMM), Water Contact Recreation (REC-1), Native American Culture (CUL), Subsistence Fishing (FISH), Tribal Tradition and Culture (CUL), Tribal Subsistence Fishing (T-SUB), and Subsistence Fishing (SUB). This approach provides the information that the state agencies and the public consider to be of highest priority. More limited monitoring focused on evaluating the aquatic life beneficial use has also been conducted, principally via a two-year study (2012 and 2013) of mercury exposure and risk in birds in California lakes (Ackerman et al. 2015). The Ackerman et al. (2015) study also provided a tool for managers that allows estimation of risk to birds based on mercury concentrations in prey fish. Prey fish monitoring was then included as an add-on in the lake sport fish sampling that was conducted in 2014-2017, and is planned to continue in future lake sampling. SWAMP bioaccumulation monitoring has not included shellfish due to funding limitations and the generally lower risks posed by contaminants in shellfish.

B. Addressing Multiple Monitoring Objectives and Assessment Questions for Fishing Beneficial Uses

The BOG has developed an overarching set of monitoring objectives and assessment questions for a statewide program evaluating the impacts of bioaccumulation on beneficial uses for fishing (Table 1). This assessment framework is consistent with frameworks developed for other components of SWAMP, and is guiding 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 is on evaluating status and trends. Bioaccumulation monitoring is a very effective and essential tool for evaluating status, and is often the most cost-effective tool for evaluating trends. Monitoring status and trends in bioaccumulation will provide some information on sources, 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, pathways, and effectiveness of management actions.

C. Addressing Multiple Habitat Types

SWAMP has defined the following categories of water bodies:

- lakes and reservoirs;
- bays and estuaries;
- coastal waters;
- large rivers;
- wadeable streams; and
- wetlands.

Due to their vast number, high fishing pressure, and a relative lack of information on bioaccumulation (Davis et al. 2007), lakes and reservoirs were identified as the highest priority for monitoring and were sampled in 2007-2008 (Davis et al. 2010). Coastal waters, including bays and estuaries, were selected as the next priority, due to their importance for sport fishing and a relative lack of past monitoring, and were sampled in 2009-2010 (Davis et al. 2012). In 2011, SWAMP conducted a statewide survey of bioaccumulation in California rivers and streams (Davis et al. 2013). Beginning with the inception of a long-term monitoring program for bass lakes in 2015, the Bioaccumulation Monitoring Program has established an overarching long-term plan for providing updated information on the status of lakes, the coast, and rivers and streams. Water bodies where obtaining updated information on the status of bioaccumulation is a high priority (such as bass lakes and the coast), are being sampled on a 10-year cycle, and other water bodies are generally being sampled on a 20-year cycle (Table 2). The plan does not include sampling of wadeable streams and wetlands because of the limited amount of human exposure from fishing in these habitats.

Some subcategories have or will have a need for annual monitoring. The Delta Regional Monitoring Program (Delta RMP), for example, has identified a need for annual sampling of black bass (a term encompassing largemouth, smallmouth, and spotted bass) in the Sacramento-San Joaquin River Delta to provide a baseline and track trends in support of the Delta Methylmercury TMDL (item 7 in Table 2). Similarly, reservoirs where actions are taken as part of the statewide mercury TMDL will need to be monitored on an annual basis to determine the effectiveness of actions that are taken to reduce mercury bioaccumulation (item 3 in Table 2).

A need for monitoring of sites within San Francisco Bay on a five-year cycle has been identified and is being met by the Regional Monitoring Program for Water Quality in San Francisco Bay (Bay RMP) (Sun et al. 2017; item 11 in Table 2).

For water bodies where bioaccumulation has been determined to be a concern, a 10-year cycle for providing updated information on status would be a practical minimum revisit frequency. This frequency of sampling represents a provides a low but valuable level of information on potential changes in food web bioaccumulation. The information generated from these updates will be useful to the state and regional boards in impairment assessments and 303(d) list updates, and to OEHHA for updating consumption advice.

Other subcategories of water bodies have been shown to generally be of lower concern with respect to bioaccumulation. These water bodies should still be revisited periodically, but can be revisited less frequently than the water bodies with contamination problems. Chief among these are numerous lake and river sites where trout have been sampled and found to have low concentrations of contaminants. A 20-year cycle would be reasonable, given limited resources for monitoring, for these water bodies.

Some of the monitoring that is needed will be provided by other programs. For example, the Delta RMP, Bay RMP, and the TMDL program are expected to provide the sampling that is needed on one-year and five-year cycles. For water bodies covered by these programs, more frequent sampling will provide a stronger basis for tracking trends and for obtaining information on trends in a shorter time-frame to support management. An appropriate role for SWAMP is to address the needs that are not being covered by other programs.

Lakes with black bass account for a large number and proportion of the water bodies that are not being covered by other programs and need to be sampled at a 10-year frequency. The regional boards have identified a group of 187 priority bass lakes that are being monitored by SWAMP on a 10-year cycle, via a rotating panel design with five panels and sampling occurring every other year (Bioaccumulation Oversight Group 2015; item 1 in Table 2).

Obtaining updated information on the status of bioaccumulation on the coast has been identified as a high priority in the SWAMP Bioaccumulation Monitoring Program, with sampling to be conducted on a 10-year cycle. Fishing pressure on the coast is high, and contaminant concentrations at levels of concern are widespread.

III. DESIGN OF THE COASTAL WATERS SURVEY

A. Management Questions for this Survey

Four management questions have been articulated to guide this second SWAMP survey of the status of bioaccumulation in sport fish on the California coast. These management questions are essentially the same as the questions that guided the initial screening effort, with a revised wording of the first management question.

Management Question 1 (MQ1)
Status: What is the status of contaminants in representative fish species in popular fishing areas?

Answering this question is critical to determining the degree of impairment of the fishing beneficial use (formally designated as "ocean, commercial, and sport fishing" and abbreviated as "COMM") along the coast due to bioaccumulation. Six other beneficial uses can also apply to fishing: Water Contact Recreation (REC-1), Native American Culture (CUL), Subsistence Fishing (FISH), Tribal Tradition and Culture (CUL), Tribal Subsistence Fishing (T-SUB), and Subsistence Fishing (SUB). This question places emphasis on characterizing the status of the fishing beneficial use through monitoring of the predominant pathways of exposure – representative fish species and popular fishing areas. This focus will provide information on the resources that water quality managers and people care most about.

The data needed to answer this question are average concentrations in representative fish species from popular coastal fishing locations. Inclusion of as many species as possible is important to understanding the nature of impairment in any areas with concentrations above thresholds. In many areas, some fish species may be safe for consumption while others are not, and this is valuable information for anglers. Monitoring species that accumulate high concentrations of contaminants ("indicator species") is valuable in answering this question: if concentrations in these species are below thresholds, this is a strong indication that an area has low concentrations.

Management Question 2 (MQ2)
Regional Distribution: What is the distribution of contaminant concentrations in fish within regions?

Answering this question will provide information that is valuable in formulating management strategies for observed contamination problems. This information will allow managers to prioritize their efforts and focus attention on the areas with the most severe problems. Information on regional distribution will also provide information on sources and fate that will be useful to managers.

This question can be answered with different levels of certainty. For a higher and quantified level of certainty, a statistical approach with replicate observations in the spatial units to be compared is needed. In some cases, managers can attain an adequate

level of understanding for their needs with a non-statistical, non-replicated approach. With either approach, reliable estimates of average concentrations within each spatial unit are needed.

Management Question 3 (MQ3)
Trends: What are the trends in contaminant concentrations in representative fish species in popular fishing areas?

Information on trends is essential to effective management of contaminants that bioaccumulate in sport fish. It is critically important to know whether the problem is getting better or worse; in other words, whether food web mercury concentrations, for example, are trending up or down on a local, regional, and statewide scale. Understanding trends at a statewide scale is essential to understanding trends at local and regional scales. A statewide increasing trend could obscure the beneficial effects of local or regional management actions to reduce bioaccumulation. On the other hand, a statewide declining trend could give the impression that local or regional actions are more effective than they actually are.

Mercury concentrations in fish are the most widespread concern, and the need for information on mercury trends is especially great. Food web mercury might 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 climate change. 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). Hypothesized causes of these regional trends include global atmospheric emissions, climate change, invasive species, and changes in food web structure. On the coast, shifts in ocean circulation (e.g., current patterns and upwelling) could also drive changes in food web mercury.

The data needed to answer this question are measurements that are repeated over time of average concentrations in indicator species at popular fishing areas. Striving for consistency in the sampling design (e.g., species and locations within zones) over time will maximize the utility of the data for long-term trend analysis. With a 10-year cycle for coastwide sampling, this approach will establish a foundation for and slowly build a long-term time series for trend evaluation.

Information on trends is being generated in a more timely manner for San Francisco Bay by a related program - the Regional Monitoring Program for Water Quality in San Francisco Bay (RMP) (Sun et al. 2017). That program samples every five years and has been in place since 1994.

Management Question 4 (MQ4)
Need for Further Sampling: Should additional sampling of bioaccumulation in sport fish (e.g., more species or larger sample size) in an area be conducted to develop more comprehensive consumption guidelines?

Consumption guidelines provide a mechanism for reducing human exposure to bioaccumulated contaminants in the short term. Based largely on the data generated in the SWAMP coastal survey of 2009-2010, OEHHA issued a statewide consumption advisory for the entire coast in 2016 (Smith et al. 2016). In developing consumption advice, 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 those species. The diversity of species on the coast demands a relatively large effort to characterize interspecific variation. The present round of coastal sampling will address data gaps identified by OEHHA in the process of developing the statewide coastal advisory. After the results of this round are reviewed, OEHHA will be able to further refine the list of data gaps related to advisory development.

Overall Approach

The overall approach to be taken to answer these four questions is to perform a statewide survey of bioaccumulation in sport fish on the California coast. Answering these questions will provide an updated assessment for decision-makers to understand the scope of the bioaccumulation problem and will provide regulators with information needed to establish priorities for both cleanup actions, if appropriate, and further development of consumption guidelines.

It is anticipated that this screening study may lead to more detailed follow-up investigations of areas where consumption guidelines and cleanup actions are needed. For example, an outcome of the 2009-2010 survey is the Strategy for a Healthy San Diego Bay, which was prompted in part by findings from SWAMP monitoring. Funding for these follow-up studies will come from other local or regional programs, rather than the statewide monitoring budget.

B. Coordination

Through coordination with other programs, SWAMP funds for this survey will be highly leveraged to achieve a much more thorough statewide assessment than could be achieved by SWAMP alone.

First, this effort will be closely coordinated with Bight '18, a comprehensive regional water quality monitoring program for the Southern California Bight (SCB). Every five years, dischargers in the SCB collaborate to perform this regional monitoring. Sport fish tissue monitoring is one element of the Bight Program that is conducted on a 10-year cycle. The collaborative approach taken in 2009 for the Bight will be followed again in 2018, with SWAMP providing sample collection and some of the chemical analyses, and the Bight Program providing a substantial amount of additional chemical analyses. The Bight Program will contribute over $200,000 worth of analytical work (analysis of polychlorinated biphenyls [PCBs], organochlorine pesticides [OCHs], and arsenic, in addition to supplemental analysis of mercury and selenium) to the joint effort. An extensive laboratory intercalibration will be another valuable contribution from the Bight Program that will promote consistency and comparability of data generated by the many labs contributing to the Bight Program and other coastal monitoring by SWAMP and other programs.

The SWAMP survey will also be coordinated with intensive sampling in San Francisco Bay by the Bay RMP. The Bay RMP conducts thorough sampling of contaminants in sport fish in the Bay on a five-year cycle (see Sun et al. [2017] for the latest results), with the next round occurring in 2019. This sampling has been conducted since 1994. The Bay RMP will provide complete and thorough coverage of the Bay, with no additional effort by SWAMP needed. Bay RMP monitoring will include an extended analyte list that includes polybrominated diphenyl ethers (PBDEs), perfluoroalkyl and polyfluoroalkyl substances (PFASs), dioxins, microplastic, and possibly other contaminants of emerging concern. The Bay RMP will benefit from this collaboration by SWAMP providing a statewide dataset and interpretive report that will serve as a valuable context for RMP data. The Bay RMP effort represents an additional $380,000 of sampling, analysis, data management, and reporting.

In addition, three regional Water Boards are providing supplemental funds for the Bight sampling. The Region 4 Water Board will supplement the statewide survey with $54,000 to provide for more thorough coverage of the SCB, specifically inclusion of sharks and beach seining of surfperch. The Region 8 Water Board is contributing another $7,000 for general support of the effort. The Region 9 Water Board is contributing another $8,000 for additional analyses of species collected in the Region, specifically PCBs, OCHs, and PBDEs.

In all, these collaborations are greatly increasing the resources available for conducting this round of the coastal bioaccumulation survey. Each of the collaborating programs will benefit from the consistent statewide assessment, increased information

due to sharing of resources, and efforts to ensure consistency in the data generated by the programs (e.g., analytical intercalibration).

The Bight group and the Bay RMP each have committees that provide oversight of these long-term monitoring programs and a history of monitoring in their regions. Consequently, the sampling design in each of these regions will vary in minor ways from the design for the rest of the state. More information on these programs and the specific designs for these regions is provided in Section L.

C. Phased Approach

This survey is being conducted over two rounds to allow coverage of the entire coast with available funds. In 2018, sampling will focus on the SCB (Water Board Regions 4, 8, and 9, and some of Region 3 – Figure 1). This will allow for coordination with Bight '18. In 2019, SWAMP will be conducting the third round of sampling for the long-term bass lake program and will also sample some coastal zones, and San Francisco Bay will be sampled by the Bay RMP. In 2020, SWAMP will sample the remaining coastal zones in the central and northern regions of the state (Regions 1, 2, and 3) (Figure 2) and any other remaining areas not covered in 2018.

A final report will present the data for all of the coastal zones sampled over the 2018-2020 period and provide a comprehensive assessment of the entire two-year coast-wide dataset (including the RMP data).

D. Spatial Considerations

California has over 3,000 miles of coastline that spans a diversity of habitats, fish populations, and dense human population centers with a multitude of popular fishing locations. Sampling this vast area with a limited budget is a challenge.

The approach being employed to sample this vast area is to divide the coast into 65 spatial units called "zones" (Figure 3). This was the approach taken for the 2009-2010 survey (Bioaccumulation Oversight Group 2009, Davis et al. 2012), and the same zones are being used for the present survey. All zones will be sampled, making a probabilistic sampling design unnecessary.

The sampling will be focused on nearshore areas, including bays and estuaries, in waters not exceeding 200 m in depth, and mostly less than 60 m deep. These are the coastal waters where most of the fishing occurs.

Several criteria were considered in drawing the boundaries of the zones.

1. Fishing pressure. Zones are smaller and more numerous in areas with more fishing pressure to provide a better characterization of human exposure. The location of fishing piers and other fishing access points was an important factor in zone delineation. On the other hand, the zones are larger in remote areas with little fishing activity.

2. Even distribution. To ensure coverage of the entire coast, the zones are generally spread evenly throughout, with adjustments made for fishing pressure as described above.
3. Homogeneity of contamination. Land use and hydrology were considered in drawing boundaries to reflect known patterns of contamination.
4. Stakeholder interest. The boundaries were reviewed by stakeholders (Water Board representatives, stakeholders in the Bight Group) and modified according to their needs.

Popular fishing locations were identified from Jones (2004) and discussions with stakeholders. Zones were developed in consultation with Water Board staff from each of the nine regions, Bight Group stakeholders, and the BOG.

Additional detail on sampling locations in San Francisco Bay is provided in Figure 4.

E. Sampling Design Within Each Zone

1. Species Targeted

Selecting fish species to monitor on the California coast is a complicated task due to the relatively high diversity of species, regional variation over the considerable expanse of the state from north to south, variation in habitat and contamination between coastal waters and enclosed bays and harbors, and the varying ecological attributes of potential indicator species. For the original statewide survey in 2009-2010 (Davis et al. 2012) the list of possibilities was narrowed down by considering the following criteria, listed in order of importance.

1. Popular for consumption
2. Sensitive indicators of problems (accumulating relatively high concentrations of contaminants)
3. Widely distributed
4. Species that accumulate relatively low concentrations of contaminants (i.e., species that are better choices for consumption)
5. Represent different exposure pathways (benthic versus pelagic)
6. Continuity with past sampling

Information relating to these criteria is presented below. Continuity with past sampling was a higher priority in this round, given the precedent set by the 2009-2010 survey.

The BOG elected not to include shellfish in this survey, due to the limited budget available and the lower consumption, lower risks to human health, and the added expense that would be required to collect shellfish. An additional consideration is that the analysis of shellfish for methylmercury (rather than total mercury) would be required for a meaningful assessment. Determination of methylmercury is more labor-intensive and costly than determination of total mercury.

Popular for Consumption

As recommended by USEPA (2000) in their document "Guidance for Assessing Chemical Contaminant Data for Use in Fish Advisories," the primary factor considered in selecting species to monitor in 2009-2010 was a high rate of human consumption. Good information on recreational fish catch was available from the Recreational Fisheries Information Network (RecFIN), a product of the Pacific States Marine Fisheries Commission (PSMFC). Established in 1992, RecFIN is designed to integrate state and federal marine recreational fishery sampling efforts into a single database to provide important biological, social, and economic data for Pacific coast recreational fishery biologists, managers and anglers. Fish catch data are available at:https://www.recfin.org/.

Tables 3 and 4 show data for two broad regions (southern and northern California) and specific data for the coast (ocean < 3 mi) and bays and harbors. Data include mass of catch in tonnes and counts in thousands. The mass and catch data were ranked for each region, then the ranks for each species were averaged to obtain an average rank. The average rank was used as the index of popularity for fish consumption. For example, in southern California coastal waters, chub mackerel was the most popular species in both coastal (within three miles of shore) and inland (bay and harbor) waters. The popular species varied between the two regions of the state (south and north) and between coastal waters and bays and harbors.

Primary and secondary target species were selected based on a combination of continuity with past sampling, continuity of sampling across the state, and popularity according to the RecFIN data.

For the SCB, the Bight Program identified kelp bass, chub mackerel, and white croaker as primary target species. These species were also the primary target species in the 2009 Bight survey, and have a combination of popularity for consumption, a broad spatial distribution that allows comparisons across Bight zones, and historic data from an extensive survey by NOAA in 2002 (NOAA 2007) and outfall monitoring going back to the early 1970s for kelp bass and the 1990s for white croaker (Davis et al. 2011). Other primary target species for the Bight were selected either as mercury indicator species, as species with wide distributions in the Bight, as species that are distributed throughout the state (allowing for comparisons among regions), or due to a combination of these attributes. Secondary species were selected using the same criteria, but generally are less abundant and have less extensive spatial distributions.

For bays and harbors in the Central Coast, maintaining long-term time series for species that have been sampled in San Francisco Bay (shiner surfperch, California halibut, white croaker, jacksmelt, and striped bass) was an important consideration. Aside from that, the general criteria of continuity with past sampling, continuity of sampling across the state, and popularity according to the RecFIN data drove the selection of species for the Central Coast and the North Coast.

The actual catch information from the 2009-2010 coastal survey was another major consideration in species selection for this round of sampling. Tables 5a-c provide a summary of which species were caught in each zone (as well as their mercury concentrations).

Sensitive Indicators

While catch data were the primary determinant of the list of target species, some adjustments were made to ensure an appropriate degree of emphasis on sensitive indicators of contamination. USEPA (2000) also recommends consideration of this (expressed as "the potential to bioaccumulate high concentrations of chemical contaminants") as a criterion of major importance. Including these species is useful in assessing the issue of safe consumption (contained in MQ1) – if the sensitive indicator species in an area are below thresholds of concern then this provides an indication that all species in that area are likely to be below thresholds.

Different contaminants have different mechanisms of accumulation; therefore a combination of species is needed to ensure inclusion of the appropriate sensitive indicators. Methylmercury biomagnifies primarily through its accumulation in muscle tissue; predators such as sharks tend to have the highest methylmercury concentrations. In contrast, the organic contaminants of concern also biomagnify, but primarily through accumulation in lipid. Concentrations of organics are therefore also influenced by the lipid content of the species, with species that are higher in lipid having higher concentrations. Species such as white croaker tend to have high lipid concentrations in their muscle tissue, and therefore usually have relatively high concentrations of organics. Other factors in addition to lipid content are also important for some organics. Trophic position and age are important for hydrophobic pollutants such as the highly chlorinated PCBs (including the major ones like PCBs 153, 138, and 180). Most studies show that there is lifetime accumulation of high log K_{ow} organohalogen compounds that are not metabolized. Sex may also be influential since the sole mechanism of excretion may be egg production in females (Ross Norstrom, personal communication; Niimi 1983, Miller 1993).

Consequently, target species in this study will include both high-lipid species such as croaker and surfperch, and long-lived predators that accumulate mercury such as kelp bass, rockfish, and sharks. These considerations had an influence on the target species list. For example, white croaker has a high potential for accumulation of organics and has been sampled extensively in past studies in both southern California and San Francisco Bay. Therefore, even though white croaker did not quite make the list of the top five most popular species in these areas, it was still included as a primary target.

Spatial Distribution

Consideration in selection of target species was also given to their spatial distribution to provide better information for answering MQ2 (regional distribution).

This is also recommended as an important criterion to consider by USEPA (2000). Due to interspecific variation in bioaccumulation, the availability of consistent species across the spatial units of interest is critical to maximizing information obtained on spatial patterns. The sampling design complies with this criterion as much as possible, with primary consideration given to the two criteria described previously. The catch data from 2009-2010 (Tables 5a-c) were valuable in assessing the likely spatial distribution of target species.

Continuity with Past Sampling

Given the information gained from the 2009-2010 survey on the availability of species with our collection methods, and the interest in building long-term time series to track trends (to answer MQ 3), continuity with past sampling was an important consideration. The information summarized in Tables 5a-c was therefore considered in the target species selection process.

Other Factors

Other factors were considered but did not have a major influence on the design due to the limited resources available.

- Species with relatively low concentrations of mercury and PCBs. Provide information useful in developing safe eating guidelines. More focused effort to obtain information on these species is left to future studies. One exception to this is blue rockfish, which are an abundant species that the 2009-2010 survey showed to have low concentrations of mercury and organics. Blue rockfish were included as a primary target species in the current survey.
- Different exposure pathways (benthic versus pelagic). This was not deemed a high priority with the limited budget.

The Target Species

Table 6 shows the lists of primary and secondary species for each region and stratum (coastal waters versus bays and harbors) based on the considerations discussed above. The available budget will allow for analysis of five species per zone. Table 6 shows more than five primary targets for each stratum in some cases, with the expectation (based on the prior survey) that the distributions of some of the primary target species will be patchy.

A summary of basic ecological attributes of the primary and secondary target species is presented in Table 7. This information will be useful in performing spatial comparisons in cases where it was not possible to collect the same species in the spatial units to be compared. In these cases, comparisons may be evaluated for species from the same guilds and with similar attributes. Information on each species was gathered from FishBase (http://www.fishbase.org/), CDFW's Marine Sportfish Identification website (https://www.wildlife.ca.gov/Fishing/Ocean/Fish-ID), Oregon State University's Marine Species with Aquaculture Potential

(http://hmsc.oregonstate.edu/projects/msap/index.html), and discussions with Jim Allen of the Southern California Coastal Water Research Project (SCCWRP) (personal communication). Species were classified into guilds based on prey items, foraging type, and habitat to identify different species along the coast with similar exposure pathways.

2. Sampling Sites

Within each zone, specific sites will be selected for sample collection. Where possible, the same sites that were sampled in 2009-2010 will be re-sampled. Criteria considered in determining the placement of sampling sites include the existence of discrete centers of fishing activity, road or boat ramp access, known patterns of spatial variation in contamination or other factors influencing bioaccumulation, and possibly other factors. The primary emphasis will be on sampling in areas that are popular for fishing. Popular fishing areas were identified through published sources (e.g., Jones [2004]) and consultation with agency staff, as described above. Sampling areas were adjusted to avoid collection in newly established MPAs where fishing is not open to the public, consistent with the goal of characterizing exposure of the fishing public.

3. Replication

There will be no replication of sites within a zone. If the sampling crew is unable to obtain sufficient samples at the first site sampled, they will move to the next site where fishing pressure is high and they are likely to obtain the needed samples. Therefore there is not a fixed number of sites within each zone.

Replicate composite samples within each zone will be collected in the SCB and San Francisco Bay. In the SCB, the Bight Group is making resources available for analyzing three replicate composites of kelp bass, white croaker, and chub mackerel within each zone. These are not necessarily site replicates, however – the replicates can be collected from a single site, if that is possible, or from multiple sites if that is necessary. These are simply multiple replicate composites of the target species from a given zone. This same basic approach will be followed in San Francisco Bay, but the Bay will be divided relatively finely into five zones (Figure 4).

