SWAMP
Surface Water Ambient Monitoring Program

Final Quality Assurance Program Plan	2009

Screening Study of Bioaccumulation on the California Coast
Quality Assurance Program Plan

May 2009 [Revision 2.1]

CALIFORNIA Water Boards

www.waterboards.ca.gov/swamp

Group A Elements: Project Management

Element 1. Title and Approval Sheets

QUALITY ASSURANCE PROJECT PLAN

SCREENING STUDY OF BIOACCUMULATION ON THE CALIFORNIA COAST

The Bioaccumulation Oversight Group (BOG)

Surface Water Ambient Monitoring Program

May 2009

Program Title	SWAMP Bioaccumulation Oversight Group Coastal Study
Lead Organization	Marine Pollution Studies Lab California Department of Fish and Game 7544 Sandholdt Road Moss Landing, CA 95039 Mark Stephenson, Project Manager Autumn Bonnema, Project Coordinator
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Effective Date	This Quality Assurance Project Plan (QAPP) is effective from May 2009 to March 2011 unless otherwise revised, approved and distributed accordingly at an earlier date.
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QAPP Preface

This Quality Assurance Project Plan (QAPP) document defines procedures and criteria that will be used for this project conducted by SWAMP Bioaccumulation Oversight Group (BOG) in association with the California Department of Fish and Game Marine Pollution Studies Laboratory (MPSL-DFG), California Dept. of Fish and Game Fish and Wildlife Pollution Control Laboratory (DFG-WPCL), and the San Francisco Estuary Institute (SFEI). Included are criteria for data quality acceptability, procedures for sampling, testing (including deviations) and calibration, as well as preventative and corrective measures. The responsibilities of SFEI, MPSL-DFG, and DFG-WPCL also are contained within. The BOG selects the sampling sites, the types and size of fish, and the number of analyses to be conducted.

This work is funded through the Surface Water Ambient Monitoring Program (SWAMP) fiscal year 08/09 Bioaccumulation funding, with coordination from Southern California Bight

(SCB), Regional Water Quality Control Board 4 (RWQCB4) and the Regional Monitoring Program in the San Francisco Estuary (RMP).

Approvals

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

Mark Stephenson
Project Manager/MPSL-DFG Laboratory Director

_____ Date_____

Rusty Fairey
Contract Manager

_____ Date_____

Jay Davis
Lead Scientist

_____ Date_____

Beverly van Buuren
SWAMP Quality Assurance Officer

_____ Date_____

Autumn Bonnema
Project Coordinator/ MPSL-DFG Quality Assurance Officer

_____ Date_____

David Crane
DFG-WPCL Laboratory Director

_____ Date_____

Loc Nguyen
DFG-WPCL Quality Assurance Officer

_____ Date_____

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Element 3. Distribution List and Contact Information

A copy of this Quality Assurance Project Plan (QAPP), in hardcopy or electronic format, is to be received and retained by at least one person from each participating entity. At least one person from each participating entity (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 PROGRAM	
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Element 4. Project Organization

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

Table 2. Positions and duties

Position	Name	Responsibilities
Contract Manager	Rusty Fairey MPSL-MLML	Approve reports and invoices for payment.
Project Manager	Mark Stephenson MPSL-DFG	Project management and oversight.
Lead Scientist	Jay Davis SFEI	Advisory Roll; Data reporting
Project Coordinator	Autumn Bonnema, MPSL-DFG	Generation of a QAPP, Project coordination; ensures all laboratory activities are completed within proper timeframes.
Program QA Officer	Beverly van Buuren QA Research Group, MLML	Approve QAPP and oversee SWAMP projects' QA/QC
Laboratory QA Officer	Loc Nguyen DFG-WPCL Autumn Bonnema, MPSL-DFG	Ensures that the laboratory quality assurance plan and quality assurance project plan criteria are met through routine monitoring and auditing of the systems. Ensure that data meets project's objective through verification of results.
Sample Collection Coordinator	Gary Ichikawa MPSL-DFG	Sampling coordination, operations, and implementing field-sampling procedures.
Laboratory Director	David Crane DFG-WPCL Mark Stephenson MPSL-DFG	Organizing, coordinating, planning and designing research projects and supervising laboratory staff; Data validation, management and reporting
Sample Custodian	Stephen Martenuk MPSL-DFG Laurie Smith DFG-WPCL additional staff	Sample storage. Not responsible for any deliverables.
Technicians	Technical staff MPSL-DFG DFG-WPCL	Conduct fish tissue dissection, digestion, and chemical analyses. Not responsible for any deliverables.

4.1. Involved parties and roles

Rusty Fairey of Marine Pollution Studies Lab - Moss Landing Marine Laboratories (MPSL-MLML) will be the Contract Manager (CM) for this project. The CM will approve reports and invoices for payment.

Mark Stephenson of MPSL-DFG will serve as the Project Manager (PM) for the project. The PM will 1) review and approve the QAPP, 2) review, evaluate and document project reports, and 3) verify the completeness of all tasks.

Jay Davis of San Francisco Estuary Institute (SFEI) is the Lead Scientist (LS) and primary contact of this project. The LS will 1) generate the Sampling and Analysis Plan (SAP), 2) approve the QAPP, and 3) provide the BOG with a final report on completion of this project.

Autumn Bonnema of MPSL-DFG is the Project Coordinator (PC). The PC will 1) prepare the QAPP, 2) ensure that laboratory technicians have processing instructions and 3) ensure all laboratory activities are completed within the proper timelines. In addition, the PC may assist field crew in preparation and logistics.

Gary Ichikawa of MPSL-DFG is in charge of directing fish collection for this project. He will 1) oversee preparation for sampling, including vehicle maintenance and 2) oversee sample and field data collection.

Stephen Martenuk is responsible for sample storage and custody at MPSL. His duties will be to oversee compositing of tissue samples. Laurie Smith will do the same for samples processed at DFG-WPCL.

David Crane will serve as the Laboratory Director (LD) for the DFG-WPCL component of this project. His specific duties will be to 1) review and approve the QAPP, 2) provide oversight for all organic chemical analyses to be done for this project, and 3) ensure that all DFG-WPCL activities are completed within the proper timelines.

Mark Stephenson will also serve as the Laboratory Director (LD) for the MPSL-DFG component of this project. His specific duties will be to 1) review and approve the QAPP, 2) provide oversight for all trace metal analyses to be done for this project, and 3) ensure that all MPSL-DFG activities are completed within the proper timelines.

The following serve in an advisory role and are not responsible for any deliverables: Terry Fleming (EPA), Bob Brodberg (Office of Environmental Health Hazard Assessment (OEHHA)), Karen Taberski (RWQCB2), Mary Adams (RWQCB3), Michael Lyons (RWQCB4), Chris Foe (RWQCB5), Cassandra Lamerdin (MPSL-MLML), Jennifer Doherty (State Water Resources Control Board (SWRCB)), Billy Jakl (MPSL-DFG), Dylan Service (MPSL-DFG), Ken Schiff (SCCWRP) and Aroon Melwani(SFEI).

4.2. Quality Assurance Officer (QAO) Role

The Laboratory Quality Assurance Officers fulfill the functions and authority of a project quality assurance officer (QAO). Autumn Bonnema is the MPSL-DFG QAO and Loc Nguyen is the DFG-WPCL QAO. The role of the Laboratory QAO is to ensure that quality control for sample processing and data analysis procedures described in this QAPP are maintained throughout the project. The Program QAO (Beverly van Buuren, MLML) acts in a consulting

role to the Laboratory QAOs and ensures the project meets all SWAMP QA/QC criteria (Puckett, 2002).

The Laboratory QAOs 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 Laboratory QAOs 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.

A conflict of interest does not exist between the Laboratory QAOs and the work outlined in this QAPP as neither Laboratory QAO participates in any of the chemical analyses of the project. There is not a conflict of interest with one person fulfilling the roles of Laboratory QAO and Project Coordinator (PC), as laboratory decisions are not made by the PC and no other duties overlap. The role of the PC is detailed above.

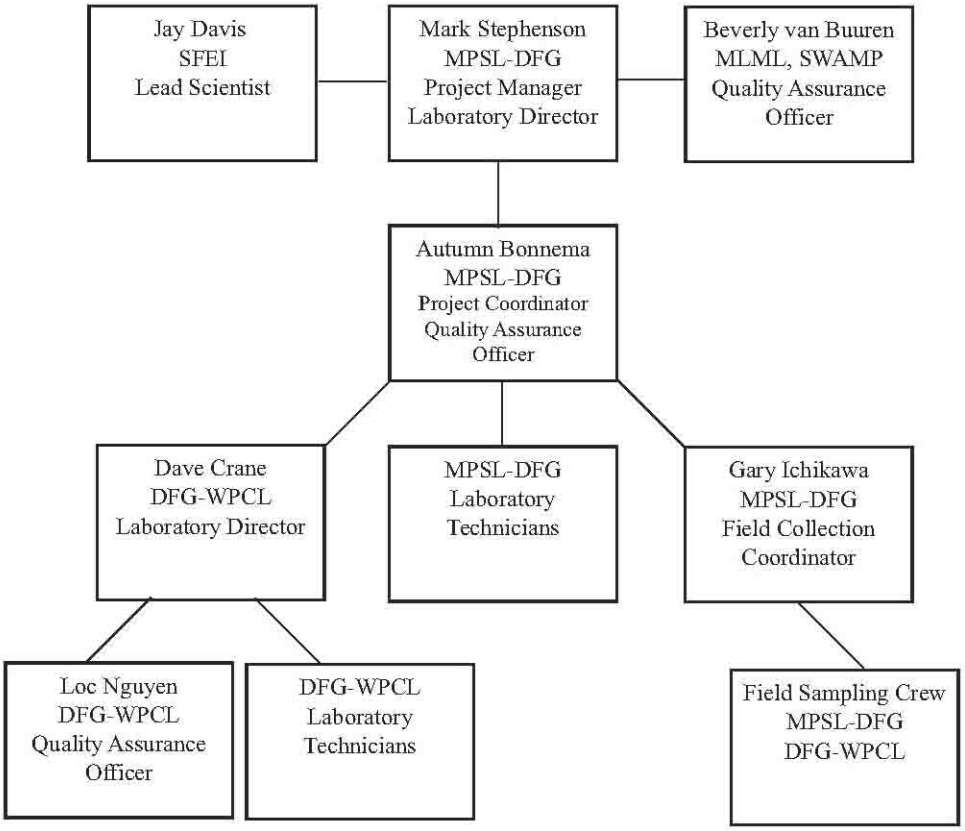
4.3. Persons responsible for QAPP update and maintenance

Revisions and updates to this QAPP will be carried out by Autumn Bonnema (PC), with technical input of the PM and the Laboratory and Program QAOs. All changes will be considered draft until reviewed and approved by the PM and the SWAMP QAO. Finalized revisions will be submitted for approval to the SWAMP QAO, if necessary.

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, DFG-WPCL and MPSL.

4.4. Organizational chart and responsibilities

Figure 1. Organizational Chart



Element 5. Problem Definition/Background

5.1. Problem statement

5.1.1. Addressing Multiple Beneficial Uses

Bioaccumulation in California water bodies has an adverse impact on both the fishing and aquatic life beneficial uses (Davis et al. 2007). The fishing beneficial use is affected by human exposure to bioaccumulative contaminants through consumption of sport fish. The aquatic life beneficial use is affected by exposure of 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. On the other hand, monitoring of status and trends in wildlife exposure can 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).

Over the long-term, a SWAMP bioaccumulation monitoring is envisioned that assesses progress in reducing impacts on both the fishing and aquatic life beneficial uses for all water bodies in California. In the near-term, however, funds are limited, and there is a need to demonstrate the value of a comprehensive statewide bioaccumulation monitoring program through successful execution of specific components of a comprehensive program. Consequently, with funds available for sampling in 2007 ($797,000) and additional funds of a similar magnitude anticipated for 2008, the BOG has decided to focus on sampling that addresses the issue of bioaccumulation in sport fish and impacts on the fishing beneficial use. This approach is intended to provide the information that the Legislature and the public would consider to be of highest priority. Monitoring focused on evaluating the aquatic life beneficial use will be included in the Project when expanded funding allows a broader scope.

5.1.2. Addressing Multiple Monitoring Objectives and Assessment Questions for the Fishing Beneficial Use

The BOG has developed a set of monitoring objectives and assessment questions for a statewide program evaluating the impacts of bioaccumulation on the fishing beneficial use (Table 3). This assessment framework is consistent with frameworks developed for other components of SWAMP, and is intended to guide the bioaccumulation monitoring program over the long-term. The four objectives can be summarized as 1) status; 2) trends; 3) sources and pathways; and 4) effectiveness of management actions.

Over the long-term, the primary emphasis of the statewide bioaccumulation monitoring program will be on evaluating status and trends. Bioaccumulation monitoring is a very effective and essential tool for evaluating status, and is often the most cost-effective tool for evaluating 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 TMDL programs) are more appropriate for addressing sources and pathways and effectiveness of management actions.

In the near-term, the primary emphasis of the statewide bioaccumulation monitoring program will be on evaluating Objective 1 (status). The reasons for this are:

1. a systematic statewide assessment of status has not been performed to date and is urgently needed;
2. we are starting a new program and establishing a foundation for future assessments of trends;
3. past monitoring of sport fish established very few time series that are useful in trend analysis.

Table 3. Bioaccumulation monitoring assessment framework for the fishing beneficial use.

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

D.1.1 What are the extent and location of water bodies with sufficient evidence to indicate that the fishing beneficial use is at risk due to pollutant bioaccumulation?
D.1.2 What are the extent and location of water bodies with some evidence indicating the fishing beneficial use is at risk due to pollutant bioaccumulation?
D.1.3 What are the extent and location of water bodies with no evidence indicating the fishing beneficial use is at risk due to pollutant bioaccumulation?
D.1.4 What are the proportions of water bodies in the State and each region falling within the three categories defined in questions D.1.1, D.1.2, and D.1.3?

D.2. ***Assess trends in the impact of bioaccumulation on the fishing beneficial use throughout the State***
D.2.1 Are water bodies improving or deteriorating with respect to the impact of bioaccumulation on the fishing beneficial use?
D.2.1.1 Have water bodies fully supporting the fishing beneficial use become impaired?
D.2.1.2 Has full support of the fishing beneficial use been restored for previously impaired water bodies?
D.2.2 What are the trends in proportions of water bodies falling within the three categories defined in questions D.1.1, D.1.2, and D.1.3 regionally and statewide?

D.3. ***Evaluate sources and pathways of bioaccumulative pollutants impacting the fishing beneficial use***
D.3.1 What are the magnitude and relative importance of pollutants that bioaccumulate and indirect causes of bioaccumulation throughout each Region and the state as a whole?
D.3.2 How is the relative importance of different sources and pathways of bioaccumulative pollutants that impact the fishing beneficial use changing over time on a regional and statewide basis?

D.4. ***Provide the monitoring information needed to evaluate the effectiveness of management actions in reducing the impact of bioaccumulation on the fishing beneficial use***
D.4.1 What are the management actions that are being employed to reduce the impact of bioaccumulation on the fishing beneficial use regionally and statewide?
D.4.2 How has the impact of bioaccumulation on the fishing beneficial use been affected by management actions regionally and statewide?

5.1.3. 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 first priority for monitoring. Coastal waters have been selected as the next priority, due to their importance for sport fishing and a relative lack of past monitoring. A Coastal Fish Contamination Monitoring Program was in initiated in 1998 (Gassel et al. 2002). This program was developed to assess the health risks of consumption of sport fish and shellfish from nearshore waters along the entire California coast. The CFCP was considered to be a critical component of a comprehensive coastal water quality protection program, and an important opportunity to build a long-term coastal monitoring database for water quality and contaminants in fish. However, the CFCP, along with the other two major state bioaccumulation monitoring programs (the Toxic Substances Monitoring Program and the State Mussel Watch Program) were discontinued in 2003 as plans for SWAMP began to take shape. Systematic monitoring of bioaccumulation in fish on the coast was therefore only in place for a few years. Given the extensive area, multiple habitats (coastline, bays and estuaries), diversity of species to be covered, and the amount of funding available ($500,000 of SWAMP funds for sampling and analysis), the coastal waters survey is also going to be a two-year effort spanning 2009 and 2010. In 2011, SWAMP will monitor bioaccumulation in California rivers and streams. In 2012, the long-term plan calls for beginning another five-year cycle of monitoring, with another two-year lake survey.

In summary, focusing on two closely associated habitat types (the coast and bays and estuaries), one objective (status), and one beneficial use (fishing) will allow us to provide reasonable coverage and a thorough assessment of bioaccumulation in California's coastal waters over a two-year period.

5.2. Decisions or outcomes

Three management questions have been articulated to guide the 2009-2010 survey of the status of bioaccumulation in sport fish on the California coast. These management questions are specific to this initial screening effort.

One major difference between this set of questions and the questions for the lakes survey is that the question regarding 303(d) listing is not included here. The 303(d) question was a major driver of the design of the lakes survey. On the coast, however, 303(d) listing is not a high priority for the Water Boards.

5.2.1. Management Question 1 (MQ1): Status of the Fishing Beneficial Use

For popular fish species, what percentage of popular fishing areas have low enough concentrations of contaminants that fish can be safely consumed?

Answering this question is critical to determining the degree of impairment of the fishing beneficial use 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 – the popular fish species and fish areas. This focus is also anticipated to enhance public and political support of the program by assessing the resources that people care most about. The determination of percentages captures the need to perform a statewide assessment of the entire California coast. The emphasis on safe consumption calls for: a positive message on the status of the fishing beneficial use; evaluation of the data using thresholds for safe consumption; and performing a risk-based assessment of the data.

The data needed to answer this question are average concentrations in popular fish species from popular fishing locations. Inclusion of as many popular 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.

5.2.2. 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. 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, good estimates of average concentrations within each spatial unit are needed.

5.2.3. Management Question 3 (MQ3): 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 consumption guidelines?

This screening survey of the entire California coast will provide a preliminary indication as to whether many areas that have not been sampled thoroughly to date may require consumption guidelines. Consumption guidelines provide a mechanism for reducing human exposure in the short-term. The California Office of Environmental Health Hazard Assessment (OEHHA), the agency responsible for issuing consumption guidelines, considers a sample of 9 or more fish

from a variety of species abundant in a water body to be the minimum needed in order to issue guidance. It is valuable to have information not only on the species with high concentrations, but also the species with low concentrations so anglers can be encouraged to target the low species. The diversity of species on the coast demands a relatively large effort to characterize interspecific variation. Answering this question is essential as a first step in determining the need for more thorough sampling in support of developing consumption guidelines.

5.2.4. Overall Approach

The overall approach to be taken to answer these three questions is to perform a statewide screening study of bioaccumulation in sport fish on the California coast. Answering these questions will provide a basis 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 and development of consumption guidelines.

It is anticipated that the screening study may lead to more detailed followup investigations of areas where consumption guidelines and cleanup actions are needed. Funding for these followup studies will come from other local or regional programs rather than the statewide monitoring budget.

5.2.5. 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 Regional Monitoring Program for Water Quality in the San Francisco Estuary (RMP), the Southern California Bight (SCB) and the Regional Water Quality Control Board 4 (RWQCB4) can be found in the Sampling and Analysis Plan (SAP) (Appendix II, p 8).

5.3. Fish tissue contamination criteria

Threshold levels for determining impairment of a body of water based on pollutants in fish tissue are listed in Tables 4 and 5. Fish Contaminant Goals (FCGs), as described by Klasing and Brodberg (2008), are “estimates of contaminant levels in fish that pose no significant health risk to humans consuming sport fish at a standard consumption rate of one serving per week (or eight ounces [before cooking] per week, or 32 g/day), prior to cooking, over a lifetime and can provide a starting point for OEHHA to assist other agencies that wish to develop fish tissue-based criteria with a goal toward pollution mitigation or elimination. FCGs prevent consumers from being exposed to more than the daily reference dose for non-carcinogens or to a risk level greater than $1x10^{-6}$ for carcinogens (not more than one additional cancer case in a population of 1,000,000 people consuming fish at the given consumption rate over a lifetime). FCGs are based solely on public health considerations without regard to economic considerations, technical feasibility, or the counterbalancing benefits of fish consumption.” For organic pollutants, FCGs are lower than Advisory Tissue Levels (ATL)s.

ATLs, as described by Klasing and Brodberg (2008), "while still conferring no significant health risk to individuals consuming sport fish in the quantities shown over a lifetime, were developed with the recognition that there are unique health benefits associated with fish consumption and that the advisory process should be expanded beyond a simple risk paradigm in order to best promote the overall health of the fish consumer. ATLs provide numbers of recommended fish servings that correspond to the range of contaminant concentrations found in fish and are used to provide consumption advice to prevent consumers from being exposed to more than the average daily reference dose for non-carcinogens or to a risk level greater than $1x10^{-4}$ for carcinogens (not more than one additional cancer case in a population of 10,000 people consuming fish at the given consumption rate over a lifetime). ATLs are designed to encourage consumption of fish that can be eaten in quantities likely to provide significant health benefits, while discouraging consumption of fish that, because of contaminant concentrations, should not be eaten or cannot be eaten in amounts recommended for improving overall health (eight ounces total, prior to cooking, per week). ATLs are but one component of a complex process of data evaluation and interpretation used by OEHHA in the assessment and communication of fish consumption risks. The nature of the contaminant data or omega-3 fatty acid concentrations in a given species in a water body, as well as risk communication needs, may alter strict application of ATLs when developing site-specific advisories. For example, OEHHA may recommend that consumers eat fish containing low levels of omega-3 fatty acids less often than the ATL table would suggest based solely on contaminant concentrations. OEHHA uses ATLs as a framework, along with best professional judgment, to provide fish consumption guidance on an ad hoc basis that best combines the needs for health protection and ease of communication for each site."

Thresholds for Total PCBs, DDTs, and Chlordanes are based on the summation of concentrations from the compounds listed in Table 6. The summations will be compared with the threshold values in Tables 4 and 5, and may lead to the identification of species which meet the beneficial uses of MQ1.

Table 4. Fish Contaminant Goals (FCGs) for Selected Fish Contaminants Based on Cancer and Non-Cancer Risk* Using an 8-Ounce/Week (prior to cooking) Consumption Rate (32 g/day) From Klasing and Brodberg (2008).**

	FCGs (ppb, wet weight)
Contaminant Cancer Slope Factor $(mg/kg/day)^{-1}$	
Chlordane (1.3)	**5.6**
DDTs (0.34)	**21**
Dieldrin (16)	**0.46**
PCBs (2)	**3.6**
Toxaphene (1.2)	**6.1**
Contaminant Reference Dose (mg/kg-day)	
Chlordane ($3.3x10^{-5}$)	100
DDTs ($5x10^{-4}$)	1600
Dieldrin ($5x10^{-5}$)	160
Methylmercury ($1x10^{-4}$)[S]	**220**
PCBs ($2x10^{-5}$)	63
Selenium ($5x10^{-5}$)	**7400**
Toxaphene ($3.5x10^{-4}$)	1100

*The most health protective Fish Contaminant Goal for each chemical (cancer slope factor- versus reference dose-derived) for each meal category is bolded.
**g/day represents the average amount of fish consumed daily, distributed over a 7-day period, using an 8-ounce serving size, prior to cooking.
[S]Fish Contaminant Goal for sensitive populations (i.e., women aged 18 to 45 years and children aged 1 to 17 years.)

Tabled values are rounded based on laboratory reporting of three significant digits in results, where the third reported digit is uncertain (estimated). Tabled values are rounded to the second digit, which is certain. When data are compared to this table they should also first be rounded to the second significant digit as in this table.

Table 5. 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). From Klasing and Brodberg (2008).

Contaminant	Three 8-ounce Servings* a Week	Two 8-ounce Servings* a Week	One 8-ounce Servings* a Week	No Consumption
Chlordane[c]	≤190	>190-280	>280-560	>560
DDTs[nc**]	≤520	>520-1,000	>1,000-2,100	>2,100
Dieldrin[c]	≤15	>15-23	>23-46	>46
Methylmercury (Women aged 18-45 years and children aged 1-17 years)[nc]	≤70	>70-150	>150-440	>440
Methylmercury (Women over 45 years and men)[nc]	≤220	>220-440	>440-1,310	>1,310
PCBs[nc]	≤21	>21-42	>42-120	>120
Selenium[nc]	≤2500	>2500-4,900	>4,900-15,000	>15,000
Toxaphene[c]	≤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.

Tabled values are rounded based on laboratory reporting of three significant digits in results, where the third reported digit is uncertain (estimated). Tabled values are rounded to the second digit, which is certain. When data are compared to this table they should also first be rounded to the second significant digit as in this table.

Table 6. Compounds summed for comparison with FCGs and ATLs levels.

Pollutant	Components	Reference
Total PCBs	Sum of all congeners analyzed	
Total PCB Aroclors	PCB AROCLOR 1248 PCB AROCLOR 1254 PCB AROCLOR 1260	SWRCB 2000
Total Chlordanes	Chlordane, cis- Chlordane, trans- Nonachlor, cis- Nonachlor, trans- Oxychlordane	USEPA 2000
Total DDTs	DDD(o,p') DDD(p,p') DDE(o,p') DDE(p,p') DDT(o,p') DDT(p,p')	USEPA 2000
Total PBDEs	Sum of all congeners analyzed	

Element 6. Project Description

6.1. Work statement and produced products

The survey is being conducted over two years to allow thorough coverage of the entire coast with available funds. 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.

In year 1, sampling will focus on the SCB (Water Board regions 4, 8 and 9 – see Figure 1) and San Francisco Bay and adjacent coastal areas (Region 2). This will allow for coordination with Bight '08 and the RMP, which are scheduled for 2009. This will also provide a basis for a report on year 1 that describes bioaccumulation in the most populated and heavily fished areas in the state near San Francisco and Los Angeles.

Sampling in year 2 will cover the other coastal regions (1 and 3) and any other remaining areas not covered in year 1. The second year report will present the data for these areas and also provide a comprehensive assessment of the entire two-year dataset.

6.2. Constituents to be analyzed and measurement techniques.

A detailed Sampling and Analysis Plan (SAP) is in Appendix II. Chemistry analytical methods are summarized in Section B13. Constituents to be analyzed are summarized in Tables 7-9a,b,c. All chemistry data will be reported on a wet weight basis.

Past studies have calculated PCB as Aroclors for comparison with older data sets and health thresholds. OEHHA no longer intends to use these data, and they will not be reported in SWAMP reports. The BOG agrees that these calculations are not as valuable as individual congener data, and will therefore cease reporting these calculated values. If necessary, these values can be calculated at a later time by the data management team using the provided congener data.

In the SWAMP Lakes Study (conducted in 2007 and 2008), PBDE data were provided at a screening level only as a free service from the analytical lab. These compounds are important emerging contaminants and will be analyzed in the Coastal Study on a subset of the samples. Two of the five species collected will be chosen for PBDE analysis. White croaker or other high lipid fish will be used.

Table 7. Constituents to be Analyzed – Fish Attributes

Fish Attributes
Total Length (mm)
Fork Length (mm)
Weight (g)
Sex
Moisture (%)
Lipid Content (%)

Table 8. Constituents to be Analyzed – Metals and Metalloids

Analyte	Analytical Method
Total Mercury	EPA 7374
Total Selenium	EPA 200.8

Table 9a. Constituents to be Analyzed – Organochlorine (OC) Pesticides

Organochlorine Pesticides (by EPA 8081BM using GC-ECD)	
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
	HCH, gamma
Others	Dacthal
	Endosulfan I
	Hexachlorobenzene
	Methoxychlor
	Mirex
	Oxadiazon
	~~Tedion~~[1]

[1]Tedion has been removed from the analyte list. This compound was discontinued from use in 1985 and has a very short residence time. Furthermore, it is a compound that is not bioaccumulated.

Table 9b. Constituents to be Analyzed – Polychlorinated Biphenyls (PCB)

Polychlorinated Biphenyl (PCB) Congeners (by EPA Method 8082M)	
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 095	PCB 189
PCB 097	PCB 194
PCB 099	PCB 195
PCB 101	PCB 198/199
PCB 105	PCB 200
PCB 110	PCB 201
PCB 114	PCB 203
PCB 118	PCB 206
PCB 126	PCB 209

Table 9c. 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
PBDE 154
PBDE 183
PBDE 190

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 10.

Five species will be collected from each of 69 zones over two years, resulting in 350 composites analyzed for the constituents found in Tables 8 and 9a, b and c.

Table 10. Project Schedule Timeline

Item	Activity and/or Deliverable	Deliverable Due Date
1	**Contracts**	
	Subcontract Development	March 2009
2	**Quality Assurance Project Plan & Monitoring Plan**	
2.1	Draft Monitoring Plan	March 2009
2.2	Final Monitoring Plan	April 2009
2.3	Draft Quality Assurance Project Plan	March 2009
2.4	Final Quality Assurance Project Plan	April 2009
3	**Sample Collection**	Yr1 April-November 2009 Yr2 April-November 2010
4	**Sample Selection and Chemical Analysis**	
4.1	Selection of Tissue for Analysis	Yr1 May-November 2009 Yr2 May-November 2010
4.2	Creation of Sample Composites	Yr1 May-December 2009 Yr2 May-December 2010
4.3	Chemical Analysis	Yr1 June 2009-March 2010 Yr2 June 2010-March 2011
5	**Interpretive Report**	
5.1	Draft Report	Yr1 June 2010 Yr2 June 2011
5.2	Final Report	Yr1 September 2010 Yr2 September 2011

6.4. Geographical setting and sample sites

California has over 3000 miles of coastline that spans a diversity of habitats and 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 69 spatial units called “zones” (SAP Figure 2, Appendix II). The use of this zone concept is consistent with the direction that OEHHA will take in the future in development of consumption guidelines for coastal areas. Advice has been issued on a pier-by-pier basis in the past in Southern California, and this approach has proven to be unsatisfactory. All of these 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.

Details on the determination of zone boundaries can be found in the SAP (Appendix II, p. 9).

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 10.

Ultimately, additional zones may be sampled pending time remaining in the sampling season and available funding within the project once cost savings from analysis has been determined.

Element 7. Quality Indicators and Acceptability Criteria for Measurement Data

Data quality indicators for the analysis of fish tissue concentrations of analytes will include accuracy (bias), precision, recovery, completeness and sensitivity. Measurement Quality Indicators for analytical measurements of organics and metals in tissue are in Table 11.

Previously collected data will not be utilized in this study, therefore specific acceptance criteria are not applicable.

Table 11. Measurement quality indicators for laboratory measurements.

Parameter	Accuracy	Precision	Recovery	Completeness	Sensitivity
Trace metals (including mercury)	CRM 75% - 125%	Duplicate RPD <25%; n/a if concentration of either sample <MDL Matrix Spike Duplicate RPD <25%	Matrix Spike 75% - 125%	90%	See Table 16
Synthetic Organics (including PCBs, pesticides, and PBDEs)	Certified Reference Materials (CRM, PT) within 70-130% of the certified 95% CI stated by provider of material. If not available then within 50-150% of reference value.	Duplicate RPD <25%; n/a if concentration of either sample <MDL	Matrix spike 50% - 150% or control limits control limits based on 3x the standard deviation of laboratory's actual method recoveries	90%	See Tables 17a,b,c

7.1. Accuracy

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 12a, b.

While reference materials are not available for all analytes, a way of assessing the accuracy of an analytical method is still required. Laboratory control samples (LCSs) provide an alternate method of assessing accuracy. 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. If an LCS needs to be substituted for a reference material, the acceptance criteria are the same as those for the analysis of reference materials. These are detailed in Tables 12a, b.

7.2. Precision

In order to evaluate the precision of an analytical process, a field sample is 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

Specific requirements pertaining to the analysis of laboratory duplicates vary depending on the type of analysis. The acceptance criteria for laboratory duplicates are specified in Tables 12a, b.

Upper and lower control chart limits (e.g., warning limits and control limits) will be continually updated at DFG-WPCL; control limits are based on 99% confidence intervals around the mean.

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 Minimum Level (ML). The U.S. Environmental Protection Agency (EPA) defines the ML as the lowest level at which the entire analytical system must give a recognizable signal and acceptable calibration point for the analyte. It is equivalent to the concentration of the lowest calibration standard, assuming that all standard operating procedure (SOP) or method-specified sample weights, volumes, and cleanup procedures have been employed.

7.2.1. Replicate Analysis

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

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

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. Bias

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

A matrix spike (MS) is prepared by adding a known concentration of the target analyte to a field sample, which is then subjected to the entire analytical procedure. If the ambient concentration of the field sample is known, the amount of spike added is within a specified range of that concentration. Matrix spikes are 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 is evaluated by calculating the percent recovery.

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

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

In order to properly assess the degree of matrix interference and potential bias, the spiking level should be approximately 2-5 times the ambient concentration of the spiked sample. If the MS or MSD is spiked too high or too low relative to the ambient concentration, the calculated recoveries are no longer an acceptable assessment of analytical bias. In order to establish spiking levels prior to analysis of samples, the laboratories should review any relevant historical data. In many instances, the laboratory will be spiking the samples blind and will not meet a spiking level of 2-5X the ambient concentration. However, the results of affected samples will not be automatically rejected.

In addition to the recoveries, the RPD between the MS and MSD is calculated to evaluate how matrix affects precision.

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

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

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

The MQO for the RPD between the MS and MSD is the same regardless of the method of calculation. These are detailed in Tables 12a, b.

7.4. 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 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. Subtracting method blank results from sample results is not permitted, unless specified in the analytical method.

7.5. Routine monitoring of method performance for organic analysis – surrogates

Surrogates are compounds chosen to simulate the analytes of interest in organic analyses. Surrogates are 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 reported concentration of each analyte is adjusted to correct for the recovery of the surrogate compound. 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 are reported with the target analyte data. Surrogate is considered acceptable if the percent recovery is within 50-150%.

7.6. Internal standards

For Gas Chromatography Mass Spectrometry (GC-MS) analysis, internal standards (i.e., injection internal standards) are 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.

7.7. 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.

7.8. 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 SAP (Appendix II). Sample site selection, sampling of relevant media (water, sediment and biota), and use of only approved/documented analytical methods will determine that the measurement data does represent the conditions at the investigation site, to the extent possible. The goal for meeting total representation of the site will be tempered by the types and number of potential sampling points (Puckett, 2002).

7.9. 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 (SAP Table 4, Appendix II) are present. Due to the variability and uncertainty of species availability in each zone, this level of completeness may not be attainable.

Laboratories will strive for analytical completeness of 90% (Table 11).

Table 12a. Measurement Quality Objectives – Inorganic 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	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 <MDL
Internal Standard	Accompanying every analytical run when method appropriate	75-125% recovery

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

Table 12b. Measurement Quality Objectives – Synthetic Organic Compounds 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 available 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 <MDL
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

Element 8. Special Training Requirements/Safety

8.1. Specialized training and safety requirements

Analysts are trained to conduct a wide variety of activities using standard protocols to ensure samples are analyzed in a consistent manner. Training of each analyst includes the use of analytical equipment and conducting analytical protocols, and other general laboratory processes including glassware cleaning, sampling preparation and processing, hazardous materials handling, storage, disposal. All laboratory staff must demonstrate proficiency in all the aforementioned and required laboratory activities that are conducted, as certified by the Laboratory QAO.

8.2. Training, safety and certification documentation

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

8.3. Training personnel

The DFG-WPCL or MPSL-DFG Lab Director (LD) trains or appoints senior staff to train personnel. The Laboratory QAO ensures that training is given according to standard laboratory methods, maintains documentation and performs performance audits to ensure that personnel have been trained properly.

8.3.1. Laboratory Safety

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

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

8.3.2. Technical Training

New employees and employees required to learn new test methods are instructed to thoroughly review the appropriate standard operating procedure(s) and are teamed up 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, Laboratory QAO and the laboratory director prior to the person independently performing the test method. Training records are retained on file for each employee by their supervisor or QAO. On-going performance is monitored by reviewing QC sample results.

Element 9. Documentation and Records

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

- Quality Assurance Project Plan (submitted to contract manager in paper and electronic formats)
- Monitoring Plan (submitted to contract manager in paper and electronic formats)
- Archived Sample Sheets (internal documentation available on request)
- Chain-of-Custody Forms (exchanged for signatures with chemistry lab, and kept on file)
- Lab Sample Disposition Logs (internal documentation available on request)
- Calibration Logs for measurements of water quality standards (internal documentation available on request)
- Refrigerator and Freezer Logs (internal documentation available on request)
- Quarterly Progress Reports (oral format to contract manager)
- Data Tables (submitted to contract manager in electronic formats)
- Draft Manuscript (produced in electronic format)
- Final Manuscript (in electronic format)
- Data Appendix (submitted to contract manager in paper and electronic spreadsheet formats)

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

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

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 PC 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 Sampling and Analysis Plan (SAP), Section III, pp. 6-17 (Appendix II). Sixty-nine Coastal "zones" will be sampled for 5 fish species each, when possible. Zones are listed in Table 13. Specific details on zone selection, boundaries and target species are found in Section III D and E1-2, pp. 9-13 of the SAP.