In the North and Central coasts, there will be only one composite sample (compositing is discussed further below) or one set of 10 individuals (for mercury indicator species) collected for each species in each zone. With the limited resources available, it is considered a higher priority to obtain information on different species than to attempt to provide a stronger basis through replication for statistical spatial comparisons among zones. It is recognized that this will make data interpretation less conclusive. Mercury will be analyzed for each of the five species collected per zone. PCBs will be analyzed in the two species with the strongest tendency to accumulate organics.

4. Size Ranges and Compositing for Each Species

Chemical analysis of trace organics is relatively expensive, and the management questions established for this survey can be addressed with good information on average concentrations, so a compositing strategy will be employed for these chemicals. Compositing yields stronger estimates of average concentration by the inclusion of multiple individuals (usually a target of five) in the composite sample.

Chemical analysis of mercury is much less expensive, and mercury concentrations are known to be closely correlated with fish size in many species. Collecting data on mercury concentrations in individual fish can provide a basis for statistical analysis to evaluate spatial or temporal patterns in a manner that takes the influence of fish size into account, an approach that has been used in all of the SWAMP sport fish surveys. Consequently, the sampling design includes analysis of mercury in individual fish for selected mercury indicator species. For the mercury indicator species, an analysis of covariance (ANCOVA) approach will be employed, in which the size:mercury relationship will be established for each location and an ANCOVA will allow the evaluation of differences in slope among the locations and the comparison of mean concentrations and confidence intervals at a standard length, following the approach of Tremblay et al. (1998). Experience applying this approach throughout the state indicates that, to provide robust regressions, 10 fish spanning a broad range in size are needed (Davis et al. 2003, Davis et al. 2008).

The 10 individual fish are divided into specific size ranges targeted for each species (Table 8). The size ranges are based on the sizes obtained in the 2009-2010 survey, and legal limits for the species where such limits are in place.

Target size ranges for the composites were based on calculation of a range using the USEPA (2000) "75% rule" (i.e., the recommendation that the smallest fish in a composite should be no less than 75% of the size of the largest fish), with one modification. OEHHA has indicated that they can accept fish in a composite that are larger than the upper size limit (derived from combining the minimum legal size with the 75% rule) –inclusion of larger fish in this manner will provide a more conservative estimate of human exposure for advisory development. Using these size guidelines will provide some control over the size and age of the fish that are analyzed, and will generate data that are more comparable over time and space.

Species with a high trophic position, relatively high mercury concentrations, and a relatively broad distribution in the 2009-2010 survey were selected for analysis of mercury in individual fish, as indicated in Table 8. The size ranges for these species were based on targeting the median of the size range in the same manner as the composites, but then also targeting fish in two smaller size ranges to provide a better basis for establishing a length versus mercury regression line.

In cases when more than five fish of one species are collected in a zone, composites will be created using the following guidelines:

1. Size: The five largest fish will be used for composites.
2. Location: Fish collected from different locations within a zone will be distributed among composites.
3. Date of Catch: Fish collected at the same or different locations on different days will be distributed among composites. This guideline will take a higher priority on fish known to be active swimmers such as mackerel.
4. Mode of Catch: Fish collected via different methods, such as hook and line, seine, or pole spear, will be distributed among composites.

When both individuals and composites are collected for a target species, the composites will be prepared from the same fish that are analyzed individually.

The sampling crew will report 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.

F. Sample Processing and Analysis

Upon collection, each fish collected will be tagged with a unique ID. 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. In addition, it is the measurement fishers and wardens use to determine whether a fish is legal size. Determining fork length at the same time is valuable because fork length is a more reliable indicator of size that is not affected by fin erosion or net damage.

Whole fish will be wrapped in aluminum foil and frozen on dry ice for transportation to the laboratory, where they will be stored frozen at -20° C. Fish will be kept frozen wrapped in foil until the time of dissection. Dissection and compositing of muscle tissue samples will be performed following USEPA guidance (USEPA 2000). At the time of dissection, fish will be placed in a clean lab to thaw. After thawing, fish will be cleaned by rinsing with ASTM Type II water, and handled only by personnel wearing polyethylene or powder-free nitrile gloves (glove type is analyte dependent). All dissection materials will be cleaned by scrubbing with Micro® detergent, rinsing with tap water, de-ionized (DI) water, and finally ASTM Type II water.

Composites will be created based on the size considerations discussed in Section E.4.. In general, fish will have the skin dissected off, and only the fillet muscle tissue will be used for analysis. This is inconsistent with the guidance of USEPA (2000) that recommends that fish with scales have the scales removed and be processed with skin on, and that skin is removed only from scaleless fish (e.g., catfish). The BOG is aware of this difference, but favors skin removal. Skin removal has been consistently used in past monitoring by SWAMP and earlier programs, and is also the preparation technique recommended by OEHHA. In addition, muscle without skin is what people commonly consume, even if the fish is prepared with intact skin, bones, and scales. Processing fish

with the skin on is very tedious and results in lower precision because the skin is virtually impossible to homogenize thoroughly. Also, skin-on preparation actually dilutes the measured concentration of mercury because there is less mercury in skin than in muscle tissue, and mercury is the most ubiquitous fish contaminant in California that leads to most of our advisories. 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. The analysis of axial fillets without skin was also advised by a national workgroup concerning the monitoring and analysis of mercury in fish (Wiener et al. 2007). Shiner surfperch samples will be an exception to this rule, as they are too small for skin removal. Procedures used in past monitoring (removing heads, tails, and viscera; leaving muscle with skin and skeleton to be included in the composites as in the Bay RMP) will be used.

Moss Landing Marine Laboratory (MLML) and Delta Environmental Laboratories (DeltaEnv) will be the primary labs analyzing samples for the second coastal survey. MLML, the SWAMP lab for trace element analysis, will analyze mercury and selenium in samples from all zones. DeltaEnv is the new SWAMP lab for trace organic analysis, and will analyze samples from zones in the Central and North coast regions.

Analyses for the SCB zones will be performed by a consortium of labs that participate in regional monitoring for the Bight Program. The Bight labs will analyze trace elements (mercury, selenium, and arsenic) and trace organics (PCBs, DDTs, chlordanes, and dieldrin). Arsenic and the legacy pesticides (DDTs, chlordanes, and dieldrin) will be analyzed only in the Bight zones (see Section G for explanation).

With a large number of labs involved, ensuring comparability is essential to generating a meaningful overall dataset. A major intercalibration exercise is being conducted to assess and achieve comparability. All of the labs that are analyzing coastal fish samples for the statewide survey are participating, along with other labs that are seizing this opportunity to assess and improve their performance. Appendix 1 summarizes the intercalibration, including the labs participating, the analytes, and other details of the exercise.

The Bight labs will be using a variety of methods. The Bight lab methods will be documented when the results of the Bight sampling are reported. The methods to be used by the SWAMP labs are summarized here.

MLML will analyze mercury 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 ±20% of the true value, or the previous 10 samples

must be reanalyzed. Three blanks, a standard reference material (DORM-4), as well as a method duplicate and a matrix spike pair will be run with each set of samples.

MLML will digest samples for selenium analysis according to USEPA Method 3052M, "Microwave Assisted Acid Digestion of Siliceous and Organically Based Matrices," modified, and analyze them according to USEPA Method 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 CCV values must be within ±20% of the true value, or the previous 10 samples must be reanalyzed. Two blanks, a standard reference material (2976 or DORM-4), as well as a method duplicate and a matrix spike pair will be run with each set of samples.

Organics analyses in the Central and North regions will be performed by DeltaEnv in Benicia, CA. Organochlorine pesticides will be analyzed according to USEPA Method 8081A, "Organochlorine Pesticides by Gas Chromatography." PCBs and PBDEs will be analyzed according to USEPA Method 8082, "Polychlorinated Biphenyls (PCBs) by Gas Chromatography."

Analysis of split samples and additional replicates for organics in the SCB will be performed by the labs that participate in Bight monitoring (see Section L below).

G. Analytes

Table 9 provides a summary of the analytes that will be measured on a wet weight (ww) basis by the SWAMP labs. Analytes and reporting limits that will apply for all of the Bight labs are listed in Table A2 of Appendix 1. Detailed analyte lists for the Bay RMP will be presented in the Sampling and Analysis Plan for that effort, to be prepared in early 2019.

Additional details on the analytes are provided below.

Ancillary Parameters

Ancillary parameters to be measured in the lab for all samples include moisture and lipid content (Table 10). Fish sex will be determined for all samples.

Methylmercury

Methylmercury is the contaminant of greatest concern with respect to bioaccumulation on a statewide basis. Based on the 2009-2010 survey (Davis et al. 2012), methylmercury is expected to continue to exceed thresholds of concern in many species and zones. Methylmercury will be measured as total mercury. Nearly all of the mercury present in edible fish muscle is methylmercury, and analysis of fish tissue for

total mercury provides a valid, cost-effective estimate of methylmercury concentration. Mercury will be analyzed in all samples.

Selenium

Selenium was analyzed in all species in the 2009-2010 survey and was found to be of low concern with regard to human exposure, exceeding only the OEHHA seven serving per week advisory tissue level (ATL) of 1 ppm (Klasing and Brodberg 2008) in 5 of the 69 zones (Davis et al. 2012). In recent years SWAMP has been analyzing selenium in all composites of fish samples that were analyzed for mercury, to provide information that could be used to take into account the interactive effect of selenium on mercury risk. This approach will be followed for this round of the coastal survey as well.

Arsenic

Arsenic was not considered a great enough concern to be included in the 2009-2010 coastal survey or in recent monitoring in San Francisco Bay. A small number of crab, clam, and white sturgeon samples were analyzed by the RMP in 2000 (Greenfield et al. 2003). Some of the dischargers in the Bight have monitoring requirements for arsenic in their discharge permits, so total arsenic will be analyzed by Bight labs in the primary target species for the Bight. Inorganic arsenic is the form of toxicological concern (USEPA 2000), but total arsenic will be measured as a conservative index of concentrations in Bight fish.

PCBs

PCBs are the contaminant of second-greatest concern with respect to bioaccumulation on a statewide basis (Davis et al. 2012). PCBs will be analyzed using congener-specific methods. A total of 53 congeners will be analyzed by the SWAMP lab (Table 10). Bight labs will analyze 39 congeners (listed in Table A2 of Appendix 1). The RMP lab will analyze all 209 congeners. Direct comparisons of sums of PCBs from the different labs will be based on the sum of the 39 Bight congeners. PCBs will be analyzed in three or four of the five composite samples in the Bight, in multiple species in San Francisco Bay, and in composites from two species per zone in the Central and North regions.

Legacy Pesticides

In the 2009-2010 survey (Davis et al. 2012), legacy pesticides exceeded thresholds of concern in a very small percentage of species and zones. However, legacy pesticides are a more significant concern in the Bight due to historical contamination. Legacy pesticides will be analyzed in all composite samples in the Bight only. The list of analytes to be included is provided in Table A2 of Appendix 1.

PBDEs

Intensive monitoring in San Francisco Bay showed that PBDEs were a rising concern in the early 2000s, but concentrations have since declined substantially (Sutton et al. 2014); as of 2014 all fish samples were well below even the lowest OEHHA ATL for seven servings per week (45 ppb) (Sun et al. 2017). PBDE monitoring will continue in the Bay in 2019, but is generally not a priority for other coastal zones. The one other exception is San Diego Bay, where samples will be analyzed for PBDEs using funding from Region 9.

Polychlorinated Dioxins and Dibenzofurans

Few data are available on polychlorinated dioxins and dibenzofurans (hereafter referred to as "dioxins") in California sport fish. The best dataset exists for San Francisco Bay, where sampling from 1994-2014 has indicated that concentrations in high-lipid species frequently exceed a published screening value of 0.14 pptr toxic equivalents (TEQs; for dioxins and furans only) (Sun et al. 2017). However, there are no known major point sources of dioxins in the Bay Area, and the concentrations measured in the Bay are comparable to those in rural areas of the US. OEHHA did not include dioxins in their development of advisory tissue levels for priority contaminants due to the lack of data for dioxins in fish throughout the state (Klasing and Brodberg 2008). Given the relatively high cost of dioxin analysis and these other considerations, dioxins generally are not included in this survey. Dioxins are considered a higher priority by the Bay RMP, so these analytes will be included for high-lipid species (white croaker and shiner surfperch) in San Francisco Bay. The RMP will analyze dioxins and dibenzofurans, but not coplanar PCBs. Analysis of dioxins and dibenzofurans has also been identified as a high priority for Humboldt Bay, so samples for Humboldt Bay will be analyzed for these chemicals. The Humboldt Bay samples will be collected in 2019 if possible (instead of 2020), the same year as the RMP, so the samples can be analyzed in the same batch with the San Francisco Bay samples.

PFAS

Perfluoroalkyl and polyfluoroalkyl substances (PFAS) have been monitored in San Francisco Bay fish since 2009 and have reached a level of some concern. All perfluorooctane sulfonate (PFOS) concentrations measured in the Bay in 2014 were well below the Minnesota one serving/week consumption threshold of 40 ppb ww. However, concentrations measured in a largemouth bass sample (14 ppb) and two striped bass samples (17 ppb and 13 ppb) collected near a major wastewater outfall fell within the Michigan eight meals per month consumption range (>13-19 ppb). OEHHA has not established ATLs for PFOS in California fish. The Bay RMP will continue to monitor PFOS and other PFASs in 2019. If levels in the Bay continue to suggest some degree of concern, monitoring of these chemicals more widely will be considered.

Other Emerging Contaminants

Other emerging contaminants are likely to be present in California sport fish. Early detection of increasing concentrations of emerging contaminants can be very valuable for managers, as evidenced by the example of PBDEs in San Francisco Bay. San Francisco Bay and Southern California (via SCCWRP) have very active emerging contaminant monitoring programs. Having these programs lead the way in assessing emerging contaminants, and then considering additions to statewide monitoring based on these pilot studies, is a cost-effective approach for monitoring emerging contaminants on the California coast, and for California water bodies more generally. Other emerging contaminants that may be included in RMP monitoring in 2019 include microplastic, the pesticide fipronil and its degradates, oxybenzone and other sunscreen ingredients, and non-targeted emerging contaminant analysis. Archives of each composite will be retained and available for analysis of emerging contaminants for five years after sample collection (Section I).

H. Quality Assurance

This effort will adhere to the quality assurance requirements established for the SWAMP. A Quality Assurance Project Plan specific to this effort is in preparation (Bioaccumulation Oversight Group 2018).

As discussed in Section F, one of the analytical challenges in this project will be coordinating among different laboratories that will be generating organics data. An intercalibration exercise is planned for the participating labs to identify any comparability problems before analysis of the field samples is initiated (Appendix 1).

I. Archiving

As described above, aliquots of homogenates of all samples analyzed will be archived for five years to provide for reanalysis in case of any mishaps or need for confirmation, as well as for analysis of emerging contaminants.

Up to three 50-g aliquots of each composite created will be archived. This will provide an integrative, representative sample for each zone 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.

Two of the three archive jars will be glass with a Teflon-lined lid (e.g., I-Chem 200 series glass jars). In addition, a separate archive aliquot will be kept in a polypropylene jar for potential analysis of perfluorinated compounds. Archived samples will be stored at -20° C.

In addition, selected San Francisco Bay samples will be archived long-term through a RMP collaboration with the National Institute of Standards and Technology (NIST). These samples will be stored in liquid nitrogen at a temperature of -150° C.

J. Non-target Species Data

In addition to the primary and secondary target species, other species will also 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 zones.

K. Timing

Sampling in 2018, and in the other years sampled, will be conducted from May through November. Seasonal variation in body condition and reproductive physiology are recognized as factors that could affect contaminant concentrations. However, sampling as many zones as possible in a given year is essential to a statewide assessment, and it will take this many months to sample the zones targeted for 2018. Humboldt Bay, and perhaps a small number of other zones, will be sampled in 2019, if possible. The remaining zones will be sampled in 2020.

L. Data Assessment

MQ1 will be assessed by comparing results from each zone to advisory tissue levels established by OEHHA in Klasing and Brodberg (2008) (Table 11). Maps, histograms, and frequency distributions will be prepared to summarize these comparisons.

MQ2 will be assessed through analysis of variance (or analysis of covariance for the species with mercury in individual fish) for the areas where replicate samples are available (SCB and San Francisco Bay). For the other areas, non-statistical methods will be used (mapping and graphing). Comparison of concentrations among regions may be performed by treating zones within each region as "replicates".

MQ3 will be assessed in later rounds of sampling after more data have been accumulated. It will take several rounds of sampling to generate enough data to begin to assess trends.

MQ4 will be assessed in consultation with OEHHA.

M. Products and Timeline

A technical report on the 2018 Bight sampling will be drafted by July 2019. The final report, incorporating revisions in response to reviewer comments, will be completed in September 2019.

The Bay RMP will produce a draft technical report on the Bay data by December 2020, and a final report by March 2021.

A final technical report will present the data for all of the coastal zones sampled over the 2018-2020 period and provide a comprehensive assessment of the entire coast-wide dataset. This report will be drafted by December 2021, and finalized by March 2022.

N. Sampling Design Summary

A summary of key elements of the sampling design is presented in Table 12.

REFERENCES

Ackerman, J.T., Hartman, C.A., Eagles-Smith, C.A., Herzog, M.P., Davis, J., Ichikawa, G., and Bonnema, A. 2015. Estimating exposure of piscivorous birds and sport fish to mercury in California lakes using prey fish monitoring—A predictive tool for managers: U.S. Geological Survey Open-File Report 2015-1106, 48 p.

Bioaccumulation Oversight Group. 2009. Sampling and Analysis Plan for a Screening Study of Bioaccumulation on the California Coast. California State Water Resources Control Board, Sacramento, CA. https://www.waterboards.ca.gov/water_issues/programs/swamp/docs/bog_coast_final_monplan_09.pdf

Bioaccumulation Oversight Group. 2015. Sampling and Analysis Plan for Long-term Monitoring of Bass Lakes and Reservoirs in California. California State Water Resources Control Board, Sacramento, CA. https://www.waterboards.ca.gov/water_issues/programs/swamp/docs/lakes_study/bass_lakes_sampling_plan.pdf

Bioaccumulation Oversight Group. 2018 in prep. Quality Assurance Project Plan: Sampling and Analysis Plan for a Second Statewide Survey of Bioaccumulation on the California Coast. Moss Landing Marine Labs. California State Water Resources Control Board, Sacramento, CA.

Chen, C.Y., C.T. Driscoll, K.F. Lambert, R.P. Mason, L.R. Rardin, C.V. Schmitt, N.S. Serrell, and E.M. Sunderland. 2012. Sources to Seafood: Mercury Pollution in the Marine Environment. Hanover, NH: Toxic Metals Superfund Research Program, Dartmouth College.

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, G. Ichikawa, A. Bonnema, C. Lamerdin, W.A. Heim, D. Crane, M. Stephenson. 2008. Draft Technical Report on Year One of a Two-Year Screening Study of Bioaccumulation in California Lakes and Reservoirs. 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 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., K. Schiff, A.R. Melwani, S.N. Bezalel, J.A. Hunt, R.M. Allen, G. Ichikawa, A. Bonnema, W.A. Heim, D. Crane, S. Swenson, C. Lamerdin, and M. Stephenson. 2011. Contaminants in Fish from the California Coast, 2009: Summary Report on Year One of 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, 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.

Drevnick, P.E., C.H. Lamborg, and M.J. Horgan. 2015. Increase in mercury in Pacific yellowfin tuna. Environ. Toxicol. Chem. 34: 931-934.

Gandhi, N., R.W.K. Tang, S.P. Bhavsar, and G.P. Arhonditsis. 2014. Fish mercury levels appear to be increasing lately: a report from 40 years of monitoring in the province of Ontario, Canada. Environ. Sci. Technol. 48: 5404–5414.

Greenfield, Ben K., J.A. Davis, R. Fairey, C. Roberts, D.B. Crane, G. Ichikawa, and M. Petreas. 2003. Contaminant Concentrations in Fish from San Francisco Bay, 2000. RMP Technical Report: SFEI Contribution 77. San Francisco Estuary Institute, Richmond, CA.

Jones, K. 2004. Pier Fishing in California. Publishers Design Group. Roseville, CA.

Klasing, S. and R. Brodberg. 2008 (Updated ATL Table November 2017). 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.

Miller, M.A. 1993. Maternal transfer of organochlorine compounds in salmonines to their eggs. Can J Fish Aquat Sci 50: 1405-1413

Monson, B.A. 2009. Trend reversal of mercury concentrations in piscivorous fish from Minnesota Lakes: 1982-2006. Environ. Sci. Technol. 43: 1750-1755.

Monson, B.A., D.F. Staples, S.P. Bhavsar, T.M. Holsen, C.S. Schrank, S.K. Moses, D.J. McGoldrick, S.M. Backus, and K.A. Williams. 2011. Spatiotemporal trends of mercury in walleye and largemouth bass from the Laurentian Great Lakes Region. Ecotoxicology 20:1555–1567.

Niimi, A.J. 1983. Biological and toxicological effects of environmental contaminants in fish and their eggs. Can J Fish Aquat Sci 40:306-312

NOAA. 2007. 2002-2004 Southern California Coastal Marine Fish Survey. National Oceanic Atmospheric and Administration. Long Beach CA 91 pp.

Smith, W., H.T. Pham, S. Klasing, M. Gassel, and S. Murphy. 2016. Statewide Advisory for Eating Fish from California Coastal Locations Without Site-Specific Advice. California Office of Environmental Health Hazard Assessment, Sacramento, CA.

Sun, J., J.A. Davis, S. N. Bezalel, J.R.M.Ross, A.Wong, D.Yee, R. Fairey, A. Bonnema, D.B. Crane, R. Grace, R. Mayfield, and J. Hobbs. 2017. Contaminant Concentrations in Fish from San Francisco Bay, 2014. SFEI Contribution #806. Regional Monitoring Program for Water Quality in San Francisco Bay, Richmond, CA.

Sutton, R., Sedlak, M.D., Yee, D., Davis, J.A., Crane, D., Grace, R. and Arsem, N. 2014. Declines in polybrominated diphenyl ether contamination of San Francisco Bay following production phase-outs and bans. Environ. Sci. Technol. 49: 777-784.

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.

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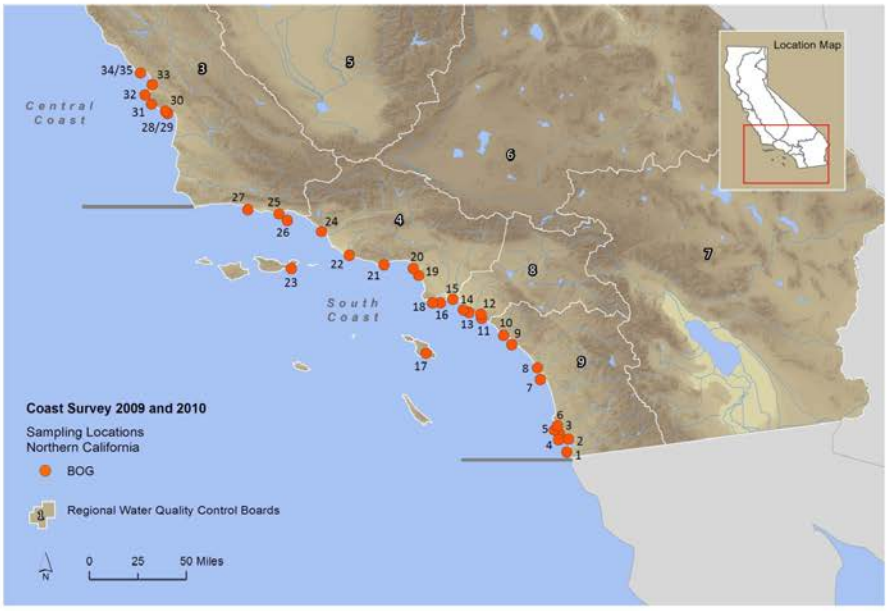
Figure 1. Fishing zones for the second SWAMP coastal bioaccumulation survey: (Southern California Bight) and southern Central Coast. The same zones sampled in the 2009 and 2010 survey are being sampled again in the second survey.



34/35	Cambria/Cayucos Coast/Northern San Luis Obispo County Coast
33	Morro Bay
32	Morro Bay Coast
31	Diablo Canyon Coast
30	Port San Luis Area
28/29	North Santa Barbara County Coast/Pismo Beach Area
27	Goleta to Pt Conception
25	Rincon to Goleta
26	Santa Barbara Channel Oil Platform
24	Ventura to Rincon
22	Pt Dume to Oxnard
21	North Santa Monica Bay
23	Northern Channel Islands
20	Middle Santa Monica Bay
19	South Santa Monica Bay
15	Long Beach
16	San Pedro Bay
18	Palos Verdes
14	Orange County Oil Platforms
13	Santa Ana River to Seal Beach
12	Newport Bay
11	Crystal Cove to Santa Ana River
10	Dana Point Harbor
9	San Onofre to Crystal Cove
17	Catalina Island
8	Oceanside Harbor
7	La Jolla to San Onofre (Agua Hedionda Lagoon)
6	Mission Bay
5	Pt Loma to La Jolla
4	Pt Loma
3	SD North Bay
2	SD South Bay
1	TJ to North Island

Figure 2. Fishing zones for the second SWAMP coastal bioaccumulation survey: northern Central Coast and North Coast.

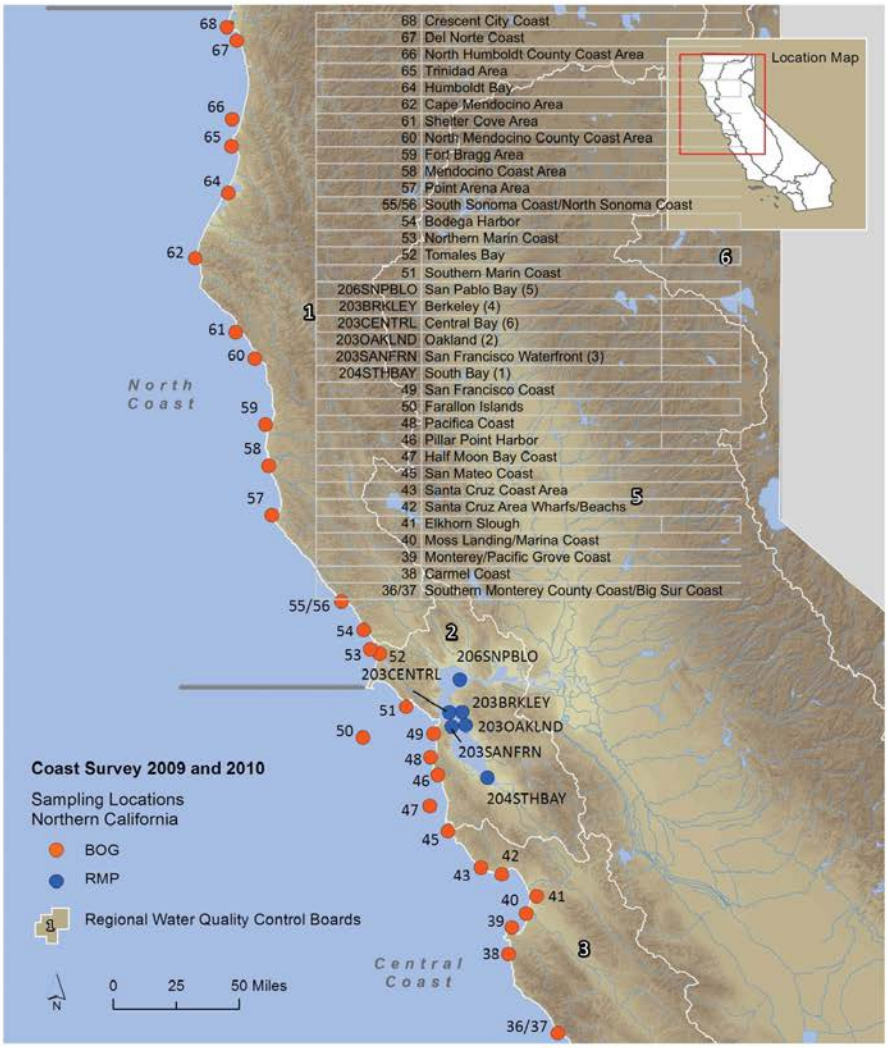


Figure 3. Fishing zones delineated for this survey. A Google Earth layer with the zones is available on the BOG website: http://www.swrcb.ca.gov/water_issues/programs/monitoring_council/bioaccumulation_oversight_group/

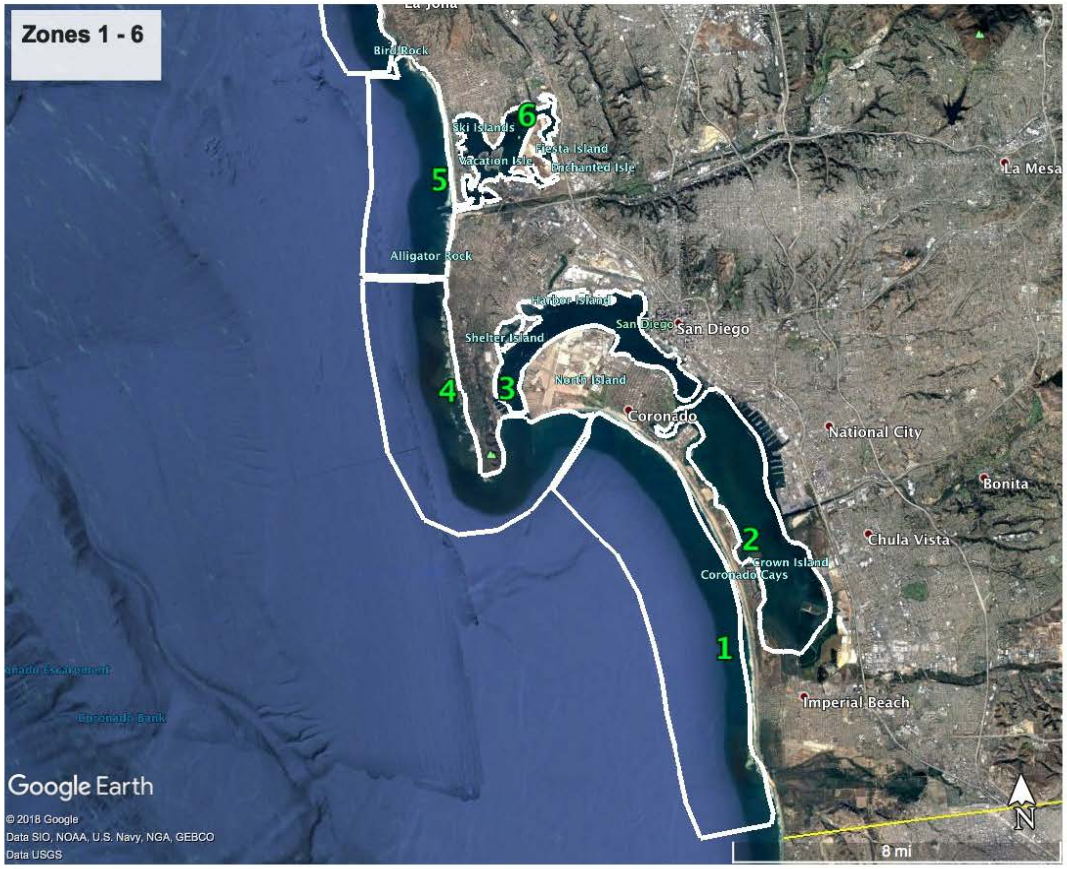


Figure 3. Zone maps (continued).

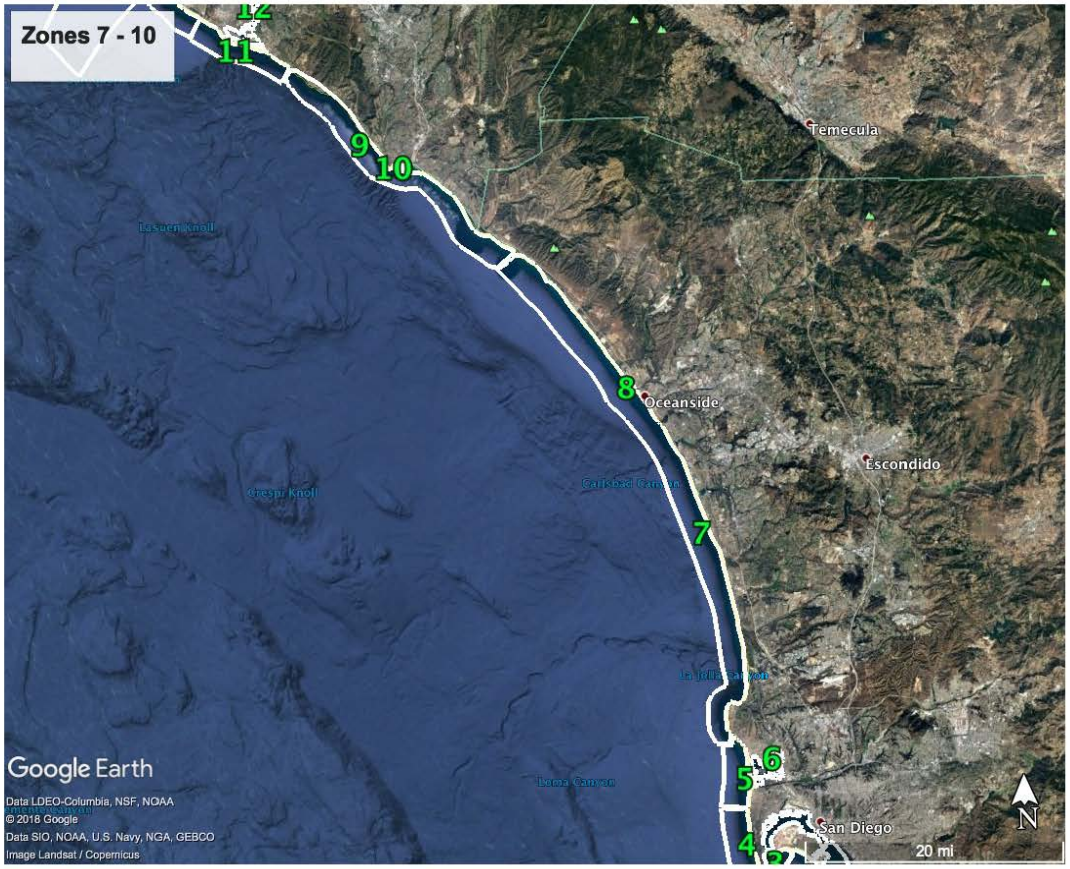


Figure 3. Zone maps (continued).

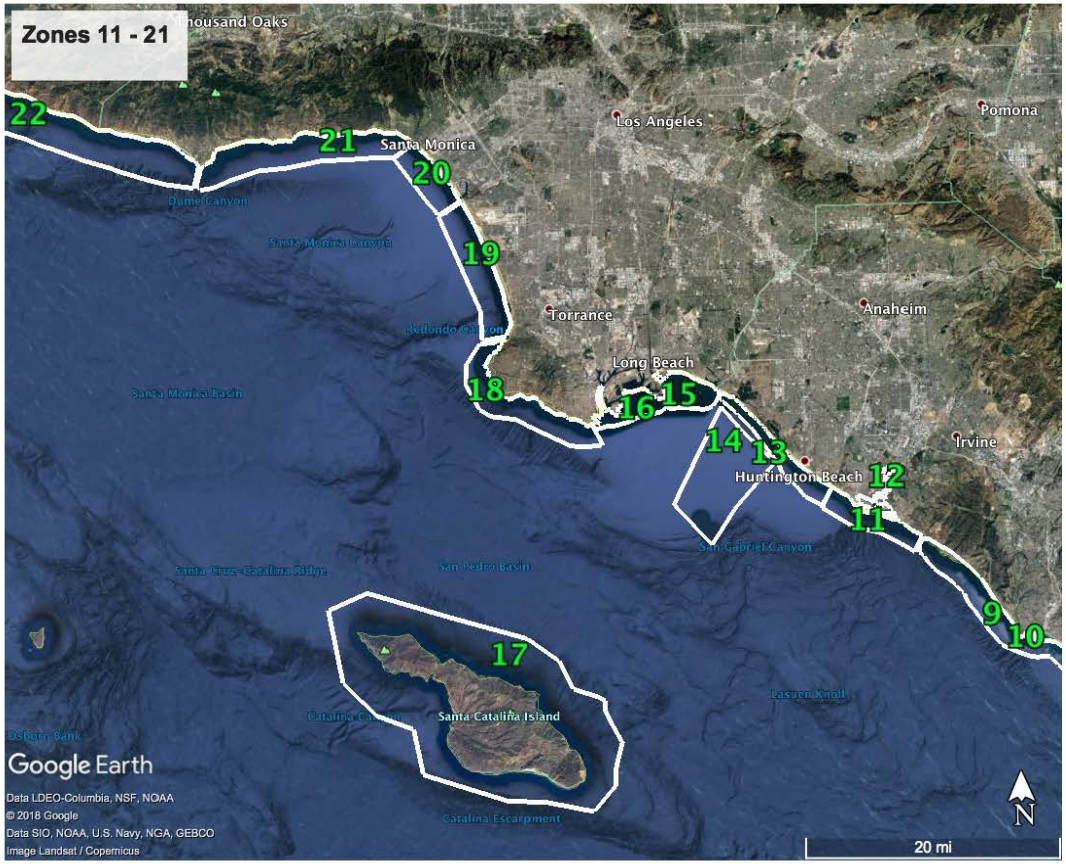


Figure 3. Zone maps (continued).

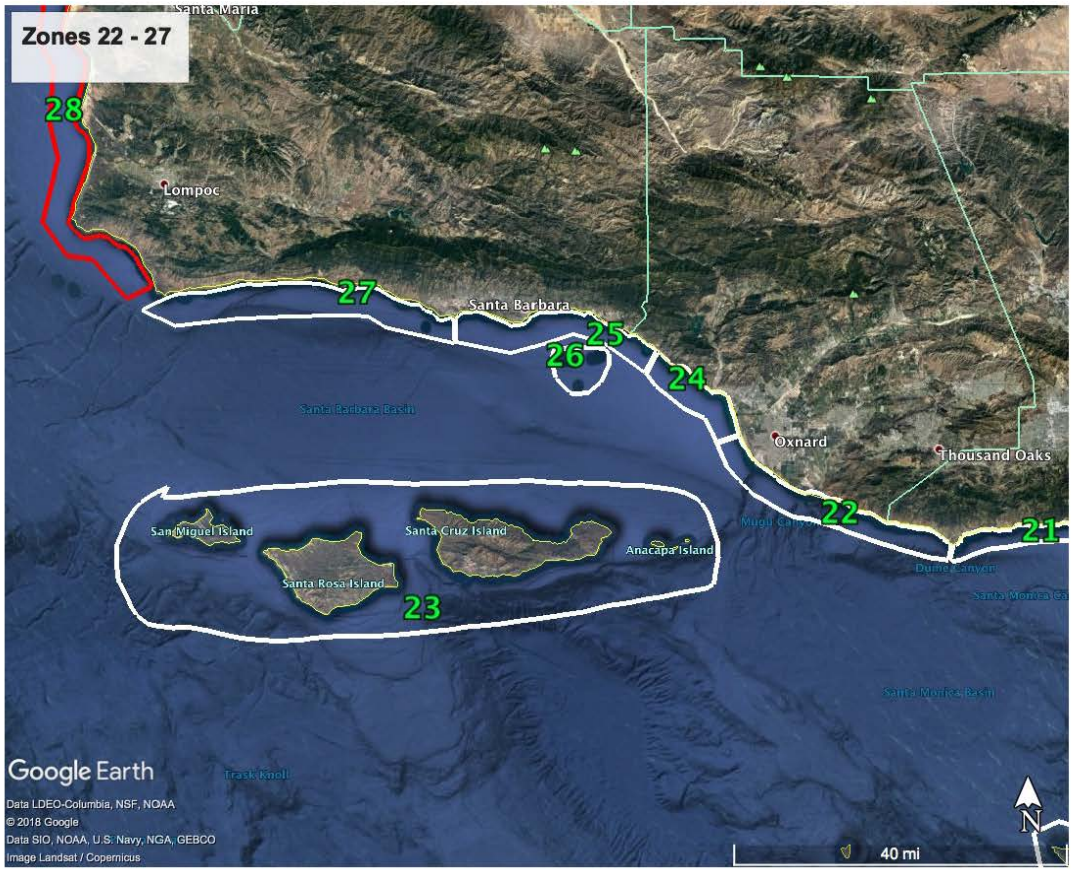


Figure 3. Zone maps (continued). Zones 28 and 29, 34 and 35, and 36 and 37 have been merged into single zones.

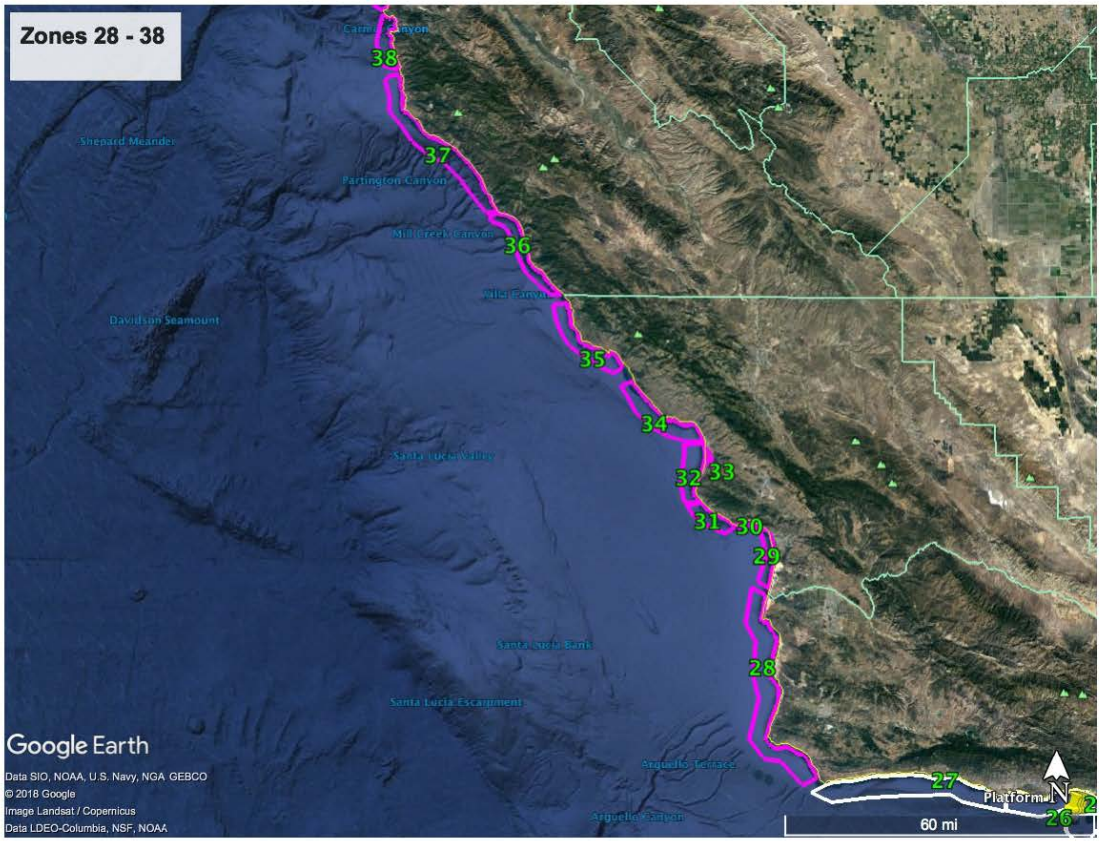


Figure 3. Zone maps (continued).

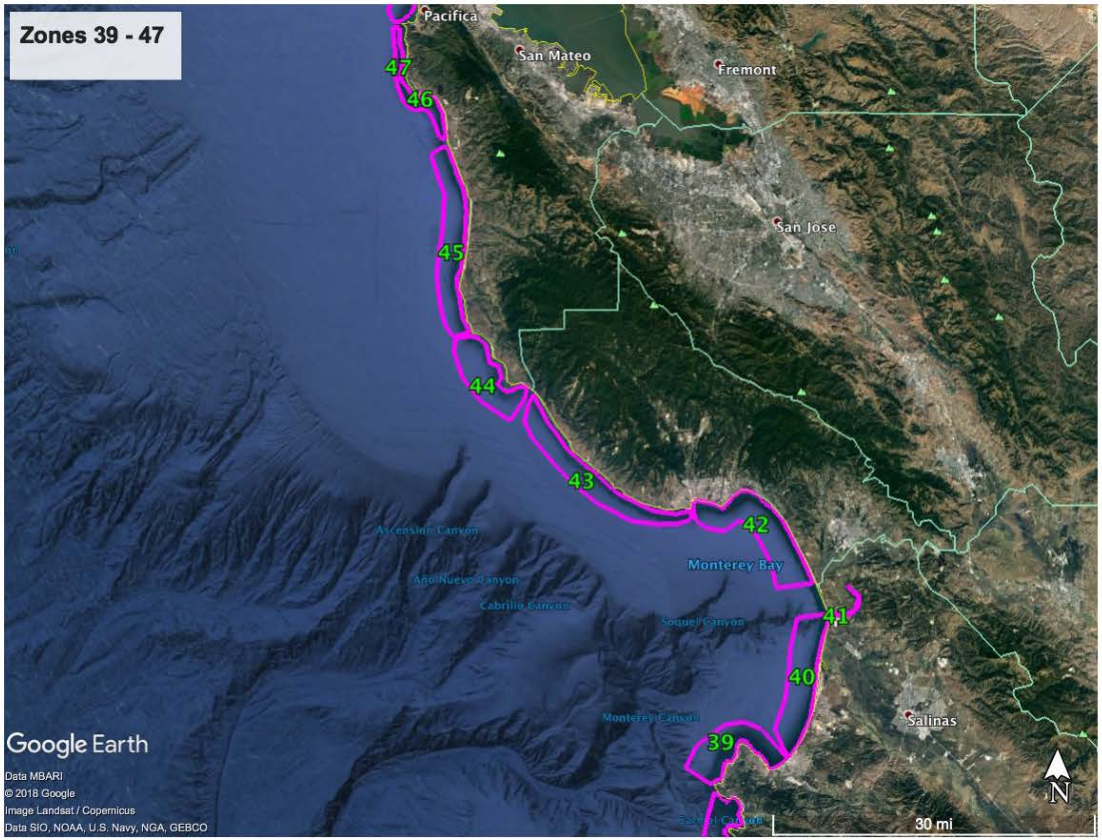


Figure 3. Zone maps (continued).

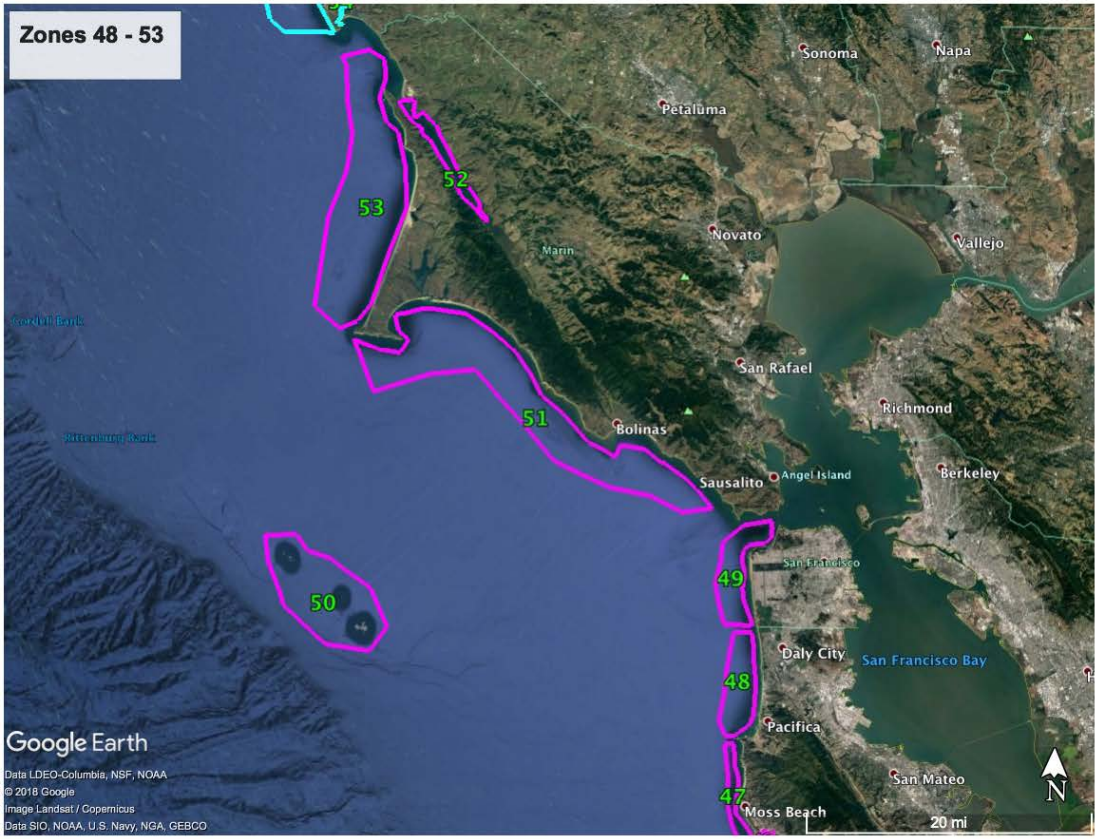


Figure 3. Zone maps (continued). Zones 55 and 56 have been merged into one zone.