Due to the large size of sampling zones, it is not anticipated any zone will become inaccessible. If a particular launch ramp or pier is not accessible, another ramp or pier within the zone will be utilized. Latitude and Longitude will be recorded wherever sampling equipment is deployed to pinpoint collection sites within each zone. Blank field data sheets are in Attachment 1.

Each zone will be sampled within 3 full field days. Potential sampling equipment and methods can be found in MPSL-102a (Appendix III). Samples collected may be stored short-term for up to 1 month prior to delivery to the laboratory for processing. Once samples have been identified for composite creation, they will be shipped to the dissection laboratory for processing and analysis according to the timeline in Table 10.

All measurements and analyses to be performed are critical to address the objectives laid out in Section III of the SAP (Appendix II), with the exception of fish weight, sex, moisture, and lipid content. These parameters may be used to support other data gathered.

Table 13. BOG Coastal Zones

Zone	Region	Station Code	Zone Name	Zone	Region	Station Code	Zone Name
1	9	91001TJNI	TJ to North Island	36	3	30836SMYC	Southern Monterey County Coast
2	9	91202SDSB	SD South Bay	37	3	30837BSUR	Big Sur Coast
3	9	91203SDNB	SD North Bay	38	3	30838CARM	Carmel Coast
4	9	90804PLMA	Pt Loma	39	3	30939MYPG	Monterey/Pacific Grove Coast
5	9	90605PLLJ	Pt Loma to La Jolla	40	3	30940MLMC	Moss Landing/Marina Coast
6	9	90606MISS	Mission Bay	41	3	30641ELKS	Elkhorn Slough
7	9	90407LJSO	La Jolla to San Onofre	42	3	30442SCWB	Santa Cruz Area Wharfs/Beachs
8	9	90208OCNH	Oceanside Harbor	43	2	30443SCCA	Santa Cruz Coast Area
9	8	90109SOCC	San Onofre to Crystal Cove	44	2	30444ANNU	Ano Nuevo Area
10	8	90110DANA	Dana Point Harbor	45	2	20245SMAT	San Mateo Coast
11	8	80111CCSA	Crystal Cove to Santa Ana River	46	2	20246PPTH	Pillar Point Harbor
12	4	80112NWPT	Newport Bay	47	2	20247HMBC	Half Moon Bay Coast
13	4	80113SASB	Santa Ana River to Seal Beach	48	2	20248PACC	Pacifica Coast
14	4	80114ORCO	Orange County Oil Platforms	49	2	20249SSFC	San Francisco Coast
15	4	40515LNGB	Long Beach	50	2	20150FARI	Farallon Islands
16	4	41116SPDB	San Pedro Bay	51	2	20151SMAC	Southern Marin Coast
17	4	40617CATI	Catalina Island	52	2	20152TBAY	Tomales Bay
18	4	40418PVER	Palos Verdes	53	2	20153NMRC	Northern Marin Coast
19	4	40419SSMB	South Santa Monica Bay	54	1	11554BDGA	Bodega Harbor
20	4	40420MSMB	Middle Santa Monica Bay	55	1	11555SSNC	South Sonoma Coast
21	4	40421NSMB	North Santa Monica Bay	56	1	11356NSNC	North Sonoma Coast
22	3	40422PTDU	Pt Dume to Oxnard	57	1	11357PTAR	Point Arena Area
23	3	31623NCHI	Northern Channel Islands	58	1	11358MENC	Mendocino Coast Area
24	3	40124VTRC	Ventura to Rincon	59	1	11359FTBG	Fort Bragg Area
25	3	31525RCGA	Rincon to Goleta	60	1	11360NMCC	North Mendocino County Coast Area
26	3	31526SBCP	Santa Barbara Channel Oil Platform	61	1	11261SHLC	Shelter Cove Area
27	3	31527GPTC	Goleta to Pt Conception	62	1	11262CMEN	Cape Mendocino Area
28	3	31028NSBC	North Santa Barbara County Coast	63	1	11063EURC	Eureka Coast Area
29	3	31029PISM	Pismo Beach Area	64	1	11064HUMB	Humboldt Bay
30	3	31030PTSL	Port San Luis Area	65	1	10865TRIN	Trinidad Area
31	3	31031DIAB	Diablo Canyon Coast	66	1	10866NHCC	North Humboldt County Coast Area
32	3	31032MRBC	Morro Bay Coast	67	1	10367DENC	Del Norte Coast
33	3	31033MRRB	Morro Bay	68	1	10368CRCC	Crescent City Coast
34	3	31034CAMB	Cambria\Cayucos Coast	69	1	10369CCHA	Crescent City Harbor
35	3	31035NSLC	Northern San Luis Obispo County Coast				

10.1. Variability

Due to potential variability of contaminant loads in individual tissue samples, samples will be analyzed in composites as outlined in the SAP (Appendix II) and MPSL-DFG SOPs (Appendix III).

10.2. Bias

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

Element 11. Sampling Methods

Fish will be collected in accordance with MPSL-102a, Section 7.4 (Appendix III) except where noted here. Because coastal habitats vary greatly, there is no one method of collection that is appropriate. 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: spear fishing, trawling, seining, gill netting, and hook and line.

Details on targeted fish species, number of individuals and size ranges can be found in the SAP (Appendix II, Tables 4 and 6).

The following adaptation to MPSL-102a, Section 7.4.5 (Appendix III) has been made: Collected fish may be partially dissected in the field. At the dock, the fish is placed on a measuring board covered with clean aluminum foil; fork and total length are recorded. Weight is recorded. Large fish such as sharks will is then be placed on the cutting board covered with a foil where the head, tail, and guts are removed using a clean cleaver (scrubbed with Micro™, rinsed with tap and deionized water). The fish cross section is tagged with a unique numbered ID, wrapped in aluminum foil, and placed in a clean labeled bag. When possible, parasites and body anomalies are noted. The cleaver and cutting board are re-cleaned with Micro™, rinsed with tap and deionized water between fish species, per site if multiple stations are sampled.

Zones are not fully segregated from other zones; therefore no special equipment cleaning will be done between zones.

Further details on sample collection and processing can be found in the SAP, Section III, E-F, pp. 10-17 (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 14 describes action to take in the event of a collection failure.

Table 14. Field collection corrective actions

Collection Failure	Corrective Action
One or more primary target species not present in fishing site within the zone	Change locations to fish another site within the zone
After 3 days effort, one or more primary target species not collected within zone	Collect one or more species from secondary target list; document the occurrence

Element 12. Sample Handling and Custody

The field coordinator will be responsible for ensuring that each field sampling team adheres to proper custody and documentation procedures. A master sample logbook of field data sheets shall be maintained for all samples collected during each sampling event. A chain-of-custody (COC, Attachment 1) form must be completed after sample collection, archive storage, and prior to sample release.

Fish samples will be wrapped in aluminum foil and frozen on dry ice for transportation to the storage freezer or laboratory, where they will be stored at -20°C until dissection and homogenization. Samples delivered to MPSL-DFG will be logged in according to MPSL-104 (Appendix III). Samples delivered to DFG-WPCL will undergo a similar handling procedure (SAMPMAN_REV_Aug08, Appendix IV).

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

Lab Homogenates will be frozen until analysis is performed. Frozen tissue samples have a 12 month hold time from the date of collection. If a hold-time violation has occurred, data will be flagged appropriately in the final results.

Element 13. Analytical Methods

Methods and equipment for laboratory analyses are listed in Table 15. EPA methods can be downloaded from www.epa.gov/epahome/index/nameindx.htm. EPA method numbers followed by "M" indicate modifications have been made. Modifications and non-EPA 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.)

An AWS brand AMW-DISC digital pocket scale, or similar, is used to weigh fish in the field and is calibrated monthly in the lab with standard weights. Fish lengths are determined using a fish measuring board that does not require calibration. No other field measurements are being taken.

Table 15. Methods for laboratory analyses

Parameter	Method	Instrument
Mercury	EPA 7473	Milestone DMA 80
Selenium	EPA 3052M EPA 200.8	CEM MARSXpress Digester Perkin-Elmer Elan 9000 ICP-MS
Organochlorine Pesticides	EPA 8081BM	Agilent 6890 GC-ECD Varian 3800 GC with Varian 1200 Triple-Quad MS
Polychlorinated Biphenyls	EPA 8082M	Varian 3800 GC with Varian 1200 Triple-Quad MS
Polybrominated Diphenyl Ethers	EPA 8081BM	Agilent 6890 GC-ECD

Mercury will be analyzed according to EPA 7473, "Mercury in Solids and Solutions by Thermal Decomposition, Amalgamation, and Atomic Absorption Spectrophotometry" (USEPA, 1998) using a Direct Mercury Analyzer (DMA 80). Samples, blanks, and standards will be prepared using clean techniques. ASTM Type II water and analytical grade chemicals will be used for all standard preparations. A continuing calibration verification (CCV) will be performed after every 10 samples. Initial and continuing calibration verification values must be within ±20% of the true value, or the previous 10 samples must be reanalyzed. Three blanks, a certified reference material (DORM-3 or similar), as well as a method duplicate and a matrix spike pair will be run with each analytical batch of samples. Reporting Limits (RL) can be found in Table 16 and Measurement Quality Objectives (MQO) in Section 7, Table 12a.

Selenium composites will be digested according to EPA 3052M, "Microwave Assisted Acid Digestion of Siliceous and Organically Based Matrices" (USEPA, 1996), modified (Appendix III), and will be analyzed according to EPA 200.8, "Determination of Trace Elements in Waters and Wastes by Inductively Coupled Plasma-Mass Spectrometry" (USEPA, 1994). 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. Two blanks, a certified reference material (2976 or DORM-2), as well as a method duplicate and a matrix spike pair will be run with each set of samples. Reporting Limits (RL) can be found in Table 16 and Measurement Quality Objectives (MQO) in Section 7, Table 12a.

All organic compounds will be extracted following EPA Methods 3545, 3640A, and 3620B. Organochlorine pesticides and PBDEs will be analyzed according to EPA 8081BM, "Organochlorine Pesticides by Gas Chromatography", modified (Appendix IV). PCBs will be analyzed according to EPA 8082M, "Polychlorinated Biphenyls (PCBs) by Gas Chromatography", modified (Appendix IV). 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 ±25% of the true value, or the previous 10 samples must be reanalyzed. One blank, a laboratory control spike (LCS), as well as a method duplicate and a matrix spike pair will be run with each set of samples. Reporting Limits (RL) can be found in Table 17a,b,c and Measurement Quality Objectives (MQO) in Section 7, Table 12b.

Table 16. Trace metal analytical parameters, reporting units, and reporting limits (RL) for tissue samples.

Parameter	Method	RL (µg/g wet wt)
Mercury	EPA 7473	0.02
Selenium	EPA 3052M, EPA 200.8	0.40

Table 17a. Trace organic analytical parameters, reporting units, and reporting limits (RL) for tissue samples. Organochlorine Pesticides by EPA 8081BM using GC-ECD.

Organochlorine Pesticides (by EPA 8081BM)		
Group	**Parameter**	**RL (ng/g wet wt)**
Chlordanes	Chlordane, cis-	1
	Chlordane, trans-	1
	Heptachlor	1
	Heptachlor epoxide	0.5
	Nonachlor, cis-	1
	Nonachlor, trans-	1
	Oxychlordane	1
DDTs	DDD(o,p')	0.5
	DDD(p,p')	0.5
	DDE(o,p')	0.5
	DDE(p,p')	1
	DDMU(p,p')	1
	DDT(o,p')	1
	DDT(p,p')	1
Cyclodienes	Aldrin	1
	Dieldrin	0.5
	Endrin	1
HCHs	HCH, alpha	0.5
	HCH, beta	1
	HCH, gamma	0.5
Others	Dacthal	0.5
	Endosulfan I	1
	Hexachlorobenzene	0.7
	Methoxychlor	1
	Mirex	1
	Oxadiazon	1
	~~Tedion~~[1]	~~2~~

[1]Tedion has been removed from the analyte list. This compound was discontinued from use in 1985 and has a very short residence time. Furthermore, it is a compound that is not bioaccumulated.

Table 17b. Trace organic analytical parameters, reporting units, and reporting limits (RL) for tissue samples. PCBs by EPA Method 8082M.

Polychlorinated Biphenyl congeners (by EPA Method 8082M)			
PCB	RL ppb (ng/g wet wt)	PCB	RL ppb (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	0.9	PCB 187	0.6
PCB 095	0.9	PCB 189	0.6
PCB 097	0.6	PCB 194	0.6
PCB 099	0.6	PCB 195	0.6
PCB 101	0.9	PCB 198/199	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

Table 17c. Trace organic analytical parameters, reporting units, and reporting limits (RL) for tissue samples. PBDEs by EPA Method 8082M.

Polybrominated Diphenyl Ethers (by EPA Method 8081BM)	
PBDE	**RL ppb (ng/g wet wt)**
PBDE 017	0.6
PBDE 028	0.6
PBDE 047	0.8
PBDE 066	0.6
PBDE 085	0.8
PBDE 099	0.8
PBDE 100	0.6
PBDE 138	0.6
PBDE 153	0.8
PBDE154	0.6
PBDE 183	1.2
PBDE 190	1.8

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 tissue analyses must be completed within the 1 year hold time. In addition, results need to be reported according to the timeline outlined in Table 10.

13.3. Sample Disposal

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

Element 14. Quality Control

MPSL-DFG and DFG-WPCL 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 Laboratory QAO 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 PI and PM via email. The PM will contact the SWAMP QAO as needed. A written report containing all corrective actions will be submitted to the SWAMP QAO on a quarterly basis.

Each aspect of laboratory quality control is listed in Tables 12a and b for frequency as well as Measurement Quality Objectives (MQO) for each.

Element 15. Instrument/Equipment Testing, Inspection and Maintenance

Laboratory instruments are inspected and maintained in accordance with lab SOPs, which include those specified by the manufacturer and those specified by the method (Tables 16 and 17a, b, and c). These SOPs have been reviewed by each respective Laboratory QAO and found to be in compliance with SWAMP criteria. DFG-WPCL and MPSL-DFG analysts are responsible for equipment testing, inspection, and maintenance. Appendices III and IV list the referenced SOPs. DFG-WPCL SOPs are available upon request from the Laboratory Director by email: dcrane@ospr.dfg.ca.gov. Likewise, MPSL-DFG SOPS are available upon request from the Laboratory QAO 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. In some cases, the cost of instruments (i.e., GC-MS, EFD, etc) prohibits the procurement of additional spare parts. However, those instruments are typically maintained and repaired by the manufacturer.

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

Element 16. Instrument/Equipment Calibration and Frequency

Laboratory instruments (listed in Table 18) are calibrated, standardized and maintained according to procedures detailed in laboratory SOPs (Appendices III and IV). Instrument manuals identify step-by-step calibration and maintenance procedures. Instruments and types of calibration required are listed in Table 18. 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) does again does not meet specifications, it will be repaired and retested until performance criteria are achieved. The maintenance will be entered in the instrument log. If sample analytical information is in question due to instrument

performance, the PM will be contacted regarding the proper course of action including reanalyzing the sample(s).

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

Table 18. Equipment maintenance and calibration frequency.

Instrument	Inspection/Maintenance Frequency	Calibration Frequency
Agilent 6890 Gas Chromatograph equipped with micro-ECD detectors and autosamplers using Enviroquant Software (Agilent)	As needed	At least once prior to each batch
Varian 3800 Gas Chromatograph with Varian 1200 Triple Quadrupole Mass Spectrometer equipped with Combi-Pal autosampler	As needed	At least once prior to each batch
Perkin-Elmer Elan 9000 Inductively Coupled Plasma - Mass Spectrometer	As needed	At least once prior to each batch
Milestone DMA-80 Direct Mercury Analyzer	As needed	At least once every 2 weeks

16.1. Analytical Instrumentation

16.1.1. Instrument calibration

Upon initiation of an analytical run, after each major equipment disruption, and whenever on-going calibration checks do not meet recommended MQOs, the system will be calibrated with a full range of analytical standards. Immediately after this procedure, the initial calibration must be verified through the analysis of a standard obtained from a different source than the standards used to calibrate the instrumentation, prepared in an independent manner, and ideally having certified concentrations of target analytes of a CRM or certified solution. Frequently, calibration standards are included as part of an analytical run, interspersed with actual samples. However, this practice does not document the stability of the calibration and is incapable of detecting degradation of individual components, particularly pesticides, in standard solutions used to calibrate the instrument. The calibration curve is acceptable if it has an R^2 of 0.990 or greater for all analytes present in the calibration mixtures. If not, the calibration standards, as well as all the samples in the batch are re-analyzed. All calibration standards will be traceable to a recognized organization for the preparation and certification of QC materials (e.g., National Institute of Standards and Technology, National Research Council Canada, US EPA, etc.).

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

16.1.2. Continuing calibration verification (CCV)

Calibration verification solutions traceable to a recognized organization are inserted as part of the sample stream. The sources of the calibration verification solutions are independent from the standards used for the calibration. Calibration verification solutions used for the CCV will contain all the analytes of interest. The frequency of these verifications is dependent on the type of instrumentation used and, therefore, requires considerable professional judgment. The required frequency for this project is listed in Table 6. All analyses are bracketed by an 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 have to 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, DFG-WPCL will contact the PM to determine proceedings, and will flag the data and note the issue in interim and final reports.

Element 17. Inspection/Acceptance of Supplies and Consumables

All supplies will be examined for damage as they are received. Laboratory ordering personnel will review all supplies as they arrive to ensure the shipment is complete and intact. All chemicals are logged in to the appropriate logbook and dated upon receipt. All supplies are stored appropriately and are discarded upon expiration date. Table 19 indicates items that are considered for accuracy, precision, and contamination. If these items are not found to be in compliance with the acceptance criteria, they will be returned to the manufacturer.

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

Project-Related Supplies (source)	Inspection / Testing Specifications	Acceptance Criteria	Frequency	Responsible Individual
Certified pre-cleaned glass (I-Chem/Fisher Scientific or similar)	Carton custody seal is inspected	Carton custody seal intact	At receipt date of shipment	MSPL-DFG or DFG-WPCL personnel
Nitrile Gloves (Fisher Scientific or similar)	Carton seal is visually inspected for damage or tampering	Carton is intact and gloves within are clean and intact	At receipt date of shipment	MSPL-DFG or DFG-WPCL personnel
Polyethylene Gloves (Fisher Scientific or similar)	Carton seal is visually inspected for damage or tampering	Carton is intact and gloves within are clean and intact	At receipt date of shipment	MSPL-DFG or DFG-WPCL personnel
Analytical Standards (Perkin-Elmer, VWR, Fisher Scientific or similar)	Solution bottles are inspected to verify factory seal	Manufacturer's seal intact	At receipt date of shipment	MSPL-DFG or DFG-WPCL personnel

Element 18. Non-Direct Measures

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

Element 19. Data Management

Field data will be entered into the SWAMP Database version 2.5 upon return to the lab. Original field sheets (Attachment 1) will be retained in a log book, and copies of the COCs (Attachment 2) will be kept by each receiving laboratory. SWAMP Authorization forms will also accompany samples sent to each laboratory (Attachment 3).

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

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

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

All raw and statistical analysis data are subject to a 100% check for accuracy by the PM and Laboratory QAOs. Data are analyzed and proofread for accuracy, and then QA checked against the QAPP and SWAMP criteria before being entered into the SWAMP database. Original hard

copies of the data are filed in a secure cabinet until requested by the PM and/or inclusion into the Final Report. Electronic copies are stored and backed up by each analyst and respective laboratory internal project manager.

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

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

Group C Elements: Assessment and Oversight

Element 20. Assessments and Response Actions

20.1. Audits

All reviews of QA data will be made by the QAO of each laboratory prior to submission of each batch to SWAMP Tissue Database 2.5. Reviews of the sampling procedures will be made by the Field Collection Coordinator and the Project Coordinator in case problems occur. As SOPs are updated and refined, additional reviews will be made. Each data technician is responsible for flagging all data that does not meet established QA/QC criteria.

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

- Initial review of analytical and field data for complete and accurate documentation, chain of custody procedures, compliance with analytical holding times, and required frequency of laboratory QA samples.
- Comparison of all spike and duplicate results with the MQOs in tables 12a and b.
- Assigning data qualifier flags to the data as necessary to reflect limitations identified by the process.

If a review discovers any discrepancy, the QAO 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.

Assessments 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 in accordance with the timeline in Table 10.

20.2. Deviations and corrective actions

Analyses are conducted according to procedures and conditions recommended by the US EPA and described in laboratory SOPs (Appendices III and IV), with the exception of those reported herein. Beyond those identified, deviations from these recommended conditions are reported to the Laboratory QAO. The PM will be notified within 24 hours of these deviations.

In the event of a SOP/QAPP deviation or corrective action, a deviation/corrective action form will be prepared, completed, signed and the PM 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 Laboratory 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

The following products are to be delivered to PM:

- Each LD shall regularly brief the PC, LS and PM on the progress of all on-going chemical analyses in monthly emails or conference calls. When deemed necessary for decision making, other BOG participants will also be notified of progress.
- The LS will provide a draft final report and a final report to the PM in accordance with the dates listed in Table 10.

Group D Elements: Data Validation and Usability

Element 22. Data Review, Verification and Validation Requirements

Data generated by project activities will be reviewed against the measurement quality objectives (MQOs) in Tables 12a and 12b, Section 7. Furthermore, the final dataset as a whole will scrutinized for usability to answer the three Management Questions.

Element 23. Verification and Validation Methods

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 laboratory QAO. Additionally, the Laboratory QAO 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 the Laboratory QAO and LD. The Laboratory QAO will be responsible for informing data users of the problematic issues that were discussed, along with the associated reconciliations and corrections. DFG-WPCL checklists and forms are in Attachment 5. MPSL-DFG does not have specific forms; comments are made on original data sheets and reports.

Data will be reported electronically to the Project Coordinator, then to the SWAMP Database Management Team (DMT) for inclusion in the SWAMP Database version 2.5. The DMT will follow SWAMP SOP Chemistry Data Verification V1.1 (Appendix V).

Data will be validated by Stacey Swenson of the DMT according to RMP Data Validation (Appendix VI) with the modifications to adjust for SWAMP requirements as in Validation of BOG Database (Appendix V). A QA narrative will be produced to be incorporated in the BOG Coastal Report. This narrative will summarize the data set from a QA standpoint. Validated data will be made available to users via the SWAMP Database 2.5 provided by the DMT on the State Water Resources Control Board website (http://www.swrcb.ca.gov/water_issues/programs/swamp/).

Element 24. Reconciliation with User Requirements

Data will be reported in the SWAMP Database version 2.5. Data that do not meet with the Measurement Quality Objectives in Tables 11a and b will be flagged accordingly as discussed in Section D23. Rejected data will not be included in data analyses while data flagged as estimated will be evaluated for inclusion on a case-by-case basis in conjunction with the associated QA data and program objectives.

The project needs sufficient data, as represented by the completeness objective (Table 10, Section 7), to address the management questions laid out in Section 5; specifically MQ1 and MQ2. A failure to achieve the number of data points cited could mean an inability to answer these questions.

To address MQ1, the concentrations from all composites will be compared with the BOG adopted thresholds presented in Tables 4 and 5. Mercury will be calculated as laid out on p.14 of the SAP (Appendix II).

In order to answer MQ2 the analytical results will be compared to the BOG adopted thresholds as described in the previous paragraph. For each analyte the percent of zones that have fish that exceeded the threshold will be calculated.

Those zones with analyte results greater than the OEHHA FCGs or ATLs in Tables 4 and 5 will be called to the attention of the California Regional Water Quality Control Boards in the technical report. It will be up to each Region to compare the measured chemistry results of this study with the appropriate regional 303(d) list requirements and to determine if further sampling is needed (MQ3).

Since this study is a screening study with primarily the two management questions as objectives, complex statistical analysis is not anticipated except as mentioned above. The data collected by this study is not intended to be used with traditional statistics.

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Appendix I: List of Associated QAPPs

CDFG MPSL MLML Laboratory QAP, Revision 5. February, 2006

CDFG WPCL Laboratory QAPP, Revision 9. August, 2006

Appendix II: Sampling and Analysis Plan

FINAL

SAMPLING AND ANALYSIS PLAN FOR A SCREENING STUDY OF BIOACCUMULATION ON THE CALIFORNIA COAST

The Bioaccumulation Oversight Group (BOG)

Surface Water Ambient Monitoring Program

September 2009

ACKNOWLEDGEMENTS

This Sampling Plan was prepared by SFEI and MLML 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

This document presents a plan for sampling and analysis of sport fish in a two-year screening survey of bioaccumulation on the California coast. This work will be performed as part of the State Water Resources Control Board's Surface Water Ambient Monitoring Program (SWAMP). This effort is part of a new long-term Bioaccumulation Monitoring Project that is providing comprehensive monitoring of bioaccumulation in California water bodies.

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

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

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

The BOG was formed and began developing a strategy for designing and implementing a statewide bioaccumulation monitoring program in September 2006. To date the efforts of the BOG have been focused on a two-year screening survey of bioaccumulation in sport fish of California lakes and reservoirs (Davis et al. 2008). Under this effort, fish were collected in the summers of 2007 and 2008. A report on results from the first year is available (http://www.waterboards.ca.gov/water_issues/programs/swamp/lakes_study.shtml). A final report covering both years of the survey will be prepared in the fall of 2009.

II. GENERAL ASPECTS OF THE SWAMP BIOACCUMULATION MONITORING PROJECT

A. Addressing Multiple Beneficial Uses

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

sport fish. The aquatic life beneficial use is affected by exposure of 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. On the other hand, monitoring of status and trends in wildlife exposure can 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).

Over the long-term, a SWAMP bioaccumulation monitoring program is envisioned that assesses progress in reducing impacts on both the fishing and aquatic life beneficial uses for all water bodies in California. In the near-term, however, funds are limited, and there is a need to demonstrate the value of a comprehensive statewide bioaccumulation monitoring program through successful execution of specific components of a comprehensive program. Consequently, the BOG has decided to focus on sampling that addresses the issue of bioaccumulation in sport fish and impacts on the fishing beneficial use. This approach is intended to provide the information that the state government and the public would consider to be of highest priority. Monitoring focused on evaluating the aquatic life beneficial use will be included in the Project when expanded funding allows a broader scope. Preliminary evaluation of impacts on the aquatic life beneficial will also be explored using the data collected to evaluate impacts on the fishing beneficial use.

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

The BOG has developed a set of monitoring objectives and assessment questions for a statewide program evaluating the impacts of bioaccumulation on the fishing beneficial use (Table 1). This assessment framework is consistent with frameworks developed for other components of SWAMP, and is intended to guide the bioaccumulation monitoring program over the long-term. The four objectives can be summarized as 1) status; 2) trends; 3) sources and pathways; and 4) effectiveness of management actions.

Over the long-term, the primary emphasis of the statewide bioaccumulation monitoring program will be on evaluating status and trends. Bioaccumulation monitoring is a very effective and essential tool for evaluating status, and is often the most cost-effective tool for evaluating 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 TMDL programs) are also needed for addressing sources and pathways and effectiveness of management actions.

In the near-term, the primary emphasis of the statewide bioaccumulation monitoring program will be on evaluating Objective 1 (status). The reasons for this are:

1. a systematic statewide assessment of status has never been performed and is urgently needed;
2. we are starting a new program and establishing a foundation for future assessments of trends;
3. past monitoring of sport fish established very few time series that are useful in trend analysis that this program could have built upon.

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 first priority for monitoring. Coastal waters have been selected as the next priority, due to their importance for sport fishing and a relative lack of past monitoring. A Coastal Fish Contamination Monitoring Program was in initiated in 1998 (Gassel et al. 2002). This program was developed to assess the health risks of consumption of sport fish and shellfish from nearshore waters along the entire California coast. The CFCP was considered to be a critical component of a comprehensive coastal water quality protection program, and an important opportunity to build a long-term coastal monitoring database for water quality and contaminants in fish. However, the CFCP, along with the other two major state bioaccumulation monitoring programs (the Toxic Substances Monitoring Program and the State Mussel Watch Program) were discontinued in 2003 as plans for SWAMP began to take shape. Systematic monitoring of bioaccumulation in fish on the coast was therefore only in place for a few years. Given the extensive area, multiple habitats (coastline, bays and estuaries), diversity of species to be covered, and the amount of funding available ($500,000 of SWAMP funds for sampling and analysis), the coastal waters survey is also going to be a two-year effort spanning 2009 and 2010. In 2011, SWAMP will monitor bioaccumulation in California rivers and streams. In 2012, the long-term plan calls for beginning another five-year cycle of monitoring, with another two-year lake survey.

In summary, focusing on two closely associated habitat types (the coast and bays and estuaries), one objective (status), and one beneficial use (fishing) will allow us to provide reasonable coverage and a thorough assessment of bioaccumulation in California's coastal waters over a two-year period.

III. DESIGN OF THE COASTAL WATERS SURVEY

A. Management Questions for this Survey

Three management questions have been articulated to guide the 2009-2010 survey of the status of bioaccumulation in sport fish on the California coast. These management questions are specific to this initial screening effort.

One major difference between this set of questions and the questions for the lakes survey is that the question regarding 303(d) listing is not included here. The 303(d) question was a major driver of the design of the lakes survey. On the coast, however, 303(d) listing is not a high priority for the Water Boards.

Management Question 1 (MQ1)
Status of the Fishing Beneficial Use
For popular fish species, what percentage of popular fishing areas have low enough concentrations of contaminants that fish can be safely consumed?

Answering this question is critical to determining the degree of impairment of the fishing beneficial use 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 – the popular fish species and fish areas. This focus is also anticipated to enhance public and political support of the program by assessing the resources that people care most about. The determination of percentages captures the need to perform a statewide assessment of the entire California coast. The emphasis on safe consumption calls for: a positive message on the status of the fishing beneficial use; evaluation of the data using thresholds for safe consumption; and performing a risk-based assessment of the data.

The data needed to answer this question are average concentrations in popular fish species from popular fishing locations. Inclusion of as many popular 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.

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)
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 comprehensive consumption guidelines?

This screening survey of the entire California coast will provide a preliminary indication as to whether many areas that have not been sampled thoroughly to date may require consumption guidelines. Consumption guidelines provide a mechanism for reducing human exposure in the short-term. The California Office of Environmental Health Hazard Assessment (OEHHA), the agency responsible for issuing consumption guidelines, considers a sample of 9 or more fish from a variety of species abundant in a water body to be the minimum needed in order to issue guidance. It is valuable to have information not only on the species with high concentrations, but also the species with low concentrations so anglers can be encouraged to target the low species. The diversity of species on the coast demands a relatively large effort to characterize interspecific variation. Answering this question is essential as a first step in determining the need for more thorough sampling in support of developing consumption guidelines.

Overall Approach

The overall approach to be taken to answer these three questions is to perform a statewide screening study of bioaccumulation in sport fish on the California coast. Answering these questions will provide a basis 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 and development of consumption guidelines.

It is anticipated that the screening study may lead to more detailed followup investigations of areas where consumption guidelines and cleanup actions are needed. Funding for these followup 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 are going to 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 bioaccumulation monitoring for Bight '08, a comprehensive regional monitoring program for the Southern California Bight (SCB). Every five years, dischargers in the SCB collaborate to perform this regional monitoring. Bioaccumulation monitoring is one element of the Bight Program. Most of the work for this most recent round of Bight monitoring was performed in 2008. The bioaccumulation element, however, was delayed to 2009 in order to allow coordination with the SWAMP survey. The Bight group wanted to conduct sport fish sampling, but lacks the infrastructure to perform sample collection. The Bight group is therefore contributing approximately $240,000 worth of analytical work (analysis of PCBs and organochlorine pesticides in 225 samples) to the joint effort. This is allowing more intensive sampling of the Bight region than either program could achieve independently.

The SWAMP survey will also be coordinated with intensive sampling in San Francisco Bay by the Regional Monitoring Program for Water Quality in the San Francisco Estuary (RMP). The RMP conducts thorough sampling of contaminants in sport fish in the Bay on a triennial basis (see Hunt et al. [2008] for the latest results). This sampling has been conducted since 1994. The RMP will provide complete and thorough coverage of the Bay, with no additional effort by SWAMP needed. In addition, to coordinate with the SWAMP effort, the RMP will analyze additional species to allow for more extensive comparisons of the Bay with coastal areas and bays in other parts of the state. The RMP will benefit from this collaboration by SWAMP contributing: 1) a statewide dataset that will help in interpretation of RMP data and 2) a statewide report that will include an assessment and reporting of Bay data that will make production of a separate report by the RMP unnecessary. The RMP effort represents $215,000 of sampling and analysis.

In addition, the Region 4 Water Board is going to supplement the statewide survey with another $110,000 to provide for more thorough coverage of the SCB.

In all, these collaborations are more than doubling the total amount of SWAMP funding available for sampling and analysis in year 1 of the coastal waters 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 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

The survey is being conducted over two years to allow thorough coverage of the entire coast with available funds. 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.

In year 1, sampling will focus on the SCB (Water Board regions 4, 8 and 9 – see Figure 1) and San Francisco Bay and adjacent coastal areas (Region 2). This will allow for coordination with Bight '08 and the RMP, which are scheduled for 2009. This will also provide a basis for a report on year 1 that describes bioaccumulation in the most populated and heavily fished areas in the state near San Francisco and Los Angeles.

Sampling in year 2 will cover the other coastal regions (1 and 3) and any other remaining areas not covered in year 1. The second year report will present the data for these areas and also provide a comprehensive assessment of the entire two-year dataset.

D. Spatial Considerations

California has over 3000 miles of coastline that spans a diversity of habitats and 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 69 spatial units called "zones" (Figure 2). The use of this zone concept is consistent with the direction that OEHHA will take in the future in development of consumption guidelines for coastal areas. Advice has been issued on a pier-by-pier basis in the past in Southern California, and this approach has proven to be unsatisfactory. All of these 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. 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.

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. 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
5. Represent different exposure pathways (benthic vs pelagic)
6. Continuity with past sampling

Information relating to these criteria is presented below.

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. Monitoring of mussels is still being performed in California by NOAA's National Mussel Watch Program (using resident mussels) and by the Department of Fish and Game at more than 20 stations (using transplanted mussels). An additional consideration is that for mercury, the analysis of shellfish for methylmercury (rather than total Hg) would be required for a meaningful assessment. Determination of methylmercury is much more labor intensive and costly than determination of total Hg.

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 was a high rate of human consumption. Fortunately, good information on recreational fish catch is 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: www.recfin.org/forms/est2004.html. Additional data were obtained from Wade Van Buskirk of the PSMFC. The data were for the period Jan 2005 to Dec 2007.

Many different taxonomic groups of fish are found on the coast (e.g., rockfish, surfperch, or sharks) and some of these groups consist of quite a diversity of species. The sampling design is based primarily on coverage of a representative of selected groups within each zone. RecFIN data were used to identify the groups to target. Table 2 shows these data for the three regions (south, central and north) and specific data for the coast (ocean < 3 mi) and bays and harbors. Data include mass of catch in tonnes and counts in thousands (parentheses). 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, the most popular groups included chub mackerel; perch; flatfish; sharks, skates, and rays; rockfish; and croaker. The popular groups varied among the three regions of the state (south, central, and north) and between coastal waters and bays and harbors.

The next task was to select species within each group that will be targeted for sampling. For these decisions, RecFIN data for individual species were considered (Table 3). For example, rockfish are a popular group along most of the coast. Data for individual rockfish species were examined to identify the most popular species in each region. In coastal waters ("ocean < 3 mi" in Tables 2 and 3) of southern California, kelp bass (which were included in the "rockfish" group), were the most popular species in this group by far. Therefore, this species was selected as the primary target species for the rockfish group in this region. Since it is not always possible to collect the species that are targeted in every zone, the sampling crew will have a prioritized menu of other potential target species. Primary target species will be given the highest priority. If primary targets are not available in sufficient numbers, secondary targets have been identified. For rockfish, in the southern California ocean region, barred sand bass were the second most abundant species, and are at the top of a list of several possible secondary target species. In this manner, the RecFIN data were used to select primary and secondary targets for all of the sampling strata along the coast.

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 and therefore a combination of species is needed to ensure inclusion of the appropriate sensitive indicators. Methylmercury biomagnifies primarily through its accumulation in muscle tissue, so 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 the highest concentrations of organics. Other factors in addition to lipid are also important for some organics. Trophic position and age are important for highly hydrophobic pollutants such as the highly chlorinated PCBs (including the major ones like PCB153, 138, 180). Most studies show that there is lifetime accumulation of high log Kow 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).

Consequently, target species in this study will include both high lipid species such as croaker and surfperch, and predators that accumulate mercury such as 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 in order 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, given the primary consideration given to the two criteria described previously. As one example, shiner surfperch were selected as a secondary target for the central California coast, even though their catch was a bit lower than walleye surfperch, in order to allow for better comparison with the shiner surfperch data for central California bays and harbors.

Other Factors

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

- Cleaner species. Provide information useful in developing safe eating guidelines. More focused effort to obtain information on these species is left to future studies.
- Different exposure pathways (benthic vs pelagic). Not a high priority with the limited budget.
- Continuity with past sampling. This was a consideration in some areas, but past sampling also focused on the popular species, so the actual influence of this was not significant.

The Target Species

Table 4 shows the lists of primary and secondary species for each region and stratum based on the considerations discussed above. The available budget will allow for analysis of five species per zone. Therefore, the Table shows five primary targets for each stratum. One exception is the coast in southern California, where (in accordance with Bight Group preferences) the fifth species to be analyzed will be determined based on what is caught in the sample collection process.

A summary of basic ecological attributes of the primary and secondary target species is presented in Table 5. 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/), CDFG's Marine Sportfish Identification website (http://www.dfg.ca.gov/marine/fishid.asp), Oregon State University's Marine Species with Aquaculture Potential (http://hmsc.oregonstate.edu/projects/msap/index.html), and discussions with Jim Allen of SCCRWP (personal communication). Species were classified into guilds based on prey items, foraging type and habitat in an attempt to identify different species across the state with similar exposure pathways.

2. Sampling Sites

Within each zone, specific sites will be selected for sample collection. Criteria to be considered in determining the placement of sampling sites will 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 will be identified through published sources (e.g., Jones [2004]) and consultation with agency staff.

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 it is likely to obtain the needed samples.

In general, there will be only one composite sample (compositing is discussed further below) 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 for statistical spatial comparisons among zones. It is recognized that this will make data interpretation less conclusive. Exceptions to this are the southern California Bight (SCB) and San Francisco Bay. In the SCB, the Bight Group is making funds available for analyzing three replicates of kelp bass, white croaker, and one other species within each zone. These are not 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 replicates 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.

4. Size Ranges and Compositing for Each Species

Size Ranges and Compositing

Chemical analysis of trace organics is relatively expensive ($519 per sample for PCB congeners and $557 per sample for organochlorine pesticides), 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.

Chemical analysis of mercury is much less expensive ($65 per sample) 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 (ANCOVA) to evaluate spatial or temporal patterns in a manner that filters out the influence of fish size (for example, see Davis et al. [2008]). Consequently, the sampling design for selected mercury indicator species includes analysis of mercury in individual fish. For the mercury indicator species, an analysis of covariance approach will be employed, in which the size:mercury relationship will be established for each location and an ANCOVA will be performed that 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 (1998). Experience applying this approach in the Central Valley indicates that to provide robust regressions 10 fish spanning a broad range in size are needed (Davis et al. 2003, Davis et al. 2008).

Specific size ranges to be targeted for each species are listed in Table 6. Kelp Bass, Olive Rockfish, Black Rockfish, Blue Rockfish, and Brown Rockfish are the key mercury indicators. These species have a high trophic position and a strong size:mercury relationship. In addition, Shiner Surfperch will be analyzed as individuals for Hg because of their cosmopolitan distribution. These species will be analyzed individually for mercury, and composites from these fish will also be prepared for analysis of organics. The numbers and sizes indicated for these species will provide the size range needed to support ANCOVA.

Size ranges for other species are based on a combination of sizes prevalent in past sampling: RMP (Greenfield et al. 2005) and the CFCP (Gassel et al. 2002) and the 75% rule recommended by USEPA (2000) for composite samples. The target ranges for each species are defined by the minimum sizes listed in Table 6 and an upper bound based on the 75% rule.

In cases when more than 5 fish of one species are collected in a zone, composites will be created using the following guidelines:

1. Size: The middle interquartile will be used for composites. This eliminates bias towards either large or small fish.
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.

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

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 and because it is the measure fishers and wardens use to determine whether a fish is legal size. Determining fork length at the same time simplifies matters, and might help with IDs later to sort out freezer mishaps.

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 cleaned by rinsing with de-ionized (DI) and 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, DI water, and finally ASTM Type II water.

Composites will be created based on the 75% rule recommended by USEPA (2000). 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 skin is only removed from scaleless fish (e.g. catfish). The BOG is aware of this difference, but favors skin removal. Skin removal has been repeatedly used in past California monitoring. All fish (with limited exceptions) in Toxic Substances Monitoring Program, the Coastal Fish Contamination Program, and the Fish Mercury Project have also been analyzed skin-off. Processing fish with the skin on is very tedious and results in lower precision because the skin is virtually impossible to homogenize thoroughly and achieving a homogenous sample is difficult. Also, skin-on preparation actually dilutes the measured concentration of mercury because there is less mercury in skin than in muscle tissue. The most ubiquitous contaminant in fish in California that leads to most of our advisories is mercury. By doing all preparation skin-off we will be getting more homogeneous samples, better precision for all chemicals, and definitely a better measure of mercury concentrations, which are our largest concern. The analysis of axial fillets without skin was also advised by a national workgroup concerning the monitoring and analysis of mercury in fish (Wiener et al. 2007). Surfperch samples will be an exception to this rule. Surfperch 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 RMP) will be used.

Mercury will be analyzed according to EPA 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-3), as well as a method duplicate and a matrix spike pair will be run with each set of samples.

Selenium will be digested according to EPA 3052M, "Microwave Assisted Acid Digestion of Siliceous and Organically Based Matrices", modified, and analyzed according to EPA 200.8, "Determination of Trace Elements in Waters and Wastes by Inductively Coupled Plasma-Mass Spectrometry". Samples, blanks, and standards will be prepared using clean techniques. ASTM Type II water and analytical grade chemicals will be used for all standard preparations. A 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. Two blanks, a standard reference material (2976 or DORM-2), as well as a method duplicate and a matrix spike pair will be run with each set of samples.

Most organics analyses will be performed by the California Department of Fish and Game Water Pollution Control Lab in Rancho Cordova, CA. Organochlorine pesticides will be analyzed according to EPA 8081AM, "Organochlorine Pesticides by Gas Chromatography". PCBs and PBDEs will be analyzed according to EPA 8082M, "Polychlorinated Biphenyls (PCBs) by Gas Chromatography". 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 ±25% of the true value, or the previous 10 samples must be reanalyzed. One blank, a laboratory control spike (LCS), as well as a method duplicate and a matrix spike pair will be run with each set of samples.

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

G. Analytes

Table 7 provides a summary of the contaminants included on the list of analytes for the study. Since the study is focused on assessing the impacts of bioaccumulation on the fishing beneficial use, the list is driven by concerns over human exposure. Contaminants were included if they were considered likely to provide information that is needed to answer the three management questions for the study (see pages 6-7).

Additional discussion of the analytes is provided below.

Ancillary Parameters

Ancillary parameters to be measured in the lab include moisture and lipid (Table 8). Fish sex will not be determined for all samples as it is not considered critical for this statewide screening study. However, determination of sex has been requested by the Bight Program for fish from that region, and this will be performed.

Methylmercury

Methylmercury is the contaminant of greatest concern with respect to bioaccumulation on a statewide basis. Based on past monitoring (Gassel et al. 2002), methylmercury is expected to exceed the threshold of concern in many coastal 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 because a substantial proportion of samples of each species are expected to exceed the threshold of concern.

PCBs

PCBs are the contaminant of second greatest concern with respect to bioaccumulation on a statewide basis (Davis et al. 2007). PCBs will be analyzed using a congener specific method. A total of 55 congeners will be analyzed (Table 8). This list includes many of those identified as additional candidates for inclusion on the congener list by Sanborn and Brodberg (2007 – "Appendix 1: Detailed Evaluation of Organic Analytes to Include in the Study"). PCBs will be analyzed in all composite samples.

Legacy pesticides

Based on past monitoring (Gassel et al. 2002), legacy pesticides are generally expected to exceed thresholds of concern in a very small percentage of California coastal zones. An exception to this would be the portion of the SCB with significant historic contamination. Pesticides will be analyzed in all composite samples.

PBDEs

Few data are currently available on PBDEs in California sport fish, and a threshold of concern has not yet been established. However, a rapid increase in concentrations in the 1990s observed in San Francisco Bay and other parts of the country raised concern about these chemicals, and led to a ban on the production and sale of the penta and octa mixtures in 2006 (Oros et al. 2005). The deca mixture is still produced commercially. A threshold of concern is anticipated to be established soon by USEPA. The most important PBDE congeners with respect to bioaccumulation are PBDEs 47, 99, and 100. It is anticipated that funds will be obtained to allow for analysis of PBDE congeners. A total of 12 congeners will be analyzed (Table 8). PBDEs will be analyzed in two composite samples from each zone (if funding allows).

Dioxins and Dibenzofurans

Few data are available on dioxins and dibenzofurans in California sport fish. Perhaps the best dataset exists for San Francisco Bay, where samples from 1994, 1997, 2000, 2003, and 2006 indicated that concentrations in high lipid species exceeded a published screening value of 0.3 TEQs (for dioxins and furans only) by five fold (Greenfield et al. 2003). 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 U.S. OEHHA did not include dioxins in their recent evaluation of guidance 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, OEHHA recommended that dioxins not be included in this screening study (Table 7). Dioxins are considered a higher priority by the 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 zones will also be analyzed for these chemicals.

Selenium

Past monitoring (Greenfield et al. 2005, Gassel et al. 2002) indicates that selenium concentrations are not likely to be above thresholds in this study, except perhaps for white sturgeon in San Francisco Bay. OEHHA has requested including selenium on the analyte list for year 1 of the Coastal Survey to confirm that

concentrations are indeed below thresholds. If this proves true, it is likely that selenium analysis will not be conducted in year 2.

Organophophates, PAHs, TBT, and Cadmium

Past monitoring (e.g., San Francisco Bay work – SFBRWQCB 1995) indicates that concentrations of these chemicals in sport fish are generally far below thresholds of concern for human exposure. Therefore, they will not be included in the present study. One exception is selenium in San Francisco Bay, where a cleanup plan is being developed and the Water Board has requested additional information on concentrations in sport fish.

Other Emerging Contaminants

Other emerging contaminants are likely to be present in California sport fish. Examples include perfluorinated chemicals, other brominated flame retardants in addition to PBDEs, and others. Thresholds do not exist for these chemicals, so advisories or 303(d) listing are not likely in the near future. However, early detection of increasing concentrations of emerging contaminants can be very valuable for managers, as evidenced by the PBDE example. Measuring emerging contaminants would not directly address the management questions guiding this study, so analysis of these chemicals is not included in the design. Archives of each composite will be retained and made available for analysis of emerging contaminants in the future (see Section G.) An exception is San Francisco Bay, where the Regional Monitoring Program will be analyzing perfluorinated chemicals (see Section L).

Omega-3 Fatty Acids

Klasing and Brodberg (2008) concluded that there is a significant body of evidence and general scientific consensus that eating fish at dietary levels that are easily achievable, but well above national average consumption rates, appears to promote significant health benefits, including decreased mortality, and that because of the unique health benefits associated with fish consumption, the advisory process should be expanded beyond a simple risk paradigm in order to best promote the overall health of the fish consumer. Much of the health benefits of fish consumption are derived from their relatively high content of key omega-3 fatty acids, eicosapentaenoic acid (EPA) and docosahexaenoic acid (DHA). When these data are available, OEHHA can take them into consideration in developing safe eating guidelines. Few data are available on the omega-3 content of wild fish. The RMP is planning on obtaining these data for San Francisco Bay fish.

H. Quality Assurance

This effort will adhere to quality assurance requirements established for the SWAMP. A QAPP specific to this effort is in preparation (Bonnema 2009).

One of the analytical challenges in this project will be coordinating among different laboratories that will be generating organics data. The Bight Group resource contribution to the study is in the form of analytical chemistry for more than 200 organics samples. Multiple labs from the Bight Group will participate. Discussions are underway to select labs that are capable of generating data of sufficient quality for the study. An intercalibration exercise is planned for the participating labs to identify any comparability problems before analysis of the field samples is initiated (see Appendix 1).

I. Archiving

As described above, aliquots of homogenates of all samples analyzed will be archived on a long-term basis to provide for reanalysis in case of any mishaps or confirmation, as well as for analysis of emerging contaminants.

Up to five 50 g aliquots of each composite created will be archived. This will provide a 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.

Four of the five 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.

J. Ancillary 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 followup studies are needed in any of the sampled zones.

K. Timing

Sampling will be conducted from May 2009 through October 2009. Seasonal variation in body condition and reproductive physiology are recognized as factors that could affect contaminant concentrations. However, sampling as many zones as possible is essential to a statewide assessment, and it will take this many months to sample the zones targeted for 2009.

L. Data Assessment

MQ1 will be assessed by comparing results from each zone to thresholds established by OEHHA in Klasing and Brodberg (2008) (Tables 9 and 10). 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, nonstatistical methods will be used (mapping and graphing). Comparison of concentrations between regions may be performed by treating zones within each region as "replicates".

MQ3 will be assessed in consultation with OEHHA.

M. Products and Timeline

A technical report on the 2009 sampling will be drafted by September 2010 and will include an assessment of data from two of the most heavily fished portions of the coast near the population centers of Los Angeles and San Francisco. The final report, incorporating revisions in response to reviewer comments, will be completed in January 2011.

A second round of sampling is planned for 2010. This work would follow the same approach described in this document, but focusing on the remaining zones in Regions 1 and 3, and any other zones not yet covered in 2009. This sampling would begin May 2010. Preliminary results from the 2009 sampling will be evaluated to determine whether any adjustments to the design are needed.

N. Regional Enhancements in San Francisco Bay and the Southern California Bight

San Francisco Bay

The Regional Monitoring Program for Water Quality in the San Francisco Estuary (RMP) is coordinating closely with the SWAMP Coastal Waters Survey. The RMP conducts thorough sampling of contaminants in sport fish in the Bay on a triennial basis (see Hunt et al. [2008] for the latest results). This sampling has been conducted since 1994. A sampling plan for the RMP effort in 2009 has been prepared (Hunt 2009). The RMP will provide complete and thorough coverage of the Bay, with no additional effort by SWAMP needed. Furthermore, to coordinate with the SWAMP effort, the RMP will analyze additional species to allow for more extensive comparisons of the Bay with coastal areas and bays in other parts of the state. The RMP will benefit from this collaboration by SWAMP contributing: 1) a statewide dataset that will help in interpretation of RMP data and 2) a statewide report that will include an assessment and reporting of Bay data that will make production of a separate report by the RMP unnecessary. The RMP effort represents $215,000 of sampling and analysis.

Some important points to note about the coordination of these two efforts include:

- The zones to be sampled for the RMP are centered around the locations shown in Figure 3.
- The RMP will sample additional species beyond the standard SWAMP list for central California bays and harbors (Table 11). The additional species are striped bass, white sturgeon, and northern anchovy.
- The RMP will also measure additional analytes beyond the standard SWAMP list (Table 11). These include dioxins and dibenzofurans, perfluorinated chemicals, and omega-3 fatty acids.
- Replication within the San Francisco Bay zones will be included for some species (Table 12). The plan for replication is based on experience from multiple rounds of previous sampling. Three replicate composites of shiner surfperch will be collected from each Bay zone. Multiple replicates of white croaker will be collected (n=12), but since this species moves throughout the Bay the samples will be collected opportunistically wherever they are found.
- Multiple white sturgeon tissue types will be analyzed for selenium. Muscle fillet, muscle biopsy and liver will be analyzed. The RMP is investigating moving towards non-lethal sampling of white sturgeon in future monitoring.
- White croaker (one of the primary organic contaminant indicators) has historically been analyzed skin-on in the RMP. Skin-on analysis of organic contaminants provides information that is the most protective of human health. However, OEHHA's current sport fish consumption advisories, for white croaker, recommend removal of skin prior to eating. Additionally, the SWAMP will be analyzing this species skin-off in the Coastal Survey. To be comparable to the SWAMP program and the OEHHA consumption advisory, the RMP is moving toward skin-off analysis of white croaker. In 1997, the RMP did a side-by-side analysis of white croaker skin-on and skin-off (n=4 composites). Average PCB concentrations were 39% lower in the skin-off analysis while DDT levels were about 40% lower. The initial side-by-side analysis, due to the small sample size, did not provide enough information to definitively establish a relationship between skin-on and skin-off contaminant levels. SFEI looked through the literature for other white croaker skin-on/off data. The Palos Verdes Shelf fish monitoring program performed a side by side skin-on/off analysis with white croaker individuals. PCB and DDT levels were highly variable between the skin-on and skin-off analyses – ranging from 2-24 times lower in the skin-off analysis. In order to continue the RMP long-term data set, the RMP will perform additional side-by-side skin-on and skin-off analysis for PCBs, PBDEs, OC pesticides, and dioxins. This additional analysis will increase the cost of dissection/compositing as well as the analysis portion for white croaker – an additional $30,360.
- In order to be comparable to the SWAMP sampling plan, three additional species were added to RMP sampling – one composite for each region (3) in San Francisco Bay: leopard shark, California halibut, and jacksmelt. These species were part of the historical RMP sport fish sampling but were discontinued after 2003.
- The RMP has traditionally published a report on each round of sport fish monitoring. In 2009, to optimize use of available funds, the RMP will rely on the

SWAMP report for disseminating information from this round of sampling. The cost savings from this arrangement is being used to collect and analyze additional samples that enhance comparability of the SWAMP and RMP designs.

The Southern California Bight

The 2008 Southern California Bight Regional Marine Monitoring Program (Bight'08) is coordinating closely with the SWAMP Coastal Waters Survey. The Bight'08 monitoring program has conducted sampling approximately every five years starting in 1994. In each of the three previous surveys, results have indicated widespread tissue bioaccumulation. At times, the levels of bioaccumulation in fish tissue have exceeded thresholds for risk to wildlife consumers (Schiff and Allen, 2001; Allen et al 2007). However, this will be the first time since 1991 that a Bightwide survey of sport fish tissues for human health risk will be conducted. These data will be used by Regional Water Boards and NPDES permittees for evaluating local permit-based monitoring requirements and could be used by OEHHA for new or updated fish advisories or closures in the southern California Bight.

The Bight'08 Monitoring Program has actively engaged SWAMP for collaboration in the tissue monitoring program. The value of the collaboration is the sharing of effort. For the Bight'08 program, the effort of SWAMP to collect fish covers resources not available to Bight'08 agencies. For the SWAMP, the effort of Bight'08 to analyze samples enables additional species and replicates per species beyond what could be accommodated within the SWAMP budget.

Below is a list of the similarities and differences between the two programs:

- Identical list of monitoring questions
- Common primary target species list
- Additional secondary species list
- Additional replicate samples per target species
- Increased number of fishing zones in the southern California Bight
- Multiple labs will analyze organics in the Bight samples, with varying methods and detection limits

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Figure 1. Water Board regional boundaries.

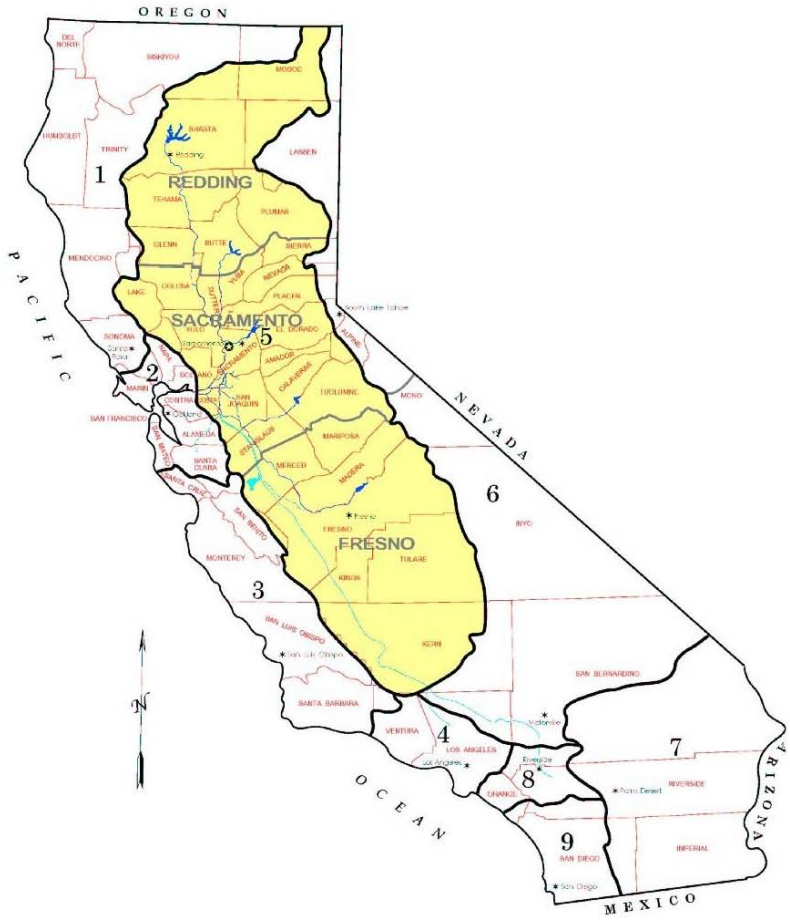

Figure 2. Fishing zones delineated for this survey. Each zone is numbered in pink and outlined in red. Fishing locations are also indicated. A Google Earth layer with the zones is available on the BOG website: http://www.swrcb.ca.gov/water_issues/programs/monitoring_council/bioaccumulation_oversight_group/

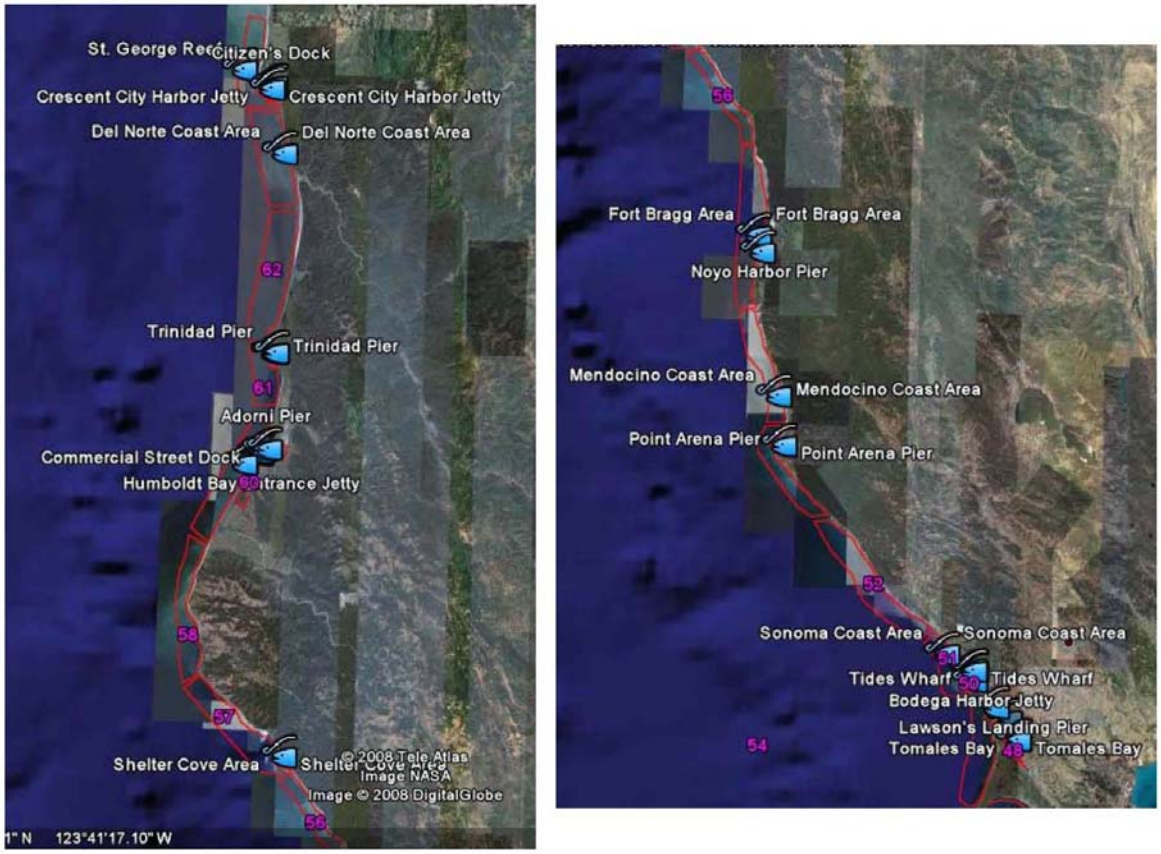

Figure 2. Zone maps (continued).

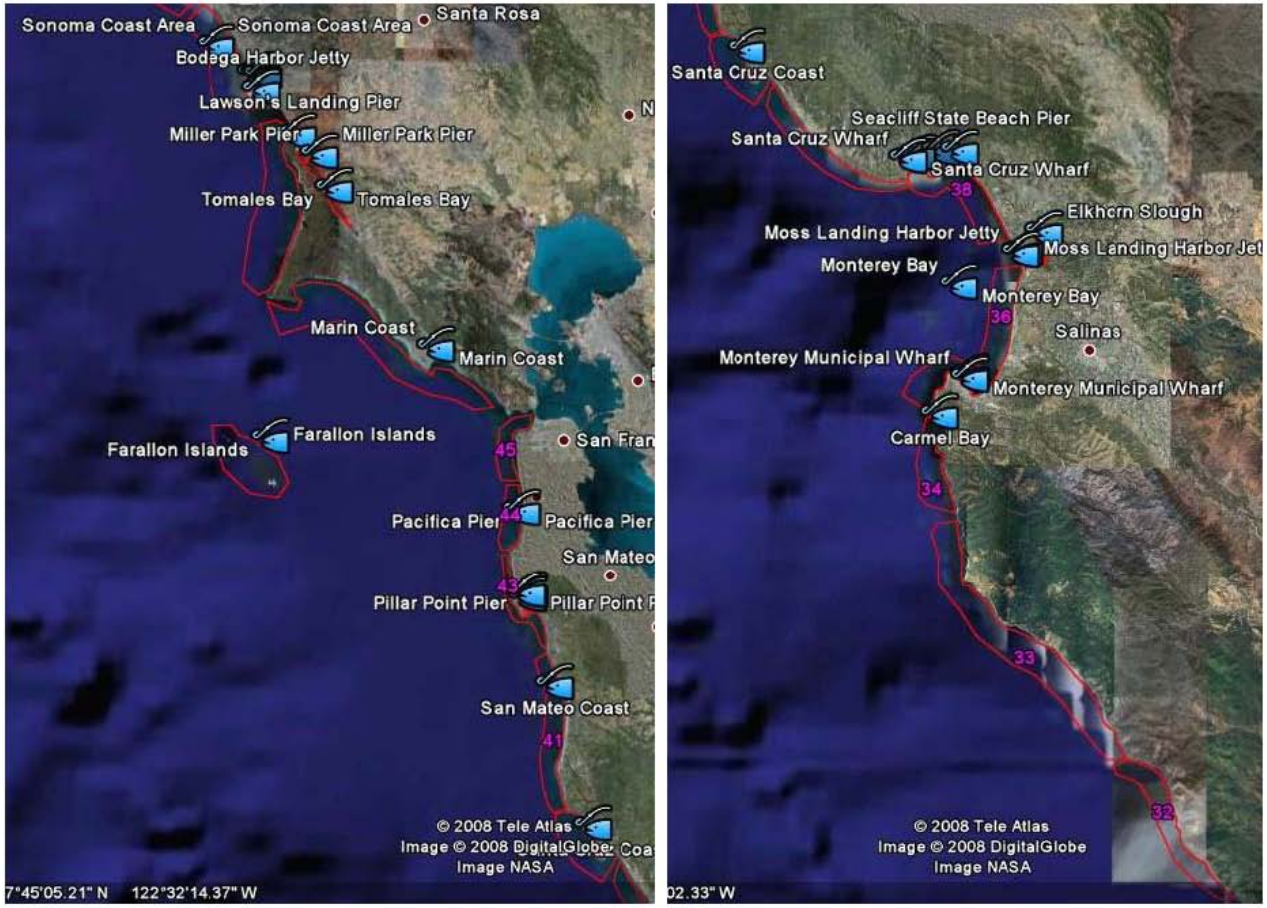

Figure 2. Zone maps (continued).

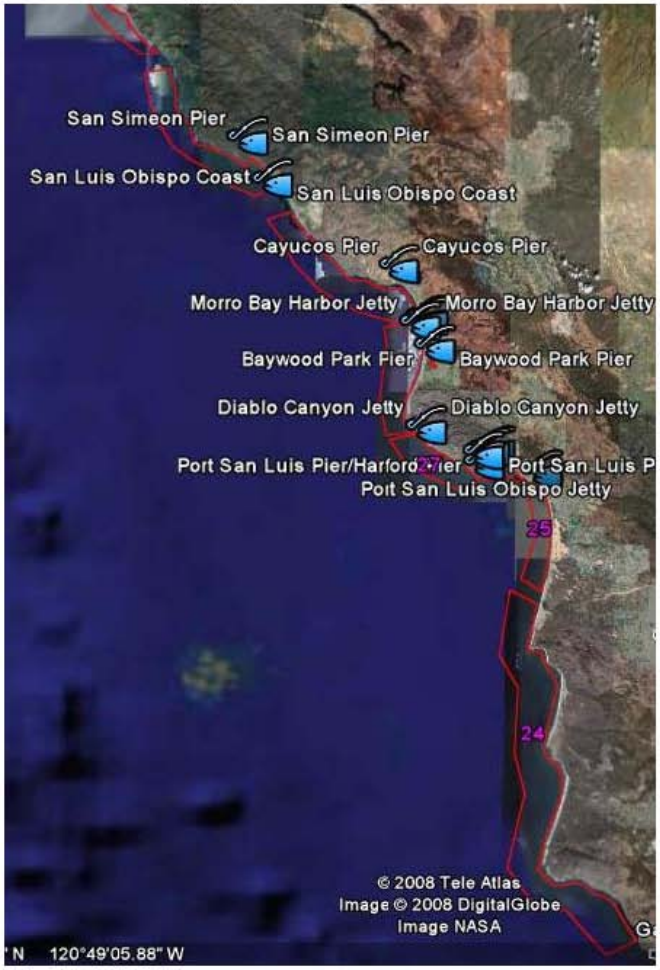

Figure 2. Zone maps (continued).

Figure 2. Zone maps (continued).

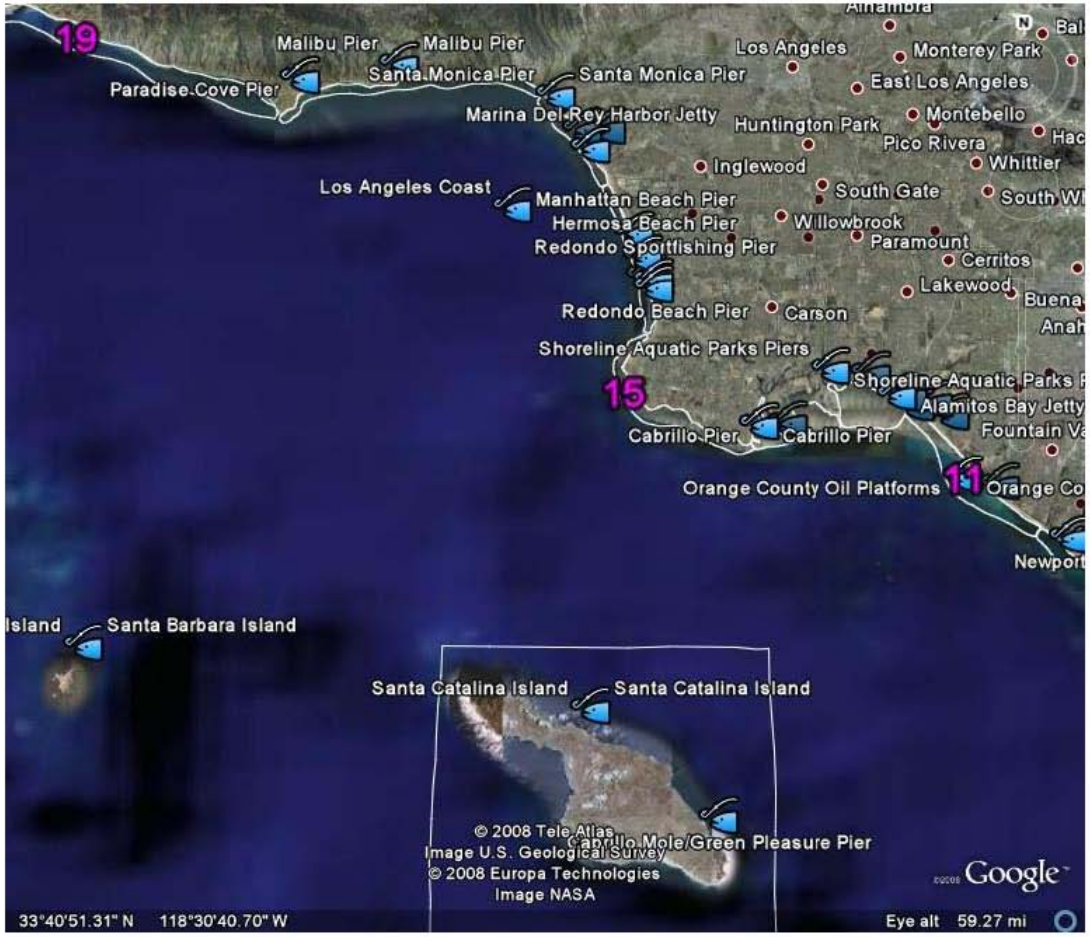

Figure 2. Zone maps (continued).

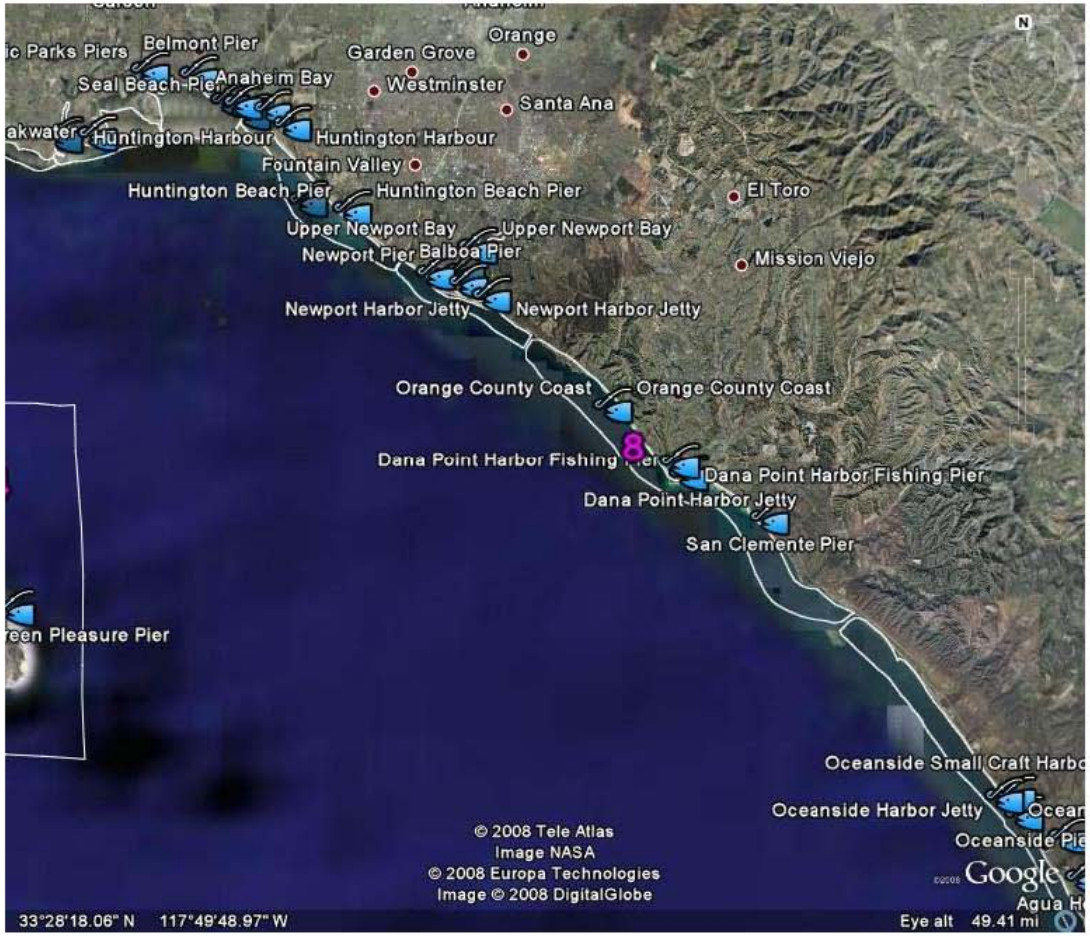

Figure 2. Zone maps (continued).