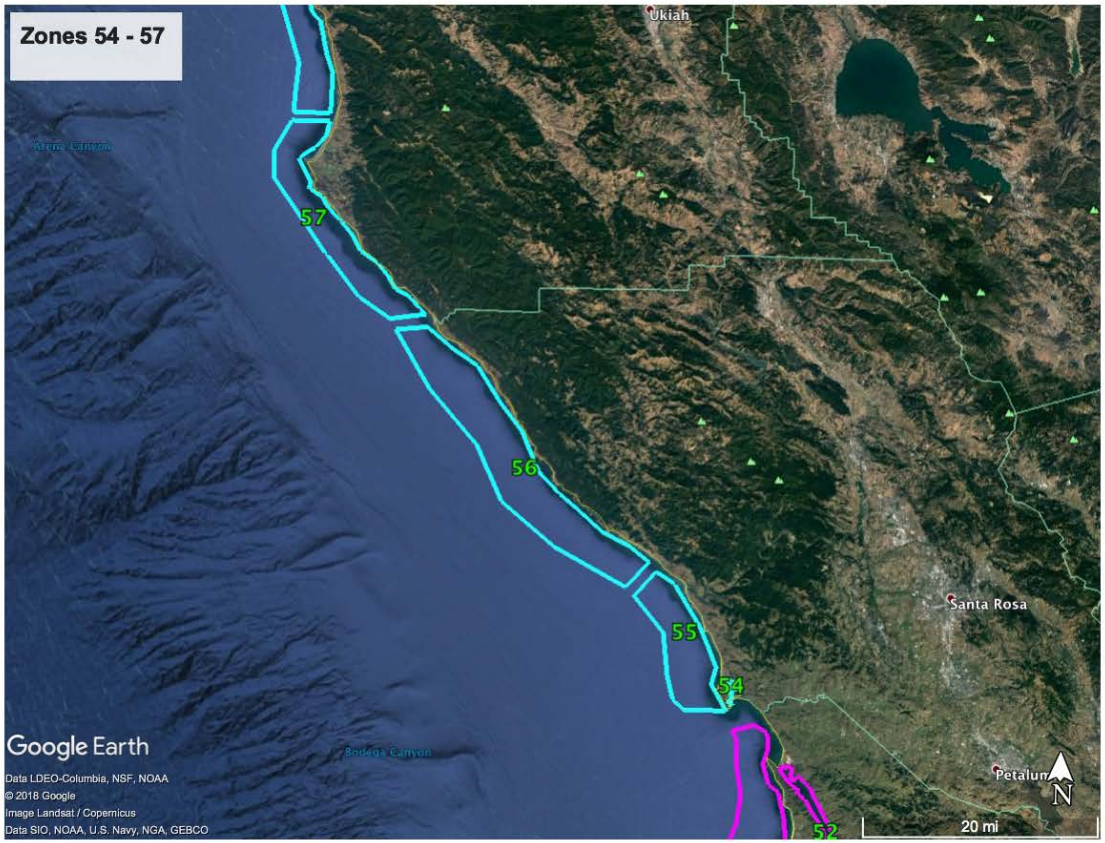


Figure 3. Zone maps (continued).

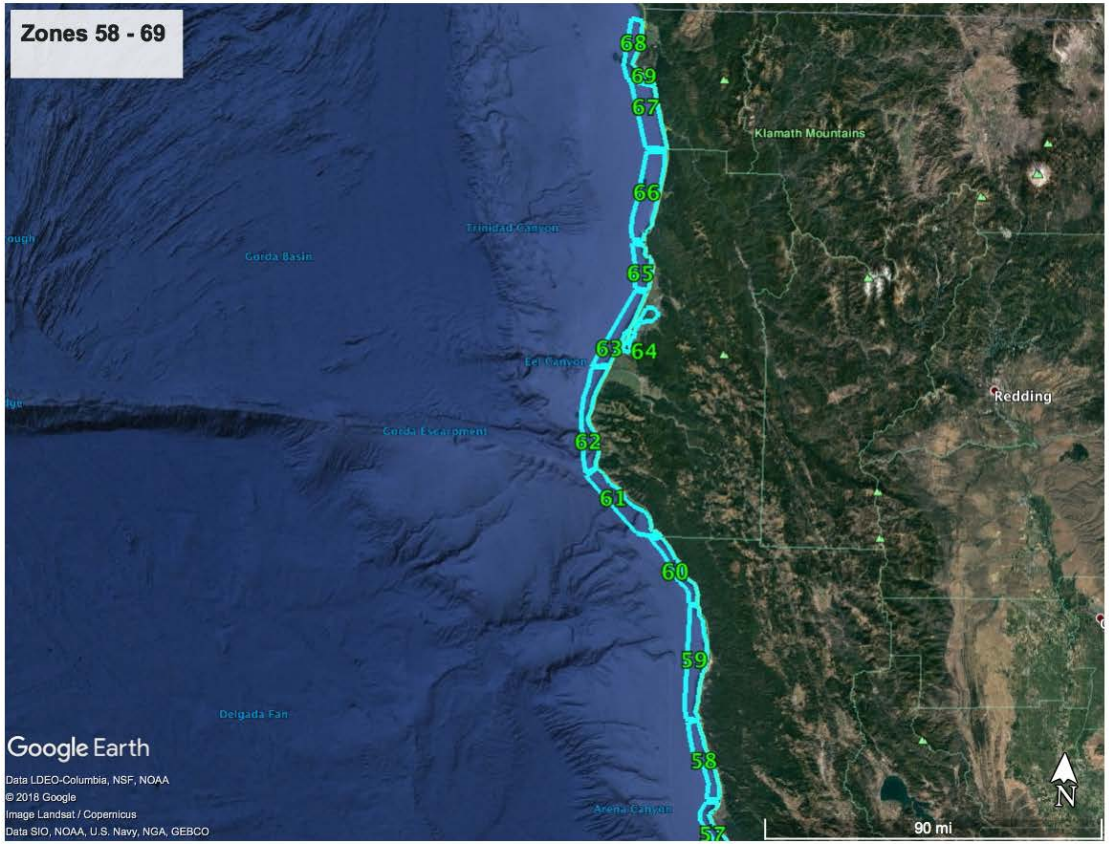


Figure 4. Zones in San Francisco Bay will be centered around five long-term monitoring sites (zones) shown on this map: San Pablo Bay, Central Bay, San Francisco Waterfront, Oakland, and South Bay. The other sites have been sampled sporadically over the years.

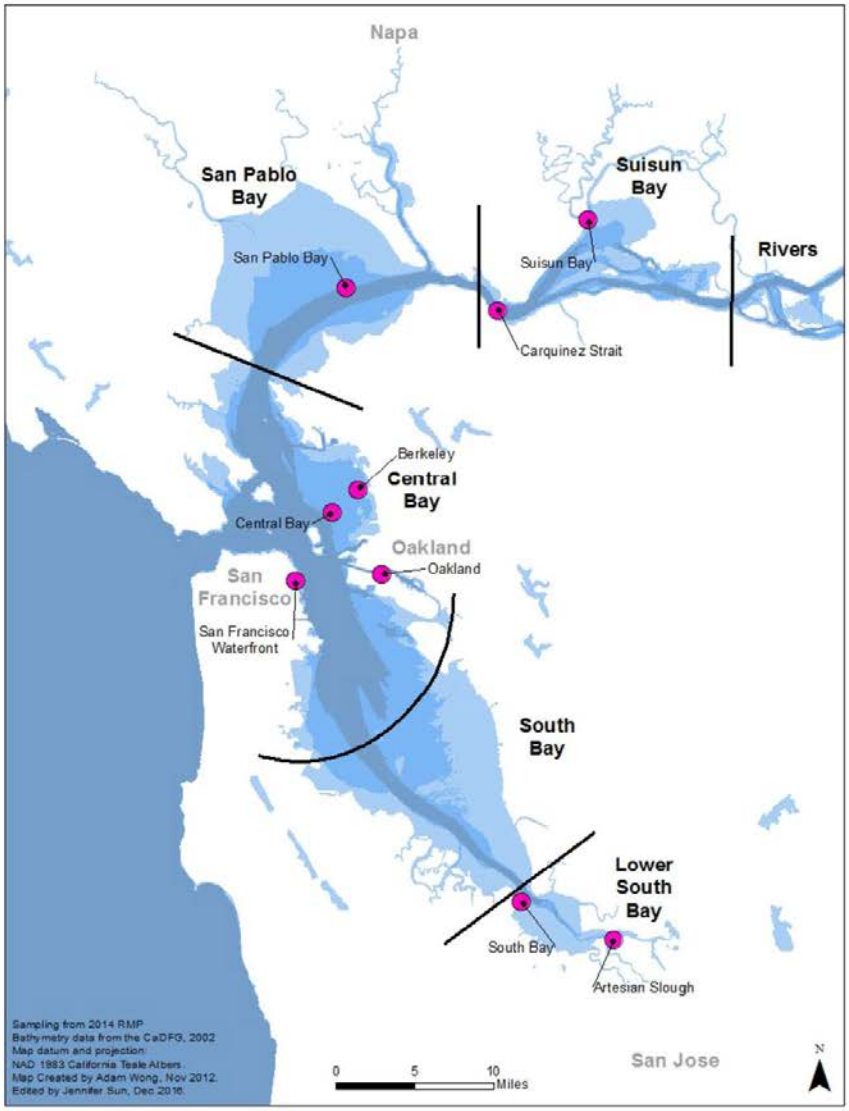


Table 1. Bioaccumulation monitoring assessment framework for fishing beneficial uses.

1. Determine the status of fishing beneficial uses throughout the State with respect to bioaccumulation of toxic pollutants

1.1 What are the extent and locations of water bodies with sufficient evidence to indicate that fishing beneficial uses are at risk due to pollutant bioaccumulation?

1.2 What are the extent and locations of water bodies with some evidence indicating fishing beneficial uses are at risk due to pollutant bioaccumulation?

1.3 What are the extent and locations of water bodies with no evidence indicating fishing beneficial uses are at risk due to pollutant bioaccumulation?

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, D.1.2, and D.1.3?

2. Assess trends in the impact of bioaccumulation on fishing beneficial uses throughout the State

2.1 Are water bodies improving or deteriorating with respect to the impact of bioaccumulation on fishing beneficial uses?

2.1.1 Have water bodies fully supporting fishing beneficial uses become impaired?

2.1.2 Has full support of fishing beneficial uses been restored for previously impaired water bodies?

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?

3. Evaluate sources and pathways of bioaccumulative pollutants impacting fishing beneficial uses

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?

3.2 How is the relative importance of different sources and pathways of bioaccumulative pollutants that impact fishing beneficial uses changing over time on a regional and statewide basis?

4. Provide the monitoring information needed to evaluate the effectiveness of management actions in reducing the impact of bioaccumulation on fishing beneficial uses

4.1 What are the management actions that are being employed to reduce the impact of bioaccumulation on fishing beneficial uses regionally and statewide?

4.2 How has the impact of bioaccumulation on fishing beneficial uses been affected by management actions regionally and statewide?

Table 2. Long-term sport fish sampling schedule. Bold indicates a firm plan.

X = funded by SWAMP, O = funded by another program

General water body category	Specific category (numbers are approximate)	Revisit frequency for each water body	2015	2016	2017	2018	2019	2020	2021	2022	2023	2024	2025	2026	2027	2028	2029	2030	2031	2032
Lakes	**1) Bass Lakes (n=190) (Statewide Core Monitoring)**	**10 yr**	**X**		**X**		**X**		**X**		**X**		**X**		**X**		**X**		**X**	
	2) "New" Bass Lakes	Screening		**X**																
	3) Bass Lakes - with mgmt actions	1 yr					O	O	O	O	O	O	O	O	O	O	O	O	O	
	4) Trout Lakes - >0.2 ppm (n=5)	10 yr												X						
	5) Trout Lakes - <0.2 ppm (n=90)	20 yr												X						
	6) "New" Trout Lakes	Screening		**X**																
Rivers and Streams	7) Bass sites in Delta (n=6)	1 yr		**O**	**O**	**O**	**O**	**O**	**O**	**O**	**O**	**O**	**O**		O		O		O	
	8) Other bass/sucker sites (n=10)	10 yr								X										X
	9) Trout Sites - <0.2 ppm (n=50)	20 yr																		
	10) Trout Sites - >0.2 ppm (n=10)	10 yr								X										X
Coast	11) SF Bay	5 yr					**O**					**O**					**O**			
	12) SC Bight (n=27)	10 yr				**XO**										O				
	13) Other coast zones (**n=35**)	10 yr				**X**	**X**	**X**								**X**	**X**	**X**		

Table 3. RecFIN catch data for major groups of species for the Southern California region (from Santa Barbara County to San Diego County) and specific data for the coast (ocean < 3 mi) and bays and harbors (inland) from January 2015 through December 2017. Data include mass of catch in tonnes and counts in thousands. The mass and catch data were sorted and ranked for each waterbody type, then the ranks for each species were averaged to obtain an average rank. The average rank was used as the index of popularity for fish consumption.

Southern California

Waterbody	SPECIES	count	mass	rank_ count	rank_ mass	avg_ rank
Inland	Pacific (Chub) Mackerel	776061	143.29	1	1	1.0
Inland	Pacific Bonito	25474	10.82	6	4	5.0
Inland	Spotfin Croaker	17799	15.76	9	3	6.0
Inland	Yellowfin Croaker	29812	9.44	4	8	6.0
Inland	Jacksmelt	36946	6.51	3	11	7.0
Inland	Kelp Bass	14053	10.58	12	5	8.5
Inland	Sargo	16193	9.82	11	6	8.5
Inland	Opaleye	23898	8.43	7	10	8.5
Inland	California Halibut	8187	28.73	16	2	9.0
Inland	Spotted Sandbass	17604	9.23	10	9	9.5
Inland	Barred Sandbass	9593	9.46	14	7	10.5
Inland	White Croaker	22920	3.76	8	13	10.5
Inland	Topsmelt	26523	2.91	5	16	10.5
Ocean <= 3 Miles	Pacific (Chub) Mackerel	2644168	473.70	1	2	1.5
Ocean <= 3 Miles	Vermilion Rockfish	415541	261.89	3	4	3.5
Ocean <= 3 Miles	Barred Surfperch	678599	254.55	2	5	3.5
Ocean <= 3 Miles	Pacific Bonito	286221	280.11	6	3	4.5
Ocean <= 3 Miles	Copper Rockfish	310574	224.84	5	7	6.0
Ocean <= 3 Miles	Ocean Whitefish	312268	125.93	4	9	6.5
Ocean <= 3 Miles	Yellowtail	160095	752.27	13	1	7.0
Ocean <= 3 Miles	Kelp Bass	257757	212.21	7	8	7.5
Ocean <= 3 Miles	Bocaccio	164265	120.79	11	10	10.5
Ocean <= 3 Miles	Blue Rockfish	188791	65.54	10	14	12.0
Ocean <= 3 Miles	Lingcod	81022	230.13	22	6	14.0
Ocean <= 3 Miles	Barred Sandbass	85652	79.31	20	12	16.0
Ocean <= 3 Miles	Jacksmelt	163316	30.62	12	20	16.0

Table 3. Continued. RecFIN catch data for major groups of species for the Northern California region (from Del Norte County to San Luis Obispo County) and specific data for the coast (ocean < 3 mi) and bays and harbors (inland) from January 2015 through December 2017. Data include mass of catch in tonnes and counts in thousands. The mass and catch data were sorted and ranked for each waterbody type, then the ranks for each species were averaged to obtain an average rank. The average rank was used as the index of popularity for fish consumption.

Northern California

Waterbody	SPECIES	count	mass	rank_ count	rank_ mass	avg_ rank
Inland	Jacksmelt	719458	122.01	1	2	1.5
Inland	California Halibut	41655	150.96	6	1	3.5
Inland	Striped Bass	61324	117.28	5	3	4.0
Inland	Pacific Herring	386269	27.35	2	7	4.5
Inland	Pacific (Chub) Mackerel	92866	11.15	3	9	6.0
Inland	Redtail Surfperch	21812	11.21	9	8	8.5
Inland	Brown Rockfish	29327	9.98	7	11	9.0
Inland	Leopard Shark	9365	52.11	18	4	11.0
Inland	Bat Ray	6545	39.72	20	5	12.5
Inland	Black Rockfish	12269	8.35	15	12	13.5
Inland	Black Perch	13999	4.35	13	15	14.0
Inland	Grass Rockfish	11161	6.20	16	13	14.5
Ocean <= 3 Miles	Lingcod	503731	1451.03	3	1	2.0
Ocean <= 3 Miles	Blue Rockfish	840404	399.75	2	3	2.5
Ocean <= 3 Miles	Black Rockfish	499847	432.27	4	2	3.0
Ocean <= 3 Miles	Barred Surfperch	985557	354.18	1	5	3.0
Ocean <= 3 Miles	Yellowtail Rockfish	337395	166.43	6	8	7.0
Ocean <= 3 Miles	Vermilion Rockfish	257933	380.83	12	4	8.0
Ocean <= 3 Miles	Brown Rockfish	292075	195.96	9	7	8.0
Ocean <= 3 Miles	Gopher Rockfish	293458	137.32	7	10	8.5
Ocean <= 3 Miles	Redtail Surfperch	279517	149.45	10	9	9.5
Ocean <= 3 Miles	Pacific (Chub) Mackerel	393056	72.83	5	14	9.5
Ocean <= 3 Miles	Copper Rockfish	183008	198.86	14	6	10.0
Ocean <= 3 Miles	Olive Rockfish	120659	85.80	16	13	14.5

Table 4. RecFin catch data for individual popular species, for two regions (south and north) and specific data for the coast (ocean < 3 mi) and bays and harbors (inland) from January 2015 through December 2017.

(table on next page)

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Northern CA

SPECIES	Name	Inland	Ocean <= 3 mi
Barred Surfperch	MT	2	354
Barred Surfperch	Num	4986	985557
Bat Ray	MT	40	5
Bat Ray	Num	6545	992
Black Perch	MT	4	6
Black Perch	Num	13999	14970
Black Rockfish	MT	8	432
Black Rockfish	Num	12269	499847
Blue Rockfish	MT	1	400
Blue Rockfish	Num	1299	840404
Brown Rockfish	MT	10	196
Brown Rockfish	Num	29327	292075
California Halibut	MT	151	40
California Halibut	Num	41655	7592
Copper Rockfish	MT	0	199
Copper Rockfish	Num	287	183008
Gopher Rockfish	MT	0	137
Gopher Rockfish	Num	429	293458
Grass Rockfish	MT	6	16
Grass Rockfish	Num	11161	24842
Jacksmelt	MT	122	50
Jacksmelt	Num	719458	239635
Leopard Shark	MT	52	3
Leopard Shark	Num	9365	455
Lingcod	MT	10	1451
Lingcod	Num	3944	503731
Olive Rockfish	MT	0	86
Olive Rockfish	Num	21	120659
Pacific (Chub) Mackerel	MT	11	73
Pacific (Chub) Mackerel	Num	92866	393056
Pacific Herring	MT	27	0
Pacific Herring	Num	386269	5166
Redtail Surfperch	MT	11	149
Redtail Surfperch	Num	21812	279517
Striped Bass	MT	117	95
Striped Bass	Num	61324	42753
Vermilion Rockfish	MT	0	381
Vermilion Rockfish	Num	118	257933
Yellowtail Rockfish	MT	0	166
Yellowtail Rockfish	Num	43	337395

Southern CA

SPECIES	Name	Inland	Ocean <= 3 mi
Barred Sandbass	MT	9	79
Barred Sandbass	Num	9593	85652
Barred Surfperch	MT	0	255
Barred Surfperch	Num	908	678599
Blue Rockfish	MT	0	66
Blue Rockfish	Num	0	188791
Bocaccio	MT	0	121
Bocaccio	Num	20	164265
California Halibut	MT	29	61
California Halibut	Num	8187	11633
Copper Rockfish	MT	0	225
Copper Rockfish	Num	110	310574
Jacksmelt	MT	7	31
Jacksmelt	Num	36946	163316
Kelp Bass	MT	11	212
Kelp Bass	Num	14053	257757
Lingcod	MT	0	230
Lingcod	Num	116	81022
Ocean Whitefish	MT	0	126
Ocean Whitefish	Num	1257	312268
Opaleye	MT	8	27
Opaleye	Num	23898	63838
Pacific (Chub) Mackerel	MT	143	474
Pacific (Chub) Mackerel	Num	776061	2644168
Pacific Bonito	MT	11	280
Pacific Bonito	Num	25474	286221
Sargo	MT	10	7
Sargo	Num	16193	12341
Spotfin Croaker	MT	16	54
Spotfin Croaker	Num	17799	60587
Spotted Sandbass	MT	9	0
Spotted Sandbass	Num	17604	499
Topsmelt	MT	3	3
Topsmelt	Num	26523	21025
Vermilion Rockfish	MT	0	262
Vermilion Rockfish	Num	74	415541
White Croaker	MT	4	4
White Croaker	Num	22920	27097
Yellowfin Croaker	MT	9	13
Yellowfin Croaker	Num	29812	52148
Yellowtail	MT	1	752
Yellowtail	Num	143	160095

Table 5a. Classification of average methylmercury concentrations for each species at each location: North Coast. Red: >0.44 ppm; Green: <0.07 ppm; Yellow: between 0.07 and 0.44 ppm.

Table 4
Classification of average methylmercury concentrations for each species at each location: North Coast. Red: >0.44 ppm . Green: <0.07 ppm . Yellow: between 0.07 and 0.44 ppm.

Coastal Species

Regional Board	Zone	Station Name	Black Rockfish	Blue Rockfish	Quillback Rockfish	Olive Rockfish	Copper Rockfish	Gopher Rockfish	China Rockfish	Vermilion Rockfish	Yellowtail Rockfish	Rosy Rockfish	Brown Rockfish	Black and Yellow Rockfish	Kelp Rockfish	Cabezon	Kelp Greenling	Lingcod	Barred Sand Bass	Kelp Bass	Spotted Sand Bass	Chub Mackerel	Spotted Scorpionfish	Pacific Barracuda	Ocean Whitefish	Rainbow Surfperch	Barred Surfperch	Black Perch	White Croaker	Black Croaker
1	68	Crescent City Coast	Yellow	Yellow	Yellow											Yellow	Yellow	Yellow												
1	67	Del Norte Coast	Yellow	Green		Green										Yellow	Yellow	Yellow												
1	66	North Humboldt County Coast Area	Yellow	Yellow	Yellow		Red	Red								Yellow		Yellow												
1	65	Trinidad Area																Yellow												
1	64	Humboldt Bay															Green													
1	62	Cape Mendocino Area	Yellow	Yellow			Red		Red							Red		Yellow												
1	61	Shelter Cove Area	Yellow	Yellow			Red	Red	Red	Yellow																				
1	60	North Mendocino County Coast Area	Yellow				Red		Red	Yellow						Red														
1	59	Fort Bragg Area	Yellow	Yellow				Red		Yellow						Red														
1	58	Mendocino Coast Area	Yellow	Yellow						Yellow	Yellow	Red				Red		Yellow												
1	57	Point Arena Area	Yellow	Yellow		Yellow		Red	Red							Yellow		Red												
1	55/56	South Sonoma Coast/North Sonoma Coast		Yellow		Yellow	Red			Yellow			Yellow																	
1	54	Bodega Harbor																								Green				
2	53	Northern Marin Coast		Green		Green							Yellow																	
2	52	Tomales Bay																												

Bay and Harbor Species

Regional Board	Zone	Station Name	Bat Ray	California Halibut	Leopard Shark	Brown Smoothhound Shark	Gray Smoothhound Shark	Spiny Dogfish	Shiner Surfperch	Pile Surfperch	White Surfperch	Yellowfin Croaker	Spotfin Croaker	Topsmelt	Jacksmelt	Striped Bass	Opaleye	Queenfish
1	68	Crescent City Coast																
1	67	Del Norte Coast																
1	66	North Humboldt County Coast Area																
1	65	Trinidad Area																
1	64	Humboldt Bay	Red	Yellow	Red				Yellow									
1	62	Cape Mendocino Area																
1	61	Shelter Cove Area																
1	60	North Mendocino County Coast Area																
1	59	Fort Bragg Area																
1	58	Mendocino Coast Area																
1	57	Point Arena Area																
1	55/56	South Sonoma Coast/North Sonoma Coast																
1	54	Bodega Harbor			Red													
2	53	Northern Marin Coast																
2	52	Tomales Bay			Red	Red			Green		Green							

Table 5b. Classification of average methylmercury concentrations for each species at each location: Central Coast. Red: >0.44 ppm; Green: <0.07 ppm; Yellow: between 0.07 and 0.44 ppm.

Table 5c. Classification of average methylmercury concentrations for each species at each location: South Coast. Red: >0.44 ppm; Green: <0.07 ppm; Yellow: between 0.07 and 0.44 ppm.

Table 6. Target species in each region for coastal waters and bays and harbors. Species in italics are those that will be analyzed as individuals for mercury as well as composited for other analytes. If the target species to be analyzed as individuals for mercury are not available, substitutions will be made. Asterisks indicate species that were in the top five in catch based on RecFIN data for each habitat by region combination. Bold indicates primary target species for the Bight Program. Based on results of the San Diego Bay Consumption Study, primary targets in San Diego Bay are chub mackerel, spotted sand bass, halibut, and topsmelt.