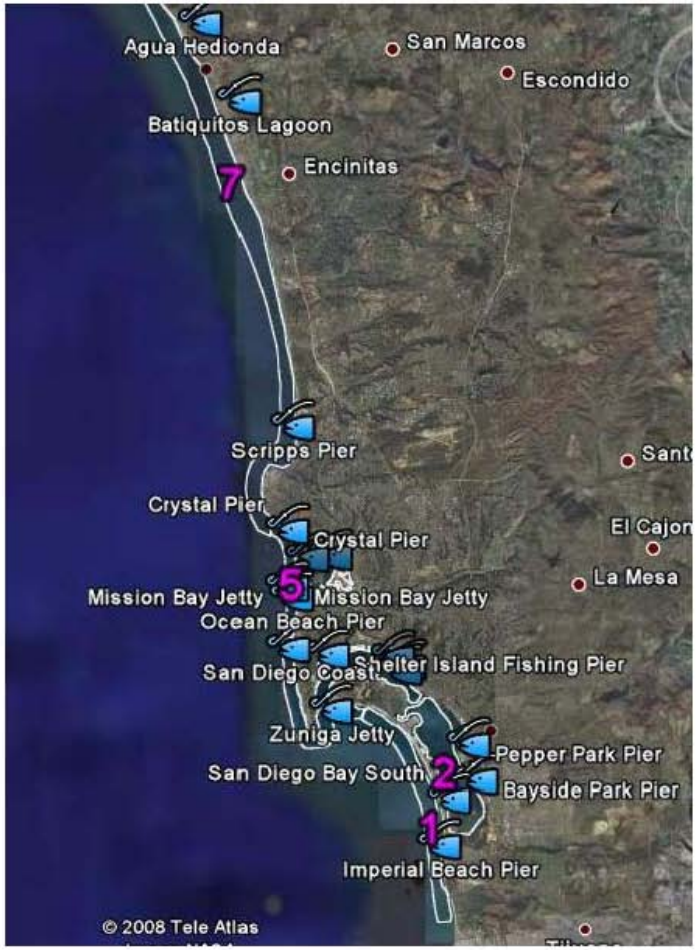

Figure 3. Zones in San Francisco Bay will be centered around the locations shown in this map.

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

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

D.1.1 What are the extent and location of water bodies with sufficient evidence to indicate that the fishing beneficial use is at risk due to pollutant bioaccumulation?

D.1.2 What are the extent and location of water bodies with some evidence indicating the fishing beneficial use is at risk due to pollutant bioaccumulation?

D.1.3 What are the extent and location of water bodies with no evidence indicating the fishing beneficial use is at risk due to pollutant bioaccumulation?

D.1.4 What are the proportions of water bodies in the State and each region falling within the three categories defined in questions D.1.1, D.1.2, and D.1.3?

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

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

D.2.1.1 Have water bodies fully supporting the fishing beneficial use become impaired?

D.2.1.2 Has full support of the fishing beneficial use been restored for previously impaired water bodies?

D.2.2 What are the trends in proportions of water bodies falling within the three categories defined in questions D.1.1, D.1.2, and D.1.3 regionally and statewide?

D.3. Evaluate sources and pathways of bioaccumulative pollutants impacting the fishing beneficial use

D.3.1 What are the magnitude and relative importance of pollutants that bioaccumulate and indirect causes of bioaccumulation throughout each Region and the state as a whole?

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

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

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

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

Table 2. RecFIN catch data for major groups of species, including data for the three regions (south, central and north) and specific data for the coast (ocean < 3 mi) and bays and harbors from January 2005 through December 2007. Data include mass of catch in tonnes and counts in thousands (parentheses). 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.

	COASTAL	BAYS HARBORS	Ocean <3mi Total			Bays/Harbors Total		
SoCal	Ocean <3mi Total	Bays/Harbors Total	Rank Mass	Rank Count	**Ave Rank**	Rank Mass	Rank Count	**Ave Rank**
Sharks, skates & rays	1069 (515)	231 (180)	2	6	4	2	5	3.5
Cabezon	74 (99)	1.75 (2.3)						
Top- & Jacksmelt	145 (925)	46 (405)	8	5	6.5	5	4	4.5
Rockfish spp	533 (1190)	20.73 (22.68)	5	4	4.5	7	7	7
Perch spp	574 (3281)	45 (212)	4	2	3	6		
Croaker spp	392 (1896)	132 (553)	6	3	4.5	4	2	3
Flatfish	625 (621)	479 (414)	3		3	1	3	2
Jack Mackerel	19.24 (153.4)	2.362 (26.18)		7			6	
Chub (Pacific) Mackerel	1359 (7648)	147 (845)	1	1	1	3	1	2
Lingcod	177.2 (106.4)	2.359 (1.364)	7	8	7.5			
Sturgeon								
Tuna (non-mackerel)	46.13 (5.247)	2.571 (0.25)						
Salmon	1.425 (0.456)							

TOP 5
RUNNERS UP
A HIGH PRIORITY EXCEPTION
TOP 5 IN OVERALL RANK

	Ocean <3mi Total			Bays/Harbors Total		
CenCal	Ocean <3mi Total	Bays/Harbors Total	Rank Mass	Rank Count	**Ave Rank**	Rank Mass	Rank Count	**Ave Rank**
Sharks, skates & rays	249.5 (127.2)	1065 (410)				1	3	2
Cabezon	48.95 (33.46)	3 (5)						
Top- & Jacksmelt	3151 (462)	111 (707)	1	3	2	3	1	2
Rockfish spp	1825 (3283)	33 (91)	2	1	1.5	6	5	5.5
Perch spp	351 (1514)	46 (427)	5	2	3.5	5	2	3.5
Croaker spp	28 (184)	13 (52)		8			6	
Flatfish	192 (259)	185 (163)	6	5	5.5	2	4	3
Jack Mackerel	3.592 (23.21)	0.389 (2.216)						
Chub (Pacific) Mackerel	60 (294)	1.842 (11.73)	8	4	6		7	
Lingcod	574 (247)	8.116 (4.083)	4	6	5	8		
Sturgeon	0.157 (0.013)	83 (10)				4	8	6
Tuna (non-mackerel)	61.19 (7.26)		7					
Salmon	730 (187)	12.2 (3.555)	3	7	5	7		

TOP 5
RUNNERS UP
A HIGH PRIORITY EXCEPTION
TOP 5 IN OVERALL RANK

Table 2. Continued. RecFIN catch data for major groups of species, including data for the three regions (south, central and north) and specific data for the coast (ocean < 3 mi) and bays and harbors from January 2005 through December 2007. Data include mass of catch in tonnes and counts in thousands (parentheses). 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.

NorCal	Ocean <3mi Total	Bays/Harbors Total	Ocean <3mi Total Rank Mass	Ocean <3mi Total Rank Count	Ocean <3mi Total Ave Rank	Bays/Harbors Total Rank Mass	Bays/Harbors Total Rank Count	Bays/Harbors Total Ave Rank
Sharks, skates & rays	14.48 (5.723)	26 (13)	7	8	7.5	1	4	2.5
Cabezon	32 (18)	0.594 (0.387)	6	4	5	8		
Top- & Jacksmelt	1 (7)	2 (16)		7		7	2	4.5
Rockfish spp	476 (599)	9 (14)	2	1	1.5	2	3	2.5
Perch spp	100 (197)	6 (23)	4	2	3	3	1	2
Croaker spp								
Flatfish	9 (7)	2.274 (0.964)	8	6	7	6	7	6.5
Jack Mackerel	0.129 (0.333)	0.009 (0.004)						
Chub (Pacific) Mackerel	0.007 (0.042)	0.019 (0.114)						
Lingcod	200 (70)	4 (2)	3	3	3	4	5	4.5
Sturgeon								
Tuna (non-mackerel)	76 (9)		5	5	5			
Salmon	480	4 (1)	1			5	6	5.5

limited fishing -
TOP 5
RUNNERS UP
A HIGH PRIORITY EXCEPTION
TOP 5 IN OVERALL RANK

Table 3. RecFin catch data for individual popular species, including data for the three regions (south, central and north) and specific data for the coast (ocean < 3 mi) and bays and harbors from January 2005 through December 2007. Green shading indicates most popular species within each group.

	SoCal				CenCal				NorCal			
	Ocean <3mi Total		Bays/Harbors Total		Ocean <3mi Total		Bays/Harbors Total		Ocean <3mi Total		Bays/Harbors Total	
	# (thou)	wt (metric tons)	# (thou)	wt (metric tons)	# (thou)	wt (metric tons)	# (thou)	wt (metric tons)	# (thou)	wt (metric tons)	# (thou)	wt (metric tons)
Olive Rockfish	42	15	1	0	171	125	1	0	8	5	0	0
Kelp Bass	1834	846	314	131	0	0	0	0				
Black Rockfish	9	3	0	0	248	135	6	3	332	283	10	7
Vermilion Rockfish	182	138	1	1	247	298	1	1	31	48	0	0
Canary Rockfish	2	1	0	0	84	31	0	0	17	9	0	0
Yellowtail Rockfish	7	3	0	0	115	52	0	0	11	7	0	0
Barred Sand Bass	1210	665	422	162	0	0	0	0				
Spotted Sand Bass	150	72	990	444	0	0	0	0				
California Scorpionfish	384	145	38	14	0	0	0	0				
Brown Rockfish	60	22	5	2	200	142	35	13	2	2	0	0
Copper Rockfish	85	44	0	0	73	72	0	0	7	10	1	0
Grass Rockfish	21	11	1	1	31	19	11	4	2	2	0	0
Gopher Rockfish	18	5	0	0	255	102	2	1	7	4	0	0
Black and Yellow Rockfish	1	0	0	0	23	10	0	0	0	0	0	0
Kelp Rockfish	22	8	0	0	15	6	1	0	0	0		
Bocaccio	85	72	0	0	18	26	0	0				
Blue Rockfish	82	30	0	0	953	402	5	2	109	58	1	1
Lingcod	106	177	1	2	247	574	4	8	70	200	2	4
Shiner Perch	154	4	20	1	156	5	233	10	2	0	2	0
Walleye Surfperch	447	38	12	1	181	21	39	4	2	0	8	1
Silver Surfperch	1	0	0	0	18	3	8	1	4	0	0	0
Spotfin Surfperch			0	0	3	0	0	0				
Black Perch	93	27	58	17	9	4	36	10				
Striped Seaperch	4	1	0	0	48	20	5	2	11	6	3	1
Rubberlip Seaperch	11	4	4	1	10	4	3	2				
Rainbow Seaperch	3	0	0	0	5	1	4	0	1	0	0	0
Barred Surfperch	1304	259	10	2	613	171	12	3				
Redtail Surfperch	0	0	0	0	68	31	5	2	148	81	6	4
Calico Surfperch	3	1	1	0	27	10	1	0	4	2		
White Seaperch	21	4	13	2	18	5	15	3				
Pile Perch	7	4	4	3	4	3	3	2	0	0	0	0
Brown Smoothhound	4	4	2	1	38	33	26	22	3	2	2	1
Gray Smoothhound	7	7	4	3	0	0	1	3	0	0		
Leopard Shark	88	124	6	10	13	31	149	338	0	0	1	2
Spiny Dogfish	10	22	0	0	7	12	4	4	2	3	5	8
Bat Ray	46	131	22	59	26	88	92	346	0	1	3	13
Pacific Sanddab	80	11	1	0	199	21	78	8	2	2		
Topsmelt	151	10	184	7	6	0	109	5	0	0		
Jacksmelt	774	135	241	39	456	80	598	109	7	1	16	2

Top
Runner Up

Table 4. Target species in each region for coastal waters and bays and harbors. Numbers indicate priorities for secondary targets. Species in italics are those that will be analyzed as individuals for Hg as well as composited for other analytes. If the target species to be analyzed as individuals for Hg are not available substitutions will be made. Asterisks indicate species that were in the top five in catch for each habitat by region combination.

Coast <3mi	SoCal	CenCal	NorCal
Primary	*Kelp Bass**		
	Olive Rockfish		
		Black Rockfish	*Black Rockfish**
		*Blue Rockfish**	*Blue Rockfish*
			Lingcod*
	Barred Surfperch*	Barred Surfperch*	
			Redtail Surfperch*
		Salmon*	Salmon*
	Chub Mackerel*		
	White Croaker	White Croaker	
Secondary	Barred Sand Bass*		
	Spotted Sand Bass		
	Scorpionfish		
		Olive Rockfish	
		Lingcod #6*	
			Cabezon #6*
	Walleye Surfperch*		Walleye Surfperch
		Shiner Surfperch	
		Jacksmelt #7*	
	Yellowfin Croaker		

Bays/Harbors	SoCal	CenCal	NorCal
Primary	*Kelp Bass**		
			*Black Rockfish**
	Spotted Sand Bass*		
	Shiner Surfperch	*Shiner Surfperch**	*Shiner Surfperch*
			Redtail Surfperch*
	Leopard Shark	Leopard Shark*	Leopard Shark
		Halibut*	
		Jacksmelt*	Jacksmelt*
	White Croaker	White Croaker	
Secondary	Barred Sand Bass*		
	Scorpionfish		
		*Brown Rockfish**	
		Black Rockfish	
			Blue Rockfish
			Lingcod #7*
	Chub Mackerel		
	Walleye Surfperch	Walleye Surfperch	Walleye Surfperch #8
		Black Perch	
	Grey Smoothound		
		Brown Smoothound	Brown Smoothound
			Spiny Dogfish #6*
	Topsmelt*		
	Jacksmelt*		

Table 5. Target species and their characteristics. Sources were from various websites and pers comm.; 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
Rockfish (Scorpaenidae)	Blue Rockfish (*Sebastes mystinus*)	2	tunicates, hydroids, jellyfishes, and larval and juvenile fishes	mid-water	deep rocky reefs ro hard, flat substrates	Bering Sea to Baja	0-100m
	Black Rockfish (*Sebastes melaops*)	3	juvenile rockfish, euphausids and amphipods (upwelling), and invertebrates (non-upwelling)	mid-water	kelp beds	Alaska to SoCal	0-366m
	California 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
	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 rocks, oil platforms, sewer pipes	Alaska to Baja	0-128m
Lingcod (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	3	polychaetes, small	benthic	Over sandy bottoms	BC to Baja	to

	(*Genyonemus lineatus*)		shrimps, crabs and mollusks				183m
	Yellowfin Croaker (*Umbrina roncador*)	3	crustaceans and fishes	benthic	coastal waters and estuaries		
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
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 perch (*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*)	2	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
New World Silversides (Atherinopsidae)	Jacksmelt (*Atherinopsis californiensis*)	2	crustaceans, fish larvae	mid-water	inshore areas, including bays	Yaquina Bay, OR to Baja	
	Topsmelt (*Atherinops affinis*)	2	zooplankton, algae	benthic/ mid-water	bays, muddy and rocky areas and kelp beds	Vancouver Island to Baja	
Mackerels (Scombridae)	Pacific Chub Mackerel (*Scomber japonicus*)	3	copepods, crustaceans, euphausids, small fishes and squids	mid-water	pelagic	Indo-Pacific	to 300m
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
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
Sculpins (Cottidae)	Cabezon (*Scorpaenichthys marmoratus*)	3	crustaceans, fish and mollusks	demersal	rocky, sandy and muddy bottoms, kelp beds	Southeastern AK to Baja	to 200m

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.
Trophic level 2: Zooplankton and benthic invertebrates.
Trophic level 3: Organisms that consume zooplankton, benthic invertebrates, and TL2 organisms.
Trophic level 4: Organisms that consume trophic level 3 organisms.

Table 6. Target species, size ranges, and numbers to include in composites.

		Primary or Secondary	Number in Composites	Size Range (mm)
Rockfish	Kelp Bass	P	5	>305 (255-350 individuals for Hg)
	Blue Rockfish	P,S	5	>305 (255-350 individuals for Hg)
	Black Rockfish	P,S	5	>305 (255-350 individuals for Hg)
	Barred Sandbass	S	5	>305
	Scorpionfish	S	5	>255
	Spotted Sandbass	S	5	>305
	Olive Rockfish	S	5	>255 (220-350 individuals for Hg)
	Brown Rockfish	P	5	>255 (220-350 individuals for Hg)
Lingcod		P,S	3	
Croaker	White Croaker	P	5	>200
	Yellow Croaker	S	5	>200
Chinook Salmon		P		
Surfperch	Barred	P	5	>150
	Redtail	P	5	>263
	Shiner	P,S	20	>100 (80-173 individuals for Hg)
	Walleye	P,S	5	>150
	Black	S	5	>150
Smelt	Jacksmelt	P	5	>220
	Topsmelt	S	5	>200
Chub Mackerel		P		
Shark	Leopard Shark	P	3	>915
	Spiny Dogfish	P	3	>610

	Brown Smoothhound	S	3	>610
	Gray Smoothound	S	3	>610
California Halibut		P	3	>558
Cabezon		S	5	>381

Table 7. Summary of analytes included in the study.

Analyte	Included in Screening Study?
Methylmercury[1]	Some individuals, all composites
PCBs	All composite samples
DDTs	All composite samples
Dieldrin	All composite samples
Aldrin	All composite samples
Chlordanes	All composite samples
PBDEs	Two composite samples per zone
Dioxins	SF Bay only
Perfluorinated chemicals	SF Bay only, archives created for remainder
Selenium	All composite samples (year 2 contingent upon year 1 results)
Omega-3 fatty acids	SF Bay only

[1] Measured as total mercury.

Table 8. Parameters to be measured.

FISH ATTRIBUTES

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

METALS AND METALLOIDS

1. Total mercury
2. Selenium

PESTICIDES

Chlordanes

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

DDTs

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

Cyclodienes

1. Aldrin
2. Dieldrin
3. Endrin

HCHs

1. HCH, alpha
2. HCH, beta
3. HCH, gamma

Table 8. Parameters to be measured (continued).

Others

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

PCBs

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

37. PCB 156
38. PCB 157

Table 8. Parameters to be measured (continued).

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

PBDEs (these would be estimated values obtained along with PCB congeners at no additional cost)

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
12. PBDE 190

Table 8. Parameters to be measured (continued).

Dioxins and Dibenzofurans

HpCDD, 1,2,3,4,6,7,8-
HpCDF, 1,2,3,4,6,7,8-
HpCDF, 1,2,3,4,7,8,9-
HxCDD, 1,2,3,4,7,8-
HxCDD, 1,2,3,6,7,8-
HxCDD, 1,2,3,7,8,9-
HxCDF, 1,2,3,4,7,8-
HxCDF, 1,2,3,6,7,8-
HxCDF, 1,2,3,7,8,9-
HxCDF, 2,3,4,6,7,8-
OCDD, 1,2,3,4,6,7,8,9-
OCDF, 1,2,3,4,6,7,8,9-
PeCDD, 1,2,3,7,8-
PeCDF, 1,2,3,7,8-
PeCDF, 2,3,4,7,8-
TCDD, 2,3,7,8-
TCDF, 2,3,7,8-

Table 9. Fish Contaminant Goals (FCGs) for Selected Fish Contaminants Based on Cancer and Non-Cancer Risk* Using an 8-Ounce/Week (prior to cooking) Consumption Rate (32 g/day)** From Klasing and Brodberg (2008).

	FCGs (ppb, wet weight)
Contaminant Cancer Slope Factor $(mg/kg/day)^{-1}$	
Chlordane (1.3)	**5.6**
DDTs (0.34)	**21**
Dieldrin (16)	**0.46**
PCBs (2)	**3.6**
Toxaphene (1.2)	**6.1**
Contaminant Reference Dose (mg/kg-day)	
Chlordane ($3.3x10^{-5}$)	100
DDTs ($5x10^{-4}$)	1600
Dieldrin ($5x10^{-5}$)	160
Methylmercury ($1x10^{-4}$)[S]	**220**
PCBs ($2x10^{-5}$)	63
Selenium ($5x10^{-3}$)	**7400**
Toxaphene ($3.5x10^{-4}$)	1100

*The most health protective Fish Contaminant Goal for each chemical (cancer slope factor- versus reference dose-derived) for each meal category is bolded.
**g/day represents the average amount of fish consumed daily, distributed over a 7-day period, using an 8-ounce serving size, prior to cooking.
[S]Fish Contaminant Goal for sensitive populations (i.e., women aged 18 to 45 years and children aged 1 to 17 years.)

Tabled values are rounded based on laboratory reporting of three significant digits in results, where the third reported digit is uncertain (estimated). Tabled values are rounded to the second digit, which is certain. When data are compared to this table they should also first be rounded to the second significant digit as in this table.

Table 10. 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). From Klasing and Brodberg (2008).

Contaminant	Three 8-ounce Servings* a Week	Two 8-ounce Servings* a Week	One 8-ounce Servings* a Week	No Consumption
Chlordane[c]	≤190	>190-280	>280-560	>560
DDTs[nc**]	≤520	>520-1,000	>1,000-2,100	>2,100
Dieldrin[c]	≤15	>15-23	>23-46	>46
Methylmercury (Women aged 18-45 years and children aged 1-17 years)[nc]	≤70	>70-150	>150-440	>440
Methylmercury (Women over 45 years and men)[nc]	≤220	>220-440	>440-1,310	>1,310
PCBs[nc]	≤21	>21-42	>42-120	>120
Selenium[nc]	≤2500	>2500-4,900	>4,900-15,000	>15,000
Toxaphene[c]	≤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.

Tabled values are rounded based on laboratory reporting of three significant digits in results, where the third reported digit is uncertain (estimated). Tabled values are rounded to the second digit, which is certain. When data are compared to this table they should also first be rounded to the second significant digit as in this table.

Table 11. Species to be collected and analytes for RMP 2009 sport fish monitoring. Numbers indicate counts of composite samples to be collected and analyzed.

	Fish Collection	Composite - Organics	Individual - Metals	OC Analyses (PCBs, Pesticides)	PBDEs	Dioxins	Hg - Indiv	Hg - Comp	Se	PFC	Omega 3s
White Croaker	12	12		24	24	24			12	3	
Placeholder (archive and other species TBD)											
Striped Bass	6	6	18	6	6		18		6	3	
Shiner Surfperch	15	5		15	15	10		15	15	3	
White Sturgeon (South Bay and San Pablo Bay)	4	4	12	4	4				36	3	
Leopard Shark	3	3		3	3		9		3	3	
Halibut	3	3		3	3			3	3	3	
Jacksmelt	4	4		4	4			4	4		
Anchovy	9	9		9	9				9	3	
Subtotals	56	46	30	68	68	34	27	22	88	21	TBD

Appendix III: MPSL-DFG SOPs

MPSL-DFG EPA Modifications and Laboratory Procedures			
Page	Procedure/Equipment	SOP Number	Revision Date
A	Modifications to EPA 3052		Feb 2006
B	Sample Container Preparation for Organics and Trace Metals, Including Mercury and Methylmercury	MPSL-101	Mar 2007
C	Sampling Marine and Freshwater Bivalves, Fish and Crabs for Trace Metal and Synthetic Organic Analysis	MPSL-102a Tis Collection	Mar 2007
E	Analysis of Mecury in Sediments and Tissue by Flow Injection Mercury System (FIMS)	MPSL-103 (formerly DFG SOP 103)	Feb 2000
D	Sample Receipt and Check-In	MPSL-104 Receipt and Check-in	Feb 2006
E	Protocol for Tissue Sample Preparation	MPSL-105 Tissue Preparation	Mar 2007

Appendix III A: 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 III B: 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 Sapphire Thermowell: CEM Part # 326280

4.40 Shoe covers: Cellucap Franklin Part # 28033

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

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

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

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

4.45 Teflon HP500+ Control Cover: CEM Part # 431255

4.46 Teflon HP500+ Cover: CEM Part # 431250

4.47 Teflon HP500+ Liner: CEM Part # 431110

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

4.49 Teflon Tape (plumbing tape)

4.50 Teflon Thermowell Nut: CEM Part #325028

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

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

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

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

4.55 Teflon Wash Bottle, 500 mL

4.56 Teflon Vent Nut: CEM Part # 431313

4.57 Titanium Cutter Screw: Büchi Analytical Part # 34376

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

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

4.60 Titanium Displacement Disc: Büchi Analytical Part # 26471

4.61 Ventilation Hood

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

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

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

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

4.66 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: Seastar Chemicals Part # BA-01

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 Seastar HNO_3 to 16.67 parts MilliQ

5.17 Nitric Acid (HNO_3), 1%: prepared by adding 1 part Seastar 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% Seastar HNO_3, cap. 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 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% Seastar 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 Teflon and Sapphire Digestion Nuts and Thermowells

7.1.9.1 Remove any rupture membranes that may still be in the Vent Nuts.

7.1.9.2 Rinse each item with a dilute Micro/Tap solution by rubbing them gently between your hands.

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

7.1.9.4 Submerge in 6% Seastar HNO_3 bath, heated for a minimum of 8 hours in a hotbath. Use a new 4milx6"x8"Zipper-closure polyethylene bag filled with acid to contain and protect these small parts in the bath. (Note: You may reuse this bag as long as it does not come in contact with unclean surfaces.)

7.1.9.5 Rinse three to five times in MilliQ.

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

7.1.9.7 Store completely dry nuts it an appropriately sized zipper-closure polyethylene bag. Label bag with the date of completion. (Note: You may use bags that have formerly contained clean nuts.)

7.1.9.8 Store thermowells in the tubes provided to reduce the chance of breakage.

7.1.10 Polyethylene Digestate Bottles

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

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

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

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

7.1.10.5 Fill with MilliQ and soak for three days.

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

7.1.10.7 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.

7.1.11 Polypropylene Centrifuge Tubes, 15 mL ("ICP Tubes")

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

7.1.11.2 Rinse three times in Tap, followed by three rinses in DI.

7.1.11.3 Submerge tubes and caps in 50% HCl cold bath for three days.

7.1.11.4 Rinse each tube and cap three times with MilliQ.

7.1.11.5 Place tubes and caps on clean crew wipers to dry.

7.1.11.6 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 and let dry completely.

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

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

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

7.2.4.1 Rinse new tubes in dilute Micro/Tap.

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

7.2.4.3 Submerge in 50% HNO_3 hot bath for 8 hours, ensuring that each tube is completely filled.

7.2.4.4 Rinse cooled tubes three to four times in MilliQ.

7.2.4.5 Let dry completely on clean Crew Wipers.

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

7.3 Methylmercury Only Sample Containers

7.3.1 Teflon Digestion or Distillation Vials

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

7.3.1.2 Rinse three times in DI.

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

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

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

7.3.2 Teflon Distillation Caps and Tubing

7.2.3.2 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 and let dry completely.

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

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

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

7.2.4.1 Rinse new tubes in dilute Micro/Tap.

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

7.2.4.3 Submerge in 50% HNO_3 hot bath for 8 hours, ensuring that each tube is completely filled.

7.2.4.4 Rinse cooled tubes three to four times in MilliQ.

7.2.4.5 Let dry completely on clean Crew Wipers.

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

7.3 Methylmercury Only Sample Containers

7.3.1 Teflon Digestion or Distillation Vials

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

7.3.1.2 Rinse three times in DI.

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

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

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

7.3.2 Teflon Distillation Caps and Tubing

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

7.3.2.2 Rinse three times in DI.

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

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

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

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

7.4 Organic Sample Containers

7.4.1 Aluminum Foil Sheets

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

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

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% Seastar 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% Seastar 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 C: 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 Apparatus and Materials

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

4.1 Anchor Chains

4.2 Backpack Shocker (electro-fishing)

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

4.4 Bone Saw

4.5 Camera, digital

4.6 Cast Nets (10' and 12')

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

4.8 Daypacks

4.9 Depth Finder

4.10 Dip Nets

4.11 Dry Ice or Ice

4.12 Gill Nets (various sizes)

4.13 GPS

4.14 Heavy Duty Aluminum Foil, prepared

4.15 Heavy Duty plastic bags, Clear 30 gallon

4.16 Inflatable Buoy

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

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

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

4.20 Otter Trawl (various widths as appropriate)

4.21 Permanent Marking Pen

4.22 Plastic bucket, 30 gallon

4.23 Plastic Ice Chests

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

4.25 Polypropylene Mesh, 76mm wide with 13mm mesh

4.26 Polypropylene Mesh, 50mm wide with 7mm mesh

4.27 Polypropylene Line, 16mm

4.28 Rods and Reels

4.29 Screw in Earth Anchor, 4-6" diameter

4.30 Scuba Gear

4.31 Seines (various size mesh and lengths as appropriate)

4.32 Stainless Steel Dive Knives

4.33 Trap Nets (hoop or fyke nets)

4.34 Teflon Forceps

4.35 Teflon Sheet, prepared

4.36 Teflon Wash Bottle, 500 mL

4.37 Wading Gear

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

5.0 Reagents

5.1 Tap water (Tap)

5.2 Deionized water (DI)

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

5.4 Micro Detergent: ColeParmer Part # 18100-20

5.5 Methanol: VWR Part # JT9263-3

5.6 Petroleum Ether: VWR Part # JT9265-3

6.0 Sample Collection, Preservation and Handling

6.1 All sampling equipment will be made of non-contaminating materials and will be inspected prior to entering the field. Nets will be inspected for holes and repaired prior to being used. Boats (including the electroshocking boat) will be visually checked for safety equipment and damage prior to being taken into the field for sample collection.

6.2 To avoid cross-contamination, all equipment used in sample collection should be thoroughly cleaned before each sample is processed. Ideally, instruments are made of a material that can be easily cleaned (e.g. Stainless steel, anodized aluminum, or borosilicate glass). Before the next sample is processed, instruments should be washed with a detergent solution, rinsed with ambient water, rinsed with a high-purity solvent (methanol or petroleum ether), and finally rinsed with MilliQ. Waste detergent and solvent solutions must be collected and taken back to the laboratory.

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

6.4 Mussels and clams to be analyzed for metals are double-bagged in zipper-closure bags. Bivalves to be analyzed for organics are wrapped in prepared aluminum foil prior to placement in zipper-closure bags.

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

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

6.7 Data is recorded for each site samples are transplanted to or collected from. Data includes, but is not limited to station name, sample identification number, site location (GPS), date collected or transplanted, collectors names, water depth, photo number, ocean/atmospheric conditions (if appropriate), description of site, and drawing if necessary.

6.8 A chain of custody form (MPSL QAP Appendix E) will accompany all samples that are brought to the lab. All samples that are processed in the lab MUST be checked in according to Method # MPSL-104.

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

7.0 Procedure

7.1 Sample collection - mussels and clams

7.1.1 The mussels to be transplanted (*Mytilus californianus*) are collected from Trinidad Head (Humboldt Bay Intensive Survey), Montana de Oro (Diablo Canyon Intensive Survey), and Bodega Head (all other statewide transplants). The freshwater clam (*Corbicula fluminea*) source is Lake Isabella or the Sacramento River. Analyze mussel and clam samples for background contaminates prior to transplanting.

7.1.2 Polyethylene gloves are worn while prying mussels off rocks with dive knives. Note: polyethylene gloves should always be worn when handling samples. Mussels of 55mm to 65mm in length are recommended. Fifty mussels are collected for each TM and each SO sample.

7.1.3 Collected mussels are carried out of collection site in zipper-closure bags placed in cleaned nylon daypacks. For the collection of resident samples where only one or two samples are being collected the mussels are double bagged directly into a labeled zipper-closure bag. Samples for SO are wrapped first in prepared aluminum foil.

7.1.4 Clams (*Corbicula fluminea*) measuring 20 to 30mm are collected by dragging the clam dredge along the bottom of the lake or river. The clams are poured out of the dredge into a 30 gallon plastic bag. Clams can also be collected by gloved hands in shallow waters and placed in labeled zipper-closure bags. 25-200 clams are collected depending on availability and necessity for analyses.

7.1.5 Data is recorded for each site samples are collected from. Data includes, but is not limited to station name, date collected, collectors names, water depth, GPS readings, photo, ocean/atmospheric conditions (if appropriate), description of site, and drawing if necessary.

7.2 Transplanted sample deployment

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

7.2.2 Once bagged, the mussels are placed in a 30 gallon plastic bag and stored in a cooler (cooled with ice) for no more than 48 hours. The ice is placed in zipper-closure bags to avoid contamination.

7.2.3 If marine samples are held for longer than 48 hours they are placed in holding tanks with running seawater at the lab. Control samples for both SO and TM are also held in the tank.

7.2.4 For freshwater clams: clams (25-200) are placed in 50mm X 7mm polypropylene mesh bags using identical procedures to those used with mussels (section 7.2.1). If clams need to be stored for more than 48 hours, the mesh bags are deployed either in a clean source or in holding tanks with running freshwater at the lab until actual sample deployment.

7.2.5 The mussels are attached to an open water transplant system that consists of a buoy system constructed with a heavy weight anchor (about 100lbs) or screw-in earth anchor, 13mm polypropylene line, and a 30cm diameter subsurface buoy. The sample bags are attached with cable ties to the buoy line about 15 feet below the water surface. In some cases the sample is hung on suspended polypropylene lines about 15 feet below the water surface between pier pilings or other surface structures. Creosote-coated wooden piers are avoided because they are a potential source of contamination. In some cases the mussels are hung below a floating dock. In shallow waters a wooden or PVC stake is hammered into the substrate and the mussel bags are attached by cable ties to the stake.

7.2.6 The clams are deployed by attaching the mesh bag with cable ties to wooden or PVC stakes hammered into substrate or screw in earth anchors. The bags containing clams are typically deployed 15cm or more off the bottom. In areas of swift water, polypropylene line is also attached to the staked bags and a permanent object (piling, tree or rock).

7.2.7 Transplants are usually deployed for 1-4 months. Ideally mussels are transplanted in early September and retrieved in late December and early January. Clams are usually transplanted in March or April and retrieved in May or June.

7.2.8 Data is recorded for each site samples are transplanted to or collected from. Data includes, but is not limited to station name, date collected or transplanted, collectors names, water depth, GPS readings, photo, ocean/atmospheric conditions (if appropriate), description of site, and drawing if necessary.

7.3 Sample Retrieval

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

7.3.2 All mussels to be analyzed for SO are wrapped in prepared aluminum foil (Method # DFG 101). The foil packet is double bagged in appropriately sized and labeled zipper-closure bags. Note: samples should only contact the dull side of the foil.

7.3.3 The bags containing samples are clearly and uniquely identified using a water-proof marking pen or pre-made label. Information items include ID number, station name, depth (if from a multiple sample buoy), program identification, date of collection, species and type of analysis to be performed.

7.3.4 The samples are placed in non-metallic ice chests and frozen using dry ice or regular ice. (Dry ice is used when the collecting trip takes more than two days.) At the lab, samples should be stored at or below -20°c until processed.

7.4 Sample Collection – Fish

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

7.4.1.1 Electro-fisher boat- The electro-fisher boat is run by a trained operator, making sure that all on board follow appropriate safety rules. Once on site, adjustment of the voltage, amps, and pulse for the ambient water is made and recorded. The stainless steel fish well is rinsed with ambient water, drained and refilled. The shocked target fish are placed with a nylon net in the well with circulating ambient water. The nylon net is washed with a detergent and rinsed with ambient water prior to use. Electro-fishing will continue until the appropriate number and size of fish are collected.

7.4.1.2 Backpack electro-fisher- The backpack shocker is operated by a trained person, making sure that all others helping follow appropriate safety rules. The backpack shocker is used in freshwater areas where an electro-fisher boat can not access. Once on site, adjustment of the voltage, amps, and pulse for the ambient water is made and recorded. The shocked target fish are captured with a nylon net and placed in a 30 gallon plastic bag. The nylon net is washed with a detergent and rinsed with ambient water prior to use. Electro-fishing will continue until the appropriate number and size of fish are collected.

7.4.1.3 Fyke or hoop net- Six-36 inch diameter hoops connected with 1 inch square mesh net is used to collect fish, primarily catfish. The net is placed parallel to shore with the open hoop end facing downstream. The net is placed in areas of slow moving water. A partially opened can of cat food is placed in the upstream end of the net. Between 2-6 nets are placed at a site overnight. Upon retrieval a grappling hook is used to pull up the downstream anchor. The hoops and net are pulled together and placed on a 30

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

7.4.1.4 Otter-trawl- A 14 foot otter trawl with 24 inch wooden doors or a 20 foot otter trawl with 30 inch doors and 80 feet of line is towed behind a boat for water depths less than 25 feet. For water depths greater than 25 feet another 80 feet of line is added to capture fish on or near the substrate. Fifteen minute tows at 2-3 knots speed are made. The beginning and ending times are noted on data sheets. The trawl is pulled over the side of the boat to avoid engine exhaust. The captured fish are emptied into a 30 gallon plastic bag for sorting. Desired fish are placed with polyethylene gloves into another 30 gallon plastic bag and kept in an ice chest with ice.