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Coast <3mi	South	Central	North
Primary	***Kelp Bass***		
	Barred Sand Bass		
	Gopher Rockfish	*Gopher Rockfish*	*Gopher Rockfish*
		Brown Rockfish	*Brown Rockfish*
		*Black Rockfish**	*Black Rockfish**
		Blue Rockfish*	Blue Rockfish*
	Lingcod	*Lingcod**	*Lingcod**
		Cabezon	*Cabezon*
		Salmon	Salmon
	Chub Mackerel*		
	White Croaker	White Croaker	
		Rainbow Surfperch	
Secondary	Scorpionfish		
	Blue Rockfish		
	Brown Rockfish		
	Copper Rockfish	Copper Rockfish	Copper Rockfish
	Vermilion Rockfish	Vermilion Rockfish	Vermilion Rockfish
		Olive Rockfish	Olive Rockfish
		Yellowtail Rockfish*	Yellowtail Rockfish*
	Barred Surfperch*	Barred Surfperch*	Barred Surfperch*
	Walleye Surfperch	Walleye Surfperch	Walleye Surfperch
		Redtail Surfperch	Redtail Surfperch
	California Halibut	California Halibut	California Halibut
	Yellowfin Croaker		
	California Sheephead (OEHHA)		
	Halfmoon/Opaleye (OEHHA)		
	Kelp Greenling (OEHHA)		
	Pacific Halibut (OEHHA)		
		Jacksmelt*	

Bays/Harbors	South	Central	North
Primary	***Kelp Bass***		
		Brown Rockfish	
	Spotted Sand Bass		
	Shiner Surfperch	Shiner Surfperch	Shiner Surfperch
			White Surfperch
		Leopard Shark	*Leopard Shark*
		California Halibut*	California Halibut*
	Jacksmelt*	Jacksmelt*	Jacksmelt*
	White Croaker	White Croaker	White Croaker
	Chub Mackerel*		
		Striped Bass*	
Secondary	*Barred Sand Bass*		
	Scorpionfish		
		Chub Mackerel*	Chub Mackerel*
		Black Rockfish	*Black Rockfish*
	Spotfin Croaker*		
	Yellowfin Croaker*		
	White Surfperch		
		Black Perch	Black Perch
	California Halibut		
	Leopard Shark		
	Gray Smoothound		
	Brown Smoothound	Brown Smoothound	Brown Smoothound
	Spiny Dogfish	Spiny Dogfish	Spiny Dogfish
	Topsmelt		
		Bat Ray	Bat Ray
	Halfmoon/Opaleye (OEHHA)		
			Redtail Surfperch
			Walleye Surperch

Table 7. Target species and their characteristics. Sources were from various websites and personal communication; primarily http://www.fishbase.org, and http://hmsc.oregonstate.edu/projects/msap/PS/masterlist/fish/

Group	Species	Trophic Level	Primary Prey	Feeding Position	Habitat	Range	Depth
Basses (Serranidae)	Kelp Bass (*Paralabrax clathratus*)	4	Small fishes (including anchovies, sardines, surfperch), squid, octopus, crabs, shrimps, and amphipods	mid-water	in or near kelp beds, but may be associated with any structure	Washington to Baja	0-50m
	Barred Sand bass (*Paralabrax nebulifer*)	3	fishes and crustaceans	demersal	sandy bottom among or near rocks	Santa Cruz, CA to Baja	0-183m
	Spotted Sand bass (*Paralabrax maculatofasciatus*)	4	small fishes and benthic crustaceans, clams	demersal	sand or mud bottom near rocks and eelgrass	Monterey, CA to Mexico	0-60m
Wrasses (Labridae)	CA Sheephead (*Semicossyphus pulcher*)	3	crustaceans, bivalves, sea urchins, polychaetes	demersal	rocky bottom usually in kelp beds	Monterey, CA to Guadalupe Island	0-55m
Sea Chubs (Scorpidinae)	Half Moon (*Medialuna californiensis*)	2-3	algae, sponges, worms, crustaceans	demersal/ benthic	rocky bottoms and kelp beds	Vancouver Island to Gulf of California	0-40m
	Opaleye (*Girella nigricans*)	2	algae, occasional invertebrates	benthic	kelp beds and shallow rocky reef with algae	San Francisco to Baja	2-30m
Rockfish (Sebastidae)	Blue Rockfish (*Sebastes mystinus*)	3	tunicates, hydroids, jellyfishes, and larval and juvenile fishes	mid-water	deep rocky reefs to hard, flat substrates	Bering Sea to Baja	0-100m
	Black Rockfish (*Sebastes melanops*)	3	juvenile rockfish, euphausids and amphipods (upwelling), and invertebrates (non-upwelling)	mid-water	kelp beds	Alaska to SoCal	0-366m
	Olive Rockfish (*Sebastes serranoides*)	3-4	fishes (particularly juvenile rockfishes), octopi, squid, copepods and crab larvae	mid-water	areas of reef or giant kelp, over hard, high relief	Northern CA to Baja (abundant SoCal to Mendocino County)	0-146m
	Brown Rockfish (*Sebastes auriculatus*)	3	small fishes, crab, shrimp, isopods and polychaetes	demersal	hard bottom; aggregate near	Alaska to Baja	0-128m

					rocks, oil platforms, sewer pipes		
	Gopher Rockfish (*Sebastes carnatus*)	3	Juvenile rockfish, crustaceans, brittle stars, mollusks, polychaetes, euphausiids	demersal	hard bottom in crevices typically within kelp beds	Eureka, CA to Central Baja	0-55m
	Copper Rockfish (*Sebastes caurinus*)	4	crustaceans, fish, octopi	demersal	rocky bottom and kelp beds	Kenai Peninsula, Gulf of Alaska to central Baja California	10-183m
Sculpins (Cottidae)	Cabezon (*Scorpaenichthys marmoratus*)	3	crustaceans, fish and mollusks	demersal	rocky, sandy and muddy bottoms, kelp beds	Southeastern AK to Baja	to 200m
Scorpionfish (Scorpaenidae)	CA Scorpionfish (*Scorpaena guttata*)	3	juvenile cancer crabs, small fishes (anchovy), octopi, isopods and shrimp	demersal	sandy and rocky areas in association with rocky reefs	Monterey Bay to Baja	0-183m
Greenlings (Hexagrammidae)	Lingcod (*Ophiodon elongatus*)	4	mostly fishes but also crustaceans, octopi and squid	demersal	near rocks	Alaska to Baja	to 475m
Croaker (Sciaenidae)	White Croaker (*Genyonemus lineatus*)	3	polychaetes, small shrimps, crabs and mollusks	benthic	over sandy bottoms	BC to Baja	to 183m
	Yellowfin Croaker (*Umbrina roncador*)	3	crustaceans and fishes	demersal	coastal waters and estuaries	Pt Conception to Gulf of CA	10-60m
	Spotfin Croaker (*Roncador stearnsii*)	3	bivalves, crabs, worms	demersal	sandy shores and bays	Pt Conception to Baja	1-22m
Surfperch (Embiotocidae)	Barred surfperch (*Amphistichus argenteus*)	3	sand crabs, clams and other inverts	benthic	surf of sand beaches, also near rocks, pilingsand other structures	Bodega Bay, CA to Baja	0-7m
	Redtail surfperch (*Amphistichus rhodoterus*)	3	Small crustaceans, small crabs, shrimp, mussels or marine worms	benthic	sand beaches in surf on exposed coasts	Vancouver Island, BC to Avila Beach, CA	0-7m
	Shiner surfperch (*Cymatogaster aggregata*)	3	calanoid copepods, crustaceans, mollusks,	mid-water/ demersal	eelgrass beds, piers and pilings	Alaska to Baja	0-146m
	Walleye surfperch (*Hyperprosopan argenteum*)	3	crustaceans, amphipods, isopods, small fish, mycids	mid-water	surf of sand beaches, and over sand near rocks	Vancouver Island to Baja	0-18m

	Black perch (*Embiotoca jacksoni*)	3	amphipods, crabs, worms	benthic	rocky areas near kelp, sand bottoms of coastal bays and around piers and pilings	Ft Bragg, CA to Baja	0-46m
	Rainbow surfperch (*Hypsurus caryi*)	3	crustaceans, bivalves, worms	demersal	rocky shores and edges of kelp beds	Cape Mendocino to Baja	1-50m
New World Silversides (Atherinopsidae)	Jacksmelt (*Atherinopsis californiensis*)	3	crustaceans, fish larvae	mid-water	inshore areas, including bays	Yaquina Bay, OR to Baja	0-30m
	Topsmelt (*Atherinops affinis*)	2	zooplankton, algae	benthic/ mid-water	bays, muddy and rocky areas and kelp beds	Vancouver Island to Baja	0-26m
Mackerels (Scombridae)	Pacific Chub Mackerel (*Scomber japonicus*)	3	copepods, crustaceans, euphausids, small fishes and squids	mid-water	pelagic	Indo-Pacific	to 300m
Sand Flounder (Paralichthyidae)	California Halibut (*Paralichthys californicus*)	3-4	fishes and squids	demersal	sandy bottoms, also in bays and estuaries	Northern WA to Baja	to 183m
Salmon (Salmonidae)	Chinook Salmon (*Onchorhynchus tshawytscha*)	4	primarily fishes, but also crustaceans and other inverts	mid-water	inshore and offshore, rivers and some lakes	Alaska to Ventura River, CA	to 375m
Hound Sharks (Triakidae)	Leopard Shark (*Triakis semifasciata*)	3	nektonic and benthic fishes, crustaceans, octopi and clams	demersal	enclosed muddy bays, estuaries and lagoons	Oregon to Baja	to 91m
	Brown Smoothhound (*Mustelus henlei*)	3	crabs, shrimp and some fishes	benthic	offshore, soft bottom	Northern CA to Baja	to 200m
	Gray Smoothound (*Mustelus californicus*)	3	mostly crabs, ghost shrimp, and small fish	benthic	inshore and offshore soft bottom, entering shallow muddy bays	Northern CA to Baja	to 200m
Dogfish Sharks (Squalidae)	Spiny Dogfish (*Squalus acantias*)	4	fishes, crustaceans, squid and octopi	benthic/ mid-water	Near bottom in enclosed bays and estuaries, also mid-water and near surface	Bering Sea to Chile	to 1460m
Eagle Rays (Myliobatidae)	Bat Ray (*Myliobatis californica*)	3	bivalves, worms, and occasional crabs	benthic	sandy or muddy bottom	Oregon to Gulf of CA	0-108m

Benthic – feeding on the bottom Demersal – feeding on or near bottom
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 and algae.
Trophic level 2: Organisms that consume mostly TL1 organisms (i.e. filter feeders and grazers).
Trophic level 3: Organisms that consume TL2 organisms.
Trophic level 4: Organisms that consume TL 3 organisms.

Table 8. Target species, size ranges, and numbers to include in composites.

	Species	Primary or Secondary	Mercury in Individuals or Composites	Number in Composites	Fishing Reg Min Size (in)	Fishing Reg Min Size (mm)	Median Size 2009-2010 (mm)	Targeted Size Range Individuals (mm)	Size Range Composites (mm)
Rockfish	Kelp Bass	P	I,C	5	14	356	316	3X(171-221) 2X(221-271) 5X(>271)	>271
	Barred Sandbass	P,S	I,C	5	14	356	346	3X(197-247) 2X(247-297) 5X(>297)	>297
	Spotted Sandbass	P	I,C	5	14	356	327	3X(180-230) 2X(230-280) 5X(>280)	>280
	Gopher Rockfish	P	I				281	3X(141-191) 2X(191-241) 5X(>241)	
	Blue Rockfish	P,S	C	5			293		>251
	Black Rockfish	P,S	I				380	3X(226-276) 2X(276-326) 5X(>326)	
	Scorpionfish	S	C	5	10	254	290		>254
	Olive Rockfish	S	C	5			322		>276
	Brown Rockfish	P,S	I				302	3X(159-209) 2X(209-259) 5X(>259)	
	Copper Rockfish	S	C	5			411		>352
Lingcod		P,S	I		22	559	682	3X(385-485) 2X(485-585) 5X(>585)	

Croaker	White Croaker	P,S	C	5			220		>189
	Yellowfin Croaker	S	C	5			195		>167
	Spotfin Croaker	S	C	5			221		>189
Chinook Salmon		P	C		24	610			>610
Surfperch	Barred	S	C	5			186		>159
	Redtail	S	C	5	10.5	267	No data		>267
	Shiner	P	C	20			110		>94
	Walleye	S	C	5			No data		>150
	Black	S	C	5			232		>199
	Rainbow	P	C	5			280		>240
	White	P,S	C	5			202		>173
Smelt	Jacksmelt	P,S	C	5			265		>227
	Topsmelt	S	C	5			128		>110
Chub Mackerel		P	C	5			240		>206
Shark	Leopard Shark	P	I		36	914	1238	3X(914-1074) 4X(1074-1234) 3X(>1234)	
	Spiny Dogfish	S	C	3			1011		>867
	Brown Smoothhound	S	C	3			978		>838
	Gray Smoothound	S	C	3			630		>540
Rays	Bat Ray	S	C	3			405		>347
Halibut	California Halibut	P,S	C	3			670		>574
	Pacific Halibut	S	C	3			No data		??
Cabezon		P,S	I		15	381	467	3X(300-350) 2X(350-400) 5X(>400)	
Halfmoon		S	C	5			No data		

Opaleye		S	C	5			221		>189
Kelp Greenling		S	C	5	12	305	360		>309
California Sheephead		S	C	5	12	305	No data		>305

Table 9. Summary of analytes included in the 2018-2020 study.

Analyte	Samples to Be Analyzed
Methylmercury[1]	Some individuals, all composites
PCBs	All composite samples in Bight and SF Bay, 2 species per zone in Central and North regions
DDTs	All composite samples in Bight only
Dieldrin	All composite samples in Bight only
Chlordanes	All composite samples in Bight only
Selenium	All composite samples, individuals in SF Bay white sturgeon
Arsenic	All composite samples in Bight only
PBDEs	Composites in selected species in Bight and SF Bay only
Dioxins	Composites in selected species in SF Bay and Humboldt Bay only
Per- and Polyfluoroalkyl Substances (PFASs)	Composites in selected species in SF Bay only
Microplastic	Composites in selected species in SF Bay only

[1] Measured as total mercury.

Table 10. Parameters to be measured by the SWAMP labs.

FISH ATTRIBUTES

1. Species
2. Total length
3. Fork length
4. Weight
5. Sex
6. Moisture
7. Lipid content

METALS AND METALLOIDS

1. Total mercury
2. Selenium

PCBs

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

Table 10. Parameters to be measured by the SWAMP labs (continued).

31. PCB 141
32. PCB 146
33. PCB 149
34. PCB 151
35. PCB 153
36. PCB 156
37. PCB 157
38. PCB 158
39. PCB 169
40. PCB 170
41. PCB 174
42. PCB 177
43. PCB 180
44. PCB 183
45. PCB 187
46. PCB 189
47. PCB 194
48. PCB 195
49. PCB 200
50. PCB 201
51. PCB 203
52. PCB 206
53. PCB 209

Organochlorine Pesticides

1. Chlordane, cis-
2. Chlordane, trans-
3. Heptachlor
4. Heptachlor epoxide
5. Nonachlor, cis-
6. Nonachlor, trans-
7. Oxychlordane
8. DDD(o,p')
9. DDD(p,p')
10. DDE(o,p')
11. DDE(p,p')
12. DDMU(p,p')
13. DDT(o,p')
14. DDT(p,p')
15. Aldrin
16. Dieldrin
17. Endrin
18. HCH, alpha
19. HCH, beta
20. HCH, gamma
21. Dacthal
22. Endosulfan I

23. Hexachlorobenzene
24. Methoxychlor
25. Mirex
26. Oxadiazon

PBDEs

1. PBDE 017
2. PBDE 028
3. PBDE 047
4. PBDE 066
5. PBDE 085
6. PBDE 099
7. PBDE 100
8. PBDE 138
9. PBDE 153
10. PBDE 154
11. PBDE 183

Table 11. Advisory Tissue Levels (ATLs) for Selected Fish Contaminants Based on Cancer or Non-Cancer Risk Using an Eight-Ounce Serving Size (Prior to Cooking) (ppb, wet weight). From Klasing and Brodberg (2008).

TABLE 2. ADVISORY TISSUE LEVELS (ATLS) FOR SELECTED FISH CONTAMINANTS BASED ON CANCER OR NON-CANCER RISK USING AN 8 OUNCE SERVING SIZE (PRIOR TO COOKING) (PPB, WET WEIGHT)

Contaminant	Consumption Frequency Categories (8-ounce servings/week)[a] and ATLs (in ppb)							
	7	6	5	4	3	2	1	0
Chlordanes[c]	≤ 80	>80-90	>90-110	>110-140	>140-190	>190-280	>280-560	>560
DDTs[**]	≤ 220	>220-260	>260-310	>310-390	>390-520	>520-1,000	>1,000-2,100	>2,100
Dieldrin[c]	≤ 7	>7-8	>8-9	>9-11	>11-15	>15-23	>23-46	>46
Mercury[nc] (Women 18-45 and children 1-17)	≤ 31	>31-36	>36-44	>44-55	>55-70	>70-150	>150-440	>440
Mercury[nc] (Women > 45 and men)	≤ 94	>94-109	>109-130	>130-160	>160-220	>220-440	>440-1,310	>1,310
PBDEs[nc]	≤ 45	>45-52	>52-63	>63-78	>78-100	>100-210	>210-630	>630
PCBs[nc]	≤ 9	>9-10	>10-13	>13-16	>16-21	>21-42	>42-120	>120
Selenium[nc]	≤ 1000	>1,000-1200	>1,200-1,400	>1,400-1,800	>1,800-2,500	>2,500-4,900	>4,900-15,000	>15,000
Toxaphene[c]	≤ 87	>87-100	>100-120	>120-150	>150-200	>200-300	>300-610	>610

[c]ATLs are based on cancer risk
[nc]ATLs are based on non-cancer risk
*Serving sizes are based on an average 160 pound person. Individuals weighing less than 160 pounds should eat proportionately smaller amounts (for example, individuals weighing 80 pounds should eat one 4-ounce serving a week when the table recommends eating one 8-ounce serving a week).
**ATLS for DDTs are based on non-cancer risk for two and three servings per week and cancer risk for one serving per week.

Table 12. Summary of key elements of the sampling design.

		Primary Target Species			
Region	**Number of Zones**	**Coast**	**Bays and Harbors**	**Analytes**	**Replicate Composites Within Zones**
North Coast	15	Gopher Rockfish Black Rockfish Blue Rockfish Lingcod Cabezon Salmon	Shiner Surfperch White Surfperch Leopard Shark California Halibut	Mercury Selenium PCBs Dioxins	No
Central Coast	20	Gopher Rockfish Brown Rockfish Blue Rockfish Lingcod Salmon White Croaker Rainbow Surfperch	Brown Rockfish Shiner Surfperch Leopard Shark California Halibut Jacksmelt White Croaker	Mercury Selenium PCBs	No
San Francisco Bay	6		Striped Bass Shiner Surfperch California Halibut Jacksmelt White Croaker White Sturgeon	Mercury Sele nium PCBs Dioxins PBDEs PFAS Microplastic Fipronil	Yes
Southern California Bight	27	Kelp Bass Chub Mackerel White Croaker Barred Sand Bass Gopher Rockfish	Kelp Bass Chub Mackerel White Croaker Spotted Sand Bass Shiner Surfperch	Mercury Selenium PCBs DDTs Dieldrin Chlordanes Arsenic PBDEs	Yes
Total	**68**				

Appendix III. MPSL-DFW SOPs

MPSL-DFW EPA Modifications and Laboratory Procedures			
Page	Procedure/Equipment	SOP Number	Revision Date
A	Sample Container Preparation for Organics and Trace Metals, Including Mercury and Methylmercury	MPSL-101	Nov 2016
B	Sampling Marine and Freshwater Bivalves, Fish and Crabs for Trace Metal and Synthetic Organic Analysis	MPSL-102a Tis Collection	Oct 2016
C	Sample Receipt and Check-In	MPSL-104 Receipt and Check-in	Oct 2016
D	Protocol for Tissue Sample Preparation	MPSL-105 Tissue Preparation	April 2018
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

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

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 Shoe covers: Cellucap Franklin Part # 28033

4.40 Steel Cutting Blade, Bottom: Büchi Analytical Part # 26907

4.41 Steel Cutting Blade, Top: Büchi Analytical Part # 26908

4.42 Syringe, 50 ml Luer Slip Norm-Ject: Air-Tite Part # A50

4.43 Teflon Centrifuge Tube, 30 mL: Nalge Nunc, Inc. Part # 3114-0030

4.44 Teflon MARS 5 Cap : CEM Part #

4.45 Teflon MARS 5 Plug : CEM Part # 212020

4.46 Teflon Vessel : CEM Part # 574125

4.47 Teflon Sheet, 0.002"x12"x1000': Laird Plastics Part # 112486

4.48 Teflon Tape (plumbing tape)

4.49 Teflon Tubing, 0.0625" ID 0.125" OD: ColeParmer Part # 06406-62

4.50 Teflon Tubing, 0.1875" ID 0.25"OD: ColeParmer Part # 06406-66

4.51 Teflon Vial with cap, 60 mL: Savillex Part # 0202

4.52 Teflon Vial with cap, 180 mL: Savillex Part # 0103L-2-2- $^{1}/_{8}$"

4.53 Teflon Wash Bottle, 500 mL

4.54 Titanium Cutter Screw: Büchi Analytical Part # 34376

4.55 Titanium Cutting Blade, Bottom: Büchi Analytical Part # 34307 DISCONTINUED

4.56 Titanium Cutting Blade, Top: Büchi Analytical Part # 34306 DISCONTINUED

4.57 Titanium Displacement Disc: Büchi Analytical Part # 26471

4.58 Ventilation Hood

4.59 Zipper-closure Polyethylene Bags, 4milx4"x6": Packaging Store Part # zl40406redline

4.60 Zipper-closure Polyethylene Bags, 4milx6"x8": Packaging Store Part # zl40608redline

4.61 Zipper-closure Polyethylene Bags, 4milx9"x12": Packaging Store Part # zl400912redline

4.62 Zipper-closure Polyethylene Bags, 4milx12"x15": Packaging Store Part # zl401215redline

4.63 Zipper-closure Polyethylene Bags, 4milx13"x18": Packaging Store Part # zl401318redline

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)

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_3), concentrated redistilled: BDH ARISTAR ULTRA Part: VWR 87003-658

5.14 Nitric Acid (HNO_3), BAKER INSTRA-ANALYZED'*, 69.0–70.0% (15N): VWR Part # JT9598-34

5.15 Nitric Acid (HNO_3), 7.5N (50%): prepared by adding 1 part Baker HNO_3 to 1 part MilliQ

5.16 Nitric Acid (HNO_3), 6%: prepared by adding 1 part BDH ARISTAR ULTRA HNO_3 to 16.67 parts MilliQ

5.17 Nitric Acid (HNO_3), 1%: prepared by adding 1 part BDH ARISTAR ULTRA HNO_3 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 (MPSL 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_3 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

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% BDH ARISTAR ULTRA HNO_3, cap. Rinse outside of bottle with MilliQ. Allow outside of bottle to dry.

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 30ml, 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.

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% BDH ARISTAR ULTRA HNO_3 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 bottle with 50% HCl, soak for three days. (Note: HCl may only be used up to 6 times before it must be appropriately discarded.)

7.1.9.4 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.

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_3 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.

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_3 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 rinse outised of bottle with MilliQ 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.

7.3 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.)

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 of 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.

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% BDH ARISTAR ULTRA HNO_3 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_3 by putting a small of 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% BDH ARISTAR ULTRA HNO_3 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 # MPSL-105.

8.2 Trace Metal and Mercury Only digestion procedures can be found in EPA 3052, modified, and Method # MPSL-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 # MPSL-103 or by DMA and EPA 7473.

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

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

9.0 Quality Control

9.1 See individual methods.

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

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 Safety

4.1 Personal protection equipment (PPE) such as life jackets, gloves, etc. are provided for each person. It is up to each individual to wear appropriate PPE for the task(s) they are performing.

4.2 Boating Safety training is required for boat operators and highly recommended for all personnel on board.

4.3 Safety equipment must be on board the vessel prior to launch. These items include, but are not limited to, the following: life jackets, fire extinguisher(s), air horn or whistle, red flare(s), oars or paddles, and first aid kit.