7.4.1.5 Gill nets- A 100 yard monofilament gill net of the appropriate mesh size for the desired fish is set out over the bow of the boat parallel to shore. The net is retrieved after being set for 1-4 hours. The boat engine is turned off and the net is pulled over the side or bow of the boat. The net is retrieved starting from the down-current end. If the current is too strong to pull in by hand, then the boat is slowly motored forward and the net is pulled over the bow. Before the net is brought into the boat, the fish are picked out of the net and placed in a 30 gallon plastic bag and kept in an ice chest with ice.

7.4.1.6 Beach seines- In areas of shallow water, beach seines of the appropriate length, height, and mesh size are used. One sampler in a wetsuit or waders pulls the beach seine out from shore. The weighted side of the seine must drag on the bottom while the float side is on the surface. The offshore sampler pulls the seine out as far as necessary and then pulls the seine parallel to shore and then back to shore, forming a half circle. Another sampler is holding the other end on shore while this is occurring. When the offshore sampler reaches shore the two samplers come together with the seine. The seine is pulled onto shore making sure the weighted side drags the bottom. When the seine is completely pulled onshore, the target fish are collected with polyethylene gloves and placed in a 30 gallon plastic bag and kept in an ice chest with ice. The beach seine is rinsed off in the ambient water and placed in the rinsed 30 gallon plastic bucket.

7.4.1.7 Cast net- A 10 or 12 foot cast net is used to collect fish off a pier, boat, or shallow water. The cast net is rinsed in ambient water prior to use and stored in a covered plastic bucket. The target fish are sampled with polyethylene gloves and placed in a 30 gallon plastic bag and kept in an ice chest with ice.

7.4.1.8 Hook and line- Fish are caught off a pier, boat, or shore by hook and line. Hooked fish are taken off with polyethylene gloves and placed in a Ziploc™ bag or a 30 gallon plastic bag and kept in an ice chest with ice.

7.4.1.9 Spear fishing- Certain species of fish are captured more easily by SCUBA divers spearing the fish. Only appropriately trained divers following the dive safety program guidelines are used for this method of collection. Generally, fish in the kelp beds are more easily captured by spearing. The fish are shot in the head area to prevent the fillets from being damaged or contaminated. Spear tips are washed with a detergent and rinsed with ambient water prior to use.

7.4.2 As a general rule, five fish of medium size or three fish of larger size are collected as composites for analysis. The smallest fish length cannot be any smaller than 75% of the largest fish length. Five fish usually provides sufficient quantities of tissue for the dissection of 150 grams of fish flesh for organic and inorganic analysis. The medium size is more desirable to enable similar samples to be collected in succeeding collections.

7.4.3 When only small fish are available, sufficient numbers are collected to provide 150 grams of fish flesh for analysis. If the fish are too small to excise flesh, the whole fish, minus the head, tail, and guts are analyzed as composites.

7.4.4 Species of fish collected are chosen for their importance as indicator species, availability or the type of analysis desired. For example, livers are generally analyzed for heavy metals. Fish without well-defined livers, such as carp or goldfish, are not collected when heavy metal analyses are desired.

7.4.5 Fish collected, too large to fit in clean bags (>500 mm) are initially dissected in the field. At the dock, the fish are laid out on a clean plastic bag and a large cross section from behind the pectoral fins to the gut is cut with a cleaned bone saw or meat cleaver. The bone saw is cleaned (micro, DI, methanol) between fish and a new plastic bag is used. The internal organs are not cut into, to prevent contamination. For bat rays, a section of the wing is cut and saved. These sections are wrapped in prepared Teflon sheets, double bagged and packed in dry ice before transfer to the freezer. During lab dissection, a subsection of the cross section is removed, discarding any tissue exposed by field dissection.

7.4.6 Field data (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, fish total lengths (fork lengths where appropriate), photo number and a note of other fish caught.

7.4.7 The fish are then wrapped in aluminum foil or Teflon sheets if thylates are analyzed. The wrapped fish are then double-bagged in zipper-closure bags with the inner bag labeled.

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

7.5 Sample Collection- Crabs

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

8.0 Analytical Procedure

8.1 Tissue Preparation procedures can be found in Method # 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.

9.0 Quality Control

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

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

10.0 References

10.1 Flegal, R.A. 1982. In: Wastes in the Ocean, Vol VI: Near Shore Waste Disposal. B.H. Ketchum (ed.). John Wiley and Sons Inc. Publishers, New York, 1982.

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10.3 Gordon, R.M., G.A. Knauer and J.H. Martin. 1980a. *Mytilus californianus* as a bioindicator of trace metal pollution: variability and statistical considerations. Mar. Poll. Bull. 9:195-198.

10.4 Hayes, S. P. and P. T. Phillips. 1986. California State Mussel Watch: Marine water quality monitoring program 1984-85. State Water Resources Control Board Water Quality Monitoring Report No. 86-3WQ.

10.5 EPA. 1995. Guidance for Assessing Chemical Contaminant Data for Use in Fish Advisories. Volume 1: Fish Sampling and Analysis. EPA 823-R-95-007.

Appendix III D: MPSL-103 Analysis of Mercury in Sediments and Tissue by Flow Injection Mercury System (FIMS)

Analysis of Mercury in Sediments and Tissue by Flow Injection Mercury System (FIMS) MPSL-103 (Formerly known as DFG SOP-103)

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Adapted from FGS 069.1 by: Mark Stephenson
February 21, 2000

1.0 SCOPE AND APPLICATION

1.1. This SOP is designed to ensure that reproducible, traceable procedures are followed in the standardization of Perkin Elmer FIMS mercury analyzer, and to establish the bounds wherein data will be considered acceptable. This SOP consists of two aspects: (1) preparation of mercury standard solutions; (2) calibration sequence of the mercury analyzer.

2.0 SUMMARY OF METHOD

Sediment and tissue are analyzed for mercury by: (1) digesting using Frontier Geoscience's methods for sediment (FGS-066) and tissue (FGS-011.2); and (2) analyzing the solutions by flow injection using the Perkin Elmer FIMS system.

3.0 INTERFERENCES **There have been some reports of interferences by chlorine gas in the digestate.**

4.0 SAFETY

4.1. **CAUTION**: The toxicity or carcinogenicity of each chemical used in this method has not been precisely determined. However, each compound should be treated as a potential health hazard. Exposure to these compounds should be reduced to the lowest possible level. Exhibit particular caution in the preparation and use of bromine monochloride, as it releases extremely irritating, corrosive fumes similar in effect to free chlorine. Always handle this reagent in an approved fume hood.

5.0 EQUIPMENT

Perkin Elmer FIMS system with autosampler

6.0 REAGENTS

6.1. Reagent Water- Reagent water (18MΩ minimum) is ultra pure deionized water starting from a pre-purified source. MilliQ water is the reagent grade water that is used for the preparation of all total mercury standards and reagents. MilliQ water must be checked weekly for total mercury concentrations. The total mercury concentration in MilliQ water must be < 0.20 ng/L.

6.2. Rinsing Water- MilliQ water is used for rinsing of analytical equipment (i.e. bubblers, frits). MilliQ water must be checked weekly for total mercury concentration. The total mercury concentration in MilliQ water must be < 1.00 ng/L.

6.3. Hydrochloric Acid- Hydrochloric acid (Baker reagent grade HCl) and must be pre-analyzed for total mercury concentration prior to use. Total mercury concentration in HCl must be < 5.00 ng/L.

6.4. Stannous Chloride (1.1%) Baker Reagent Grade

7.0 PROCEDURES

7.1. Preparation of Mercury Standard Solutions

7.1.1. Working standards are prepared by diluting 1000 ppm mercury (II) oxide (VWR, 0.1% Hg^{o} in dilute nitric acid (w:v)) with blank water (0.5% HCl in MQ, (v:v)) to final concentrations from 150 ppt to 5 ppb by weight. Method blanks are drawn from the same batch of blank water used in the preparation of working standards. PACS (2) and NBS 1944 are used as the Standard Reference Material (SRM) for sediments, and DORM (2) is the SRM used for tissues. SRM's are digested and prepared for analysis identically to samples. Aliquots are drawn from digested mercury samples and diluted with blank water until sample concentrations fall within working standard end member concentrations.

7.2. Mercury Analyzer Calibration Sequence

7.2.1. The following sequence will be used for all projects, except in cases where project specific additional requirements are stated. The sequence starts with a 5 point standard calibration curve which must cover greater than the entire range expected from the samples that are to be analyzed that day. If a sample is higher than the largest standard run, a higher standard or a smaller sample aliquot must be run.

7.2.2. Following the standard calibration curve, an initial calibration verification (ICV), and an initial calibration blank (ICB) are run followed by a minimum of 3 prep

blanks. A standard reference material (SRM) of the same matrix and digested in the same manner as the samples to be analyzed should follow the prep blanks. In the event that an SRM does not exist, a blank spike is prepared instead.

7.2.3. A maximum of 10 samples, including the above preparation blanks and SRM/blank spike, are run between ICV/ICB and the continuing calibration verification (CCV)/continuing calibration blank (CCB) pair. There may not be more than 10 samples between any CCV/CCB sets. In each batch of 20-25 samples, various matrix QC may be performed: Matrix Duplicate (MD); Matrix Spike (MS) and a matrix spike duplicate (MSD). The analysis day should end with a CCV/CCB.

7.3. ANALYZING SAMPLES

7.3.1. When running either water or solid samples, it is imperative to check the project sheets. Within these sheets the analyst will find a summary of all the information to run the samples properly. Determine if the samples are to be analyzed in a "High QA" format, the QC required by the project, as well as gathering any information about spiking levels and suggested aliquot size. Be aware that all samples considered to be High QA need to be run prior to any Standard QA samples that are to be analyzed on the same analytical day. Also, it is important for the analyst to note on the project sheets the date that the samples are analyzed and if any reruns are required. If at all possible, analyze the samples in the order that they appear on the COC's for water samples, or in the order that they were digested. The first samples analyzed should always be the blanks then the SRM, followed by actual samples. After every 10 samples including the Blanks and SRM, perform a CCV/CCB pair, and close out the run with a CCV/CCB.

Mercury Samples are analyzed by Atomic Spectroscopy using a Perkin Elmer Flow Injection Mercury System (FIMS-100) with the software application AA WinLab. A peristaltic pump set to 85 mL/min is used to transport various liquids through the system. The peristaltic pump, in conjunction with an autosampler (Perkin Elmer AS-90) draws a 4 mL aliquot of the sample solution into the mixing block. The reducing reagent (1.1 % Tin (II) chloride in 3 % HCl (v:v)) is pumped simultaneously mixing with the sample and a spontaneous reaction takes place, reducing the ionic mercury to metallic mercury. The carrier gas (liquid argon) then carries the mercury vapor to the gas/liquid separator at a flow rate of ~50 mL/min. The liquid is pumped to waste, the gas phase continues on to the FIMS-cell, which is the radiation beam of the spectrometer. The radiation source is a low pressure mercury lamp. The detector is a photocell with maximum sensitivity at 254 nm. The FIMS-cell has an inner diameter of 4 mm and an optical pathlength of 260mm. The cell is heated to 50 °C.

8.0 QUALITY ASSURANCE

8.1. The acceptable recoveries must be met in order to consider a data set valid. All data points noted on the analysis day's spreadsheet as invalid for know reasons may be discarded, if rerun during the same analysis day. In the event that the system becomes out of control during the analysis day, all results between valid QC data points shall still be considered valid.

Condition	Corrective Action
r for the calibration curve is less than 0.995	3-5, 10-13
Instrument blank is 10% greater than the IDL	3-5, 10-13
Continuing Calibration Check value differs by more than 20% from the most recent calibration.	1,3-6,10-13
QC Check Sample differs by > 30% form its expected value	1-6,10-13
The current method blank is greater than the MDL	2-6,10-13
The Percent Recovery of the current Spiked Method Blank falls outside the PQL control limits	2-6,10-13
The Relative Percent Difference of the current sample duplicate pair exceeds 30%	2-6,10-13
Percent Recovery for either of the current Matrix Spike (MS) and MS Duplicate (MSD) falls outside ±30% of the mean.	2-6,10-13
The sample matrix interfered with the ability to make an accurate determination	report with Data Qualifier "J"
The value determined is less than the MDL	= MDL with Data Qualifier "U"
The value determined is less than the PQL, but is greater than the MDL.	= PQL with Data Qualifier "I"

9.0 CORRECTIVE ACTION

Key to Corrective Actions in Table 8.1:

1. Re-analyze the samples of the analytical set
2. Re-process (re-extract or digest) the samples of the analytical set
3. Perform corrective maintenance
4. Re-calibrate instrument
5. Prepare fresh standards and re calibrate
6. Repeat the analysis of those samples analyzed since last acceptable check of this kind
7. Check calculations
8. Re-evaluate system
9. Qualify reported results
10. Determine cause of contamination/failure
11. Check water blank source

12. Clean Instrument
13. Check reagent sources

9.1. If insufficient amounts remain to repeat analysis for samples analyzed after the last acceptable CCV, use best professional judgment to estimate values. Bracket those samples from previous acceptance QC check, report the results with the Data Qualifier "J", and provide a narrative explanation.

10.0 EQUATIONS

10.1. Mercury in sediment and tissue

Concentration=(1/slope)*(peak height sample-peak height blank)*DF

% Recovery SRMs = (Observed concentration*100)/ certified concentration

Spike % recovery=
(ng Hg in spiked sample-ng Hg in unspiked sample)*100/ng Hg added

Equation assumes that sample weights of unspiked and spiked are equal.
Care should be taken to equalize these weights when aliquoting.
If the weights are unequal then the dry weight in the unspiked sample is used to calculate the ng in the sample portion of the spiked sample by the equation:
(conc. Hg unspiked*sample weight of spiked sample)=ng in unspiked sample

RPD duplicates = absolute value of$((X_1-X_2)/((X_2+X_1)/2))*100$

Appendix III E: 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 Probe thermometer: Fisher Part # 15-077-32

4.5 3-Ring Binder

4.6 Copy Machine

5.0 Reagents

5.1 Not Applicable

6.0 Sample Collection

6.1 Water Samples are collected according to EPA 1669, modified, according to analytical or project specific methods.

6.2 Tissue samples are collected according to Method MPSL-102a, or according to analytical or project specific methods.

6.3 Sediment samples are collected according to Method MPSL-102b, or according to analytical or project specific methods.

7.0 Procedure

7.1 Samples accompanied by a Chain of Custody Record (COC) are delivered to the laboratory from the field crew. Samples may be hand delivered or shipped via FedEx or another overnight shipping service provided the samples maintain the appropriate temperatures during shipment.

7.2 Cooler temperature is measured prior to the removal of any sample. The probe of the digital thermometer is placed amongst the samples. Temperature is allowed to equilibrate prior to recording on the COC and logbook. It is noted when samples were delivered by the field crew and placed directly into the refrigerator or freezer, rendering a cooler temperature unobtainable.

7.3 The COC is reviewed for preservation and requested handling of the samples.

7.4 A new page in the log book is used for each COC. Entries MUST include the following:

7.4.1 Date of entry.

7.4.2 Project Name and Number

7.4.3 Unique 9-digit Lab Number

7.4.3.1 The first four digits are the year in which the sample was received.

7.4.3.2 The second four digits are sequential numbers beginning with 0001. Each successive sample receives the next number.

7.4.3.3 A single letter is appended to each Lab Number to indicate the matrix type (-w = water, -s = sediment, -t = tissue, -c = chlorophyll a).

7.4.4 Date and time (if provided) of sample collection. Time shall be recorded using a 24-hour clock.

7.4.5 Sample Identification; station information taken directly from the COC

7.4.6 Analyte of suite of analytes requested for each sample.

7.4.7 At the end of the entry, the following are recorded:

7.4.7.1 Type- Specify the matrix of the samples. List all that apply.

7.4.7.2 Preservation/Storage- List for each matrix/analyte combination.

7.4.7.3 From- the name of the person last in possession of the samples (signed the COC)

7.4.7.4 Received by- the name of the person at the lab who first received the samples

7.4.7.5 Date and Time of sample receipt as well as cooler temperature upon arrival.

7.4.7.6 Checked by- the name of the person that verified the contents of the cooler with the COC and assigned the lab numbers.

7.4.7.7 Any comments pertaining to the samples (special instructions, anomalies, etc.).

7.5 Water samples are preserved according to the specific analytical methods (EPA 1630, 1631E and 1638). Preserved samples are given to the analysts along with copies of the COC and log-book entry.

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

7.7 At least one copy is made of each COC and log book entry. One copy MUST be kept in the COC binder. Other copies may be stored with the samples themselves, or given to the analyst.

7.8 All entries are entered and maintained in a MS Access database.

8.0 Analytical Procedure

8.1 Trace Metal tissue and sediment digestions are performed according to EPA 3052M, modified.

8.2 Mercury Only tissue and sediment digestion procedures can be found in Method # 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 MS Access database does not allow duplicate Lab Numbers

9.2 Each COC, along with a copy of the pertinent portion of the logbook, is retained for reference.

Appendix III E: 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, Teflon 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 Teflon 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 IV: DFG-WPCL SOPs

DFG-WPCL EPA Modifications and Laboratory Procedures			
Page	Procedure/Equipment	SOP number	Revision Date
A	Procedure for the Management of Samples Received for Checmical Analysis	SAMPMAN_Rev	Aug 2008
B	Determination of OC and PCB in Sediment and Tissue – Modifications to EPA 8081B and 8082	SO-TISS_SED	Mar 2005
C	Procedures for Disposal of Waste	HAZMAT_Rev4	Mar 2009
D	Protocol for Corrective Action Procedures	CORR_ACTION	Sept 2006

Appendix IV A: Procedure for the Management of Samples Received for Chemical Analysis

CDFG FISH AND WILDLIFE WATER POLLUTION CONTROL LABORATORY STANDARD OPERATING PROCEDURE FOR THE MANAGEMENT OF SAMPLES RECEIVED FOR CHEMICAL ANALYSIS

1.0 Scope and Application

This method describes the procedures to be followed for the receipt, handling, scheduling, storage, and disposal of samples received by the laboratory.

2.0 Summary of Method

The WPCL sample receiving area is located in the sample log-in room at the back of the main laboratory. All samples are immediately unpacked, checked for temperature, logged-in using the sample receipt log book, entered in Labworks (LIMS), labeled, checked for required preservation and preserved as necessary, checked for appropriate holding time limitations and properly stored (refrigerated or frozen). If samples are delivered frozen, they should be immediately transferred to the freezer after they are logged-in. DFG request for analysis and chain-of-custody records (Form FG 1000 Rev. 9/01) or chains of custody submitted with samples are completed and then given to the appropriate section leader for scheduling. After the analyses are completed, samples are stored until data review and reporting have been completed. Enforcement samples are held

Samples are then disposed using the evaporation pond (non-hazardous samples only) or logged into the hazardous waste storage area for scheduled pickup by a licensed hazardous waste contractor.

3.0 Sample Receipt

3.1 Samples are delivered to the Laboratory by DFG personnel, United Parcel Service (UPS), U.S. Postal Service, Federal Express, and by other commercial courier companies. Samples are shipped in Pollution Action Kit (PAK) boxes (non-hazardous samples only), hazardous materials shipping containers, and various sizes of ice chests.

3.2 Samples received by the Laboratory should be **immediately** taken by qualified Laboratory personnel to the sample log-in station located in the sample storage room at the back of the main laboratory building for unpacking. Samples should **not** be left in the sample receiving area or removed to office areas or laboratories. Samples addressed to individuals should not be left unattended if the addressee is not available.

3.3 All samples received by the Laboratory should be considered to be potentially hazardous and **caution** should be used when opening packages containing samples. Even non-hazardous samples can be a safety hazard to the person

unpacking the samples (ie. broken glass sample containers). All samples shipped in hazardous material shippers should be considered hazardous and should always be unpacked in a well ventilated area (under a fume extractor) or in a fume hood. Personnel unpacking hazardous samples should wear appropriate protective clothing, which at a minimum would include:

safety glasses
nitrile gloves
laboratory coat or apron
closed toe shoes

3.4 Cut packaging tape with a knife or scalpel. Open package or ice chest and remove paperwork which should be on top. Open laboratory log book and record the letter "L" followed by the next consecutive number followed by the last two numbers of the current year. Below the laboratory "L" number, write the date and below the date write your initials. The "L" number will be used for the entire set of samples. Record this number on the line labeled laboratory number in the upper right hand corner of the Form FG 1000 Rev. 9/01, on the chain-of-custody form (if applicable), and any other paperwork that accompanied the samples. All writing must be in ink (preferably ball point pen).

3.5 Remove samples from the shipping container and line them up on the counter. Check each sample container for cracks or breakage. Make sure that each sample is labeled. Labworks LIMS can be used to print labels for sample containers. See 3.14 for instructions. Labels may be handwritten by writing the laboratory number on each container with waterproof marker followed by consecutive numbers. For example:

sample 1 = L-345-07-01
sample 2 = L-345-07-02

3.6 Record the following information about the samples in the log book next to the laboratory number:

number of containers
type of sample (eg. sediment, water, oil, etc.)
condition of sample (broken, leaking, etc.) if necessary
where samples were collected
person requesting analyses and DFG region or other agency name
type of analysis
Index-PCA code, if given by collector

3.7 Check the Form FG 1000 Rev. 9/01 for sample descriptions (Identification/Location), if this is not filled out, do so using the information on the sample labels. Check the Form FG 1000 Rev. 9/01 for analysis requested. If instructions are unclear, contact the person who collected the samples.

3.8 If any problems are found with the samples when they are received (eg. broken containers, missing samples, samples have been shipped that are not recorded on the chain-of-custody documents), notify the individual who shipped the samples immediately and inform them of the problem.

3.9 Sign, print name and date the Form FG 1000 Rev. 9/01 and chain of custody card (if used) next to "received by". Give the submitter the goldenrod copy. Put the pink copy in the binder at the log-in area. The pink copy is used to enter all sample information into the laboratory information management system (LIMS)..

3.10 Check the holding time/sample preservation table for the analysis requested and record the holding time expiration on the Form FG 1000 Rev. 9/01 if applicable. If preservation is required, do so immediately and record the type of preservative, date preserved, and initial the Form FG 1000 Rev. 9/01. Preservation of inorganics samples is also entered in a separate log book located in the inorganic lab (this is done by inorganic lab staff). Each container should receive a label indicating that the sample was preserved and the type of preservative.

3.11 Determine where the samples will be stored and record refrigerator or freezer number in the space provided on the Form FG 1000 Rev 09/01. Keep samples together as a set. Samples should be stored as follows:

VOA samples only – Elmo (front lab, left side)
petroleum samples – Grover (front lab, left side)
inorganic samples – WPCL R5, R7, WPCL R2 (machine room), TSM F2 (sample storage room), walk in R2
ambient monitoring samples – TSM R3 (left side), TSM F2, F4, WPCL F1 (sample storage room), walk in F1
pesticides (F&W Loss) – WPCL F3 (sample storage room), WPCL R1, R2, R3, R4 (back lab), walk in R3, walk in F1

F = freezer; R = refrigerator

Enforcement and regulatory samples must be stored in a locked refrigerator/freezer.

3.12 Give all paperwork to the appropriate lead analyst:

Petroleum/semi-VOA/VOA - Bob Todd
Inorganic/hatchery monitoring - Patty Bucknell
Pesticide (F&W Loss) investigations - Abdou Mekebri
Pesticides (pesticides contracts) - Abdou Mekebri
Tissue/sediment monitoring-(PCBs, PBDEs, OC pesticides)-Kathleen Regalado

3.13 If samples were received in a PAK, photocopy the Form 1000 and write "PAK" in large letters on the front of the photocopy or if a Form FG 1000 Rev 09/01 was used, check the box labeled "PAK Requested" if not already done. Give the photocopy to Bob Todd so that a new PAK will be sent out to replace the one received.

3.14. Using Labworks for log-in samples

1. Go into Multilog
2. Load the project pre-log in group and enter site location, collection date, collection time and checking analytes, matrix, report address against COC
3. Labels can be printed from Labworks with L#, site location, analyses, collection date and time
   - 3a. Go to Labworks explore and pick L #'s for the labels
   - 3b. Highlight the accession for labels. Choose LABEL 30251.CEF and click OK
   - 3c. The program is automatically connected to access. Pick report feature and click LABEL-30251-inorg
4. Place the labels on samples

4.0 Scheduling

4.1 Laboratory analyses are scheduled by priority. Priorities are ranked as follows:

1-Enforcement samples with regulatory holding time
2-Spill and/or wildlife loss in progress
3-Routine samples with regulatory holding time
4-Enforcement samples with completion date requested
5-Routine samples with completion date requested
6-Enforcement samples with no holding time
7-Routine samples with no holding time

4.2 Samples should be completed as soon as possible after receipt. If a delay is anticipated for the completion of an analysis, the person requesting the analysis should be advised of the delay.

5.0 Sample Storage

5.1 Samples remain refrigerated or frozen until they are needed for analysis. Samples are removed from storage for analysis and then are returned to refrigerated storage. Tissue samples are always returned to a freezer after they are analyzed. Samples remain refrigerated/frozen until results are reported.

5.2 Samples that will not spoil may be moved to locked non-refrigerated storage after the report has been completed. Retain chain-of-custody cards or the original copy of the Form FG 1000 Rev. 09/01 for all stored samples.

5.3 Samples retained by the laboratory for six months that are not required as physical evidence should be disposed using a hazardous materials disposal contractor. The person submitting the samples should be contacted and told the samples will be disposed unless a request is made to store the samples at the laboratory for a longer period of time. When samples are disposed, the word "Disposed" and the disposal date are recorded next to the logbook entry (lab accession number) for that sample with the person's initials that authorized the disposal. Inorganics samples are also entered in separate disposal log book located in the inorganic lab (this is done by inorganic lab staff).

QA Officer Approval:_____ Date: _____

SOP Final Approval:_____ Date: _____

Appendix IV B: SO-TISS Determination of OC and PCB in Sediment and Tissue (Modifications to EPA 8081B and 8082)

ANALYSIS OF EXTRACTABLE SYNTHETIC ORGANIC COMPOUNDS IN TISSUE AND SEDIMENT
(Organochlorine Pesticides, Polychlorinated Biphenyls and Polybrominated Diphenyl Ethers)

1.0 Scope and Application

1.1 This method describes the sample preparation using an automated extraction system for the determination of trace residue levels of a selected list of organochlorine (OCs) pesticides, polychlorinated biphenyls (PCBs) and polybrominated diphenyl ethers (PBDEs) in fish and shellfish tissues and sediments. Dual column gas chromatography with dual electron capture detectors (GC-ECD) and/or gas chromatography with triple quadrupole mass spectrometry (GC-MSMS) are used to analyze OC pesticides, PCBs and PBDEs. Table 1 lists the target OC pesticide compounds currently analyzed with their method detection limits and reporting limits. Table 2 lists the PCB congeners and Aroclor mixtures analyzed with their reporting limits. Table 3 lists the PBDE congeners analyzed with their method detection limits and reporting limits.

1.2 These procedures are applicable when low parts per billion analyses are required to monitor differences between burdens in organisms and sediment concentrations from relatively uncontaminated reference areas and contaminated areas. In addition, the procedures are applicable when low detection limits are required for the estimation of potential health effects of bioaccumulated substances.

Table 1. Organochlorine Compounds Analyzed and their Minimum Detection Limits (MDL) and Reporting Limits (RL) in Tissue (ng/g, wet wt.) and Sediment (ng/g dry weight), based on 50 % moisture.

	Tissue		Sediment	
	MDL, ng/g wet wt.	RL, ng/g wet wt.	MDL, ng/g dry wt.	RL, ng/g dry wt.
aldrin	0.414	1.00	0.800	2.00
chlordane, cis	0.400	1.00	0.800	2.00
chlordane, trans	0.450	1.00	0.900	2.00
chlorpyrifos	0.204	1.00	0.400	2.00
dacthal	0.096	1.00	0.200	2.00
DDD, o,p'	0.096	1.00	0.200	2.00
DDD, p,p'	0.124	1.00	0.250	2.00
DDE, o,p'	0.178	2.00	0.400	4.00
DDE, p,p'	0.480	2.00	1.00	4.00
DDMU, p,p'	0.108	3.00	0.200	6.00
DDT, o,p'	0.216	3.00	0.400	6.00
DDT, p,p'	0.156	5.00	0.300	10.0
diazinon	4.80	20.0	10.0	40.0
dieldrin	0.432	0.500	1.00	1.00
endosulfan I	0.560	2.00	1.00	4.00
endosulfan II	0.682	5.00	1.40	10.0
endosulfan sulfate	0.546	5.00	1.00	10.0
endrin	0.180	2.00	0.400	4.00
HCH, alpha	0.262	0.500	0.500	1.00
HCH, beta	0.210	1.00	0.400	2.00
HCH, gamma	0.144	0.500	0.300	1.00
heptachlor	0.356	1.00	0.700	2.00
heptachlor epoxide	0.246	1.00	0.500	2.00
hexachlorobenzene	0.346	0.692	0.700	1.40
methoxychlor	0.146	3.00	0.300	6.00
mirex	0.300	1.50	0.600	3.00
nonachlor, cis	0.308	1.00	0.600	2.00
nonachlor, trans	0.194	1.00	0.400	2.00
oxadiazon	0.544	1.00	1.00	2.00
oxychlordane	0.474	1.00	1.00	2.00
parathion, ethyl	0.524	2.00	1.00	4.00
parathion, methyl	0.756	4.00	1.50	8.00
tedion	1.07	2.00	2.00	4.00
DBOB(surrogate)	NA	NA	NA	NA
DBCE(surrogate)	NA	NA	NA	NA
DDD*deuterated (surrogate)	NA	NA	NA	NA

Table 2. PCB Congeners and Aroclor mixtures Analyzed and their Reporting Limits (RL) in Tissue (ng/g, wet weight) and Sediment (ng/g, dry weight).

NIST PCB Congeners:	
PCB Congener 8	PCB Congener 128
PCB Congener 18	PCB Congener 138
PCB Congener 28	PCB Congener 153
PCB Congener 44	PCB Congener 170
PCB Congener 52	PCB Congener 180
PCB Congener 66	PCB Congener 187
PCB Congener 87	PCB Congener 195
PCB Congener 101	PCB Congener 206
PCB Congener 105	PCB Congener 209
PCB Congener 118	PCB Congener 209 C^{13}(surrogate)

Additional PCB Congeners:	
PCB Congener 27	PCB Congener 141
PCB Congener 29	PCB Congener 146
PCB Congener 31	PCB Congener 149
PCB Congener 33	PCB Congener 151
PCB Congener 49	PCB Congener 156
PCB Congener 56	PCB Congener 157
PCB Congener 60	PCB Congener 158
PCB Congener 64	PCB Congener 169
PCB Congener 70	PCB Congener 174
PCB Congener 74	PCB Congener 177
PCB Congener 77	PCB Congener 183
PCB Congener 95	PCB Congener 189
PCB Congener 97	PCB Congener 194
PCB Congener 99	PCB Congener 198_199
PCB Congener 110	PCB Congener 200
PCB Congener 114	PCB Congener 201
PCB Congener 126	PCB Congener 203
	PCB Congener 137

All individual PCB Congener reporting limits (RL) are 0.2 ng/g (wet weight) or 0.4 ng/g (dry weight, based on 50 % moisture). Estimated Aroclor concentrations calculated from the congener concentrations have the following RLs:

Aroclors:	RL ng/g (wet wt.)	RL ng/g (dry wt.)
Aroclor 1248	25	50
Aroclor 1254	10	20
Aroclor 1260	10	20

Table 3. Polybrominated Diphenyl Ethers (PBDEs) and their Minimum Detection Limits (MDL) and Reporting Limits (RL) in Tissue (ng/g, wet wt.) and Sediment (ng/g, dry wt., based on 50 % moisture.)

	Tissue		Sediment	
	MDL, ng/g wet wt.	RL, ng/g wet wt.	MDL, ng/g dry wt.	RL, ng/g dry wt.
BDE 17	0.139	0.600	0.278	1.20
BDE 28	0.148	0.600	0.296	1.20
BDE 47	0.196	0.800	0.391	1.60
BDE 66	0.135	0.600	0.269	1.20
BDE 100	0.157	0.600	0.314	1.20
BDE 99	0.197	0.800	0.394	1.60
BDE 85	0.177	0.800	0.354	1.60
BDE 154	0.165	0.600	0.329	1.20
BDE 153	0.185	0.800	0.370	1.60
BDE 138	0.200	0.800	0.400	1.60
BDE 183	0.297	1.20	0.594	2.40
BDE 190	0.437	1.80	0.874	3.60
BDE 209	1.00	10.0	2.00	20.0

2.0 Summary of Method

2.1 Sets of 10-18 homogenized tissue or sediment samples are scheduled for extraction by the project lead chemist. Extraction method employed was developed and validated by the Water Pollution Control Laboratory (WPCL) and is a modification of EPA Method 3545A Pressurized Fluid Extraction (PFE). Extract cleanup and partitioning methods are modifications of EPA Methods 3640A Gel Permeation Cleanup and 3620C Florisil Cleanup and the multi-residue methods for fatty and non-fatty foods described in the U.S. Food and Drug Administration, Pesticide Analytical Manual, Vol. 1, 3rd Edition 1994, Chapter 3, Multi-residue Methods, Section 303-C1.

Homogenized tissue or sediment samples are removed from the freezer and allowed to thaw. A separate extraction bench sheet is initiated for each set of samples which are distinguished by project, sample matrix type and analysis type.

2.2 A 1-5 g (tissue or sediment homogenate) sample is weighed into a pre-weighed aluminum planchet and placed in a 70°C oven for 48 hours to determine moisture content. A 10 g sample is mixed using a clean glass stirring rod with approximately
7 g of pre-extracted Hydromatrix® in a 250 mL Trace Clean Wide Mouth Jar until the mixture is free flowing. The mixture is then poured into a 33 mL stainless steel Dionex Accelerated Solvent Extractor (ASE 200) extractor cell and packed by tamping the mixture. A solution containing pesticide, PCB and

PBDE surrogate compounds is added to the cell and the cap is screwed onto the cell. The extractor cells (maximum of 24) are placed on the ASE 200 autosampler rack and the samples are extracted twice with a 50/50 mixture of acetone/dichloromethane (DCM) using heat and pressure. The extracts are automatically collected in two 60 mL VOA vials.

2.3 The combined extracts (~100 mL) are dried using sodium sulfate, evaporated to approximately 1.0 mL using Kuderna-Danish (K-D) glassware equipped with 3-ball Snyder columns and micro-Snyder apparatus and diluted to 10 mL using DCM. The extracts are then filtered through a 0.45 µm syringe filter into J_2 Scientific AccuPrep 170 (GPC) autosampler tubes. If the lipid content needs to be determined, two milliliters each of the filtered extracts are removed and placed in a pre-weighed aluminum planchet.

2.4 The GPC autosampler tubes are then placed on the GPC autosampler for initial sample cleanup by gel permeation (size exclusion) chromatography.

2.5 The cleaned-up extracts are evaporated using K-D apparatus and solvent exchanged into petroleum ether. The extracts are then fractionated using 5 grams of Florisil® in a 11 mm x 300 mm column with a 250 mL reservoir. The Florisil® columns prepared for tissue samples are eluted with 6% diethyl ether/PE (Fraction 1), 15% diethyl ether/PE (Fraction 2), and 50% diethyl ether/PE (Fraction 3). Florisil® columns prepared for sediment samples are eluted with 6% diethyl ether/PE (Fraction 1) and 50% diethyl ether/PE (Fraction 2).The fractions are concentrated to an appropriate volume using K-D/micro K-D apparatus prior to analysis by dual column high resolution gas chromatography and/or GC-MSMS. The distribution of synthetic organic compounds in the fractions is listed in Table 4.

Table 4. Distribution of Synthetic Organic Compounds Among the Three Fractions of a Standard Florisil® Column.

6% Fraction 1/	**15% Fraction** 2/	**50% Fraction** 3/
aldrin	dacthal	endosulfan II 5/
chlordane (cis-)	DBCE*	endosulfan sulfate
chlordane (trans-)	dieldrin	
DBOB*	endosulfan I 4/	
DDE, o,p'	endosulfan II 5/	
DDE, p,p'	endrin	
DDD, o,p'	oxadiazon	
DDD, p,p'/DDD-d10*,p,p'	tetradifon	
DDMU, p,p'		
DDT, o,p'		
DDT, p,p'		
endosulfan I 4/		
heptachlor		
heptachlor epoxide		
hexachlorobenzene		
HCH-alpha		
HCH-beta		
HCH-gamma		
methoxychlor		
nonachlor (cis-)		
nonachlor (trans-)		
oxychlordane		
polybrominated diphenyl ethers (PBDEs)		
polychlorinated biphenyls (PCBs)/PCB 209*(C^{13})		
toxaphene		

* surrogate
1/ 6% ethyl ether in petroleum ether (analysis by GC-MSMS)
2/ 15% ethyl ether in petroleum ether (analysis by GC-ECD)
3/ 50% ethyl ether in petroleum ether (analysis by GC-ECD).
4/ In both 6% and 15% fractions.
5/ In both 15% and 50% fractions.

3.0 Interferences

3.1 Solvents, reagents, glassware, and other sample processing hardware may cause GC artifacts and/or elevated baselines, resulting in the misinterpretation of chromatograms. 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. Specific selection of reagents and purification of solvents by distillation in all-glass systems are required. High-purity, distilled-in-glass solvents are commercially available.

An effective way of cleaning laboratory glassware is by rinsing with polar and non-polar solvents before use. The cleaning procedure used must be tested by analyzing procedural blanks prior to analyzing samples.

3.2 Phthalates are common laboratory contaminants that are used widely as plasticizers. Sources of phthalate contamination include plastic lab-ware, plastic tubing, plastic gloves, plastic coated glassware clamps, and have been found as a contaminant in Na_2SO_4. Polytetrafluoroethylene (PTFE) can be used instead of polypropylene or polyethylene to minimize this potential source of contamination. However, use of PTFE lab-ware will not necessarily preclude all phthalate contamination.

3.3 Interferences co-extracted from tissue and sediment samples limit the method detection and quantitation limits. For this reason, sample extract cleanup is necessary to yield reproducible and reliable analyses of low level contaminants.

4.0 Apparatus and Materials

4.1 Wide mouth, borosilicate glass, pre-cleaned and certified, 250 mL, Qorpak or equivalent.

4.2 Chromatographic Column - (300 mm x 11 mm) borosilicate glass chromatography column with 250 mL reservoir and Teflon stopcock.

4.3 Glass wool, Pyrex - solvent washed prior to use.

4.4 Kuderna-Danish (K-D) Apparatus

4.4.1 Concentrator tube - 10 mL, graduate (Kontes K0570050-1025, or equivalent). A ground stopper, 19/22 joint, is used to prevent evaporation of extracts.

4.4.2 Evaporation flask - 500 mL (Kontes K-570050-0500, or equivalent), attached to concentrator tube with blue clamp (Kontes K-662750-0012).

4.4.3 Snyder column - three ball (Kontes K-503000-0121, or equivalent).

4.4.4 Micro-Snyder column - (Kontes VWR KT569261-0319 or equivalent).

4.4.5 Boiling stones, Chemware® Ultra-Pure PTFE, extracted with acetone and petroleum ether. Note that boiling chips can be a significant source of contamination if not properly cleaned.

4.5 Water bath, Organomation Assoc. Inc.(OA-SYS/S-EVAP-KD), 115 V, thermostatically controlled with stainless steel cover to fit 5 K-D apparatus, installed in a fume hood. Water bath is equipped with solvent recovery system.

4.6 Extractor, automated, Dionex Accelerated Solvent Extractor (ASE 200), Dionex P/N 047046.

4.6.1 Extraction Cells, 33 mL, Dionex P/N 049562

4.6.2 Filters, cellulose for ASE extraction cells, Dionex P/N 049458.

4.6.3 VOA Vials, 60 mL, pre-cleaned and certified.

4.7 Sample vials - glass, 2.5 mL with PTFE-lined screw cap.

4.8 Analytical balance - capable of weighing 0.1 mg.

4.9 Drying oven.

4.10 Balance - capable of 100 g to the nearest 0.01 g.

4.11 Disposable Pasteur Pipettes - (rinsed with solvents before use).

4.12 Aluminum dishes for moisture and lipid determination.

4.13 Desiccator with indicating desiccant.

4.14 Glass funnel, 75 mm.

4.15 Graduated cylinder, 250 mL and 100 mL.

4.17 Culture tubes, 13 x 100mm and 16 x 100 mm, with PTFE lined cap.

4.18 Centrifuge tubes, 15 mL, graduated to 0.1 mL and calibrated to 1.0 mL.

4.19 Gas chromatographs (GC) (3): Hewlett-Packard HP 6890 plus, equipped with dual micro-ECD. All are equipped with split-splitless injector with EPC and autosampler.

4.20 GC Capillary columns, 60 meter DB5 and 60 meter DB17MS (J&W Scientific) (0.25 mm I.D. and 25 µm film thickness) connected to a single injection port using a "Y" press fit connector.

4.21 GC Data System, Hewlett-Packard, to collect and record GC data, generate reports, and compute and record response factors for multi-level calibrations.

Data system should be capable of calibrating a method using a minimum of 5 concentrations of analytical standards.

4.22 Gas chromatograph-mass spectrometer (triple quadrupole), Varian Model 1200L with Varian Model 3800 gas chromatograph, split-splitless injector with EPC and Combi-Pal autosampler.

4.23 Homogenizer, Bucchi Model B-400 (Brinkman P/N 16-07-200-1) or equivalent equipped with titanium knife assembly (Brinkman P/N 16-07-222-2) and glass sample vessel (Brinkman P/N 16-07-245-1).

4.24 Homogenizer, Brinkman Polytron or equivalent equipped Teflon and titanium generator assembly (for homogenization of small sample amounts).

4.25 Gel Permeation (size exclusion) Chromatograph, automated, J2 Scientific AccuPrep 170, equipped with 70 g S-X3 BioBeads J_2 Scientific P/N C0070G (100% DCM).

5.0 Reagents

5.1 Petroleum ether (PE), Burdick and Jackson, distilled in glass and pesticide residue or HRGC grade or equivalent.

5.2 Acetone. (Same as above).

5.3 Iso-Octane. (Same as above).

5.4 Diethyl ether preserved with 2% ethanol.(Same as above).

5.5 Dichloromethane (DCM). (Same as above).

5.6 Chem Elut-Hydromatrix®, Varian P/N 0019-8003. Pre-extracted on ASE-200 with acetone/DCM prior to use.

5.7 Sodium sulfate. Anhydrous granular reagent grade, rinsed with PE prior to use.

5.8 Florisil®, 60/100 mesh, PR grade, U.S. Silica.

5.9 Nitrogen, pre-purified grade (99.9999%) or better (used for ASE and GPC).

5.10 Nitrogen, ultra-pure (99.99999%) for ECD makeup.

5.11 Helium, ultra-pure (99.99999%) for GC carrier gas.

5.12 Air, compressed, breathing quality, for ASE pneumatics.

5.13 OC/PCB/PBDE Surrogate Mix containing: 40 ppb of deuterated p,p'-DDD-d10, PCB 209(C^{13}), and dibutylchlorendate (DBCE).

5.14 Standard Reference Material (SRM), National Institute of Standards and Technology (NIST): SRM 1588b (Organics in Cod Liver Oil) and SRM 1944 (New York/New Jersey Waterway sediment).

CAUTION

The toxicity or carcinogenicity of each compound or reagent used in this method has not been precisely determined. However, each chemical compound should be treated as a potential health hazard. Exposure to these compounds should be reduced to the lowest possible level. The laboratory is responsible for maintaining a current awareness file of OSHA regulations regarding the safe handling of the chemicals specified in this method. A reference file of data handling Material Safety Data Sheets should also be made available to all personnel involved in these analyses.

6.0 Sample Collection, Preparation, and Storage

6.1 In the field, sources of contamination include sampling gear, grease from ship winches or cables, ship and/or motor vehicle engine exhaust, dust, and ice used for cooling. Efforts should be made to minimize handling and to avoid sources of contamination. This will usually require that resection (i.e., surgical removal) of tissue be performed in a controlled environment (e.g., a laboratory). The samples should be double wrapped in aluminum foil and immediately frozen with dry ice in a covered ice chest. Ice should be in water tight plastic bags for transporting live shellfish.

6.2 To avoid cross-contamination, all equipment used in sample handling should be thoroughly cleaned before each sample is processed. All instruments must be 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 tap water, rinsed with a high-purity acetone, and finally rinsed with Type II water.

6.3 Resection should be carried out by or under the supervision of a competent biologist. Each organism should be handled with clean high carbon steel, titanium, quartz, or Teflon instruments (except for external surfaces). The specimens should come into contact with pre-cleaned glass surfaces only. Polypropylene and polyethylene surfaces are a potential source of contamination and should not be used. To control contamination when resecting tissue, separate sets of utensils should be used for removing outer

tissue and for resecting tissue for analysis. For fish samples, special care must be taken to avoid contaminating target tissue (especially muscle) with slime and/or adhering sediment from the fish interior (skin) during resection. The incision "troughs" are subject to such contamination; thus, they should not be included in the sample. In case of muscle, a "core" of tissue is taken from within the area bordered by the incision troughs, without contacting them. Unless specifically sought as a sample, the dark muscle tissue that may exist in the vicinity of the lateral line should not be mixed with the light muscle tissue that constitutes the rest of the muscle tissue mass.

6.4 The resected tissue sample should be placed in a clean glass or PTFE container which has been washed with detergent, rinsed twice with tap water, rinsed once with distilled water, rinsed with acetone, and, finally, rinsed with high-purity petroleum ether.

6.5 The U.S. EPA has published a guidance document containing specific recommendations regarding holding times and temperatures for tissue samples to be analyzed for semi-volatile organic compounds. The following holding conditions should be observed. Tissue samples should be maintained at $\leq$-20° C and analyzed as soon as possible, but within 12 months of sample receipt.

6.6 Sediment samples may be refrigerated at 4°C for up to 14-days maximum or must be stored frozen at minus (-) 20°C for up to 12 months maximum.

7.0 Sample Extraction

7.1 Remove homogenized tissue or sediment samples from freezer and allow to thaw. Prior to extraction, the tissue samples are homogenized using a Bucchi B-400 mixer equipped with a titanium knife assembly or for small samples a Brinkman Polytron® equipped with a titanium and Teflon generator. Decant any excess water from the sediment samples prior to thoroughly mixing by hand using a clean glass rod or may be homogenized using a Polytron homogenizer equipped with stainless steel generator equipped with Teflon bearings. Sample sets of 10-18 should be extracted when possible. The ASE-200 extractor will extract 24 cells. Be sure to reserve enough cells for method blanks, matrix spikes, and laboratory control spikes.

7.2 A separate extraction bench sheet is initiated for each project, sample matrix type, and analysis type. Several bench sheets may be used for an extraction set.

7.3 Prepare a glass rod or Teflon spatula for each sample to be weighed by rinsing 3 times with petroleum ether using a Teflon wash bottle.

7.4 Label 60 mL VOA vials for the collection of the sample extract. The labels must be placed between 1.5" and 3" from the top of the VOA cap; if they are placed outside of this area, they will interfere with the ASE optical sensor. Use two VOA vials for each sample. Label the first VOA vial with the ASE position number, bench sheet number and the sample name. Label the second VOA vial the same but add "RE" to distinguish between the two vials. Label and weigh aluminum planchets for lipid and moisture determinations (write sample ID on the bottom of planchets using a ball point pen).

7.5 Tare a 250 mL glass jar. Using a clean (solvent rinsed) glass rod, stir the tissue or sediment so that the mixture is homogeneous. Weigh 10 g of sample into the jar, record the weight on the bench sheet, and add the twice-extracted Hydromatrix® from one ASE cell. Stir the mixture thoroughly and go on to the next sample. After approximately 15 minutes stir the sample again. Repeat this at 15 minute intervals two more times or until the sample mixture is free flowing.

7.6 Weigh 1-5 g of additional sample into a pre-weighed and tared aluminum planchet for % moisture analysis. Place planchets in 70°C oven for 48 hours and re-weigh dry weight.

7.7 Place a pre-rinsed powder funnel on top of a 33 mL ASE cell containing a pre-extracted cellulose filter *(the filter is the one that was used to pre-extract the Hydromatrix®)*.

7.8 Pour the tissue or sediment/Hydromatrix® mixture through the powder funnel back into the extraction cell that the Hydromatrix® was poured from. Tap the cell against the counter top to settle the contents. The mixture will fill the cell and it may be necessary to pack it slightly using the glass rod and the end of the powder funnel. The cells used for the method blank and laboratory control spike and its duplicate *(if used)* will contain only Hydromatrix®.

7.9 All of the extraction cells are spiked with the OC/PCB/PBDE pesticide surrogate standard. Spike each cell with exactly 0.5 mL of the appropriate surrogate solution. Surrogate spikes must be witnessed, recorded and dated on the extraction bench sheet.

7.10 The extraction cells used for the matrix spike (MS) and duplicate matrix spike (MSD) and laboratory control spike (LCS) and its duplicate (LCSD) (if used) are spiked with exactly 0.5 mL of the OC/PCB/PBDE matrix spike solution (40 ng/mL). A separate MS/MSD and LCS/LCSD (if used) is required for each class of compounds being analyzed. Matrix spikes must be witnessed, recorded and dated on the extraction bench sheet.

7.11 The extraction cells are capped *(Firmly tightened but do not overtighten)* and placed on the ASE 200 carrousel. The first set of labeled VOA collection vials

are placed on the ASE 200 collection carrousel with the position numbers corresponding to the position numbers of the extraction cells. Make sure that the solvent reservoir contains enough solvent for the extraction.

7.12 Samples are extracted with acetone/methylene chloride (DCM) 50:50 using the following conditions:

Pre-heat	0 min.
Heat	5 min.
Static	5 min.
Flush	60%
Purge	300 sec.
Cycles	1
Pressure	1500 psi
Temp	100 °C
Sol A Other	100%

7.13 After the initial extraction is complete, remove full VOA vials and place in a Wheaton rack. Place the second set of collection VOA vials labeled "RE" on the ASE carrousel. Check each of the extraction cells to make sure that the caps are *(firmly tightened)* as they tend to loosen with the first extraction. Make sure that the replacement vials are in the correct order. Make sure that the solvent reservoir contains enough solvent for the re-extraction. Re-start the ASE-200.

7.14 When extraction is completed, place VOA vials in a Wheaton rack with the "RE" vials next to the vials from the first extraction. The extracts should be re-capped with solid green caps (Qorpak) and placed in a refrigerator for storage until they are removed for the GPC cleanup procedure.

8.0 Gel Permeation Chromatography

IMPORTANT: ***All glassware, glass wool, and sodium sulfate must be triple-rinsed with petroleum ether before they are used for this procedure.***

8.1 Remove VOA vials containing the sample extracts from the refrigerator. Make sure the vials are capped with the green Qorpak caps. Allow them to sit out until they are at room temperature.

8.2 Set up and label pre-cleaned K-D flasks (4-6) with concentrator tubes attached on ring stands in the fume hood. Place a funnel containing a plug of pre-cleaned glass wool in the bottom of the funnel and place the funnel in the top of the K-D flask. Add about two inches of pre-rinsed sodium sulfate to the funnel. Make sure that the level of the sodium sulfate is uniform across the

funnel to prevent any possible splashing out.

8.3 Pour sample extracts from the VOA vials through sodium sulfate into the K-D flask. Add about 10 mL of DCM to the VOA vial, cap and shake and add this rinse to the sodium sulfate. Repeat with another 10 mL DCM rinse. Rinse the sodium sulfate with an additional portion of DCM (~50 mL) by pouring from a clean and rinsed 400 mL beaker. After the solvent has completely drained through the sodium sulfate add one more additional rinse of DCM (~50 mL) from the beaker of clean DCM. Allow the DCM to completely drain through the sodium sulfate (~3-5 minutes).

8.4 Add 0.5 mL Iso-Octane using a macro-pipetter and a solvent rinsed boiling chip to each K-D flask. Place a Snyder column on the K-D flask, clamp with a green clamp and place the flask on the hot water bath set at 80-82°C. Drop down the inverted Hopkins condenser from the solvent recovery system and attach it to the top of the Snyder column. Turn the water supply on to the solvent recovery system until the water flow is between 1500-2000 cc/min. Evaporate the solvent until the apparent volume is 2-5 mL. Remove the inverted Hopkins condenser and secure using the set clamps so that it is out of the way. At this point there should be between 2-5 mL visible in the concentrator tube while the K-D apparatus is still on the hot water bath and 10 mL or less of the solvent remaining after the K-D flask is removed from the hot water bath and the solvent drains from the Snyder column. Dry off the water using a WyPall X60 towel to remove any water from around the ground glass union of the concentrator tube and the K-D flask to prevent any of it from entering the concentrator tube upon removal.

8.5 After the K-D apparatus has cooled and all of the solvent has drained from the Snyder column, remove the Snyder column, label the concentrator tube and then remove the concentrator tube from the flask and place the tube in a test tube rack and cover with pre-rinsed aluminum foil. Rinse the Snyder column with petroleum ether and place back in the column rack for storage. After all of the flasks have been removed from the hot water bath, repeat steps 2-5 for the remaining samples extracted with this set.

8.6 Add a new micro-boiling stone and place a clean micro-Snyder column on the concentrator tube with a blue clamp and place in a 400 mL beaker containing hot water heated to approximately 75°C on a hot plate. If the solvent does not begin to boil, remove the tube from the bath immediately, allow it to cool slightly, add a new micro boiling stone to prevent it from bumping and place it back in the bath. Evaporate the solvent until only 1.0 mL remains in the concentrator tube. Four or five tubes can be evaporated at one time.

8.7 When the solvent has been evaporated to 1.0 mL remove the tube from the bath and allow it to cool in a test tube rack. Remove the micro-Snyder column and add DCM to the concentrator tube to reach a final volume of 10.0 mL.

8.8 Gelman filter (0.45 µm) the sample into a 12 mL culture tube. Using a volumetric pipette remove 2.0 mL of the filtered sample and place it in a pre-weighed aluminum planchet if lipid determination is needed. Cap the culture tube with the Teflon-insert style caps. Mark the bottom of the meniscus with a pen in case of evaporation before clean-up on GPC.

8.9 All samples are cleaned using a J_2 Scientific GPC (Autoinject 110, AccuPrep 170, DFW-20 Fixed Wavelength Detector, 1" ID glass column with 70g Bio-Beads SX-3 in 100% DCM)

8.9.1 From the desktop double click on the AccuPrep.exe shortcut to open the program. Click on the Use Injector button and allow the instrument time to initialize. Activate the pump by using the top left hand button. A solvent Control Pump window will open up. Click on the Apply Defaults button and then OK on the Selected Pressure Limit 30 psi. The pump should audibly be heard coming on and the green light should show that the system is on line and status flowing. Make sure that the bottle of clean DCM is full and the waste bottle is empty. Allow the system to pump for about 5 minutes before switching the column in-line (gray button next to Column that has 'Put in line' on it). The pressure will be observed to normally go up to the 12-16 psi range. Turn the power on to the detector to allow it at least 30 minutes of time to warm up before use. Because the scale is auto-adjusted in the software now it is no longer necessary to manually adjust the range on the unit itself.

8.9.2 While the system is equilibrating, the sequence can be entered. Click on the Seq button next to the Pump button. An 'Editing new sequence' window will pop up. This gives a view of the instrument which clearly shows the sample tray locations and the corresponding sample collection locations. By clicking on the sample tray position, a new window 'Adding sample at tray position #' will pop up. This allows information to be included about each specific sample. Sample position 1 will always be a calibration standard (CLP-340) which is run prior to any sequence of runs to verify instrument integrity. In the Sample ID field just type in 'CLP-340'. In the Descrip (optional), information pertaining to the project, laboratory control number, bench sheet number and date are typically added. The Method File needs to be changed to 'ZGPC Calib' for only this sample and in the Sample Type field the 'Calibration' type can be chosen. After this information is completed click on the OK to continue. This returns you back to the main sequence window but now the first position will be highlighted in green. Continue by adding the next sample information to tray position 2, again following the same steps as before. By default the Method File will be on the program SOPAH which is used for both pesticides (SO) and petroleum (PAH) clean-up. Also by default, the Sample Type field will already be set at 'Sample'. This will not need to be changed until a duplicate sample (Duplicate), matrix spike (Matrix Spike), matrix spike duplicate (Spike Duplicate), laboratory control spike (Spiked

Blank), and the SRM (Lab Control Std) are encountered. After all the samples have been added to the sequence, save it as the bench sheet number (BS###). From the Editing sequence window print out the sample list. Compare the information to your original bench sheet to insure there are no mistakes. Make sure the ZGPC method is being used for the calibration standard and the SOPAH method is being used for the samples. Next verify that the samples are still at the marked line on the culture tubes (add DCM to the marked line if they are not). Place a tube with the GPC Calibration Standard Solution (CLP-340) in sample tray position 1 and then follow as the sequence was made in the remaining positions.

8.9.3 Get two boxes of the 125 mL Trace Clean amber bottles for sample collection. A bottle does not need to be placed in collection position #1 because that is the GPC Calibration Std (all goes to waste). Remove the white caps from the bottles and place them on top of the detector (so that Teflon side is not exposed to possible contamination). Label the boxes with bench sheet and laboratory control numbers and keep them for the post-GPC samples to be stored in. Now that the pump as had plenty of time to equilibrate the system and the detector has had plenty of time to warm up, in the Signal field click to adjust the setting to 'Absorbance Units' and click on the 'Zero Signal' button to set the baseline.

8.9.4 If the pressure seems to be pretty stable between the 12-16 psi range and all the sample positions and collection positions have been loaded, then click on the large button with the stop watch to begin the program. A window will pop up asking if the correct column method is loaded (100%DCM). Click on 'yes' to engage the syringe pump to begin priming. The sample probe will move over to sample position #1 and aspirate the sample. After the samples have all been processed (~1 hour per sample), remove the label from the sample position and place it on the bottle in corresponding collection position. Cap the bottle and place it back in the box that was retained for their storage. At the end of the sequence there will be a window that pops up saying that the 'Sequence has been successfully completed'. The column will switch offline and the pump will automatically shut down. The only thing that has to manually be turned off is the power to the detector. Empty the waste container into a 4L waste bottle labeled with a hazardous waste label.

8.10 Pour the GPC eluate into a rinsed K-D flask. Rinse the bottle with some DCM and add that to the K-D flask. Add 0.5 mL Iso-Octane and a micro boiling chip to each K-D flask. Attach a Snyder column to the flask and place in the hot water bath. Attach the inverted Hopkins condenser to the top of the Snyder column and turn to water on to the solvent recovery system (~1500-2000 cc/min). When the volume of the solvent in the concentrator tube is level with the base of the K-D flask, remove the inverted Hopkins condenser and secure out of the way. Lift the K-D apparatus up enough to be able to angle it slightly and add 40-50 mL Petroleum Ether through the top of the Snyder column. By

holding the K-D apparatus at an angle, it allows the solvent to more easily drain back into the flask. Return to the K-D apparatus back into the hot water bath. Repeat this step 2 more times to successfully solvent exchange the sample from DCM to Petroleum Ether. When the apparent volume in the concentrator tube is 5-10 mL remove it from the hot water bath. Wipe down the K-D apparatus with a WyPall X60 towel especially around the ground glass junction. Remove the Snyder column from the K-D apparatus and allow to completely drain into the concentrator tube. After it has finished cooling, remove the concentrator tube and bring to a final volume of 10 mL in DCM. Split the sample using a 5 mL volumetric pipette. One aliquot is transferred to a labeled 13 x 100 mm test tube. Add a new micro boiling chip to the remaining aliquot and place it in a 400 mL beaker containing water heated to approximately 75°C on a hot plate (4-5 tubes can be evaporated at one time). Evaporate the solvent down to 1-2 mL. Remove it from the water bath and allow it to cool. Exactly one-half of the extract is removed and placed in a GC autosampler vial for PAH silica/alumina column cleanup or for archive if PAHS are not requested.

8.11 Transfer the solution to a 13 x 100 culture tube with a Pasteur pipette, rinse the concentrator tube with 0.5 ml of Petroleum Ether, vortex, and transfer the rinse to the culture tube. Repeat the rinse step two more times, and add each rinse to the culture tube. Cap the culture tube with a Teflon faced cap.

8.12 SEDIMENT SAMPLES ONLY: Check the GPC chromatogram for a sulfur peak. If a sulfur peak is present, add acid rinsed copper to the culture tubes to remove any residual sulfur from the extract.

9.0 Florisil® Column Fractionation

IMPORTANT: ***All glassware, glass wool, and sodium sulfate must be triple-rinsed with petroleum ether (PE) before they are used for this procedure.*** **Florisil®** ***must be activated in an oven at 130°C for at least 24 hours prior to use.***

9.1 This procedure is performed after the GPC cleanup procedure for all tissue and sediment samples analyzed for pesticides and PCBs.

9.2 **PCB ONLY:** When the samples are to be analyzed for only PCBs prepare only the 6% ethyl ether in petroleum ether Florisil column eluant. Make an amount slightly in excess of what is actually needed to allow for any loss which may occur during solvent transfer. The required volume is 40 mL per sample for the 6% eluant.

9.3 **TISSUE:** Prepare the reagents to be used for Florisil® cleanup <u>for tissue</u>: 6% ethyl ether in petroleum ether, 15% ethyl ether in PE, and 50% ethyl ether in

PE. Make an amount slightly in excess of what is actually needed to allow for any loss which may occur during solvent transfer. The required volume is 40 mL per sample for the 6%, 50 ml per sample for the 15% (F2), and 40 ml per sample for the 50% (F3) fractions.

9.4 **SEDIMENT:** Prepare the reagents to be used for Florisil® cleanup for sediment: 6% ethyl ether in petroleum ether and 50% ethyl ether in PE. Make an amount slightly in excess of what is actually needed to allow for any loss which may occur during solvent transfer. The required volume is 40 mL per sample for the 6% and 40 ml per sample for the 50% fraction.

9.5 Prepare the chromatography columns. Place a small piece of PE rinsed glass wool in the bottom of the column and tap into place with a PE rinsed glass rod. Cover with a small portion (0.5 inch) of sodium sulfate. Fill the column with 5 grams of Florisil® that has been measured using a dedicated pre-calibrated culture tube. Tap column with rubber "mallet" to firmly settle the Florisil®. Top the column with 3/4-1 inch of sodium sulfate. This will prevent the column from being disrupted when solvent is added and will remove any residual water.

9.6 Place a 600 mL beaker under the column and pre-wet the column with about 25 mL of petroleum ether.

IMPORTANT: ***From this point and through the elution process, the solvent level should never be allowed to go below the top of the sodium sulfate layer.***

9.7 When approximately 1 inch of PE remains above the surface of the column, add 0.5 mL of iso-octane to a K-D flask and place it under the column making sure that the stopcock is in the full open position. This will allow for a flow rate of about 2 to 3 mL/min. When the meniscus of the PE rinse reaches the column bed surface, decant the sample onto the column. Immediately add approximately 0.5 mL of PE to the tube, vortex, and add the rinse to the sample extract on the column. Add another 0.5 ml of PE to the tube, vortex, and add this final rinse to the sample extract on the column. Start the columns in a sequential fashion, and the lag time will be adequate to perform the necessary tasks for up to six columns.

9.8 When the combined sample and rinses reach the sodium sulfate layer, add 40 mL of 6% diethyl ether/petroleum ether that has been carefully measured out using a graduated cylinder to the column reservoir. Make sure that the stopcock is fully open in order to achieve the desired flow rate of 2 to 3 mL per minute. Place a 50 mL clean, dry, petroleum ether rinsed beaker over the top of the reservoir to prevent evaporation during the elution process. If only **PCB** analyses are requested, allow the column to completely drain and stop here.

TISSUE SAMPLES

9.9 Just as the last of the 6% diethyl ether/PE solvent reaches the top of the sodium sulfate layer, add 0.5 mL of iso-octane to a new K-D flask and exchange it for the K-D containing the 6% elution, add 50 mL of the 15% diethyl ether/PE mixture to the column reservoir, replace the 50mL beaker, and elute as before. Add a micro boiling stone and attach a Snyder column with a green clamp to the K-D flask containing the 6% diethyl ether/PE fraction and place vessel in the hot water bath with the temperature set at 80-82 °C and reduce volume to an apparent volume of 1 mL. Tap the Snyder column to make sure solvent is not trapped between the balls then remove the vessel from the bath and place in the vessel stand to cool.

9.10 Repeat the above adding 0.5 mL of iso-octane to a new K-D flask and exchange it for the K-D flask containing the 15% eluant. Add 40 mL of 50% diethyl ether/PE mixture to the solvent reservoir. Allow all of the eluant to drain into the K-D flask.

SEDIMENT SAMPLES

9.11 Just as the last of the 6% diethyl ether/PE solvent reaches the top of the sodium sulfate layer, add 0.5 mL of iso-octane to a new K-D flask and exchange it for the K-D flask containing the 6% eluant, add 40 mL of the 50% diethyl ether/PE mixture to the column reservoir, replace the 50mL beaker, and elute as before. Add a micro boiling stone and attach a Snyder column with a green clamp to the K-D flask containing the 6% diethyl ether/PE fraction and place vessel in the hot water bath with the temperature set at 80-82 °C and reduce volume to an apparent volume of 1 mL. Tap the Snyder column to make sure solvent is not trapped between the balls then remove the vessel from the bath and place in the vessel stand to cool.

9.12 When the vessels are cool, remove the concentrator tube from the K-D flask add a new micro boiling stone and attach a clean micro-Snyder column to the concentrator tube with a blue clamp and place in a 400 mL beaker containing hot water heated to approximately 75°C on a hot plate. Evaporate the solvent until only 0.5-1 mL remains in the concentrator tube. Four or five tubes can be evaporated at one time.

9.13 When the solvent has been evaporated to 0.5-1 mL remove the tube from the bath and allow it to cool in a test tube rack. Remove the micro-Snyder column and transfer the contents to a calibrated centrifuge tube rinsing the concentrator tube with a small amount of PE and adding the rinsate to the centrifuge tube. If the volume in the centrifuge tube is greater than 1 mL, evaporate to 1 mL using nitrogen. Mix the tube contents by tapping the bottom of the tube causing a vortex which will rinse the sides of the tube. A Vortex

Genie mixer may be used for this step. Transfer the extract to a clean labeled culture tube and cap.

9.14 Repeat for 15% (tissue only) and 50% extracts. The extracts are ready for analysis by GC-ECD and GC-MSMS.

10.0 Analytical Procedure

10.1 Before the sample extracts can be analyzed, a sequence listing the order of calibration standards, second source check standards, initial and continuing calibration blanks, initial and continuing calibration verification standards and sample extracts is written using Agilent Chemstation (GC) or Varian (GC-MSMS) Software.

10.2 Each sequence includes a minimum of seven calibration standards. The calibration curve concentration for chlorinated hydrocarbons differs for different analytes, but in general the range is 0.5 ppb to 500 ppb. The calibration curve concentration range for polychlorinated biphenyl congeners (PCBs) is 0.5 ppb to 100 ppb. Higher concentrations of PCB standards (50 ppb to 1000 ppb) are analyzed with samples containing higher concentrations of PCBs.

10.3 To verify the calibration standards, second source pesticide check standards (Radian Corp., Pesticide Check Standard Mix A, ERP-009L; Pesticide Check Standard Mix B, ERP-011L) and PCB congener check standard (Ultra Scientific, RPC-EPA) are analyzed. The second source analytes and their concentrations are listed in Table 5 (pesticides) and Table 6 (PCB congeners).

Table 5. Radian Pesticide Calibration Check Standards (Mix A and B)

Mix A	Certified Concentration (ng/µL)
Aldrin	10.0
Gamma-HCH	5.00
DDT, p,p'	20.0
Dieldrin	10.0
Endosulfan I	10.0
Endosulfan II	20.0
Heptachlor	10.0
Heptachlor epoxide	10.0
Methoxychlor	80.0
Mix B	
Alpha-HCH	5.00
Beta-HCH	20.0
Delta-HCH	10.0
Cis-chlordane	10.0

Trans-chlordane		10.0
DDD, p,p'		20.0
DDE, p,p'		10.0
Endosulfan sulfate	20.0	
Endrin		20.0

Table 6. Ultra Scientific PCB Congener Check Standard

RPC-EPA	Certified Concentration (ng/µL)*
PCB 8	4.0
PCB 18	4.0
PCB 28	4.0
PCB 52	4.0
PCB 44	4.0
PCB 66	4.0
PCB 101	4.0
PCB 118	4.0
PCB 153	4.0
PCB 105	4.0
PCB 138	4.0
PCB 187	4.0
PCB 128	4.0
PCB 180	4.0
PCB 170	4.0
PCB 195	4.0
PCB 206	4.0
PCB 209	4.0

* Initial concentration of RPC-EPA is 0.2 µg/mL in iso-octane. This solution is diluted 2:100 in iso-octane

10.4 An initial calibration blank and initial calibration verification standard is analyzed after the calibration standards and prior to the first sample extract. For the 6% Fraction and 15% Fraction runs, continuing calibration blanks (CCBs) and calibration verification standards (CCVs) are analyzed after ten sample extracts have been analyzed. The 50% Fraction extracts contain more lipid material and can cause the CCVs to fail to meet the % recovery criteria, therefore the CCBs and CCVs are analyzed after every five sample extracts. If a CCV fails, the five samples prior to the failed CCV and the five samples after the failed CCV are re-analyzed after a new calibration curve is analyzed.

10.5 The CCV analyte concentrations are mid-range of the calibration curve (5 – 10 ppb).

10.6 As the run proceeds, sample extracts are monitored for analyte concentrations that are greater than the calibration curve and need dilution.

10.7 Instrumentation

Gas Chromatographs with Electron Capture Detectors:

10.7.1 Agilent 6890*plus* gas chromatograph equipped with two ^{63}Ni micro-electron capture detectors with EPC and autosampler. Two 60 meter, 0.25 mm ID, 0.25 µm (film thickness) fused silica columns (J&W) are used. A 5 meter length of DB-5 column is connected to a press fit "Y" union which splits the column effluent into two 60 m columns, a DB-5 and a DB-17MS. The injector is a split-splitless injector with EPC.

10.7.2 Chromatograph conditions:
The injector is operated isothermal at 240°C. The oven has an initial temperature of 80°C which is held for 1 minute and then temperature programmed to 210°C at a rate of 15°C/min and held for 10 min. It is then programmed to 280°C at a rate of 2°C/min and is held for 51 min (for PBDE analysis the oven is held at 280°C for 110 min). Helium is used as the carrier gas at a linear velocity of 35 cm/sec. Nitrogen is used for the detector makeup at 30 mL/min.

10.7.3 Sample volume:
Three microliters of samples and standards are injected and split approximately 50/50 onto the 60 m DB-5 and the 60 m DB-17MS.

10.7.3 Instrument calibration:
External standard calibration is used.

10.7.4 Data acquisition and processing:
Detector signals are acquired and processed with a Agilent 3365 Series II Chemstation. Data processing may also be done using Enviroquant Software.

Gas Chromatograph-Triple Quadrupole Mass Spectrometer:

10.7.5 Varian Model 3800/1200L gas chromatograph/triple quadrupole mass spectrometer equipped with a Model 1177 split-splitless injector with EPC and CombiPal autosampler. A J&W 60 meter, 0.25 mm ID, 0.25 µm (film thickness) XLB fused silica columns (J&W) is used. The injector is a split-splitless injector with EPC.

10.7.6 Chromatograph Conditions:
The injector is operated isothermal at 280°C in splitless mode with pressure pulse (45 psi for 1.05 min). The oven has an initial temperature of 80°C which is held for 1 minute and then temperature programmed to 210°C at a rate of 15°C/min and held for 10 min. It is then programmed to 280°C at a rate of 2°C/min and is held for 8 min.

Helium is used as the carrier gas at a constant column flow of 1 mL/min.

10.7.7 Mass Spectrometer Conditions:
The mass spectrometer is operated in electron impact (EI) ionization and MSMS mode using argon as the CID gas. A collision energy of 10 to 30 volts is used depending on the analyte. Q1 and Q3 mass fragments were selected to optimize selectivity and sensitivity. See Table 7.

Table 7. Varian 1200 MS collision energies and mass fragments (Q1 and Q3) for targeted analytes.