5.0 Apparatus and Materials

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

5.1 Anchor Chains

5.2 Backpack Shocker (electro-fishing)

5.3 Boats (electro-fishing and/or for setting nets)

5.4 Boat Box containing safety equipment

5.5 Bone Saw

5.6 Camera, digital

5.7 Cast Nets (10' and 12')

5.8 Data Sheets (see MPSL QAP Appendix E for example)

5.9 Daypacks

5.10 Depth Finder

5.11 Dip Nets

5.12 Dry Ice or Ice

5.13 Gill Nets (various sizes)

5.14 Gloves, leather or neoprene coated for hauling lines

5.15 Gloves, polyethylene for sample collection

5.16 GPS

5.17 Heavy Duty Aluminum Foil, prepared

5.18 Heavy Duty plastic bags, Clear 30 gallon

5.19 Buoys

5.20 Labels, gummed waterproof: Diversified Biotech Part #: LCRY-1258

5.21 Life Jackets

5.22 Nylon Cable Ties, 7/16" wide x 7" long

5.23 Other (minnow traps, set lines, throw nets, etc)

5.24 Otter Trawl (various widths as appropriate)

5.25 Permanent Marking Pen

5.26 Plastic bucket, 30 gallon

5.27 Plastic Ice Chests

5.28 Polyethylene Gloves: VWR Part # 32915-166, 32915-188, and 32915-202

5.29 Polypropylene Mesh, 76mm wide with 13mm mesh

5.30 Polypropylene Mesh, 50mm wide with 7mm mesh

5.31 Polypropylene Line, 16mm

5.32 Rods and Reels

5.33 Screw in Earth Anchor, 4-6" diameter

5.34 Scuba Gear

5.35 Seines (various size mesh and lengths as appropriate)

5.36 Stainless Steel Dive Knives

5.37 Trap Nets (hoop or fyke nets)

5.38 Teflon Forceps

5.39 Teflon Wash Bottle, 500 mL

5.40 Wading Gear

5.41 Zipper-closure Polyethylene Bags, 4milx13"x18": Packaging Store Part # zl401318redline

6.0 Reagents

6.1 Tap water (Tap)

6.2 Deionized water (DI)

6.3 Type II water (ASTM D1193): Use Type II water, also known as MilliQ, for the preparation of all reagents and as dilution water.

6.4 Micro Detergent: ColeParmer Part # 18100-20

6.5 Methanol: VWR Part # JT9263-3

6.6 Petroleum Ether: VWR Part # JT9265-3

6.7 10% Bleach

7.0 Sample Collection, Preservation and Handling

7.1 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.

7.2 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. Boats/nets should be pre-cleaned with 10% bleach to prevent introducing invasive species from one water body to another water body.

7.3 Samples are handled with polyethylene-gloved hands only. The samples should be sealed in appropriate containers immediately.

7.4 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.

7.5 Fish are wrapped in part or whole in prepared aluminum sheets and subsequently placed into zipper-closure bags.

7.6 Crabs analyzed for metals and/or organics are double-bagged in plastic zipper-closure bags.

7.7 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.

7.8 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.

7.9 Samples are maintained at -20°c and extracted or digested as soon as possible.

8.0 Procedure

8.1 Sample collection - mussels and clams

8.1.1 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 San Joaquin River at Big Break.

8.1.2 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.

8.1.3 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.

8.1.4 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.

8.1.5 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.

8.2 Transplanted sample deployment

8.2.1 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.

8.2.2 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.

8.2.3 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.

8.2.4 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.

8.2.5 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.

8.2.6 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).

8.2.7 Transplants are usually deployed for 1-6 months. Ideally mussels are transplanted in September and retrieved in January/February. Clams are usually transplanted in March or April and retrieved in May or June.

8.2.8 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.

8.3 Sample Retrieval

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

8.3.2 All mussels to be analyzed for SO are wrapped in prepared aluminum foil (Method MPSL-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.

8.3.3 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.

8.3.4 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.

8.4 Sample Collection – Fish

8.4.1 Fish are collected using the appropriate gear for the desired species and existing water conditions.

8.4.1.1 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. 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.

8.4.1.2 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. The shocked target fish are captured with a nylon net and placed in a 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.

8.4.1.3 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 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.

8.4.1.4 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.

8.4.1.5 Gill nets- Various lengths of monofilament gill nets of the appropriate mesh size for the desired fish are set out over the bow of the boat parallel to shore. The net is retrieved after being set for 1-4 hours. If necessary, nets are left overnight to collect the desired fish. 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 plastic bag and kept in an ice chest with ice.

8.4.1.6 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.

8.4.1.7 Cast net- A 6, 8, 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 plastic bag and kept in an ice chest with ice.

8.4.1.8 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 plastic bag and kept in an ice chest with ice.

8.4.1.9 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.

8.4.2 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.

8.4.3 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.

8.4.4 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.

8.4.5 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 aluminum foil 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.

8.4.6 When fish are large enough, individuals are tagged with a unique numbered tag, such as a Floy Tag. Smaller fish are foil wrapped and placed in individual bags if it is necessary to keep them individually.

8.4.7 Field data (MPSL 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, tag or bag number, fish total lengths (fork and standard lengths where appropriate), fish weight, photo number and a note of other fish caught.

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

8.5 Sample Collection- Crabs

8.5.1 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.

9.0 Analytical Procedure

9.1 Tissue Preparation procedures can be found in Method # MPSL-105.

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

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

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

9.5 Methylmercury tissue samples are extracted and analyzed according to Method # MPSL-109.

10.0 Quality Control

10.1 Field Replicates: project specific requirements are referenced for field replication.

10.2 A record of sample transport, receipt and storage is maintained and available for easy reference.

11.0 Method Performance

11.1 See individual analytical methods.

12.0 References

12.1 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.

12.2 Goldberg, E.D., ed. 1980. The International Mussel Watch. National Academy of Sciences Publ., Washington, D.C.

12.3 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.

12.4 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.

12.5 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

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 or Laser thermometer: Fisher Part # 15-077-32 or 15-077-967

4.5 3-Ring Binder

4.6 Copy Machine

4.7 Computer with Microsoft Excel and internet access

5.0 Reagents

5.1 Not Applicable

6.0 Sample Collection

6.1 Water Samples are collected according to EPA 1669, modified, MPSL-111, or 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.

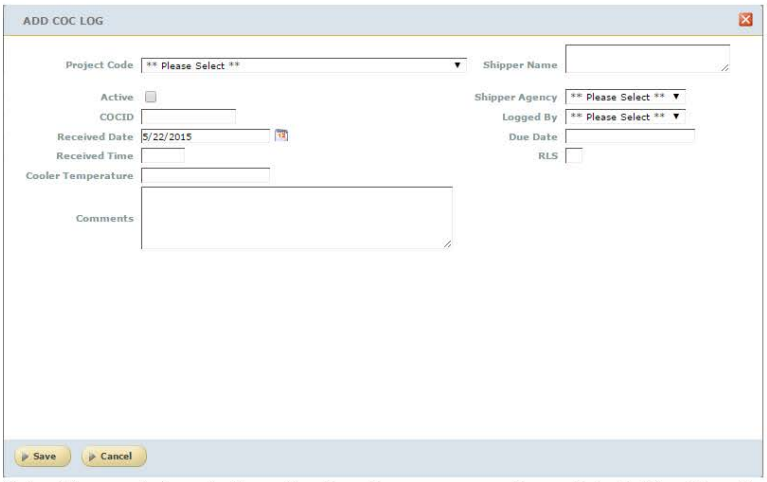
7.2 Cooler temperature is measured prior to the removal of any sample and recorded on the COC. The probe of the digital thermometer is placed amongst the samples. If a laser thermometer is used, the beam is aimed at a sample container. Temperature is allowed to equilibrate prior to recording on the COC 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 Each COC is assigned a unique COCID in the following format: YYYYMMDD_HHMM (ex 20150522_0945)

7.5 A new COC Log record is created in the MPSL database (http://rdc-gamma:8080/Security/SignIn.aspx)

7.5.1 Click Lab Logs, COC Log, then the add new button  . You'll get the following pop-up:



7.5.2 Select the project code from the dropdown menu and populate fields. Required fields are: Project Code, Active, COCID, Received Date, Received Time, Cooler Temperature, Shipper Name, Shipper Agency, Logged By, and RLS.

7.5.2.1 If there are multiple projects per COC, a unique COCID must be created for each.

7.6 A New Lab Log file is created using the LabLogTemplate.xls.

7.6.1 Begin by filling out the LabLog tab.

7.6.1.1 Required fields are in yellow: COCID, Active, QA Requested, LabNumber, GroupCode, AnalyteGroup, Preparation Preservation Name, Preparation Preservation Date, RLS and Collected Date.

7.6.1.1.1 LabNumbers are unique 9 digit numbers beginning with the year in which the sample was received. The last digits are sequential numbers beginning with 0001 (ex 2016-0001)

7.6.1.1.2 LookUp values are found in the LookUp tabs at the end of the workbook.

7.6.1.2 At least one purple field is required: Station Code or Station Name. If both are provided, please populate both fields.

7.6.2 For waters and sediments, fill out the LabDigestion tab.

7.6.2.1 Each LabNumber is assigned constituent codes based on the analyses requested. Use the ConstituentLookUp to populate template.

7.6.2.1.1 There are separate codes for total and dissolved fractions

7.6.2.1.2 Field or LabBlanks have specific codes beginning with Wb_.

7.6.2.1.3 Sediment Moisture is its own constituent.

7.6.2.2 Digestion Number = NA for all waters, chlorophyll, and mercury sediments; TBD for metals and methylmercury sediments

7.6.3 Save as a new file using a standard naming scheme to easily recall this file.

7.6.4 Print a hard copy of the LabLog and attach it to the COC and place in the COC binder.

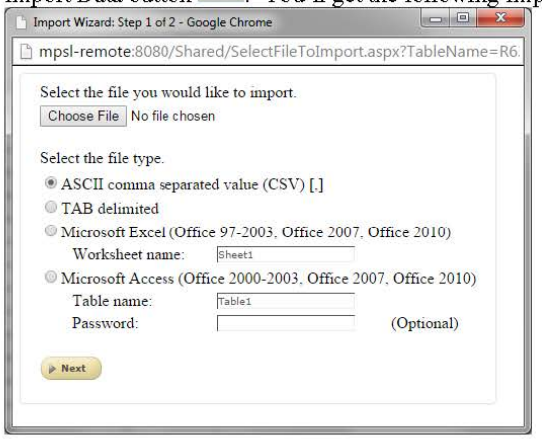
7.6.5 A second copy is firmly affixed in the bound logbook.

7.6.5.1 The date received and project name is written in indelible ink at the top of the page.

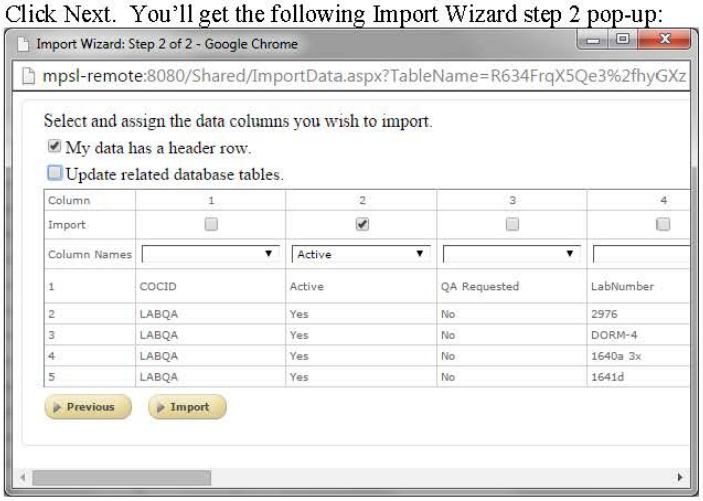
7.6.5.2 Sample Type, Preparation/Preservation, shipper name, received by name and checked by name are recorded in indelible ink.

7.6.6 Upload the LabLog

7.6.6.1 Navigate in the online database to Lab Logs, Lab Log, Lab Log - View, then the Import Data button  . You'll get the following Import Wizard step 1 pop-up:



7.6.6.2 Click Choose File. Select Microsoft Excel and type LabLog in the worksheet name. Click Next. You'll get the following Import Wizard step 2 pop-up:



7.6.6.3 Verify the first few rows of data look like what you want to import.

7.6.6.4 Uncheck Update related database tables, then click Import. If everything goes well, you'll get a response of XX records were successfully added, otherwise it will ask to save the skipped records file.

7.6.6.4.1 If a skipped records file is generated, determine and correct the error and reload the file.

7.6.7 Upload the LabDigestion for water and sediment samples. (For Tissues, see below.)

7.6.7.1 Navigate to Pre-Analyses, Water/Sediment, Lab Digestion – View, then the Import Data button  . You'll get the Import Wizard step 1 pop-up.

7.6.7.2 Click Choose File. Select Microsoft Excel and type LabDigestion in the worksheet name. Click Next. You'll get the Import Wizard step 2 pop-up.

7.6.7.3 Verify the first few rows of data look like what you want to import.

7.6.7.4 Uncheck Update related database tables, then click Import. If everything goes well, you'll get a response of XX records were successfully added, otherwise it will ask to save the skipped records file.

7.6.7.4.1 If a skipped records file is generated, determine and correct the error and reload the file.

7.6.8 For tissue samples, email the LabLogTemplate you created to the Project Manager. He or she will use CompositeTemplate.xls to add the tissue records to the online database with the following steps:

7.6.8.1 Assign Parts in the Parts tab

7.6.8.1.1 Required field are in yellow: Lab Number, Active, TissueID, Tissue Code, and RLS

7.6.8.1.2 Lab Numbers must match the LabLog previously uploaded.

7.6.8.1.3 Assign Assign a unique TissueId to each component of the Lab Number. If this is fish, use the tag number provided. If this is mussels, use the Lab Number but create individual records for A B and C

7.6.8.1.4 Use the TissueLookUp tab to identify the appropriate Tissue Code for each Part.

7.6.8.2 Assign CompositeIDs in the Composite tab.

7.6.8.2.1 Required fields are in yellow: Active, CompositeID, Homogenized Date, and RLS

7.6.8.2.2 Using either the station name, code or other identifier, assign a unique CompositeID for each composite or individual to be analyzed.

7.6.8.2.2.1 SuperComposites are assigned IDs here as well.

7.6.8.2.3 Enter 1/1/1950 under Homogenized Date. This is a placeholder only, and will be updated once homogenization has actually taken place.

7.6.8.3 Pair CompositeID and TissueID in the CompositeJunction tab. All fields are required.

7.6.8.4 If SuperComposites are created, use the CompositeDetail tab to identify the components.

7.6.8.4.1 Required fields are in yellow: Active, CompositeID, CompositeID Components, and RLS

7.6.8.5 Assign constituents to be analyzed for each composite using the CompositeDigestion tab

7.6.8.5.1 Required fields are in yellow: Active, LabSampleID, CompositeID, Constituent Code, Digestion Number, and RLS.

7.6.8.5.1.2 Assign unique LabSampleIDs to each sample to be used by the analyst in instrument software. For fish, this may be a tag number or a truncated version of the CompositeID.

7.6.8.5.1.3 Use the LookUp tabs in the workbook to identify Constituent Codes and Digestion Numbers. Moisture has its own constituent code.

7.6.8.5.1.4 Default digestion numbers are NA for mercury and TBD for all other in-house analyses. Digestion numbers for samples being shipped to other labs are lab specific and can be found in the LookUp.

7.6.8.6 Upload the new tables by navigating to each of the appropriate tables under Pre-Analyses, Tissue – Edit

7.6.8.6.1 Click the Import Data button  . You'll get the Import Wizard step 1 pop-up.

7.6.8.6.2 Click Choose File. Select Microsoft Excel and type the appropriate tab in the worksheet name. Click Next. You'll get the Import Wizard step 2 pop-up.

7.6.8.6.3 Verify the first few rows of data look like what you want to import.

7.6.8.6.4 Uncheck Update related database tables, then click Import. If everything goes well, you'll get a response of XX records were successfully added, otherwise it will ask to save the skipped records file.

7.6.8.6.4.5 If a skipped records file is generated, determine and correct the error and reload the file.

7.7 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.

7.8 Tissue, sediment and chlorophyll a samples are stored in a walk-in freezer at -20°C until dissection and/or digestion can occur.

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 # MPSL-106 and Method # MPSL-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 # MPSL-103 or by DMA and EPA 7473.

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 The online database does not allow duplicate COCIDs or 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

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 # MPSL-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

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_3), BAKER INSTRA-ANALYZED'*, 69.0–70.0%: VWR Part # JT9598-34

5.10 Nitric Acid (HNO_3), 50%: prepared by adding 1 part Baker HNO_3 to 1 part MilliQ

6.0 Sample Collection, Preservation and Handling

6.1 Samples should be collected according to Method # MSPL-102a, # MPSL-102b, and EPA 1669, modified.

6.2 All dissection equipment and containers must be prepared according to Method # MPSL-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 # MPSL-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.

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_3 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 DI.

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_3, 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.

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 # MPSL-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 # MPSL-103 or by DMA and EPA 7473.

8.4 Methylmercury tissue samples are extracted and analyzed according to Method # MPSL-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.

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

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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 pressure
Sediment 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

SWAMP IQ Procedures			
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. BOG Data Validation SOP

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 (<=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[1]

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:

[1] 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-lower limit of the range))
   - Upper Rejection Limit = 100+(2*(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	QACode	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 QACode "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 elements 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 (≤25%), or marginally fail (25%<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	QACode	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. Bight ’18 Chemistry Quality Assurance Manual

Southern California Bight
2018 Regional Marine Monitoring Survey
(Bight ‘18)

Chemistry Quality Assurance Manual

Prepared by:
Bight ’18 Chemistry Technical Committee

DRAFT February 12, 2018

V. ANALYSIS OF CHEMICAL CONTAMINANTS IN SEDIMENTS

A. Overview

There are many aspects to assuring the quality of chemical measurements. This section presents Bight '18 QA/QC protocols and requirements covering a wide range of activities, from sample collection and laboratory analysis, to the final validation of the resultant data. There have been five previous Bight surveys (1994, 1998, 2003, 2008 and 2013) and each subsequent Quality Assurance Manual has been built upon the previous manual. Guidance for the original Quality Assurance Manual was based on USEPA SW846 and protocols developed for the EMAP-E Virginian Province, as well as those developed over many years by the National Oceanic and Atmospheric Administration's (NOAA) National Status and Trends (NS&T) Program. The protocols described herein are applicable to low parts-per-billion analyses of marine sediment samples unless, otherwise noted.

The Bight '18 survey will measure a variety of organic and inorganic contaminants in marine sediment samples (Table 5-1). In addition, this survey requires that the participating analytical laboratories demonstrate comparability continuously through strict adherence to common QA/QC procedures, routine analysis of Certified Reference Materials (CRMs), and regular participation in interlaboratory comparison exercises (round-robin analyses). The QA/QC program has adopted a "performance-based" approach to achieving quality assurance of low-level contaminants. Laboratories are not required to use the same analytical methods for each type of analysis. Instead, each laboratory is free to choose the best, or most feasible method available within the constraints of cost and equipment, and provided that the resulting data meets all of the specified QA/QC criteria for accuracy, precision, and sensitivity.

Each laboratory must demonstrate its capability to meet the stated measurement quality objectives (MQOs) for each of the target analytes, in each respective matrix. Initially, each laboratory should establish a method detection limit (MDL) for each target analyte following the MDL protocol cited in 40 CFR Part 136. Laboratories must participate in any available on-going intercalibration exercises, and meet the performance criteria prior to analysis of the survey samples.

The participating laboratories must review their laboratory performance on a continuous basis and make corrections if QA/QC criteria are not met. The comparability in performance among laboratories is continuously evaluated based on analysis of certified reference materials (CRMs), selected intercalibration samples, spiked samples, sample duplicates, and laboratory reagent blanks.

B. Sample Collection, Preservation and Holding Time

Field personnel must strictly adhere to Bight '18 protocols to insure the collection of representative, uncontaminated sediment chemistry samples. These sample collection protocols

are described in detail in the Field Operations Manual. Briefly, the key aspects of quality control associated with chemistry sample collection are as follows:

- Field personnel must be thoroughly trained in the proper use of sample collection gear, and must be able to distinguish acceptable versus unacceptable sediment grab samples in accordance with pre-established criteria.

- Field personnel must be thoroughly trained to recognize and avoid potential sources of sample contamination (e.g., engine exhaust, winch wires, deck surfaces, ice used for cooling).

- Samplers and utensils that come in direct contact with the sample should be made of non-contaminating materials (high-quality stainless steel only) and should be thoroughly cleaned between sampling stations.

- Sample containers should be of the recommended type (Table 5-2) and must be free of contaminants (i.e., carefully pre-cleaned).

- Conditions for sample collection, preservation and holding times should be followed (Table 5-2).

C. Laboratory Operations

Overview

The Bight '18 survey will involve the distribution of sediment chemistry samples among several different laboratories. Each participating laboratory will analyze samples using existing methodology and report results for the constituents listed in Table 5-1.

The QA/QC requirements presented in the following sections are intended to provide a common foundation for the protocols used by each laboratory. The resultant QA/QC data will facilitate assessment of the comparability of results among the different laboratories and for the different analytical procedures. The QA/QC requirements specified in this plan represent the minimum requirements for any given analytical method. Additional method-specific requirements should always be followed, as long as the minimum requirements presented in this document have been met.

The performance-based Bight '18 QA program for analytical chemistry laboratories is based on an initial demonstration of laboratory capability (e.g., performance evaluation) and an ongoing demonstration of capability. Control limit criteria and recommended frequency of analysis for each QA/QC element or sample type required in the Bight '18 program are summarized in Tables 5-3 to 5-6. The following sections discuss general aspects of the QA/QC elements.

Prior to the analysis of samples, each laboratory should calculate nominal MDLs for each analyte, establish an initial calibration curve for all analytes, and demonstrate acceptable performance on a known or blind accuracy-based material. Following a successful first phase, the laboratory must demonstrate its continued capabilities by participating in an on-going series of interlaboratory comparison exercises, repeated analysis of certified reference materials (CRMs), laboratory control standards, and analysis of laboratory method blanks and spiked samples. These steps are detailed in the following sections.

The results for the various QA/QC samples should be reviewed by laboratory personnel immediately following the analysis of each sample batch. The results should then be used to determine whether any control limit criteria have not been met, and if corrective actions must be taken before any further sample analyses.

To accomplish the objectives of the Bight '18 study, three criteria must be met for any analytical methods used:

- Sufficient sensitivity must be obtained to achieve the required data reporting objectives for any target analytes (Table 5-1). The confidence of these reporting requirements is estimated by assessing the analytical variation resulting from repeated analyses of spiked samples close to these levels (sensitivity criteria).

- Performance of each laboratory must be consistent with that of the other laboratories. Laboratories analyzing the Bight '18 samples must participate in the on-going intercalibration exercises. The results must be within specified limits agreed upon by the chemistry committee.

- Analyses of certified reference materials must yield values within the specified range of the certified values. However, due to the inherent variability in analyses near the method detection limit, control limit criteria for relative accuracy will only apply to analytes having certified values that are >10 times the MDL established by the laboratory (accuracy criteria).

The on-going intercalibration exercises are used to provide an initial check on the performance of the participating laboratories against these criteria. Any laboratory that fails to meet these criteria should repeat analyses of the intercalibration samples before commencing analyses of actual Bight '18 survey samples.

Continuous performance evaluation against these criteria can be achieved by analyses of sample duplicates, spiked blanks, matrix spikes, reporting level spikes, laboratory control standards, and certified reference materials. The data quality requirements for the Bight '18 study are summarized in Tables 5-3 to 5-6. Discussion of each component is detailed below.

Initial calibration

Equipment should be calibrated prior to the analysis of each sample batch, after each major equipment disruption, and whenever on-going continuing calibration checks do not meet recommended control limit criteria (Table 5-1).

Organics. The calibration range must be established for each constituent from a minimum of five analytical standards of increasing concentration. The calibration range should be well characterized and must be established prior to the analysis of samples. Only data resulting from quantification within the demonstrated working calibration range may be reported by a laboratory without annotation (i.e., quantification based on extrapolation outside the calibration range is not acceptable). Samples with measured concentrations above the calibration range should be diluted as appropriate, and reanalyzed. For results below the lowest calibration point or reporting limit (RL), samples may be further concentrated, or the results must be "flagged" (annotated) as <RL. The latter is acceptable only if: (1) sample extraction/concentration steps were sufficient to meet the target analyte RL goals of the study, or (2) matrix problems have required sample dilution.

Trace metals. ICP/AES instruments are calibrated with a calibration blank and a minimum of one calibration standard. ICP/MS and the atomic absorption spectrometers including flame atomic absorption (FAA), graphite furnace (GFAA), hydride generation, and cold vapor are calibrated using a minimum of 1 blank and three calibration standards. The linear coefficient of the calibration curve must be at least 0.995 to be acceptable.

Initial documentation of method detection limits

In the Bight '18 program, the MDL will be used to demonstrate the capability of a laboratory to reach the sensitivity required to measure a specific constituent and demonstrate acceptable precision. The MDL represents a quantitative estimate of low-level response detected at the maximum sensitivity of a method. The Code of Federal Regulations (40 CFR Part 136) gives the following rigorous definition: *"The MDL is 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."* The calculated MDL is a function of method precision at low analyte concentrations.