	Segment	Q1	Q3	Collision Energy	Internal Standard
DBOB	1	296	246	-20	HCH, alphaC^{13}
HCH, alpha	2	219	183	-10	HCH, alphaC^{13}
HCH, alphaC^{13}	2	223	187	-10	Internal Std
HCB	3	284	214	-30	HCB C^{13}
HCBC^{13}	3	290	220	-30	Internal Std
HCH, gamma	4	219	183	-15	HCH, alphaC^{13}
HCH, beta	4	219	183	-15	HCH, alphaC^{13}
Heptachlor	5	272	237	-15	HeptachlorC^{13}
HeptachlorC^{13}	5	277	242	-15	Internal Std
Chlorpyrifos	6	314	258	-10	ChlorpyrifosC^{13}
ChlorpyrifosC^{13}	6	325	260	-15	Internal Std
Aldrin	6	293	258	-10	ChlorpyrifosC^{13}
Oxychlordane	7	387	263	-10	Nonachlor, transC^{13}
Heptachlor epoxide	7	387	353	-10	HeptachlorC^{13}
DDE, o,p'	8	318	246	-10	DDE, p,p'C^{13}
DDMU, p,p'	9	284	212	-15	DDE, p,p'C^{13}
Chlordane, trans	9	373	266	-15	Nonachlor, transC^{13}
Chlordane, cis	9	373	266	-15	Nonachlor, transC^{13}
Nonachlor, trans	10	409	310	-15	Nonachlor, transC^{13}
Nonachlor, transC^{13}	10	418	310	-15	Internal Std
DDE, p,p'	12	318	246	-15	DDE, p,p'C^{13}

DDE, p,p'C^{13}	12	329	258	-15	Internal Std
DDD, o,p'	13	235	165	-20	DDE, p,p'C^{13}
DDT, o,p'	15	235	165	-15	DDT, p,p'C^{13}
Nonachlor, cis	16	409	275	-15	Nonachlor, transC^{13}
DDD, p,p-deuterated	16	243	173	-20	DDT, p,p'C^{13}
DDD, p,p'	16	235	165	-15	DDT, p,p'C^{13}
DDT, p,p'	17	235	165	-25	DDT, p,p'C^{13}
DDT, p,p'C^{13}	17	248	177	-20	Internal Std
Methoxychlor	18	227	169	-20	DDT, p,p'C^{13}
Mirex	20	272	237	-15	DDE, p,p'C^{13}

10.7.8 Instrument Calibration:

Internal standard calibration is used. Internal standards are added to the standards and sample extracts just prior to analysis. The following internal standards are used at 1.0 ng/µL:

PCB Internal Standards
PCB 52 (C^{13}) – 4Cl congeners
PCB 97 (C^{13}) – 5Cl congeners
PCB 128 (C^{13}) – 6Cl and 7Cl congeners
PCB 194 (C^{13}) – 8Cl congeners
PCB 206 (C^{13}) – 9Cl congeners
PCB 209 (C^{13}) – 10Cl congeners

OC Internal Standards
HCH, alpha (C^{13})
HCB, (C^{13})
Heptachlor, (C^{13})
Chlorpyrifos, (C^{13})
Nonachlor, trans (C^{13})
DDE, p,p' (C^{13})
DDT, p,p' (C^{13})

Nine target analyte calibration levels are used (0.25, 0.50, 1.0, 2.0, 5.0, 10.0, 20.0, 50.0, 100 ng/µL).

10.7.9 Sample volume:

Two microliters of samples and standards are injected.

10.7.10 Data processing:
Mass spectrometer signals are acquired and processed using Varian 1200L software .

11.0 References

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Appendix IV C: Procedure for the Handling, Storage and Disposal of Hazardous and General Laboratory Waste

File: HAZMAT_Rev4_SOP
Date: 3/23/09
Revision #: 4
Prepared By: DBC
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CDFG Fish and Wildlife Water Pollution Control Laboratory (WPCL) and Petroleum Chemistry Laboratory (PCL) Standard Operating Procedure for the Handling, Storage, and Disposal of Hazardous and General Laboratory Waste

1. Scope and Application

1.1 Federal and state regulatory control over hazardous waste has become extraordinarily stringent in recent years. These changes have dramatically increased the complexity of handling the hazardous waste produced by laboratories.

1.2 These procedures are provided to ensure safe, efficient, and legally compliant handling and disposal of hazardous waste.

2. Summary of Hazardous Waste Disposal

2.1 **NEVER DISPOSE OF LIQUIDS, SOLID CHEMICALS, LABORATORY SAMPLES, HAZARDOUS WASTE OR HAZARDOUS MATERIALS IN THE LABORATORY TRASH RECEPTICALS OR DUMPSTERS. NEVER DISCHARGE LIQUID HAZARDOUS WASTE TO THE SANITARY SEWER (bathroom drains) OR EVAPORATION POND (laboratory sinks, fume hood drains, floor drains).** Non-hazardous aqueous laboratory waste can be disposed of by discharging to the evaporation pond.

2.2 Characteristics of Hazardous Waste (these definitions apply to waste potentially generated by WPCL, for complete definitions see Title 22 Article 2 section 66261.10)

Ignitability – Hazardous Waste Number **D001**

- is liquid, other than an aqueous solution containing less than 24 percent alcohol by volume, with flash point less than 60ºC (140ºF);
- is not a liquid and is capable of causing fire through friction, absorption of moisture or spontaneous chemical changes and, when ignited burns so vigorously and persistently that it creates a hazard;
- is an ignitable compressed gas;
- is an oxidizer defined in 49 CFR section 173.151.

Corrosivity – Hazardous Waste Number **D002**

- is aqueous and has a pH less than or equal to 2 or greater than or equal to 12.5;
- is not aqueous and, when mixed with an equivalent weight of water, produces a solution having a pH less than or equal to 2 or greater than or equal to 12.5.

Reactivity – Hazardous Waste Number **D003**

- is normally unstable and readily undergoes violent change without detonating;
- reacts violently with water;
- forms potentially explosive mixtures with water;
- when mixed with water, generates toxic gases, vapors or fumes in a quantity sufficient to present a danger to human health or the environment;
- is a cyanide or sulfide bearing waste which, when exposed to pH conditions between 2 and 12.5, can generate toxic gases, vapors or fumes in a quantity sufficient to present a danger to human health or the environment;
- is capable of detonation or explosive reaction if it is subjected to a strong initiating source or if heated under confinement;
- is readily capable of detonation or explosive decomposition at STP;
- is a Class A explosive.

Toxicity – Hazardous Waste Number **D004-D043**

See attached definitions of toxicity.

2.3 All chemical hazardous waste must be properly identified, labeled, segregated, and stored prior to removal by a qualified and licensed hazardous waste contractor.

2.4 Maximum Storage Times

- The maximum length of time that hazardous waste may be stored by the laboratory is 270 days from the initial date of accumulation.
- On the date that 55 gallons of waste have accumulated, the laboratory has 90 days to have the waste removed.
- Hazardous waste should be transferred from the laboratory to the hazardous material storage building within 6 months of the initial date of accumulation. The date that the waste is transferred to the hazardous material storage building, that date must be entered on the hazardous waste label under "Accumulation Start Date". Waste must be removed within 90 days of the Accumulation Start Date.
- Any hazardous waste container stored over 270 days is a violation.

2.5 Labeling Hazardous Waste Containers

- All hazardous waste containers must be labeled properly.
- Hazardous waste labels must be completely filled out.
- The first date of accumulation (WPCL waste log-in code) must always be entered on the upper right corner of the label with the individual's initials. **The date that the waste is transferred from the lab to the hazardous waste storage building must be entered on the label in the "Accumulation Start Date" section and that starts the 90 day removal requirement.**

2.6 Neutralization of Aqueous Acids and Bases

Small volumes of aqueous corrosive hazardous waste may be neutralized and discharged to the evaporation pond if the following procedures are followed. A hazardous waste facilities permit or other grant of authorization is not required for treatment of laboratory hazardous waste (neutralization of aqueous acid and base waste) generated onsite, if all of the following requirements are met:

- The hazardous waste is treated in containers using recommended procedures and quantities for treatment of laboratory wastes published by the National Research Council (NRC) or procedures for treatment of laboratory wastes published in peer-reviewed scientific journals.
- The waste is treated at a location that is as close as practical to the location where the laboratory hazardous waste is generated, **and the treatment is conducted within 10 calendar days after the date that the waste is generated.**
- The amount of laboratory hazardous waste treated in a single batch does not exceed the quantity limitation specified in subparagraph (A) or (B), whichever is the smaller quantity:
  - (A) **Five gallons** or 18 kilograms, whichever is greater.
  - (B) (i) Except as otherwise provided in clause (ii), the quantity limit recommended in the procedures published by the NRC or in other peer reviewed scientific journals for the treatment procedure being used. (ii) a qualified chemist has demonstrated that a larger quantity can be safely treated and documentation to that effect is maintained onsite.
- The laboratory hazardous waste treated is from a single procedure, or set of procedures that are part of the same laboratory process.
- The person performing the treatment has knowledge of the laboratory hazardous waste being treated, including knowledge of the procedure that generated the laboratory waste, and has received hazardous waste training, including how to conduct the treatment, manage treatment residuals, and respond effectively to emergency situations.
- Training records for all persons performing treatment of laboratory hazardous wastes pursuant to this subdivision are maintained for a minimum of three years.
- All records maintained by the laboratory pertaining to treatment conducted pursuant to this subdivision are made available for inspection upon request by a representative of the department or the CUPA or other authorized agency.

3. Source Reduction and Waste Minimization

3.1 Whenever possible, experiment protocols should include provisions to both reduce the volume of the source, and minimize the generation of hazardous waste.

3.2 Waste such as motor oil, paper, corrugated boxes, toner cartridges etc. that is eligible for recycling should be recycled.

4. Storage of Waste in the Laboratory

4.1 Each laboratory should have a designated location in which to store hazardous materials to be discarded. This location should be out of the way of the normal lab activities, but should be easily accessible and recognizable. This space should be properly labeled. Fume hoods may be used temporarily to store small quantities of materials being generated but hazardous materials should not be allowed to accumulate in hoods because it could block air flow. Cabinets under fume hoods are appropriate storage locations for small quantities of hazardous waste.

4.2 All waste materials must be kept in secondary containers and segregated by hazard class (i.e., oxidizing agents such as potassium permanganate or hydrogen peroxide should be separated from organics or corrosives, acids should be separated from bases, etc.). Secondary containers can be lab trays, or any such device that will contain 110% of the largest container.

5. Labeling

5.1 Containers must be labeled prior to being used as receptacles for hazardous waste. Printed Hazardous Waste Labels must be used and filled out completely with all mandatory information including (see attached examples and summary of hazardous waste labeling codes commonly used at WPCL):

- the words "Hazardous Waste"
- starting date of accumulation in upper right corner and initials of person labeling waste container (waste identification number)
- CDFG Fish and Wildlife Water Pollution Control Lab
  2005 Nimbus Road (916) 358-2858
  Rancho Cordova, CA 95670
- WPCL's EPA ID Number (**CAD980815401**)
- the "Accumulation Start Date" or the date the waste is transferred to the hazardous materials storage building which starts the 90 day storage period (satellite storage rule)
- the composition (name of the waste) and physical state (gas, liquid, solid, sludge)
- a description of the hazardous properties of the waste (i.e. flammable, reactive, toxic, corrosive)
- EPA waste code and California waste code
- Approved D.O.T. Shipping Name and "UN" number (proper shipping names must be written exactly as listed in the D.O.T. regulations)

5.2 Handwritten labels made from tape or unprinted labels are **not** acceptable .

5.3 Chemical names must be specific. Nonspecific labels such as "organic waste", "waste solvents", "acid waste", etc., are not sufficient.

5.4 Chemical formulas or abbreviated chemical names are **not** acceptable.

5.5 Specific waste identification labels may be used **in addition to** the Hazardous Waste Label, such as PCB waste labels.

5.6 Hazardous Waste Labels are available in the cabinet in the photocopier room in the main laboratory.

6. Documentation (Hazardous Waste Inventory Logs)

6.1 When a waste container is started in the laboratory or moved to the hazardous materials storage building, it must be logged-in on laboratory's (lab room or hazmat storage building) Hazardous Waste Inventory Log. Each laboratory room where hazardous waste is generated and the hazardous materials storage building must have a logbook for recording the information described in 6.2.

6.2 The log must include the following information:
Container Log # (Identification No. – month/day/year, initials)
- -Date In (date that waste accumulation started)
- -Date Out (date that waste is transferred to the hazardous material storage building)
- -Waste Description
- -Waste Amount (size of container)
- -Hazard (Toxic, Flammable, etc)
- -First initial and surname of the person starting the waste

6.3 When the hazardous waste container is moved to the hazardous materials storage building, the waste must be logged out of the laboratory and logged into the hazardous materials storage building. The date that the waste is transferred must be entered on the Hazardous Waste label in the "Accumulation Start Date" section.

7. Containers

7.1 Containers must be properly labeled and in good condition (i.e., structurally sound and leak-proof) and **kept closed** unless you are adding or removing wastes. Liquids must be in a screw-capped container that will not leak if tipped over. Corks, parafilm, lab beakers, or other open containers are **not** acceptable. If waste is not in a proper container, transfer the waste.

7.2 The size of the container should correspond with the quantity of materials discarded. For example, it is not cost effective to ship 50 mL of material in a 4 L container, etc.

7.3 Contaminated lab ware such as glassware, gloves, paper towels, etc., must not have liquid in them. They must be placed in clear, double plastic bags and properly labeled with a Hazardous Waste Label. For disposal of broken glassware and sharps see Section 11.

7.4 Glass, paper, or plastic must not be placed in liquid waste containers.

7.5 The material must be compatible with the container - acids or bases cannot be stored in metal containers or solvents in plastic.

7.6 Containers must be inspected weekly for leaks and deterioration (**this must be documented with any deficiencies and corrective action**).

7.7 Hazardous waste storage building must be inspected weekly by the WPCL safety officer or his designate. **This inspection must be documented with any deficiencies and corrective action.**

8. Waste Segregation

8.1 Proper segregation of waste chemicals in the laboratory helps facilitate waste disposition options such as recycling. This can also result in cost savings for disposal. Any questions about waste segregation should be directed to the WPCL safety officer.

8.2 Examples of responsible and cost-effective segregation include:

- Separating halogenated from non-halogenated solvents
- Isolating metals from other wastes
- Keeping waste acetone separate from other solvents

9. Empty Containers

9.1 Empty (nothing can be poured out if the container is inverted) chemical containers of **five gallons or less** that have had the caps removed and labels defaced or removed may be disposed of as regular refuse. **Full or partially full containers should never be thrown in the regular trash.**

9.2 Empty containers that held extremely hazardous materials (waste) must be triple rinsed prior to disposal. **All rinsate** must be handled as hazardous waste.

10. Unknowns

10.1 Unknowns must be characterized prior to disposal. If the Laboratory can

not characterize the material it will have to be characterized by the hazardous waste contractor or another lab specializing in the characterization of hazardous waste prior to disposal.

11. Sharps

11.1 Broken glass, pipets, and any other sharp material that is not contaminated with hazardous material must be disposed of in a rigid sharps or broken glassware container.

11.2 Sharps that have been contaminated with hazardous materials should either be triple rinsed and discarded with non-contaminated sharps or should be discarded in a separate rigid sharps container designated and labeled as hazardous waste.

12. Tissue and Sediment Waste

12.1 Tissue waste resulting from dissection of fish should remain frozen until it is to be discarded. The frozen fish tissue should be bagged and transported to the sanitary landfill where it is discarded in a designated area used for that purpose. The county landfill should be contacted ahead of time to find out dates that these types of refuse are accepted.

12.2 Waste sediment and soils that meet the definition of hazardous waste must be labeled and treated accordingly. Waste sediment and soils that are not hazardous waste must be disposed of as non-hazardous laboratory waste and removed by the laboratory's hazardous waste contractor.

13. Hazardous Materials Storage Building Weekly Inspections

13.1 The hazardous materials storage building must be inspected weekly. A permanent record of the inspections, inspection log and deficiency and corrective action reports must be kept. Examples of the checklist, inspection log and deficiency report are attached.

14. References

California Environmental Protection Agency, Department of Toxic Substances Control (DTSC) Fact Sheets January 2002 and December 2006.

CCR Title 22, sections 66261.10, 66262.20-24, 66262.34

California Health and Safety Code, section25200.3.1

Safety Officer	Signature	Date
Laboratory Director	Signature	Date

Appendix IV D: Protocol for Corrective Action Procedures

California Department of Fish and Game
Fish and Wildlife Water Pollution Control Laboratory
2005 Nimbus Road
Rancho Cordova, CA 95670

STANDARD OPERATING PROCEDURE

NONCONFORMANCE, CORRECTIVE ACTION AND PREVENTIVE ACTION

1.0 SCOPE AND APPLICATION

1.1 The purpose of this document is to describe the procedure used by the DFG Water Pollution Control Laboratory (WPCL) for the identification and documentation of nonconforming events, items, or procedures and the assessment of their impact on the quality of data generated by the laboratory.

1.2 This procedure also describes the laboratory's corrective action and preventive action procedures and monitoring.

1.3 This SOP is applicable to all laboratory systems involved in the quality system and analytical processes in the laboratory, including but not limited to, sample receiving and logging, storage, preparation, analysis, reporting, auditing, and proficiency testing.

1.4 This SOP also addresses instances of nonconformance for which no corrective action is possible or appropriate, but documentation of the nonconformance is still required.

1.5 Documentation of all nonconformances is required by WPCL and is maintained on file by the QC Officer.

1.6 This procedure also requires documentation of nonconformances resulting from errors made by persons submitting samples to the laboratory. The documentation required is maintained and archived with the appropriate data set.

1.6.1 Errors made by persons submitting samples to the laboratory (i.e. errors on chain of custody documents, sample labeling, etc.) are documented and resolved by immediately contacting the sampler or person responsible for the samples.

1.6.2 Errors that are identified following receipt of analytical results that do not result from any laboratory mistake (i.e. incorrect sample identifier, test method requested, etc.) are resolved using directions taken from consultation with the data user.

2.0 DEFINITIONS

2.1 **Nonconformance** – An item, event, or procedure which does not comply or agree with the governing documents, procedures, policies or requirements (e.g. QAPP, QAMP, WPCL QA Manual, etc.).

2.2 **Corrective action** – A twofold measure that is taken to correct a nonconforming event and to eliminate or severely restrict the reoccurrence of the same type of nonconformance.

2.3 **Preventive action** – A measure taken to address needed improvements and potential sources of nonconformances.

3.0 RESPONSIBILITIES

3.1 Any individual within the laboratory can stop an analysis when nonconformance occurs (i.e. nonconformance not caused by sample matrix or similar unpreventable condition, etc.). Problems will be identified, documented and resolved prior to continuing the analysis.

3.1.1 The quality assurance officer also possesses the authority and responsibility to stop any work that does not meet quality standards of the laboratory and to take all necessary steps to return the system in question to a state of control.

3.1.2 The Laboratory Director is responsible for the determination of "official" work stoppages and for notifying all parties of concern regarding work stoppages, redistribution, subcontracting, if necessary, and subsequent work resumption.

3.1.2.1 The Laboratory Director, in conjunction with the Section Lead Chemist and QC Officer is responsible for redistributing workload during work stoppages to ensure that requirements are met with respect to hold and turnaround times.

3.1.2.2 The Section Lead Chemist, QC Officer, or Laboratory Director are responsible for notifying the data user of significant problems requiring work stoppages.

3.2 Each employee who detects a deficiency is responsible for initiating documentation of the nonconformance and forwarding the documentation to the appropriate Section Lead Chemist and /or QA Officer for review and assessment.

3.3 The Section Lead Chemist, in conjunction with the QA Officer and Laboratory Director, is responsible for analyzing the source of the nonconforming item, determining the impact of the nonconformance on the quality of the data and /or operations and implementing corrective actions to correct and/or restrict the noted deficiency according to the requirements detailed in the project QAPP or laboratory QA manual.

3.4 The Quality Assurance Officer is responsible for maintaining nonconformance/corrective action records and aiding personnel in the identification of nonconforming items, determining the extent of the nonconformance, and planning corrective action.

3.5 Laboratory personnel are responsible for participating in cause analysis and implementing corrective actions in response to nonconformances and for timely written response(s).

3.5.1 Generally, corrective actions, taken in response to nonconformances and cause analysis, are to be initiated immediately upon the identification of the event.

3.5.2 Some nonconformances, cause analyses and appropriate corrective actions may require additional time due to external factors, including purchasing new materials, obtaining additional training etc.

3.5.3 The QA Officer is responsible for participating in and monitoring nonconformance identification, cause analysis and corrective actions to ensure that each nonconformance is addressed quickly and effectively.

3.6 The Laboratory Director is ultimately responsible for assuring that laboratory procedures are performed in accordance to written instructions.

4.0 NONCONFORMANCE PROCEDURE AND DISCUSSION

4.1 Each performance event is documented by the individual who identifies the event. Documentation is effected using the Nonconformance/Corrective Action Record (NC/CAR). The NC/CAR is completed following the instructions on the form and is forwarded to the Section Lead Chemist followed by the QA Officer for review.

4.1.1 All laboratory staff have access to electronic NC/CAR forms.

4.1.2 The person(s) identifying the nonconformance will complete the Set ID, Sample Matrix, Analysis, Date Documented, and Date of Occurrence along with a brief description of the nonconformance.

4.1.2.1 If a specific data set is not appropriate to identify the nonconformance, complete a descriptive title in the space marked Set ID to allow for clear and concise identification of the nonconformance addressed.

4.1.3 The identifier will also sign the initiated form and forward it to the Section Lead Chemist who will pass it on to the QA Officer.

4.1.4 The Section Lead Chemist and/or QA Officer will assess the impact of the nonconformance on the data generated and will formulate a cause analysis study, if necessary.

4.2 Nonconformances may also be generated by the QA Officer in response to specific Measurement Quality Objectives and Method Quality Objectives. These reports do not require the Section Lead Chemist's review.

4.3 One NCR is completed for each nonconformance identified; however multiple sample sets may be documented on one NCR form if the deficiency is the same for each set listed.

4.4 The Section Lead Chemist and QA Officer review the NCR for assignment of cause analysis investigation and potential corrective actions. If following the determination of cause, a corrective action is deemed appropriate; procedures are followed as described in Section 6.

5.0 ROOT CAUSE ANALYSIS PROCEDURE AND DISCUSSION

5.1 Root cause analysis may involve any number of people, from 1 or 2 to the entire laboratory staff, and may involve informal conversations to lengthy reports to various individuals but must include a sufficient number of people to effectively and efficiently identify what happened and more importantly the cause of the nonconforming event (why it happened) or item and all related factors that contribute to the nonconformance.

5.1.1 The root cause of the nonconformance may not always be the obvious source of the problem.

5.1.2 Root causes that result in nonconformances can include: staff skills and training, client requirements, sample composition, methods requested, equipment, calibration, supplies, etc.

5.1.3 Investigations include historical sample performance (i.e. samples from the same site submitted by the same client), method performance, analyst training, and any other factors relating to system performance that could make an impact on the nonconformance identified.

5.1.4 Additionally upon the identification of cause of the nonconformances, internal audits may be performed where appropriate areas of activity are audited as soon as possible if the identification of the nonconformance casts doubts on the laboratory's compliance with its own policies and procedures or project QAMP or QAPP.

5.2 Root cause analysis studies must be appropriate to the scope and severity of the nonconformance identified.

5.3 Root cause analysis is generally undertaken by the QA Officer, in conjunction with the specific Section Lead Chemist and staff, and is monitored by the QA Officer for effectiveness in addressing the original nonconformance identified.

5.4 Upon identification of the root cause, the QA Officer and/or Section Lead Chemist will complete the Cause of Nonconformance section on the NC/CAR report and will then decide if a corrective action is needed, what steps should be performed to implement that corrective action to remedy and restrict the reoccurrence of the nonconformance and will designate the laboratory personnel who will be assigned to implement the steps required.

6.0 CORRECTIVE ACTION PROCEDURE AND DISCUSSION

6.1 Upon identification of the root cause, the QA Officer and Section Lead Chemist then decide if a corrective action is needed, what steps should be performed to correct the nonconformance and to severely restrict reoccurrence and will also determine the personnel who will be assigned to implement the steps selected.

6.2 Common corrective actions include: recalibration, instrument maintenance, sample repreparation, analysis of spiking solutions for degradation, etc. but must be appropriate to the scope and the magnitude of the nonconformance identified.

6.3 The corrective action portion of the NC/CAR form is then completed and retained by the QA Officer for further review and a copy is placed with the documentation for the project affected by the nonconformance/corrective action.

7.0 PROCEDURES FOR CORRECTIVE ACTION AND FOLLOW-UP

7.1 Allowing an appropriate period of reasonable time to fully implement the corrective action, the QA Officer will then perform a review of the subsequent implementation and effectiveness of all corrective actions.

7.1.1 This follow-up is usually performed within 3-5 days, but the time frame may vary depending on the complexity of the corrective action required.

7.1.2 Following this review, the NC/CAR form is then completed by the QA Officer.

7.1.2.1 If corrective action has been successful, the NC/CAR form is copied and the copy placed in the appropriate sample set for archive and the original is retained by the QA Officer in the NC/CAR file.

7.1.2.2 If corrective actions have not been successful, the Section Lead Chemist and QA Officer will conduct another review to determine other possible courses of action and repeat procedures in Sections 5 – 6.

7.1.2.3 If no corrective action has been taken by the individual assigned to implement the corrective action, the issue will be reported to the Laboratory Director for further action.

8.0 PREVENTIVE ACTIONS DISCUSSION AND PROCEDURE

8.1 Preventive action is a pro-active process to determine the areas where potential improvements can be made to reduce the likelihood of problems or complaints.

8.2 Preventive actions may originate with any member of the laboratory, from analyst to Laboratory Director, and should be brought to the attention of the Section Lead Chemist and/or QA Officer for consideration.

8.2.1 It may be necessary for the originator to prepare a short report regarding the type of improvement needed and potential improvements to be made to provide ample information for a thorough discussion among the laboratory's lead chemists and director.

8.2.2 Preventive actions generally result from the Section Lead Chemists or the Laboratory Director as a result of conversations with laboratory staff or daily activities.

8.2.3 Preventive actions can result from needed changes as instrumentation or procedures become outdated, newer technology is created to improve the laboratory's throughput and data quality, or as a result of trends identified during control charting or data analysis/review, etc.

8.3 Once issues are identified for possible preventive actions and the QA Officer is informed, the issues are discussed with the Section Lead Chemist and analysts.

8.4 The issue will be discussed with the laboratory staff affected by the proposed preventive actions, including possible benefits and costs, for formulation of an action plan.

8.4.1 If subsequent investigations are necessary, they will be assigned to specific personnel and will be monitored by the QA Officer for resolution by the date assigned for completion of the investigation.

8.5 Following the reception of all required supporting information, the Laboratory Director is responsible for determining the need for the proposed preventive action, for assigning personnel to perform the preventive action duties, and for determining the time frame in which the duties will be completed.

8.6 If the situation becomes an actual nonconformance or the result of nonconformances prior to the resolution of the preventive action; the preventive actions taken will be assistive, but the issue is then addressed using the procedure for nonconformance/cause analysis/corrective action and that procedure will take precedence over the preventive action activities.

9.0 APPENDICIES

9.1 NC/CAR Report (2 pages)

10.0 REFERENCES

10.1"General Requirements for the Competence of Testing and Calibration Laboratories," ISO/IEC 17025:1999(E).

California Department of Fish and Game
Fish and Wildlife Water Pollution Control Laboratory
2005 Nimbus Road
Rancho Cordova, CA 95670

SOP Contract QA Officer Approval: _____ Date: _____

SOP Final Approval: _____ Date: _____

SOP QA Officer Approval: _____ Date: _____

California Department of Fish and Game
Fish and Wildlife Water Pollution Control Laboratory
2005 Nimbus Road
Rancho Cordova, CA 95670

Date: 9-18-06
SOP# CORR_ACTION
Revision #0
Prepared by: DBC
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Appendix 9.1 NC/CAR Report

CDFG WATER POLLUTION CONTROL LABORATORY
NONCONFORMANCE/CORRECTIVE ACTION REPORT

NC/CAR# _____
(assigned by QA Officer)

Directions: Fill in all information in the top box and briefly explain the nonconformance. Forward this document to the QA Officer for review/follow-up and archive in the project folder.

SUBMIT THIS REPORT TO THE QA OFFICER WITHIN ONE WORKING DAY AFTER NONCONFORMANCE HAS BEEN DOCUMENTED

Set ID#(s): _____

Sample Matrix: _____ **Analysis:** _____

Date Documented: _____ **Date of Occurrence:** _____

Briefly describe nonconformance: *(Check all that apply)*

1. _____ LCS compounds outside warning/control limits.
2. _____ Contamination in blank outside warning/control limits
3. _____ MS/MSD compounds outside warning/control limits.
4. _____ Surrogate outside warning/control limits.
5. _____ Calibration curve/check standard outside warning/control limits.
6. _____ SRM outside warning/control limits.
7. _____ Other: (describe) _____

_____

Was client contacted? _____ Yes (If yes, complete the following information) _____ No

Client contact: _____ **Organization:** _____

Date: _____ **Time:** _____

Signature: _____ **Date:** _____

Supervisor's Signature: _____ **Date:** _____

QUALITY ASSURANCE USE ONLY

Date NCR received by QA: _____

Signature: _____ **Date:** _____

California Department of Fish and Game
Fish and Wildlife Water Pollution Control Laboratory
2005 Nimbus Road
Rancho Cordova, CA 95670

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CDFG WATER POLLUTION CONTROL LABORATORY
NONCONFORMANCE/CORRECTIVE ACTION REPORT

NC/CAR# _____
(assigned by QA Officer)

Directions: Cause and corrective action are to be completed by the Section Lead Chemist with the aid of any responsible parties. A two-week (or appropriate) follow-up to the corrective action will be given to resolve the issue by the personnel assigned to address the nonconformance. If corrective action is not addressed, the report will be forwarded to the Lab Director for resolution.

Section Lead Chemist

Cause of Nonconformance:

_____ Matrix Effect.
_____ Spiking solution/Standard mix degradation.
_____ Instrument malfunction.
_____ Preparation error.
_____ Other (describe): _____
_____

Section Lead Chemist

Corrective Action: (Check all that apply)

_____ Sample was re-prepared and reanalyzed.
_____ Standards were re-prepared and reanalyzed.
_____ Instrument maintenance was performed.
_____ Spiking solution/standard solution was analyzed for degradation.
_____ Other (describe): _____
_____
_____ No action necessary/possible. Why? _____

Person assigned to correct nonconformance: _____

Date corrective action is to be initiated: _____

Assigned by: _____

QA USE ONLY (Follow Up Comments)

Was corrective action initiated? _____ Yes _____ No* _____ Not Required

Did corrective action correct nonconformance? _____ Yes _____ No*

** (if no to either, forward to Laboratory Director for further action)*

Comments:

Signature: _____ **Date:** _____

Laboratory Director (if applicable)

Comments:

Signature: _____ **Date:** _____

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Appendix V: MPSL-MLML SOPs

MPSL-MLML Procedures			
Page	Procedure/Equipment	SOP Number	Revision Date
A	SWAMP SOP Chemistry Data Verification V1.1		March 2005
B	Validation of BOG Database (Modifications to RMP Data Validation)		

Appendix V A: SWAMP SOP Chemistry Data Verification v1.1

This document is an official SWAMP SOP and can be found at:
http://mpsl.mlml.calstate.edu/SWAMP_SOP_Chemistry_Data_Verification_v1.1.pdf

Appendix V B: Validation of BOG Database

(Modifications to RMP Data Validation)

Validation of Bog Database

Verified and validated using the SWAMP MQOs, following the SWAMP verification SOPs and SWAMP Classification SOP. Rejection points and blank contamination were based on SFEI documentation.

Accuracy

MS/MSD %R, LCS, CRM

Following SWAMP MQOs, one QC element is allowed to fail for accuracy in a batch and still be compliant. When more than one QC element fails, the analyte in each QC element is given a QAcode (EUM, GBC, GB) and then all in batch for that analyte is classified as Est. In these cases, a "VIU" QAcode was applied to the field samples.
Rejection Point: VRIU was applied to the field samples when %R was above 200 in the highest failed QC element SRM>MS/MSD>LCS and the compliance code was changed to Rej. In the case where there is only one QC element reported in the batch and the %R was above 200 then the compliance code would be rejected.

Surrogates

Following SWAMP MQOs, failed surrogates and associated analytes are given a QACode of GN and classified Est in the given sample

Precision

MS/MSD and DUP

When precision is out, the QC sample is given a QACode (IL) and all in batch for the analyte is classified as Est. In these cases, a "VIL" QACode was applied to the field samples.
Rejection Point: VRIL was applied to the field samples when the RPD in either the MS/MSD or DUP was >50 and the compliance code was changed to Rej.

Blank Contamination

A 3X rule was used for blank contamination. When field samples results were <3X the associated blank results a QACode of VRIP was applied to the samples and given a compliance code Rej. Field sample results >/= 3X the blank were given a QACode of VIP and compliance code remained Com.

Holding Times

Samples outside HT were given a QACode of H and a compliance code of Est.

Screening Results

Sample results that were screened as in the case of Aroclors were given a QACode of HT,SCR and a compliance code of Est

Missing QC elements
Batches where the required QC elements were not performed were given a BatchVer code of "VI" and a compliance code of Est.

Appendix VI: SFEI Procedures

SFEI Procedures			
Page	Procedure/Equipment	SOP Number	Revision Date
A	RMP Data Validation		

Appendix VI A: RMP Data Validation

Don's note: I think SWAMP/BOG have to write their own data validation rules, as their QAPP may be more specific about certain things that the RMP QAPP does not specify, e.g. needing failures on 2 of 3 recovery criteria to flag, etc. These internal applications of the checks for RMP were made based on historical data and hierarchies based on judgment of what measures were most important, since the RMP QAPP did not always specify (how bad things needed to be to censor, which measures took priority). In the future we are planning on revision of the RMP QAPP to be as specific as possible about everything we can think of, but until then a cleaned up variant of this will have to be a de facto SOP.

RMP Data Validation

Blank checks

1) Calculate Average of "Method Blank" grouped by LabBatch for various analytes (if all results are blank corrected, rather than average the blanks, we calculate and compare the standard deviation of the blank to its MDL and the field sample results)

1. Compare average of blank to its MDL, if AverageOfBlank < AverageOfMDL then no further action for that analyte is required
2. If AverageOfBlank is > MDL, then there is blank contamination. The next step will be to compare the field results to the blank results. Be sure that the blank results, MDLs, and field sample results are all in the same units and basis.
    a. If blank result is reported on a mass basis rather than a concentration (e.g ng rather than ng/g) then you will need to convert the blank to a concentration. If field samples are always the same size, convert the blank result to ng/g assuming the blank was the same size (e.g. if the sample mass was 2g then divide the blank result by 2g). This needs to be done even if the blank has no true mass like field samples. If the field sample sizes are variable – then determine the lowest field sample size and use this to convert blank mass to a concentration. Be sure to scale MDLs, RLs, as well, using the same method above, to get agreeing values and units.
    b. Scaling MDLs: If field sample sizes vary (e.g. 10g wet sediment samples might range 2-8g dry weight, with results reported in dry weight concentration) then you will need to scale blank value vs each individual sample. Often this can be done by scaling the MDL, if individual result MDLs are scaled to sample size; Generally labs develop MDL on a per analysis basis, i.e. X ng in an extract, regardless of what original sample mass that extract represents. If the sample has Y ng of analyte, for a sample with sample weight (WS), Y/WS is the concentration, X/WS is the sample specific MDL. Because the blank often has no true weight, it is often assigned an arbitrary blank weight (WB). The blank extract, with Z ng of analyte, will then be reported as concentration Z/WB, with MDL of X/WB.
        i. If WB=WS, blank MDL = sample MDL, and no scaling is needed, compare blank and sample results directly.
        ii. If they are not equal, the concentration reported for the blank (Z/WB) must be scaled for the specific sample of weight WS. Since the sample weight used in analysis is generally NOT reported in the LabResults, we scale the blank using the values we

DO have reported: sample concentration (Y/WS), sample MDL (X/WS), blank concentration (Z/WB) and blank MDL (X/WB). What we want to derive is Z/WS, the concentration that the blank would have been if it were exactly the same weight as the sample. We can do that from the 4 reported values with some algebra, let us know if you need the equation written out.

c. If the field result (in ng/g) <3*AverageOfBlank (scaled) then flag field sample with VRIP, (censored result- blank is likely too large a component of field result to be quantitative), superceding any existing IP. (We discussed in yesterday's phone call using 5*AverageOfBlank – this would be more conservative and would result in more rejected data – SWAMP should make the decision on using 3* or 5*).
d. If field result (in ng/g) >3*AverageOfBlank flag field sample with VIP if not already IP flagged.

Accuracy check

Keep in mind that if the BOG QAPP specifies other range and/or failure requirements (e.g. BOTH CRMs and MSs must fail to get results flagged/censored), BOG should follow its stated requirements.

In RMP we use a hierarchy for accuracy checks,

SRM > MS > BlankSp

Table 1. (Table 11 from BOG QAPP) shows BOG Data Quality Objectives

Parameter	Accuracy	Precision	Recovery	Completeness	Sensitivity
Synthetic Organics (including PCBs, pesticides, and PBDEs)	Certified Reference Materials (CRM, PT) within 95% CI stated by provider of material. If not available then within 50% to 150% of true value	Duplicate RPD ± 25%	Matrix spike 50% - 150% or control limits at ± 3 standard deviations based on actual lab data	90%	See Tables 16a,b,c
Trace metals (including mercury)	CRM 75% to 125%	Duplicate RPD ± 25%	Matrix Spike 75% - 125%	90%	See Table 14

If there is a higher priority measure (e.g. SRM) within an acceptable range then the analyte passes or fails regardless of the outcome of the remaining measures (e.g. MS).

We average across LabBatches for a project submission (e.g. one year of data for one lab considered together). Moderate failure (> target range but <2x the range – see table 1 above) of the highest priority usable measure get a VIU assigned to each analyte that fails the test, a bad failure (>2x outside target range) gets VRIU, which gets applied to the field samples, indicating that this reporting batch (which may be several LabBatches) has suspect values for the given analyte.**

**(Using a linear 2x range may not make sense for an acceptance range of +/- 50% (e.g. organics matrix spikes), as that would mean accepting 0-200% recovery, you would never censor for low recovery unless negative recoveries (MS < Native?) are reportable. Otherwise you might use a geometric 2x range, (50%)^2 to (150%)^2, i.e. 25%-225% as a censoring threshold.)

We average among LabBatches for a reporting submission because there may be noise in the analysis, so if SRM recoveries are say 70%, 85%, 78%, 80%, there is no particular reason only the samples in the batch with the SRM at 70% might be suspect.

Any target measure (ExpectedValue) must be at least 3xMDL otherwise it doesn't count. Even with our hierarchy, if an ExpectedValue is > 10x the average field result, it falls in priority if the next highest priority measure (e.g. MS etc.) is <10x field result.

Matrix spikes also need to be a minimum of 2x the native (unspiked) sample, otherwise a lot of the error in recovery is only noise in the analytical measurement. Test this by calculating the ratio of the OriginalFieldResult/MSExpectedValue, if the ratio is <0.5 then ignore the recovery result and preferentially use the next best measurement (e.g. Blank spike, or SRM previously ignored for being >10x field samples).

After all is said and done, there will be some analytes that will have nothing to verify accuracy; their SRM values are not certified or are <3xMDL, the matrix spikes are <2x the field sample, and/or the analyte is not one of the compounds spiked in the matrix or blank spikes. We presume innocence until proven guilty, those analytes are left unflagged.

Precision check

In RMP again we have a hierarchy, if we have results for all/multiple:

Lab/field replicates > SRM ~ MS > BlankSpike. See table one above for benchmarks.

For lab/field replicates, derive averages for each sample. Generally we use lab duplicates, except for some matrices/analyte types, where due to sample size issues we can only get field replicates. Operationally that means we average by SampleID (= one sample jar = unique sample location, time, matrix, SampleReplicate). If there are no SampleIDs with more than one LabResult (e.g. lab replicate = 1) record for the analyte, then we change that condition to average by sample location, time, matrix only.

1) Check that for each SampleID the average of the replicates > 3xAverageOfMDL. If AverageOfResult >3xAverageOfMDL then include RPD or RSD in lab submission evaluation. If one result were ND and the second 100xMDL, the average result (assume the ND=0) is 50xMDL, you would have a serious problem, so it would be a mistake to ignore the precision of that (just because one result was ND). Repeat for SRMs, MSs, and any other samples with replicates.
    a. For MSs, since the ExpectedValues are often not exactly the same (due to slight variations in spiked amount, sample size), rather than set some threshold for defining the "same" expected value (e.g. how many decimal places), we just always calculate RPD/RSD on samples that are within

usable range (if MS ExpectedValue > 3xMDL, and Native/MS ExpectedValue < 0.5)***

2) Average across LabBatches for a project submission (e.g. one year of data considered together) for each SampleType that has replicates. Results that average <3xMDL for an analyte are ignored

3) Use the hierarchy for replicates to choose the SampleType for assigning QAcodes If there is a higher priority measure (e.g. lab/field duplicate) within an acceptable range then the analyte passes or fails regardless of the outcome of the remaining measures (e.g. SRM or MS). Similar to accuracy, we flag on a project/event/submission basis for one lab, as there will be some variation in average concentration and noise around the RPDs/RSDs- a higher RPD in one batch may just be a result of the sample chosen for that rep having lower concentration, or just the odds of heterogeneity in subsamples of a grab sample, etc. If on average there is often a problem with precision for an analyte, then we have a problem that should be noted/flagged.

4) If RPD or RSD for an analyte (averaged across batches for a lab submission, for the SampleType chosen for evaluation) is >25% AND <50% (>1x to <2x target range) apply VIL to that analyte for the entire submission. If RPD or RSD >50% (>2x range) then apply VRIL to that analyte for the entire lab submission.

*** SWAMP outlines a method for doing an MS/MSD RPD check by different methods depending on whether the spiking value is the same or different- if spiking value is different, RPD is calculated on %recovery, if spiking value is the same, RPD is calculated on the raw values. For RMP we just go for calculating on %recovery always (worst case scenario, avoids some problems of trying to judge when to go for one calculation method versus the other)- e.g. you have a parent sample with 5 ng/g, and you make an MS/MSD spiking 10ng each sample, which turn out to 0.9 and 1.0g- are the expected values the same or not? Depends on how much you round – one you expect a concentration of 5ng/g*1g = 5ng +10ng = 15ng in 1g (expectedvalue=15ng/g.) The other you get 5ng/g*0.9g = 4.5ng +10ng = 14.5ng in 0.9g = 16.1ng/g. What if the MS vs MSD is 0.95 vs 1.0 g mass? There will often be some inexactness in the MS/MSD masses and spiking volumes, always going by RPD on %recovery avoids those questions. Otherwise where is the cutoff where the ExpectedValue is considered equal? Within 1%? 2%? 5%?

Range check

This may not be possible with the lakes data as they are distinct water bodies, so expected concentrations my not fall within some predictable range like in SF Bay where conditions are similar and waters mix.

Nonetheless, differences in concentration of >100x within a species for similar water bodies might be something of a concern. What the review threshold should be is a bit of professional judgement. We generally contact the reporting lab to make sure if they stand by the number. Sometimes they fix the number (seeing matrix interference, ion ratio issues, peak drift, etc on closer examination of the raw data), other times they say it should remain as is. Then it is a matter of professional judgment whether we believe it, if not, we flag VQ or VRQ depending on whether we think the number is possible/realistic, or so far out of range that it should be censored.

Year to year variation of >10x for the same water body would be a bit alarming, for us a difference of even 2x for a station or a bay segment between years gets a bit concerning although not entirely out of the realm of possible. Again, it is up to the PI what threshold they want to look at. This check may not be relevant for the lakes data since there is only one year of data for most lakes studied, although literature/data for similar lakes previously studied may provide indication if results are several orders of magnitude off.

Inconsistencies

It may seem inconsistent that we group within LabBatch for blank checking and within collection event/submission for precision and accuracy, why not both by the same grouping? We had considered that, but often blank subtraction is done on a LabBatch basis, almost never do you get blank correction across batches, so for the uncorrected blanks it seemed the evaluation should also have to be within batch. On the other hand, for the recovery checks, results are often tracked across batches (e.g. on control charts) so it seemed that cross batch evaluation within the group of data analyzed for submission for an event/ at one time, would be more appropriate, more an indicator of general problems than the luck of the draw whether a particular field sample was analyzed in the LabBatch with a low recovery on SRM, or where the native sample used for the MS was high or low. Likewise the precision measurement, to flag on the basis of within LabBatch results only subjected results to too much chance dependent on the concentrations in the field sample that was randomly chosen for a replicate. If SWAMP feels otherwise they should flag on different grouping bases, hopefully documenting why they made those choices.

Appendix VII: Approval Signatures

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Attachment 1: Chain of Custody Forms

DFG REQUEST FOR ANALYSIS AND CHAIN OF CUSTODY RECORD Page ____ of ____

Fiscal Year: 08	Project ID: 08SWSBG1		Contact Person:		Phone:
Field Crew			Autumn Bonnema		831-771-4175
Mailing Address			Address		
7544 Sandholdt Road			7544 Sandholdt Road		
City	State	Zip	City	State	Zip
Moss Landing	CA	95039	Moss Landing	CA	95039
			email: bonnema@mlml.calstate.edu		

Sample Identification/Location			Analysis Requested		BOG Analyte List	Sample Type	# of Containers	Preservation
						Fish	Foil Wrap in plastic	Temp
StationCode	StationName	BagID	Collection Date					

Comments/Special Instructions

Samples Reliquished By (Signature)	Print Name and Date	Received By (Signature)	Print Name and Date

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

Fiscal Year: 08
Region:
Field Crew:

Project ID: SWAMP_SB_BOG
Season:
Date:

Contact Person: Autumn Bonnema
Phone: 831-771-4175
email: bonnema@mlml.calstate.edu
Mailing Address: 7544 Sandholdt Rd.
Moss Landing, CA 95039

Trip#	StationCode	Station Name	BagID	Species	Sample Date	Tissue Organics	Tissue THg*	Tissue Se*	# of Containers Foil wrap in Plastic	# of Containers 60ml Glass	Preservation Frozen
					TOTAL	0	0	0	0	0	0

Comments: Please see attached AA form for explicit instructions and data reporting format.
* Analysis will be perfomed by MPLS-DFG; dissect and send homogenate

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

Attachment 2: Field Data Sheets

SWAMP Tissue Sampling - Non-Trawl (Event Type = TI) BOG Coastal		Entered in d-base (initial/date)			Pg of Pgs
***StationCode:** __ __ __ __ __ __ __ __ __	*StationName:		*Trip:		**Agency:**
***FundingCode1:** __ __ __ __ __ __ __ __ __	*Date (mm/dd/yyyy): / /				
***FundingCode2:** __ __ __ __ __ __ __ __ __	ArrivalTime:	***Purpose** (circle all that apply): Tissue Habitat			*Purpose Failure Code:
*Sampling Crew:	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 Location1					
DOMINANTSUBSTRATE:	Concrete,Cobble,Gravel,Sand,Mud,Other________,unk				1: (RB / LB / BB / US / DS / ##)
OTHER PRESENCE:	Foam, OilySheen, None, Trash, MacroAlgae, Other___________				
Comments:					2: (RB / LB / BB / US / DS / ##)
					3: (RB / LB / BB / US / DS / ##)

OCCUPATIONMETHOD: Boat (RV___________________________), Walk-In			GPS Model:			accuracy	Datum: **NAD83** Other _________		
Location:	OpenWater/Bank/MidChan	#_____	*StationDepth (m):	DistanceFromBank(m):	Coord	(ft / m)	Lat (dd.ddddd)	Long (-ddd.ddddd)	Depth (m)
COLLECTION METHOD:	Hook, Net, Seine, Spear, Trap, Shock			Start Time	1				
COLLECTIONDEVICE:	Hook/Line, Gill Net (mesh size)________, CastNet, Seine, 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				
Location:	OpenWater/Bank/MidChan	#_____	*StationDepth (m):	DistanceFromBank(m):	Coord	(ft / m)	Lat (dd.ddddd)	Long (-ddd.ddddd)	Depth (m)
COLLECTION METHOD:	Hook, Net, Seine, Spear, Trap, Shock			Start Time	1				
COLLECTIONDEVICE:	Hook/Line, Gill Net (mesh size)________, CastNet, Seine, 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				
Location:	OpenWater/Bank/MidChan	#_____	*StationDepth (m):	DistanceFromBank(m):	Coord	(ft / m)	Lat (dd.ddddd)	Long (-ddd.ddddd)	Depth (m)
COLLECTION METHOD:	Hook, Net, Seine, Spear, Trap, Shock			Start Time	1				
COLLECTIONDEVICE:	Hook/Line, Gill Net (mesh size)________, CastNet, Seine, 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				
Location:	OpenWater/Bank/MidChan	#_____	*StationDepth (m):	DistanceFromBank(m):	Coord	(ft / m)	Lat (dd.ddddd)	Long (-ddd.ddddd)	Depth (m)
COLLECTION METHOD:	Hook, Net, Seine, Spear, Trap, Shock			Start Time	1				
COLLECTIONDEVICE:	Hook/Line, Gill Net (mesh size)________, CastNet, Seine, 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				

Failure Codes: Dry (no water), Instrument Failure, No Access, Non-sampleable, Pre-abandoned, Other

Collection Comments:

Modified9/24/2009

SWAMP Tissue Sampling - Trawl (Event Type = TI) BOG Coastal

Entered in d-base (initial/date) | Pg of Pgs

*StationCode: ___ ___ ___ ___ ___ ___ ___ ___ ___ ___	*StationName:		*Trip:		*Purpose Failure Code:	Agency
*FundingCode1: ___ ___ ___ ___ ___ ___ ___ ___	*Date (mm/dd/yyyy): / / 2009					
*FundingCode2: ___ ___ ___ ___ ___ ___ ___ ___	ArrivalTime:	*Purpose (circle all that apply): Tissue Habitat				
*Sampling Crew:	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 Location1						
DOMINANTSUBSTRATE:	Concrete,Cobble,Gravel,Sand,Mud,Other________,unk					1: (RB / LB / BB / US / DS / ##)
OTHER PRESENCE:	Foam, OilySheen, None, Trash, MacroAlgae, Other__________					
Comments:						2: (RB / LB / BB / US / DS / ##)
						3: (RB / LB / BB / US / DS / ##)

Tissue Collection (MethodCode: Trawl)

OCCUPATIONMETHOD: Boat RV______________________	*GPS/DGPS	Lat (dd.ddddd)	Long (-ddd.ddddd)
COLLECTION DEVICE: MPSL-DFG_OtterTrawl, other _________________________	Target:	NA	NA
	GPS Model:		
GEOSHAPE: Line / Point	Datum: NAD83 WGS84 Other ______		

Location	#	Start Time	Latitude (dd.ddddd)	Longitude (-ddd.ddddd)	Depth (m)	WireOut (m)	End Time	Latitude (dd.ddddd)	Longitude (-ddd.ddddd)	Accuracy (ft / m)
OpenWat/ Bank/ MidChan	1									
StationWaterDepth(m):		DistanceFromBank(m):		HydroMod: None, Bridge, Pipes, Pier, Breakwater, Other __________				HydroModLoc: US / DS / WithIn		
OpenWat/ Bank/ MidChan										
StationWaterDepth(m):		DistanceFromBank(m):		HydroMod: None, Bridge, Pipes, Pier, Breakwater, Other __________				HydroModLoc: US / DS / WithIn		
OpenWat/ Bank/ MidChan										
StationWaterDepth(m):		DistanceFromBank(m):		HydroMod: None, Bridge, Pipes, Pier, Breakwater, Other __________				HydroModLoc: US / DS / WithIn		
OpenWat/ Bank/ MidChan										
StationWaterDepth(m):		DistanceFromBank(m):		HydroMod: None, Bridge, Pipes, Pier, Breakwater, Other __________				HydroModLoc: US / DS / WithIn		
OpenWat/ Bank/ MidChan										
StationWaterDepth(m):		DistanceFromBank(m):		HydroMod: None, Bridge, Pipes, Pier, Breakwater, Other __________				HydroModLoc: US / DS / WithIn		
OpenWat/ Bank/ MidChan										
StationWaterDepth(m):		DistanceFromBank(m):		HydroMod: None, Bridge, Pipes, Pier, Breakwater, Other __________				HydroModLoc: US / DS / WithIn		
OpenWat/ Bank/ MidChan										
StationWaterDepth(m):		DistanceFromBank(m):		HydroMod: None, Bridge, Pipes, Pier, Breakwater, Other __________				HydroModLoc: US / DS / WithIn		
OpenWat/ Bank/ MidChan										
StationWaterDepth(m):		DistanceFromBank(m):		HydroMod: None, Bridge, Pipes, Pier, Breakwater, Other __________				HydroModLoc: US / DS / WithIn		

Comments: Failure Codes: Dry (no water), No Access, Non-sampleable, Other

Modified 5/8/2009

SWAMP Tissue Sampling - Non-Trawl (Event Type = TI) Continued		Entered in d-base (initial/date)		Pg of Pgs
*StationCode: ___ ___ ___ ___ ___ ___ ___ ___ ___ ___	*StationName:	Trip:		Agency
*FundingCode: ___ ___ ___ ___ ___ ___ ___ ___ ___	*Date (mm/dd/yyyy): / /			

Tissue Collection

Location:	OpenWater/Bank/MidChan	#_____	*StationDepth (m):	DistanceFromBank(m):	Coord	Accuracy (ft / m)	Latitude (dd.ddddd)	Longitude (-ddd.ddddd)	Depth (m)
COLLECTION METHOD:	Hook, Net, Seine, Spear, Trap, Shock			Start Time	1				
COLLECTIONDEVICE:	Hook/Line, Gill Net (mesh size)________, CastNet, Seine, 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				
Location:	OpenWater/Bank/MidChan	#_____	*StationDepth (m):	DistanceFromBank(m):	Coord	(ft / m)	Latitude (dd.ddddd)	Longitude (-ddd.ddddd)	Depth (m)
COLLECTION METHOD:	Hook, Net, Seine, Spear, Trap, Shock			Start Time	1				
COLLECTIONDEVICE:	Hook/Line, Gill Net (mesh size)________, CastNet, Seine, 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				
Location:	OpenWater/Bank/MidChan	#_____	*StationDepth (m):	DistanceFromBank(m):	Coord	(ft / m)	Latitude (dd.ddddd)	Longitude (-ddd.ddddd)	Depth (m)
COLLECTION METHOD:	Hook, Net, Seine, Spear, Trap, Shock			Start Time	1				
COLLECTIONDEVICE:	Hook/Line, Gill Net (mesh size)________, CastNet, Seine, 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				
Location:	OpenWater/Bank/MidChan	#_____	*StationDepth (m):	DistanceFromBank(m):	Coord	(ft / m)	Latitude (dd.ddddd)	Longitude (-ddd.ddddd)	Depth (m)
COLLECTION METHOD:	Hook, Net, Seine, Spear, Trap, Shock			Start Time	1				
COLLECTIONDEVICE:	Hook/Line, Gill Net (mesh size)________, CastNet, Seine, 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				
Location:	OpenWater/Bank/MidChan	#_____	*StationDepth (m):	DistanceFromBank(m):	Coord	(ft / m)	Latitude (dd.ddddd)	Longitude (-ddd.ddddd)	Depth (m)
COLLECTION METHOD:	Hook, Net, Seine, Spear, Trap, Shock			Start Time	1				
COLLECTIONDEVICE:	Hook/Line, Gill Net (mesh size)________, CastNet, Seine, 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				
Location:	OpenWater/Bank/MidChan	#_____	*StationDepth (m):	DistanceFromBank(m):	Coord	(ft / m)	Latitude (dd.ddddd)	Longitude (-ddd.ddddd)	Depth (m)
COLLECTION METHOD:	Hook, Net, Seine, Spear, Trap, Shock			Start Time	1				
COLLECTIONDEVICE:	Hook/Line, Gill Net (mesh size)________, CastNet, Seine, 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				

Comments:

Modified 9/24/2009

SWAMP Tissue Sampling - Fish Abundance			Entered in d-base (initial/date)						Pg: of Pgs			
*StationCode: __ __ __ __ __ __ __ __ __ __			StationName:						Date (mm/dd/yyyy): / /			
Storage	Location/ Collection Method #	Organism ID	Tag #	Species Name/Code	TL (mm)	FL (mm)	Weight (g)	Count	Sex	Range (mm)	Count Est.	Anom	Condition
									M F Unk LAB				D W L
									M F Unk LAB				D W L
									M F Unk LAB				D W L
									M F Unk LAB				D W L
									M F Unk LAB				D W L
									M F Unk LAB				D W L
									M F Unk LAB				D W L
									M F Unk LAB				D W L
									M F Unk LAB				D W L
									M F Unk LAB				D W L
									M F Unk LAB				D W L
									M F Unk LAB				D W L
									M F Unk LAB				D W L
									M F Unk LAB				D W L
									M F Unk LAB				D W L
									M F Unk LAB				D W L
									M F Unk LAB				D W L
									M F Unk LAB				D W L
									M F Unk LAB				D W L
									M F Unk LAB				D W L
									M F Unk LAB				D W L
									M F Unk LAB				D W L
									M F Unk LAB				D W L
									M F Unk LAB				D W L
									M F Unk LAB				D W L
									M F Unk LAB				D W L

Location/CollectionMethod #: Match fish with LocationName, Collection Method, and # from Tissue Collection sheet
Organism ID: Combine BAG # and FISH # (e.g., fish 1 of composite WC01 is WC01-1) to be unique **Tag #:** Use if applicable; must be unique **Species Code**: see attached list for codes
Stage: Adult (A), Juvenile (J), Subadult (SA), Not Recorded (NR) **Count Est**: If appropriate, add < or > if count is estimated **Condition:** Note whether individual is Dead (D), Weak (W), or Live (L)
Anomalies: Fin Erosion (**FinEro**), Gill Erosion (**GillEro**), Lesion (**Les**), Parasite (**Par**), Popeye (**PE**), Tumor (**Tum**), Hemmorage (**Hem**), Skeletal Deformity (**SkDef**)

Comments: BOG_Coastal 1) Bag Numbers must be consecutive by previous trips per StationCode, 2) Smaller fish that are bagged by species but not tagged must be bagged by location #.

Modified 9/24/2009

Coastal Fish Species Code List Author Gary Ichikawa 9/24/2009

Coastal Fish List	Code
Barred sand bass	BSB
Barred surfperch	BRS
Bat ray	OBR
Black & yellow rockfish	BYR
Black croaker	BKC
Black surfperch	BLS
Black rockfish	BLR
Blacksmith	BKS
Blue rockfish	BUR
Bocaccio	BOC
Bonefish	OBF
Brown rock crab	BRC
Brown rockfish	BRR
Brown smoothhound	BSH
Calico surfperch	CSP
California corbina	OCC
California halibut	OCH
California lizardfish	CLF
California sheephead	CSH
Canary rockfish	CNR
Chilipepper rockfish	CPR
China rockfish	CHR
Chinook salmon	CHS
Coho salmon	COS
Copper rockfish	CPR
Diamond turbot	ODT
Dungeness crab	ODC
Dwarf surfperch	DWS
English sole	OES
Fantail sole	OFS
Gaper clam	OGC
Gopher rockfish	OGR
Grass rockfish	GRR
Grass shrimp	OGS
Greenstriped rockfish	GSR
Grey smoothhound Shark	GSS
Halfmoon	HFM
Horseneck clam	HNC

Coastal Fish List	Code
Jack mackerel	OJM
Jack smelt	OJS
Kelp bass	OKB
Kelp greenling	OKG
Kelp rockfish	KPR
Kelp perch	OKP
Leopard shark	OLS
Lingcod	OLC
Littleneck clam	LNC
Longfin sanddab	LFS
Northern Anchovy	ONA
Pacific halibut	OPH
Chub mackerel	OCM
Ocean whitefish	OWF
Olive rockfish	OLR
Opaleye	OPE
Pacific angel shark	PAS
Pacific hake	PCH
Pacific sandad	PSD
Pacific sardine	PSR
Pile surfperch	PSP
Queenfish	QUF
Quillback rockfish	QBR
Rainbow surfperch	RBS
Red rock crab	RRC
Redtail surfperch	RSP
Reef surfperch	RFS
Rosethorn rockfish	RTR
Round stingray	ORS
Rubberlip surfperch	RLS
Sargo	SAR
Shiner surfperch	SHS
Shovelnose guitarfish	SGF
Silver surfperch	SSP
Speckled sanddab	SSD
Spiny dogfish	SDF
Splitnose rockfish	SPN
Spotfin croaker	SFC

Coastal Fish List	Code
Spotfin surfperch	SFS
Spotted sand bass	SSB
Spotted scorpionfish	SSF
Spotted turbot	STR
Starry flounder	OSF
Striped bass	STB
Striped mullet	OSM
Striped surfperch	STS
Tapes clam	OTC
Top smelt	TPS
Vermillion rockfish	OVR
Walleye surfperch	WSP
White croaker	OWC
White Sturgeon	WST
White surfperch	WHS
Wolf eel	OWE
Yellow rockcrab	YRC
Yellowfin croaker	OYC
Yellowtail rockfish	YTR

New Species	Code

CollectionDeviceName	Datasheet
MPSL-DFG_CastNet_Bait	Bait
MPSL-DFG_CastNet_Mullet	Mullet
MPSL-DFG_GillNet_1ComboPanel	1
MPSL-DFG_GillNet_2ComboPanel	2
MPSL-DFG_GillNet_3(100m, 8.5")	3
MPSL-DFG_GillNet_4ComboPanel	4
MPSL-DFG_GillNet_5(100m, 6.0")	5
MPSL-DFG_GillNet_6(100m, 3.75")	6
MPSL-DFG_OtterTrawl_12	12
MPSL-DFG_OtterTrawl_16	16
MPSL-DFG_PoleSpears	Spears

Notes to Standardize SWAMP Field Data Sheets (For in the field use)

Key Reminders to identify samples:

1. **Sample Time** is the SAME for all samples (Water, Sediment, & Probe) taken at the sampling event. Use time of FIRST sample; important for COC.
2. **Group;** many diffrent ways to do a group, one suggestion is to create groups which assign trips to assess frequency of field QA

Collection Details

1. **Personnel**: S. Mundell, G Ichikawa (first person listed is crew leader)
2. **Location:** Use "openwater" in bay/estuary/harbor only if no distinguishable channel exists
3. **GRAB vs INTEGRATED**: GRAB samples are when bottles are filled from a single depth; INTEGRATED sample are taken from MULTIPLE depths and combined.
   a. GRAB: use 0.1 for subsurface samples; if too shallow to submerge bottle; depth =0
   b. INTEGRATED: -88 in depth sampled, record depths combined in sample comments
4. **TARGET LAT/LONG**: Refers to the existing station location that the sampling crew is trying to achieve; can be filled out prior to sampling
5. **ACTUAL LAT/ LONG**: is the location of the current sample event.
6. **HYDROMODIFICATION:** Describe existing hydromodifications such as a grade control, drainage pipes, bridge, culvert
7. **HYDROMOD LOC**: if there is an IMMEDIATE (with in range potentially effecting sample) hydromodification; Is the hydromodification upstream/downstream/within area of sample; if there is no hydromodification, NA is appropriate
8. **STREAM WIDTH and DEPTH**: describe in meters at point of sample.

FIELD OBSERVATIONS: (each one of these observations has a comment field in the database so use comment space on data sheet to add information about an observation if necessary)

1. **PICTURES:** use space to record picture numbers given by camera; be sure to rename accordingly back in the office. (StationCode_yyyy_mm_dd_uniquecode)
2. **WADEABILITY**: in general, is waterbody being sampled wadeable to the average person AT the POINT of SAMPLE
3. **DOMINANT SUBSTRATE**: if possible; describe DOMINANT substrate type; use UNK if you cannot see the dominant substrate type
4. **BEAUFORT SCALE**: use scale 0-12; refer to scales listed below.
5. **WIND DIRECTION**: records the direction from which the wind is blowing
6. **OTHER PRESENCE**: VASCULAR refers to terrestrial plants or submerged aquatic vegetation (SAV) and NONVASCULAR refers to plankton, periphyton etc. These definitions apply to vegetation IN the water at the immediate sampling area.
7. **OBSERVED FLOW**: Visual estimates in cubic feet/ second.
8. **WATER COLOR**: This is the color of the water from standing creek side
9. **WATER CLARITY**: this describes the clarity of the water while standing creek side; clear represents water that is clear to the bottom, cloudy may not be clear to bottom but greater than 4" can be seen through the water column.
10. **SedimentComp**: generally described sediments used for chemistry sample

Note: these reminders do not give all details needed to maintain equivalent SWAMP sampling protocols, they are strictly for "infield" use to help insure comparability of field observations.

BEAUFORT SCALE: Specifications and equivalent speeds for use at sea

FORCE	EQUIVALENT SPEED 10 m above ground		DESCRIPTION	SPECIFICATIONS FOR USE AT SEA
	miles/hour	knots		
0	0-1	0-1	Calm	Sea like a mirror
1	1-3	1-3	Light air	Ripples with the appearance of scales are formed, but without foam crests.
2	4-7	4-6	Light breeze	Small wavelets, still short, but more pronounced. Crests have a glassy appearance and do not break.
3	8-12	7-10	Gentle breeze	Large wavelets. Crests begin to break. Foam of glassy appearance. Perhaps scattered white horses.
4	13-18	11-16	Moderate breeze	Small waves, becoming larger; fairly frequent white horses.
5	19-24	17-21	Fresh breeze	Moderate waves, taking a more pronounced long form; many white horses are formed. Chance of some spray.
6	25-31	22-27	Strong breeze	Large waves begin to form; the white foam crests are more extensive everywhere. Probably some spray.
7	32-38	28-33	Near gale	Sea heaps up and white foam from breaking waves begins to be blown in streaks along the direction of the wind.
8	39-46	34-40	Gale	Moderately high waves of greater length; edges of crests begin to breakinto spindrift. The foam is blown in well-marked streaks along the direction of the wind.
9	47-54	41-47	Severe gale	High waves. Dense streaks of foam along the direction of the wind. Crests of waves begin to topple, tumble and roll over. Spray may affect visibility.
10	55-63	48-55	Storm	Very high waves with long over-hanging crests. The resulting foam, in great patches, is blown in dense white streaks along the direction of the wind. On the whole the surface of the sea takes on a white appearance. The 'tumbling' of the sea

Source:

Last edited on 09 January, 1999 Dave Wheeler weatherman@zetnet.co.uk
Web Space kindly provided by Zetnet Services Ltd, Lerwick, Shetland.

heavy and shock-like. Visibility affected.

BEAUFORT SCALE: Specifications and equivalent speeds for use on land

FORCE	EQUIVALENT SPEED 10 m above ground		DESCRIPTION	SPECIFICATIONS FOR USE ON LAND
	miles/hour	knots		
0	0-1	0-1	Calm	Calm; smoke rises verticall.
1	1-3	1-3	Light air	Direction of wind shown by smoke drift, but not by wind vanes
2	4-7	4-6	Light Breeze	Wind felt on face; leaves rustle; ordinary vanes moved by wind
3	8-12	7-10	Gentle Breeze	Leaves and small twigs in constant motion; wind extends light flag
4	13-18	11-16	Moderate Breeze	Raises dust and loose paper; small branches are moved.
5	19-24	17-12	Fresh Breeze	Small trees in leaf begin to sway crested wavelets form on inland waters
6	25-31	22-27	Strong Breeze	Large branches in motion; whistling heard in telegraph wires umbrellas used with difficulty
7	32-38	28-33	Neargale	Whole trees in motion; inconvenience felt when walking against the wind
8	39-46	34-40	Gale	Breaks Twigs and generally impedes progress

Source:

Last edited on 09 January, 1999 Dave Wheeler weatherman@zetnet.co.uk
Web Space kindly provided by Zetnet Services Ltd, Lerwick, Shetland.

Attachment 3: Analysis Authorization Forms

Analysis Authorization
Fiscal Year: 08/09
Region:

Project ID: SWAMP_SB_BOG
Season:
Date:

Contact Person: Autumn Bonnema
Phone: 831-771-4175
email: bonnema@mlml.calstate.edu
Mailing Address: 7544 Sandholdt Road
Moss Landing, CA 95039

				Dissect and Analyze Tissue Flesh						Dissect and Send to WPCL Tissue Flesh					Archive	Archive
Trip	Station	SpeciesCode	CompositeIDText	Hg	Comp Hg	Comp Se	%Moisture		Weight/Sex	OC	PCB	PBDE	%Moisture	%Lipid	Individuals	Comp
Total				0	0	0	0		0	0	0	0	0	0	0	0

Analysis Authorization
Fiscal Year: 08/09
Region:

Project ID: SWAMP_SB_BOG
Season:
Date:

Contact Person: Autumn Bonnema
Phone: 831-771-4175
email: bonnema@mlml.calstate.edu
Mailing Address: 7544 Sandholdt Road
Moss Landing, CA 95039

				Dissect and Analyze						Dissect and Send to MPSL				
				Tissue Flesh						Tissue Flesh			Archive	
Trip	Station	SpeciesCode	CompositeIDText	OC	PCB	PBDE	%Moisture	%Lipid	Weight/Sex	Ind Hg	Comp Hg	Comp Se	Individuals	Location Comp
Total				0	0	0	0	0	0	0	0	0	0	0

Attachment 4: Laboratory Data Sheets

SWAMP Lab Data Sheet - FISH	ProjectID: SWAMP_SB_BOG	PrepPres:	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 / L / O		
2									M / F / Unk	T / L / O		
3									M / F / Unk	T / L / O		
4									M / F / Unk	T / L / O		
5									M / F / Unk	T / L / O		
6									M / F / Unk	T / L / O		
7									M / F / Unk	T / L / O		
8									M / F / Unk	T / L / O		
9									M / F / Unk	T / L / O		
10									M / F / Unk	T / L / O		
11									M / F / Unk	T / L / O		
12									M / F / Unk	T / L / O		
13									M / F / Unk	T / L / O		
14									M / F / Unk	T / L / O		
15									M / F / Unk	T / L / O		
16									M / F / Unk	T / L / O		
17									M / F / Unk	T / L / O		
18									M / F / Unk	T / L / O		
19									M / F / Unk	T / L / O		
20									M / F / Unk	T / L / O		
21									M / F / Unk	T / L / O		
22									M / F / Unk	T / L / O		
23									M / F / Unk	T / L / O		
24									M / F / Unk	T / L / O		
25									M / F / Unk	T / L / O		

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

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

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

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

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

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

Modified 06/08/07

SWAMP Lab Data Sheet - FISH	ProjectID: SWAMP_SB_BOG	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

Composite/Individual ID:	**Composite/Individual ID**:	**Composite/Individual ID**:
Analysis: Mercury Organics Archive	**Analysis:** Mercury Organics Archive	**Analysis:** Mercury Organics Archive
Jar Weight Full (g):	Jar Weight Full (g):	Jar Weight Full (g):
Jar Weight Empty (g):	Jar Weight Empty (g):	Jar Weight Empty (g):
Comp Tissue Wt (Jar Full - Empty; g):	Comp Tissue Wt (Jar Full - Empty; g):	Comp Tissue Wt (Jar Full - Empty; g):
Duplicate: Yes / No **DUP ID:**	**Dup**: Yes / No **DUP ID:**	**Duplicate**: Yes / No **DUP ID:**
Composite/Individual ID:	**Composite/Individual ID**:	**Composite/Individual ID**:
Analysis: Mercury Organics Archive	**Analysis:** Mercury Organics Archive	**Analysis:** Mercury Organics Archive
Jar Weight Full (g):	Jar Weight Full (g):	Jar Weight Full (g):
Jar Weight Empty (g):	Jar Weight Empty (g):	Jar Weight Empty (g):
Comp Tissue Wt (Jar Full - Empty; g):	Comp Tissue Wt (Jar Full - Empty; g):	Comp Tissue Wt (Jar Full - Empty; g):
Duplicate: Yes / No **DUP ID:**	**Dup**: Yes / No **DUP ID:**	**Duplicate**: Yes / No **DUP ID:**
Composite/Individual ID:	**Composite/Individual ID**:	**Composite/Individual ID**:
Analysis: Mercury Organics Archive	**Analysis:** Mercury Organics Archive	**Analysis:** Mercury Organics Archive
Jar Weight Full (g):	Jar Weight Full (g):	Jar Weight Full (g):
Jar Weight Empty (g):	Jar Weight Empty (g):	Jar Weight Empty (g):
Comp Tissue Wt (Jar Full - Empty; g):	Comp Tissue Wt (Jar Full - Empty; g):	Comp Tissue Wt (Jar Full - Empty; g):
Duplicate: Yes / No **DUP ID:**	**Dup**: Yes / No **DUP ID:**	**Duplicate**: Yes / No **DUP ID:**
Composite/Individual ID:	**Composite/Individual ID**:	**Composite/Individual ID**:
Analysis: Mercury Organics Archive	**Analysis:** Mercury Organics Archive	**Analysis:** Mercury Organics Archive
Jar Weight Full (g):	Jar Weight Full (g):	Jar Weight Full (g):
Jar Weight Empty (g):	Jar Weight Empty (g):	Jar Weight Empty (g):
Comp Tissue Wt (Jar Full - Empty; g):	Comp Tissue Wt (Jar Full - Empty; g):	Comp Tissue Wt (Jar Full - Empty; g):
Duplicate: Yes / No **DUP ID:**	**Dup**: Yes / No **DUP ID:**	**Duplicate**: Yes / No **DUP ID:**
Composite/Individual ID:	**Composite/Individual ID**:	**Composite/Individual ID**:
Analysis: Mercury Organics Archive	**Analysis:** Mercury Organics Archive	**Analysis:** Mercury Organics Archive
Jar Weight Full (g):	Jar Weight Full (g):	Jar Weight Full (g):
Jar Weight Empty (g):	Jar Weight Empty (g):	Jar Weight Empty (g):
Comp Tissue Wt (Jar Full - Empty; g):	Comp Tissue Wt (Jar Full - Empty; g):	Comp Tissue Wt (Jar Full - Empty; g):
Duplicate: Yes / No **DUP ID:**	**Dup**: Yes / No **DUP ID:**	**Duplicate**: Yes / No **DUP ID:**

Comments: Keep archive tissue if possible; If a duplicate is made, use Dup ID as identification for analysis

Modified 06/08/07