Each laboratory is to follow the procedure specified in 40 CFR Part 136 (Federal Register, Oct. 28, 1984) to calculate nominal MDLs for each target analyte and each analytical method employed. Briefly, at least seven replicates of each representative matrix should be spiked at a concentration between one and five times the estimated detection limit (except for certain trace metals; see below for details), or at RL as a default. The amount of sample (i.e., mass of sediment or tissue) used in calculating the MDL should match, as closely as possible, the amount of sample typically used. The mean and standard deviation of the replicates are used to compute the MDL by multiplying the standard deviation by the Student t value for the 99% confidence interval (for n=7, t=3.143).

Trace metals. The MDLs for aluminum, antimony, arsenic, barium, beryllium, cadmium, chromium, copper, iron, lead, mercury, nickel, selenium, silver, and zinc should be determined on a certified reference material or be calculated from a spiked clean matrix.

Reporting levels

In the Bight '18 program, each laboratory will report results down to at least their established reporting limits. Each laboratory RL must be at or below the concentrations listed in Table 5-1. Results should be flagged if the they are between the RL and the MDL.

Calibration verification

An initial calibration verification standard is analyzed at the beginning of each analysis following the calibration procedure to check the accuracy of the calibration. For all the analytical techniques, one initial calibration verification standard is required from a source different from the source that is used for the calibration standards. The initial calibration verification standard is near the mid-range of the calibration and must be within ±10% of the true value when analyzed. ICP/AES also requires a second initial calibration check standard of a substantially different concentration than the first initial calibration check standard; the second initial calibration check standard must also be within ±10% of the true value when analyzed.

For continuing trace metal measurements, the continuing calibration verification (CCV) verifies that the instrument stays in calibration throughout the analysis. The CCV is prepared in the same acid matrix as the calibration standard. It is analyzed after every ten samples and at the end of the run. The CCV can come from any source that is near the mid-range of the calibration and must be within the ranges specified in Table 5-3.

Calibration blanks (trace metals)

Laboratories need to analyze calibration blanks (pure matrix used to prepare calibration standard solutions) prior to analysis of samples to ensure that the instrument is free of contamination. Concentrations of all target analytes obtained from analysis of the calibration blanks should be below MDLs.

Method blanks

Method blanks (also called procedural blanks) are used to assess laboratory contamination during all stages of sample preparation and analysis. For both organic and inorganic analyses, one laboratory reagent blank should be run in every sample batch. The method blank should be processed through the entire analytical procedure in a manner identical to the samples. Control limits for blanks (Tables 5-4 to 5-6) are based on the laboratory's maximum acceptable method detection limits (trace metals) or reporting levels (trace organics and TOC) as documented prior to the analysis of samples. For trace metals, it is preferable that the level of any analyte in the method blank be below the MDL. Alternatively, the concentration of any target analyte must be less than 5% of the ERL for those constituents that have an ERL established, or less than 5% of the concentration of the analyte in the sample for those analytes without established ERL values. A reagent blank concentration equal to or greater than three times the MDL for one or more of the analytes of interest requires definitive corrective action to identify and eliminate the source(s) of contamination before proceeding with sample analysis.

For trace organics, if the method blank contains any analyte with a measured concentration greater than RL, all samples for that batch should be re-analyzed if the analyte is detected in samples. Concentrations lower than RL should be reported, but not used to correct concentrations in the field samples.

Sample duplicates

Analysis of sample duplicates is used to assess the precision of an analytical method in quantifying target analytes and not required for all methods. Samples collected in the field have the potential to be highly heterogeneous. It is incumbent on the laboratory to make a reasonable effort to homogenize the samples prior to analysis but it is still possible for sample homogeneity to have a large effect on the variability of the results. The relative percent difference (RPD) between the sample and sample duplicate results is calculated as follows:

$$RPD = \frac{(C1 - C2)}{(C1 + C2)/2} \times 100$$

Where: C1 = the larger of the duplicate results for a given analyte, and
C2 = the smaller of the duplicate results for a given analyte.

The data from this process are typically used to establish a statistical range with which the precision of subsequent analyses can be assessed.

Matrix spikes and matrix spike duplicates

A laboratory spiked sample matrix (commonly called a matrix spike or MS) and a laboratory spiked sample matrix duplicate (commonly called a matrix spike duplicate or MSD) will be used both to evaluate the effect of the sample matrix on the recovery of the compound(s) of interest and to provide an estimate of analytical precision. A minimum of one MS/MSD should be analyzed for 10% of samples. The matrix spike solution should contain all the analytes of interest. The final spiked concentration of each analyte in the sample should be between 10 and 100 times the MDL for that analyte, as previously calculated by the laboratory. If the unspiked sample contains more than this amount, then the sample should be spiked with one to five times the preexisting concentration in the sample.

Recovery data for the fortified compounds ultimately are intended to provide a basis for determining the prevalence of matrix effects in the samples analyzed during the project. However, these data may not reflect the true magnitude of matrix interference with the analyses since recently spiked analytes often do not permeate the sample matrix to the same extent as in field contaminated sediments. This is particularly true for measurements of trace organics in complex matrices. Therefore, it is recommended that recovery data from analyses of MS and MSD samples be used only as an evaluation tool for methods measuring trace organics.

For trace metals, the spike control limits are presented in Table 5-3 for all elements except iron and aluminum due to their high concentrations. If the percent recovery for any

analyte in the MS or MSD is lower than the control limits, the raw data quantitation reports should be reviewed. If the reason for a low percent recovery value is not identified, the instrument response may be checked using a calibration standard. Low matrix spike recoveries may be a result of matrix interference and further instrument response checks may not be warranted, especially if the low recovery occurs in both the MS and MSD, and the other QC samples in the batch indicate that the analysis was "in control". An explanation for low percent recovery values for MS/MSD results should be given in the cover letter accompanying the data package. Corrective actions taken and verification of acceptable instrument response must be included. These corrective actions can include re-analysis of the samples associated with the MS/MSD.

Analysis of the MS/MSD also is useful for assessing laboratory precision. The RPD between the MS and MSD results should be within the control limits (Tables 5-3 to 5-6) for at least one result per batch. If results for any analytes do not meet the control limit criteria, calculations and instruments should be checked. A repeat analysis may be required to confirm the results.

Certified reference materials

Certified reference materials (CRMs) generally are the most useful QC samples for assessing the accuracy of a given analysis (i.e., closeness of a measurement to the "true" value). CRMs can be used to assess accuracy because they have "certified" concentrations of the analytes of interest, as determined through replicate analyses by a reputable certifying organization using two independent measurement techniques for verification. In addition, the certifying organization may provide "non-certified" or "informational" values for other analytes of interest. Such values are determined using a single measurement technique, which may introduce unrecognized bias. Therefore, non-certified values must be used with caution in evaluating the performance of a laboratory using a method which differs from the one used by the certifying organization. A list of reference materials used for the Bight '18 study is presented in Table 5-7.

A laboratory control material (LCM) may be used in addition to, but not as a replacement for, CRMs. A LCM is similar to a CRM in that it is a homogeneous matrix that closely matches the samples being analyzed. Although the concentrations of the target analytes in these materials are not certified, they can be used to assess the precision (i.e., consistency) of a single laboratory, and to determine the degree of comparability among different laboratories. In practice, LCMs may be preferred for routine (i.e., day to day) analysis because CRMs are relatively expensive. Moreover, as-collected (i.e., wet) LCMs from the study area are more representative of the types of samples that will be delivered to the laboratories during the actual study. However, for the Bight '18 study the specified CRMs must be analyzed with every sample batch to provide a check on analytical performance.

Routine analysis of CRMs and LCMs is a vital aspect of the "performance-based" Bight '18 QA philosophy. For the organic analyses, one CRM (NIST 1944) must be analyzed along with each batch of samples. For the metals analyses, CRM 540 must be analyzed with each batch of samples. However, only one of these CRM (540) will be used for determination data

acceptability criteria. For CRMs, both the certified and non-certified concentrations of the target analytes should be known to the analyst(s) and should be used to provide an immediate check on performance before proceeding with a subsequent sample batch. Performance criteria for both precision and accuracy have been established for analysis of CRMs and LCMs (Tables 5-3 to 5-6).

If the laboratory fails to meet either the precision or accuracy control limit criteria for a given analysis of the CRM, the data for the entire batch of samples is suspect. Calculations and instruments should be checked; the CRM may have to be reanalyzed to confirm the results. If the values are still outside the control limits in the repeat analysis, the laboratory is required to find and eliminate the source(s) of the problem and repeat the analysis of that batch of samples until control limits are met, before continuing with further sample processing. The results of the CRM or LCM analysis should never be used by the laboratory to "correct" the data for a given sample batch.

Surrogate standards

Recovery surrogates are compounds chosen to simulate the analytes of interest in organic analyses. The recovery surrogate represents a reference analyte against which the signal from the analytes of interest is compared directly for the purpose of determining extraction efficiency. Recovery surrogates must be added to each sample, including QA/QC samples, prior to extraction. The reported concentration of each analyte should NOT be adjusted to correct for the recovery of the surrogate standards. The surrogate recovery data should be monitored; each laboratory must report the percent recovery of the surrogate(s) along with the target analyte data for each sample. If possible, isotopically labeled analogs of the analytes should be used as recovery surrogates for GC/MS analyses.

Control limit criteria for surrogate recoveries are provided in Tables 5-4 to 5-5. Each laboratory should set its own control limit criteria based on the experience and best professional judgment of the analyst(s). It is the responsibility of the analyst(s) to demonstrate that the analytical process is always "in control" (i.e., highly variable surrogate recoveries are not acceptable for repeat analyses of the same certified reference material and for the matrix spike/matrix spike duplicate).

Internal standards (organics)

Internal standards are added to each sample extract just prior to instrumental analysis to enable optimal quantification, particularly of complex extracts subject to matrix effects or 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 also can be used to detect and correct for problems in the instrument. The elements or compounds used as internal standards must be different from those already used as recovery surrogates. The analyst(s) should monitor internal standard retention times and recoveries to determine if instrument maintenance or repair, or changes in analytical procedures, are indicated. Corrective action should be initiated based on the experience of the analyst(s) and not solely because warning or control limits are exceeded. Instrument problems that may have affected the

data or resulted in the reanalysis of the sample should be documented properly in logbooks and internal data reports and used by the laboratory personnel to take appropriate corrective action.

D. Data Evaluation Procedures

It is the responsibility of the Project Manager or his designee to acknowledge initial receipt of the data package(s), verify that the four data evaluation steps (see below) are completed. The analytical laboratory must be notified of any additional information or corrective actions deemed necessary after the data evaluation. Following satisfactory resolution of all "corrective action" issues, the final action is to notify the laboratory in writing that the submitted results have been officially accepted as complete. It may be necessary or desirable for a team of individuals (e.g., the QA Coordinator, Lab Coordinator and/or staff analytical chemists) to assist the Project Manager in technical evaluation of the submitted data packages. While the Project Manager has ultimate responsibility for maintaining official contact with the analytical laboratory and verifying that the data evaluation process is completed, it is the responsibility of the QA Coordinator to closely monitor and formally document each step in the process as it is completed. This documentation should be in the form of a data evaluation tracking form or checklist that is filled in as each step is completed. This checklist should be supplemented with detailed memos to the project file outlining any concerns with data omissions, analysis problems, or descriptions of questionable data identified by the laboratory.

Evaluation of the data package should begin as soon as possible following its receipt, since delays increase the chance that information may be misplaced or forgotten. In addition, if holding times have been exceeded, options for reanalysis may be limited. The following steps are to be followed and documented in evaluating Bight '18 chemistry data:

- Checking data completeness (verification)
- Assessing data quality (validation)
- Assigning data qualifier codes
- Taking final actions

Checking data completeness

The first part of data evaluation is to verify that all required information has been provided in the data package. For the Bight '18 survey, this should include the following steps:

- Project personnel should verify that the package contains the narrative explanations signed by the laboratory manager, hard copies of all results (including QA/QC results), and accompanying computer diskettes.

- The electronic data file(s) should be parsed and entered into the Bight '18 chemistry database to verify that the correct format has been supplied.

- Once the data have been entered into the appropriate Bight '18 database, automated checks should be performed to verify that results have been reported

for all expected samples and all analytes.

The Project Manager should contact the laboratory and request any missing information as soon as possible after receipt of the data package. If information was omitted because required analyses were not completed, the laboratory should provide and implement a plan to correct the deficiency. This plan may include submittal of a revised data package and possible reanalysis of samples.

Assessing data quality

Data validation, or the process of assessing data quality, can begin after Bight '18 personnel have determined that the data package is complete. Normally, the first major part of validation involves checking 100% of the data for any possible errors resulting from transcription of tabulated results, misidentification or miscalculations. However, Bight '18 laboratories are expected to submit data that has been tabulated and checked thoroughly for accuracy; the raw data reports needed to perform these checks (e.g., chromatograms, original quantitation reports) are not submitted as part of the data package. The laboratory is required to maintain this raw data in an orderly manner and to have these records available for review by Bight '18 personnel upon request. The first-step validation checks performed by Bight '18 personnel will be limited to the following:

1. A check to verify that all reporting units and numbers of significant figures are correct.

2. A check to verify that all of the laboratory's calculated percent recovery values (for calibration check samples, Laboratory Control Materials, and matrix spikes) and relative percent difference values (for duplicates) are correct.

3. A check to verify that the reported concentrations for each analyte fall within "environmentally-realistic" ranges, determined from previous studies and expert judgment. In addition, past studies indicate that the different compounds in each class of chemicals being measured on Bight '18 (e.g., PAHs, PCBs, DDTs and other chlorinated pesticides) typically occur in the environment in more or less fixed ratios to one another. For example, the DDT breakdown products p,p-DDD and p,p-DDE typically occur at higher concentrations than p,p-DDT in marine sediments in off Southern California. If anomalous departures from expected relative concentrations are found, it may indicate a problem in the measurement or data reduction, which in turn warrants further investigation.

The second major aspect of data validation is to compare the QA/QC data against established criteria for acceptable performance. This will involve the following steps:

1. Results for QA/QC samples should be tabulated, summarized and evaluated. A set of summary tables should be prepared from the database showing the percent recovery values and relative percent difference values (where applicable) for the CRMs, LCMs and matrix spike/matrix spike duplicate samples. The tables should indicate the percent

recovery values for each individual batch of samples, as well as the average, standard deviation, coefficient of variation, and range for all batches combined.

2. Similar summary tables should be prepared for the laboratory reagent blank QA/QC samples.

3. The summary results, particularly those for the CRMs and/or LCMs should be evaluated by comparing them against the QA/QC warning and control limit criteria for accuracy, precision, and blank contamination.

4. Method detection limits reported by the laboratory for each analyte should be tabulated.

There are several possible courses of action to be taken if the reported data are deficient (i.e., warning and/or control limits exceeded) during the assessment of data quality. The laboratory's cover letter (narrative explanation) should be consulted to determine if the problems were satisfactorily addressed. If only warning limits were exceeded, then it is appropriate for the laboratory to report the results. Violation of control limits, however, will result in one of the following courses of action. Either all associated results will be qualified in the database as estimated values (explained in the following section), or the data will be rejected and deleted from the database because the analysis was judged to be out of control (based on the professional judgment of the reviewer).

Assigning data qualifier codes

Data qualifier codes are notations used by laboratories and data reviewers to briefly describe, or qualify, data and the systems producing data. Bight '18 data reviewers will assign data qualifier codes in situations where there are violations of control limit criteria. The most typical situation is when a laboratory fails to meet the accuracy control limit criteria for a particular analyte in a CRM or matrix spike sample. In these situations, the QA reviewer should verify that the laboratory did meet the control limit criteria for precision. If the lack of accuracy is found to be consistent (i.e., control limit criteria for precision were met), then it is likely that the laboratory experienced a true bias for that particular analyte. In these situations, all reported values for that particular analyte will be qualified with a code that has the following meaning: *"The reported concentration is considered an estimate because control limits for this analyte were exceeded in one or more quality control samples."*

Because some degree of expert judgment and subjectivity typically is necessary to evaluate chemistry QA/QC results and assign data qualifier codes, data validation will be conducted only by qualified personnel. It is the philosophy of the Bight '18 that data which are qualified as estimates because of minor violation of a control limit in a QA/QC sample are still usable for most assessment and reporting purposes. However, it is important to note that all QA/QC data will be readily available in the database along with the results data, so that interested data users can make their own estimation of data quality.

Taking final action

Upon completion of the above steps, a report summarizing the QA review of the data package should be prepared, samples should be properly stored or disposed of, and laboratory data and accompanying explanatory narratives should be archived both in a storage file and in the database. Technical interpretation of the data begins after the QA review has been completed.

Reports documenting the results of the QA review of a data package should summarize all conclusions concerning data acceptability and should note significant quality assurance problems that were found. These reports are useful in providing data users with a written record on data concerns and a documented rationale for why certain data were accepted as estimates or were rejected. The following items should be addressed in the QA report:

1. Summary of overall data quality, including a description of data that were qualified.

2. Brief descriptions of analytical methods and the method(s) used to determine detection limits.

3. Description of data reporting, including any corrections made for transcription or other reporting errors, and description of data completeness relative to objectives stated in the QA Project Plan.

4. Descriptions of initial and ongoing calibration results, blank contamination, and precision and bias relative to QA plan objectives (including tabulated summary results for CRMs, LCMs and matrix spike/matrix spike duplicates).

The chemistry QA results will be presented in the appropriate Bight '18 technical reports,and will also become a permanent part of the database documentation (i.e., meta data). The QA/QC data collected by the Bight '18 will be used not only to assess the accuracy and precision of individual laboratory measurements, but ultimately to assess the comparability of data generated by multiple laboratories.

Table 5-1. Bight '18 Marine Monitoring Survey Target Analyte List and Reporting Limits For Sediments

Metals	Reporting Limit (ng/g dry wt)
Aluminum	NA
Antimony	10,000
Arsenic	1,600
Barium	NA
Beryllium	200
Cadmium	90
Chromium	16,000
Copper	7,000
Iron	NA
Lead	9,300
Mercury	30
Nickel	4,200
Selenium	1,000
Silver	200
Zinc	30,000

PBDEs	Reporting Limit (ng/g dry wt)
BDE-17	0.1
BDE-28	0.1
BDE-47	0.1
BDE-49	0.1
BDE-66	0.1
BDE-85	0.1
BDE-99	0.1
BDE-100	0.1
BDE-138	0.1
BDE-153	0.1
BDE-154	0.1
BDE-183	0.1
BDE-190	0.1

Pesticides	Reporting Limit (ng/g dry wt)
4,4'-DDT	0.5
2,4'-DDT	0.5
4,4'-DDD	0.5
2,4'-DDD	0.5
4,4'-DDE	0.5
2,4'-DDE	0.5
4,4'-DDMU	0.5
alpha-Chlordane	0.5
gamma-Chlordane	0.5
cis-nonachlor	0.5
trans-nonachlor	0.5
oxychlordane	0.5

Pyrethroids	Reporting Limit (ng/g dry wt)
Bifenthrin	0.5
Cyfluthrin (total)	0.5
Cypermethrin (total)	0.5
lambda-Cyhalothrin (total)	0.5
cis-Permethrin	0.5
trans-Permethrin	0.5
Deltamethrin	0.5
Esfenvalerate	0.5

Fipronils	Reporting Limit (ng/g dry wt)
Fipronil	0.5
Fipronil Desulfinyl	0.5
Fipronil Sulfide	0.5
Fipronil Sulfone	0.5

Table 5-1 (Cont.). Bight '18 Marine Monitoring Survey Target Analyte List and Reporting Limits For Sediments

PCBs	Reporting Limit (ng/g dry wt)
PCB-8	3
PCB-18	3
PCB-28	3
PCB-37	3
PCB-44	3
PCB-49	3
PCB-52	3
PCB-66	3
PCB-70	3
PCB-74	3
PCB-77	3
PCB-81	3
PCB-87	3
PCB-99	3
PCB-101	3
PCB-105	3
PCB-110	3
PCB-114	3
PCB-118	3
PCB-119	3
PCB-123	3
PCB-126	3
PCB-128	3
PCB-138	3
PCB-149	3
PCB-151	3
PCB-153	3
PCB-156	3
PCB-157	3
PCB-158	3
PCB-167	3
PCB-168	3
PCB-169	3
PCB-170	3
PCB-177	3
PCB-180	3
PCB-183	3
PCB-187	3
PCB-189	3
PCB-194	3
PCB-195	3
PCB-201	3
PCB-206	3

PAHs	Reporting Limit (ng/g dry wt)
1,6,7-Trimethylnaphthalene	20
1-Methylnapthalene	20
1-Methylphenanthrene	20
2,6-Dimethylnaphthalene	20
2-Methylnapthalene	20
Acenaphthene	20
Acenaphthylene	20
Anthracene	20
Benz[a]anthracene	80
Benzo[a]pyrene	80
Benzo[b]fluoranthene	80
Benzo[e]pyrene	80
Benzo[g,h,i]perylene	80
Benzo[k]fluoranthene	80
Biphenyl	20
Chrysene	80
Dibenz[a,h]anthracene	80
Fluoranthene	80
Fluorene	20
Indeno[1,2,3-c,d]pyrene	80
Naphthalene	20
Perylene	80
Phenanthrene	20
Pyrene	80

Table 5-2. Summary of chemistry sample collection and holding time conditions. (Maximum holding time for mercury is 6 months.)

Parameter	Container Type	Container Size (mL)	Preservation Requirements	Maximum Holding Time[a]	Data Submittal Time
Sediment Grain Size	plastic	125 (80% full)	cold (4 °C)	6 months	6 months
Sediment Total Organic Carbon	amber glass	250 (80% full)	frozen (-20 °C)	1 year	6 months
Trace Metals	amber glass	250 (80% full)	frozen (-20 °C)	1 year	6 months
Trace Organics	amber glass	2 x 125 (80% full)	frozen (-20 °C)	1 year	6 months

a Holding time starts from sampling date for sediment and from compositing date for tissue.

Table 5-3
Summary of Data Quality Objectives for the
Trace Metal Measurements

MEASUREMENT	FREQUENCY	CONTROL LIMIT
Method Blank	1/batch	<MDL or <5% of the measured concentration in samples
Certified Reference Materials ERA Soil #540 LOT#099	1/batch	Within PT performance acceptance limits of certified values for all 15 analytes
ICP-AES		
Calibration	Initial setup	Minimum 1 blank and one calibration standard
Interference check	1/run	±20% true value
Initial calibration verification (ICV)	2 points/batch	±10% true value
Continuing calibration verification (CCV)	10%	±10% true value
Matrix spike	10%	At least one matrix spike per batch must be within 30% true value. Should all spiked sample recoveries be outside 30% of true value, add a post-digestion spike to the unspiked sample and analyze. If all spike recoveries are outside 30% of true value, note matrix caused poor spike recovery. If all spike recoveries are within 30% of true value, repeat digestion. Spike duplicate results must have an RPD ≤ 20% if MSD is analyzed.
Spiked blank	1/batch	±25% true value
Duplicate sample or matrix spike sample		10% Statistical process control analyses (within 3σ)
ICP-MS		
Calibration	Initial setup	Minimum 1 blank and three calibration standards
Initial calibration verification (ICV)	1 point/batch	±10% true value
Continuing calibration verification (CCV)	10%	±10% true value
Calibration Blank	10%	<MDL. If > MDL, run two more times, the average must be <MDL. If average > MDL, reanalyze.
Matrix spike	10%	At least one matrix spike per batch must be within 30% true value; ≤ 30% RPD for over 10 times MDL. If ≥ 30% RPD and

Table 5-3
Summary of Data Quality Objectives for
Trace Metal Measurements (Cont.)

		post-digestion spike recovery is > 25% note matrix problem. If > 20% RPD and post-digesiton spike recovery is ≤ 25% repeat digestion and analysis
Spiked blank	1/batch	±25% true value
Duplicate sample or matrix spike sample	10%	Within ±30% RPD
Atomic Absorption (AA, GFAA, Hydride Generation, Cold Vapor)		
Calibration	Initial setup	Minimum 1 blank and three calibration standards; linear coefficient ≥ 0.995
Initial calibration verification (ICV)	1/batch	±10% true value
Continuing calibration verification (CCV)	10%	±20% true value
Calibration Blank	10%	<MDL. If > MDL, run two more times, the average must be <MDL. If average > MDL, reanalyze.
Matrix spike	10%	At least one matrix spike per batch must be within 30% true value. If all matrix spike analyses are ≥ 20%, interference test must be conducted
Spiked blank	1/batch	15% true value
Duplicate sample or matrix spike sample	10%	Within ±30% RPD
Interference check	As required	(a) Dilution test: Select typical sample with concentration 25 times the MDL. Dilute sample 5 times. The concentration of the undiluted sample and 5 times the concentration of the diluted sample must be within 10%. If > 10% or all samples are below 10 times the MDL, then proceed to (b). (b) Post-digestion spike: Spike sample to bring concentration to 2 to 5 times the original concentration or 20 times the MDL. The recovery must be within 15%. If not, perform the standard addition procedure described in USEPA SW846

Table 5-4
Summary of Data Quality Objectives for
Polynuclear Aromatic Hydrocarbon[a] Measurements

MEASUREMENT	FREQUENCY	CONTROL LIMIT
Initial calibration		Relative standard deviation (RSD) of the response factor within ± 25% for 80% of the analytes. Or correlation coefficient ($r^2 > 0.990$) for linear and non-linear curves. First or second order curves allowed.
Initial calibration verification	1/batch	Initial calibration verification should be performed immediately following the initial calibration. Relative percent difference (RPD) compared to initial calibration should be less than 30% of all analytes. Second source of calibration standards is used.
Cont. calibration verification	1 set/batch	Continued calibration verification should be performed at the beginning and end of each batch. The one in the middle is optional for long batches or long run times. Relative percent difference (RPD) compared to intial calibration should be less than 20% for 80% of the analytes. The same source of standards to initial calibration is used.
Method Blank	1/batch	< 10 times the MDL for all analytes
Matrix spikes/MS duplicates	1/batch	60-140% recovery of spiked mass for >80% of analytes; RPD <40% for >70% of analyte
Certified reference material	1/batch	Within ± 40% of specified value for ≥ 80% of analytes selected by and agreed to by the Chemistry Technical Subcommittee
Surrogate spikes	1/sample	Laboratories develop their own control limits; all surrogate recovery data should be reported
Internal standards (Optional)	1/sample	Laboratories develop their own

[a]maximum of 20 samples per extraction batch and a reasonable number of sample extracts per instrument batch.

Table 5-5
Summary of the Data Quality Objectives for
PCBs, Chlorinated Pesticides, Pyrethroids, PBDEs, and Fipronil Measurements[a]

MEASUREMENT	FREQUENCY	CONTROL LIMIT
Initial calibration		Relative standard deviation (RSD) of the response factor within ± 25% for 80% of the analytes. Or correlation coefficient ($r^2 > 0.990$) for linear and non-linear curves. First or second order curves allowed.
Initial calibration verification	1/batch	Initial calibration verification should be performed immediately following the initial calibration. Relative percent difference (RPD) compared to initial calibration should be less than 30% of all analytes. Second source of calibration standards is used.
Cont. calibration verification	1 set/batch	Calibration verification should be performed at the beginning and end of each batch. The one in the middle is optional for long batches or long run times. Relative percent difference (RPD) compared to intial calibration should be less than 25% for 80% of the analytes. The same source of standards to initial calibration is used.
Method Blank	1/batch	< 10 times the MDL for all analytes and also < RL
Certified reference material[b]	1/batch	Within ±40% of the certified value for ≥ 70% of the analytes selected by and agreed to by the Chemistry Technical Subcommitee
Matrix spikes/duplicates (MS/MSD)	1 set/batch	60-140% recovery of spiked mass for > 70% of analyte within each class; RPD <40% for >70% of analyte
Surrogate spikes	1/sample	Laboratories develop their own control limits
Internal standards (Optional)	1/sample	Laboratories develop their own

[a] maximum of 20 samples per extraction batch and a reasonable number of sample extracts per instrument batch.
[b] pertains to analytes where certified values are available and documented

Table 5-6
Summary of the Data Quality Objectives for Total Organic Carbon[a] Measurements

MEASUREMENT	FREQUENCY	CONTROL LIMIT
Initial calibration		RSD < 20%
Calibration verification	1/batch	RPD compared to inital calibraiton should be less than 20%
Calibration blank	1/batch	Below MDLs
Method blank	1/batch	< 10 Times the MDL
Sample duplicates	1/batch	RPD < 30%
Certified reference material	1/batch	Within ±20% of certified value

[a] maximum of 20 samples per extraction batch and a reasonable number of sample extracts per instrument batch.

Table 5-7
Certified Reference Materials Recommended by the Chemistry Technical Committee

Calibration solution	
SRM 1491	Aromatic hydrocarbons in hexane/toluene
SRM 1492	Chlorinated pesticides in hexane
SRM 1493	Polychlorinated biphenyl congeners in 2,2,4-trimethylpentane
Environmental matrix (Organics)	
CRM-SRM 1944 (NIST)	PCBs, PAHs, chlorinated hydrocarbons, and PBDEs in marine sediment
FRMs (Field Sediments)	Marine Sediment from Port of Los Angeles for pyrethroids and fipronils; Marine Sediment from Palos Verdes for PCBs, PAHs, CHCs, and PBDEs
CRM-SRM 1946 (NIST)	PCBs, Chlorinated hydrocarbons, and PBDEs in Lake Superior fish tissue
FRMs (Field Tissue)	PCBs, Chlorinated hydrocarbons, and PBDEs in Palos Verdes Shelf fish tissue
Environmental matrix (Trace Metals)	
CRM-ERA 540	Priority Pollutant Soil Certified Standard
FRMs (Field Sediment)	Marine Sediment from Palos Verdes
CRM-DORM-4	Metals in fish protein (NRC Canada)
FRMs (Field Tissue)	PCBs, Chlorinated hydrocarbons, and PBDEs in Palos Verdes Shelf fish tissue
Environmental matrix (total organic carbon)	
SRM 1944 or PACS-2 (NRC Canada)	TOC in marine sediment

VI. ANALYSIS OF CHEMICAL CONTAMINANTS IN FISH

A. Overview

This section presents Bight '18 quality assurance/quality control (QA/QC) protocols and requirements for bioaccumulation assessment covering sample composite and laboratory analysis. There has been one previous fish bioaccumulation surveys (2008) and current QA manual has been built upon the previous manual. The protocols described herein are applicable to low parts-per-billion analyses of fish tissue samples unless, otherwise noted.

The Bight '18 survey will measure several organic and inorganic contaminants in fish tissue samples (Table 6-1). In addition, this survey requires that the participating laboratories demonstrate comparability through strict adherence to common QA/QC procedures and participation in the intercalibration exercise prior to start of field sample analysis. The QA/QC program has adopted a similar "performance-based" approach to assess chemical contaminants in sediments to achieving quality assurance of low-level contaminants in fish tissue.

B. Tissue composite samples

Upon collection, each fish will be tagged with a unique identification number and measured for 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. During dissection, each fish will be sexed and the weight of fillet is recorded.

Dissection and compositing of muscle tissue samples will be performed following USEPA guidance (USEPA 2000). There will be a total of three composite samples per species per zone. A total of five specimens will be collected per composite sample. All specimens should be legal size or larger. If more than five specimens are collected, then the middle 75% of the length distribution will be used for the composite. Specimens from this interquartile range will be selected at random for inclusion in each composite.

Filet muscle tissue with the skin off will be used for analysis. Muscle filets are recommended by the USEPA (U.S. EPA. 2000). Skin removal has been repeatedly used in past California monitoring including the Toxic Substances Monitoring Program, the Coastal Fish Contamination Program, and most southern California NPDES monitoring programs. If some species are too small to be filleted, fish are processed whole but with head, tail, and viscera removed.

C. Contaminants

Tissue samples will be analyzed for PCB congeners, DDTs, Chlordanes, PBDEs, , mercury, selenium, and arsenic (Table 6-1). Reporting levels should be equivalent to or below Office of Environmental Health Hazard Assessment advisory tissue levels (OEHHA ATLs) for comparative purposes. Quality assurance activities shall focus on accuracy, precision, sensitivity, and comparability (Table 6-2 and 6-3).

Table 6-1. List of constituents and reporting limit in tissue

Analyte	Reporting Limit (ng/wet g)
Total PCB [a]	1
Total DDT [b]	1
PBDEs	0.6
Chlordane [c]	1
Mercury [d]	20
Selenium	400
Arsenic	300

a Congeners 8, 18, 28, 37, 44, 49, 52, 66, 70, 74, 77, 81, 87, 99, 101, 105, 110, 114, 118, 119, 123, 126, 128, 138, 149, 151, 153, 156, 157, 158, 167, 168, 169, 170, 177, 180, 183, 187, 189, 194, 195, 201, 206
b o,p'- and p,p'- isomers of DDT, DDE, and DDD, plus p,p'-DDMU
c cis- and trans-chlordane, cis and trans-nonachlor, and oxychlordane
d Can be measured as total Hg

Table 6-2
Summary of Data Quality Objectives for the
Metal (mercury, selenium, and arsenic) Measurements in Tissue

MEASUREMENT	FREQUENCY	CONTROL LIMIT
Method Blank	1/batch	<MDL or < 5% of measured concentration in the sample
Certified Reference Materials DORM-4	1/batch	Within ±30% of certified value for all analytes
Calibration	Initial setup	
Initial calibration verification (ICV)	1 points/batch	±20% true value
Continuing calibration verification (CCV)	1/10 samples	±20% true value
Matrix spike/MS duplicate	1/batch	RPD ≤ 25%, 75-125% recovery
Spiked blank	1/batch	±25% true value
Duplicate sample	1/batch	Within ±25% RPD

Table 6-3
Summary of the Data Quality Objectives for
PCBs, Chlorinated Pesticides, and PBDEs Measurements[a] in Tissue

MEASUREMENT	FREQUENCY	CONTROL LIMIT
SRM 1946	1/batch	Within ±50% of the certified value for ≥ 70% of the analytes selected by and agreed to by the Chemistry Technical Subcommitee
Initial calibration		Relative standard deviation (RSD) of the response factor within ± 25% for 80% of the analytes. Or correlation coefficient ($r^2 > 0.990$) for linear and non-linear curves. First or second order curves allowed.
Initial calibration verification	1/batch	Initial calibration verification should be performed immediately following the initial calibration. Relative percent difference (RPD) compared to initial calibration should be less than 30% of all analytes. Second source of calibration standards is used.
Cont. calibration verification	1 set/batch	Calibration verification should be performed at the beginning and end of each batch. Relative percent difference (RPD) compared to intial calibration should be less than 25% for 80% of the analytes
Method blank	1/batch	< 10 times the MDL for all analytes
Matrix spikes/MS duplicates (MS/MSD)	1 set/batch	50-150% recovery of spiked mass for >70% of analyte within each class; RPD <50% for >70% of analyte
Surrogate spikes	1/sample	Laboratories develop their own control limits
Internal standards (Optional)	1/sample	Laboratories develop their own

[a] maximum of 20 samples per extraction batch and a reasonable number of sample extracts per instrument batch.

Literature cited

U.S. EPA. 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, DC.

Appendix VI. Signature Pages

Attachment 1. Field Data Sheet Example

SWAMP Tissue Sampling - Trawl (Event Type = TI) SWB_FishCoast_2018
Entered in d-base (initial/date)
Pg of Pgs

***StationCode:** ___ ___ ___ ___ ___ ___ ___ ___ ___
*StationName:
Agency: MPSL-DFW
*Sampling Crew:
*Date (mm/dd/yyyy): / / 2018
*Purpose Failure Code: Non-sampleable__________ Equipment Failure__________ Other __________
ArrivalTime:
DepartureTime:
BEAUFORT SCALE (see attachment):
WIND DIRECTION (from): N W E S
PHOTOS (RB & LB assigned when facing downstream; RENAME to StationCode_yyyy_mm_dd_uniquecode):
1: (RB / LB / BB / US / DS / ##)
HabitatObs (CollectionMethod= Not App.) associated with general area trawled
DOMINANT SUBSTRATE: Concrete,Cobble,Gravel,Sand,Mud,Other________,unk
Swell (ft/m)
OTHER PRESENCE: Foam, OilySheen, None, Trash, MacroAlgae, Other___________
Comments:

Tissue Collection (MethodCode: Trawl)
*GPS/DGPS
OCCUPATION METHOD: RV: WeeG, New 17' Whaler, Field Monkeys , Other__________(circle one)
GPS Model:
COLLECTION DEVICE: MPSL-DFG_OtterTrawl, other ____________________________
Datum: **NAD83** WGS84 Other ________

Location	#	Start Time	Latitude (dd.ddddd)	Longitude (-ddd.ddddd)	End Time	Latitude (dd.ddddd)	Longitude (-ddd.ddddd)	Accuracy (ft/m)	Fish in trawl
OpenWat/ Bank/ MidChan									
StationWaterDepth(m):		HydroMod: None, Bridge, Pipes, Pier, Breakwater, Other ____					HydroModLoc: US / DS / WithIn		
OpenWat/ Bank/ MidChan									
StationWaterDepth(m):		HydroMod: None, Bridge, Pipes, Pier, Breakwater, Other ____					HydroModLoc: US / DS / WithIn		
OpenWat/ Bank/ MidChan									
StationWaterDepth(m):		HydroMod: None, Bridge, Pipes, Pier, Breakwater, Other ____					HydroModLoc: US / DS / WithIn		
OpenWat/ Bank/ MidChan									
StationWaterDepth(m):		HydroMod: None, Bridge, Pipes, Pier, Breakwater, Other ____					HydroModLoc: US / DS / WithIn		
OpenWat/ Bank/ MidChan									
StationWaterDepth(m):		HydroMod: None, Bridge, Pipes, Pier, Breakwater, Other ____					HydroModLoc: US / DS / WithIn		
OpenWat/ Bank/ MidChan									
StationWaterDepth(m):		HydroMod: None, Bridge, Pipes, Pier, Breakwater, Other ____					HydroModLoc: US / DS / WithIn		
OpenWat/ Bank/ MidChan									
StationWaterDepth(m):		HydroMod: None, Bridge, Pipes, Pier, Breakwater, Other ____					HydroModLoc: US / DS / WithIn		
OpenWat/ Bank/ MidChan									
StationWaterDepth(m):		HydroMod: None, Bridge, Pipes, Pier, Breakwater, Other ____					HydroModLoc: US / DS / WithIn		

Comments:
Bycatch: (spp/est #)

SWAMP Tissue Sampling - Non-Trawl (Event Type = TI) SWB_FishCoast_2018			Entered in d-base (initial/date)		Pg of Pgs
***StationCode:** ___ ___ ___ ___ ___ ___ ___ ___ ___ ___	*StationName:				**Agency: MPSL-DFW**
*Sampling Crew:	*Date (mm/dd/yyyy): / / 2018				*Purpose Failure Code: Non-sampleable_________ Equipment Failure_________ Other _________
	ArrivalTime:				
	DepartureTime:	BEAUFORT SCALE (see attachment):		WIND DIRECTION (from): N W E S	**PHOTOS** (RB & LB assigned when facing downstream; RENAME to StationCode_yyyy_mm_dd_uniquecode):
HabitatObs (CollectionMethod= Not App.) associated with general area fished					
DOMINANT SUBSTRATE: Concrete,Cobble,Gravel,Sand,Mud,Other________,unk		Swell (ft/m)			1: (RB / LB / BB / US / DS / ##)
OTHER PRESENCE: Foam, OilySheen, None, Trash, MacroAlgae, Other___________					
Comments:					
OCCUPATION METHOD: RV: WeeG, New 17' Whaler, Field Monkeys , Other_________, Walk-In (circle one)		GPS Model:	accuracy	Datum: **NAD83** Other _________	

Location:	OpenWater/Bank/MidChan	#______	*StationDepth (m):		Coord	(ft / m)	Lat (dd.ddddd)	Long (-ddd.ddddd)	Depth (m)
COLLECTION METHOD:	Hook, Net, Seine, Spear, Trap			Start Time	1				
COLLECTION DEVICE:	Hook/Line, Gill Net, CastNet, Seine, net#___, Other_______				2				
HYDROMODIFICATION:	None, Bridge, Pipes, Concrete Channel, Pier, Breakwater			End Time	3				
HYDROMODLOC(to sample):	US / DS / NA/ WI	Other __________	GEOSHAPE: Line Poly Point		4				
Fish Collected:									
Location:	OpenWater/Bank/MidChan	#______	*StationDepth (m):		Coord	(ft / m)	Lat (dd.ddddd)	Long (-ddd.ddddd)	Depth (m)
COLLECTION METHOD:	Hook, Net, Seine, Spear, Trap			Start Time	1				
COLLECTION DEVICE:	Hook/Line, Gill Net, CastNet, Seine, net#___, Other_______				2				
HYDROMODIFICATION:	None, Bridge, Pipes, Concrete Channel, Pier, Breakwater			End Time	3				
HYDROMODLOC(to sample):	US / DS / NA/ WI	Other __________	GEOSHAPE: Line Poly Point		4				
Fish Collected:									
Location:	OpenWater/Bank/MidChan	#______	*StationDepth (m):		Coord	(ft / m)	Lat (dd.ddddd)	Long (-ddd.ddddd)	Depth (m)
COLLECTION METHOD:	Hook, Net, Seine, Spear, Trap			Start Time	1				
COLLECTION DEVICE:	Hook/Line, Gill Net, CastNet, Seine, net#___, Other_______				2				
HYDROMODIFICATION:	None, Bridge, Pipes, Concrete Channel, Pier, Breakwater			End Time	3				
HYDROMODLOC(to sample):	US / DS / NA/ WI	Other __________	GEOSHAPE: Line Poly Point		4				
Fish Collected:									

Comments:	Bycatch: (spp/est #)

SWAMP Tissue Sampling - Fish Abundance SWB_FishCoast_2018 | Entered in d-base (initial/date) | Pg: of Pgs

*StationCode: __ __ __ __ __ __ __ __ | StationName: | Date (mm/dd/yyyy): / /

Location #	OrganismID	Tag #	Species Name	TL (mm)	FL (mm)	Weight (g)	Count	Sex	Anomaly & Body Loc.	Size Range(mm)	Count Est.
								M F U L			
								M F U L			
								M F U L			
								M F U L			
								M F U L			
								M F U L			
								M F U L			
								M F U L			
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								M F U L			
								M F U L			
								M F U L			
								M F U L			

Location #: Match fish with Location # from Tissue Collection sheet | **OrganismID:** Combine bag # and fish # (e.g., fish 1 of bag KPB01 is KPB01-01) to be unique | **Tag #**: Use if applicable

Size Range: use only if individuals are not measured and recorded | **Count Est**: If appropriate, add < or > if count is estimated

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), Popeye (PE), Tumor (T), Ulceration (U), White Spots (W), and any combination | **BodyLocation**: Branchial Chamber(BRC), Buccal Cavity(BC), Eyes(E), Musculoskeleton(M), Skin/Fins(SF) | **Sex**:unk(**U**),taken at Lab(**L**)

Comments: 1) Bag Numbers must be consecutive by previous trips per StationCode, 2) OrganismID fish # starts at 1 for each bag, 3) Smaller fish that are bagged by species but not tagged must be bagged by location #.

Attachment 2. Chain of Custody Form Example

Analysis Authorization		**Project ID:**	**SWB_FishCoast_2018**	Contact Person:	Autumn Bonnema	Kim Pham	
Contract:		**Season:**	Spring through Fall	Phone:	831-771-4175	916-322-8429	
Region:	Statewide	**Date:**	2018	email:	bonnema@mlml.calstate.ec	kimberly.pham@waterboards.ca.gov	
Protocol Code:	BOG-Coast						

StationCode	StationName	BagID	Sample Date	Comments	Field Preparation Preservation	Tissue Mercury THg total	Tissue Selenium Se total	Tissue Arsenic As total	Tissue Organics total	
					FieldFrozen					
					FieldFrozen					
					FieldFrozen					
					FieldFrozen					
					FieldFrozen					
					FieldFrozen					
					FieldFrozen					
					FieldFrozen					
					FieldFrozen					
					FieldFrozen					
					FieldFrozen					
					FieldFrozen					
					FieldFrozen					
					FieldFrozen					
					FieldFrozen					
					FieldFrozen					

Comments:

Please wait for authorization instructions from Autumn

Samples Relinquished by (Print and Sign):	**Date & Time**	**Samples Received by (Print and Sign):**	**Date & Time**

SWAMP REQUEST FOR ANALYSIS AND CHAIN OF CUSTODY (COC) RECORD

Analysis Authorization		**Project ID:**	**SWB_FishCoast_2018**	Contact Person:	Autumn Bonnema	Kim Pham
Contract:		**Season:**	Spring through Fall	Phone:	831-771-4175	916-322-8429
Region:	Statewide	**Date:**	2018	email:	bonnema@mlml.calstate.edu	kimberly.pham@waterboards.ca.gov
Protocol Code:	BOG-Coast					

StationCode	StationName	CompositeID	Sample Date	Comments	Field Preparation Preservation	Tissue Mercury THg total	Tissue Selenium Se total	Tissue Arsenic As total	Tissue PCBs total	Tissue OCs total
					FieldFrozen					
					FieldFrozen					
					FieldFrozen					
					FieldFrozen					
					FieldFrozen					
					FieldFrozen					
					FieldFrozen					
					FieldFrozen					
					FieldFrozen					
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					FieldFrozen					
					FieldFrozen					
					FieldFrozen					
					FieldFrozen					
					FieldFrozen					
					FieldFrozen					
					FieldFrozen					
					FieldFrozen					
					FieldFrozen					

Comments:

Please report %Moisture and %Lipid with other analytical results in the Results template provided.

Samples Relinquished by (Print and Sign):	**Date & Time**	**Samples Received by (Print and Sign):**	**Date & Time**

Attachment 3. Laboratory Data Sheet Example

SWAMP Lab Data Sheet - FISH	ProjectID: SWB_FishCoast_2018	PrepPres: Skin OFF	LabID:	Pg: 1 of 2 Pgs
StationCode:	Tissue: Fillet		Entered d-base (initial/date)	
StationName:	Homog. Method: BUCCHI POLYTRON OTHER_____		Staff: Diss. Homog.	
Species Name:	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									M / F / Unk	T		
2									M / F / Unk	T		
3									M / F / Unk	T		
4									M / F / Unk	T		
5									M / F / Unk	T		
6									M / F / Unk	T		
7									M / F / Unk	T		
8									M / F / Unk	T		
9									M / F / Unk	T		
10									M / F / Unk	T		
11									M / F / Unk	T		
12									M / F / Unk	T		
13									M / F / Unk	T		
14									M / F / Unk	T		
15									M / F / Unk	T		
16									M / F / Unk	T		
17									M / F / Unk	T		
18									M / F / Unk	T		
19									M / F / Unk	T		
20									M / F / Unk	T		
21									M / F / Unk	T		
22									M / F / Unk	T		
23									M / F / Unk	T		
24									M / F / Unk	T		
25									M / F / Unk	T		

OrganismID: xxxxxxxxLLXX##YYYzz-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

SWAMP Lab Data Sheet - FISH	**ProjectID:** SWB_FishCoast_2018	PrepPres: Skin OFF	LabID:	Pg: 1 of 2 Pgs
StationCode:	Tissue: Fillet	Entered d-base (initial/date)		
StationName:	Homog. Method: BUCCHI POLYTRON OTHER___________	Staff: Diss. Homog.		
Species Name:	Date Diss. (mm/dd/yyyy): / /	Date Homog. (mm/dd/yyyy): / /		

CHEMISTRY JARS

Individual ID:
Analysis: Mercury
Jar Weight Full (g):
Jar Weight Empty (g):
Comp Tissue Wt (Jar Full - Empty; g):

Individual ID:
Analysis: Mercury
Jar Weight Full (g):
Jar Weight Empty (g):
Comp Tissue Wt (Jar Full - Empty; g):

Individual ID:
Analysis: Mercury
Jar Weight Full (g):
Jar Weight Empty (g):
Comp Tissue Wt (Jar Full - Empty; g):

Individual ID:
Analysis: Mercury
Jar Weight Full (g):
Jar Weight Empty (g):
Comp Tissue Wt (Jar Full - Empty; g):

Individual ID:
Analysis: Mercury
Jar Weight Full (g):
Jar Weight Empty (g):
Comp Tissue Wt (Jar Full - Empty; g):

Individual ID:
Analysis: Mercury
Jar Weight Full (g):
Jar Weight Empty (g):
Comp Tissue Wt (Jar Full - Empty; g):

Individual ID:
Analysis: Mercury
Jar Weight Full (g):
Jar Weight Empty (g):
Comp Tissue Wt (Jar Full - Empty; g):

Individual ID:
Analysis: Mercury
Jar Weight Full (g):
Jar Weight Empty (g):
Comp Tissue Wt (Jar Full - Empty; g):

Individual ID:
Analysis: Mercury
Jar Weight Full (g):
Jar Weight Empty (g):
Comp Tissue Wt (Jar Full - Empty; g):

CompositeID:
Analysis: PCBs, OCs, PBDEs
Jar Weight Full (g):
Jar Weight Empty (g):
Comp Tissue Wt (Jar Full - Empty; g):
Glass 125mL

CompositeID:
Analysis: Selenium
Jar Weight Full (g):
Jar Weight Empty (g):
Comp Tissue Wt (Jar Full - Empty; g):
Plastic 30mL

CompositeID:
Analysis: Archive1
Jar Weight Full (g):
Jar Weight Empty (g):
Comp Tissue Wt (Jar Full - Empty; g):
Glass 60mL

CompositeID:
Analysis: Archive2 (PFAs)
Jar Weight Full (g):
Jar Weight Empty (g):
Comp Tissue Wt (Jar Full - Empty; g):
Plastic 30mL

CompositeID: C1_516TP0045BOG17LMB
Analysis: Archive3
Jar Weight Full (g):
Jar Weight Empty (g):
Comp Tissue Wt (Jar Full - Empty; g):
Glass 60mL

Comments: