

# **Sampling and Analysis Plan for Sport Fish in Artesian Slough**

Approved by RMP Lead Scientist on 7/12/15  
Revised 8/21/15

## **Introduction**

The Regional Monitoring Program for Water Quality in San Francisco Bay (RMP) monitors concentrations of contaminants in sport fish tissue as an indicator of water quality impairment. In 2014, the RMP collected sport fish samples from various locations in the Bay as part of routine Status and Trends Monitoring.

The San Jose-Santa Clara Regional Wastewater Facility (SJSCRWF) discharges treated effluent to Artesian Slough in San Jose, CA. SJSCRWF monitors fish populations in Artesian Slough through a contract with UC Davis.

For this study, the RMP and SJSCRWF are partnering to analyze contaminants in fish that UC Davis collects from Artesian Slough using the methods for the RMP Status and Trends monitoring. The fish will be collected during the 2015 summer season. The target species will be striped bass, largemouth bass, and carp. The samples will be processed by staff at the Marine Pollution Studies Lab at Moss Landing Marine Laboratory (MPSL) and then analyzed for mercury (at MPSL), PCBs (at the California Department of Fish & Wildlife's Water Pollution Control Laboratory [WPCL]), PBDEs (at WPCL), and PFCs (at AXYS Analytical Laboratories). The field, laboratory, and quality assurance methods used will conform to the RMP Quality Assurance Project Plan (SFEI, 2014). The results of this study will be included in the sport fish monitoring final report that will be prepared by the RMP by the end of 2015.

The purpose of this Sampling and Analysis Plan is to clearly outline the sampling design, methods, and archiving strategy to make it easy for project partners to coordinate.

## **Sampling Design**

Three species of fish will be collected from Artesian Slough according to the following sampling design:

**Table 1: Artesian Slough Fish Sampling Design**

<b>Species</b>	<b>Striped Bass</b>	<b>Largemouth Bass</b>	<b>Carp<sup>1</sup></b>
Target size range (cm, total length)	45-59 cm (9 fish) 60-82 cm (9 fish)	20-24.9 cm (2 fish) 25-30.4 cm (2 fish) 30.5-40.7 cm (6 fish) >40.7 cm (2 fish)	45-60 cm
Target # Fish	18	12	9
Target # fish per composite <sup>2</sup>	3	5 <sup>3</sup>	3
<b>Number of fish caught as of 8/21/15</b>	<b>8</b>	<b>12</b>	<b>1</b>
# of Composites <sup>4</sup>	6	1 <sup>3</sup>	3
Tissue analyzed	Muscle without skin	Muscle without skin	Muscle without skin

1 -- PFC analyses will be conducted on these samples. Avoid using any Teflon coated equipment when handling these fish.

2 -- Composites will include equal masses from each individual fish. Composites will be formed from fish within the same size class, and the length of the smallest fish in the composite should be no less than 75% of the length of the largest fish.

3 -- Only one composite composed of samples from 5 individual fish from the 30.5-40.7 cm size range will be analyzed for PCBs in largemouth bass. Samples from each of 12 individual fish will be processed for mercury analysis. See Table 2 for analytical plan details.

4 -- Composite samples will be analyzed as follows: striped bass - PCBs; largemouth bass - PCBs; carp - PCBs (2 composites only), PBDEs, and PFCs. Percent moisture and percent lipid will be measured in all composites. Individual striped bass and largemouth bass samples will be analyzed for percent moisture and mercury.

### **Sampling Methods**

Fish will be collected by UC Davis using gill nets and hook-and-line sampling.

The coordinates of the actual sampling site will be determined using a hand-held or shipboard global positioning system (GPS) and reported on field sheets provided by SFEI. Other pertinent information will also be recorded, including the sampling method, device, depth, and descriptive location.

At the dock, the fish will be placed on a measuring board covered with a clean plastic bag. Fork length, total length, and weight will be measured and recorded on field sheets provided by SFEI.

An AWS brand AMW-DISC digital pocket scale, or similar, will be used to measure fish weights in the field and will be calibrated in the lab with standard weights. Length measurements will be conducted on a fish measuring board that does not require calibration. No other field measurements are being measured.

When possible, sex, parasites, and body anomalies will be noted on the field sheet.

### **Sample Handling and Custody**

Each fish will be tagged with a unique ID, individually wrapped whole in aluminum foil, placed in a clean labeled bag, and then frozen on dry ice immediately. While on dry ice, the UC Davis field staff and SJSCRWF staff will check on the samples periodically to ensure that they are appropriately protected and there is sufficient dry ice.

After each sampling date, SJSCRWF staff will transport the samples on dry ice to a -20C storage freezer at SJSCRWF.

Once all the samples for the study have been collected, SJSCRWF will arrange for a courier to transport the samples on dry ice to MPSL. The samples will then be stored at -20C until dissection and homogenization. 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. The study is planned to be completed well before the 12 month hold time.

All samples will be accompanied by a chain of custody form (COC) (provided by SFEI). The COC form will include the sample unique ID, site name, collection date, sample type, analysis required, and other remarks. Shipping information is provided in Attachment 2.

Field sheets will also be sent by the field staff to MPSL for digitization and entry into SWAMP database format. MPSL will provide SFEI with a digital record of the field sheets and collections information at the end of the study.

### **Sample Processing**

MPSL will process the samples to create the individual and composite samples for laboratory analyses. Each individual fish sample will be analyzed for percent moisture, and each composite fish sample analyzed for organics will be analyzed for percent lipids. Mercury analyses will be conducted on samples of individual fish, while analyses of organic compounds will be conducted on composite samples. The number of composites or individuals to be analyzed for each parameter, and the number of fish per composite, are listed below in Table 2. All samples will be processed as muscle fillets with the skin off. MPSL will dissect and homogenize fish tissue samples according to method MPSL-105 (SWAMP 2014).

Composites will include equal masses from each individual fish in the composite. Composites will be formed from fish within the same size class, and the length of the smallest fish in the composite should be no less than 75% of the length of the largest fish.

**Table 2: Artesian Slough - Reportable Parameters**

Revised based on number of fish caught as of 8/21/15

Species	Number of fish per composite	Number of Samples (composites or individuals) <sup>1</sup>			
		PCBs (comp.)	PBDEs (comp.)	Hg (indiv.)	PFCs (comp.)
Striped bass	3	3	3	8	3
Largemouth Bass	5	1	1	12	1
Carp	3	1	1		1
Analytical Lab	--	WPCL	WPCL	MPSL	AXYS Analytical Labs
Reporting Units	--	ng/g ww	ng/g ww	ug/g ww	ng/g ww
Sample Container	--	60 mL glass jar, class 200 or 300	60 mL glass jar, class 200 or 300	pre-cleaned 60 mL glass or 30 mL PP jar	pre-cleaned 30 mL PP jar

1 -- Percent moisture and percent lipid will be measured in all composites analyzed for organic compounds. Individual samples will be analyzed for percent moisture.

For samples that are to be analyzed by PFCs, the compositing lab (MPSL) and analytical lab (AXYS) will ensure that no Teflon coated equipment, containers, or wrappings will be used when handling these fish and corresponding samples. AXYS will deliver pre-cleaned containers for PFCs to MPSL for sample transport.

For the remaining samples, sample containers will be pre-cleaned and prepared by MPSL according to MPSL-101. For samples that are to be analyzed for mercury, only Teflon or glass/quartz containers with Teflon-lined caps will be used to store and transport these samples (SWAMP 2014).

Samples prepared for PCB and PBDE analysis will be sent by MPSL to the CDFW-WPCL laboratory, and samples prepared for PFC analysis will be sent by MPSL to AXYS Analytical Inc. Samples prepared for Hg analysis will be analyzed on-site. MPSL will be responsible for

ensuring that all samples are maintained at -20C during transport to the respective laboratories. Any deviation should be noted and reported to SFEI.

Project samples will not be disposed of until all analyses are complete and analytical and QC results have been reviewed and approved by the SFEI Project Manager and QA Officer. Once all sampling processing is complete, MPSL will provide SFEI with a record of the compositing information in CEDEN database format.

### **Analytical Methods**

Detailed analytical method and QA/QC protocols can be found in Elements 13 and 14 of the 2014 Quality Assurance Program Plan for The Regional Monitoring Program for Water Quality in San Francisco Bay (RMP 2014).

The number of samples of each species that will be analyzed for each parameter is listed in Table 2. Lists of compounds that will be included in each organic compound parameter group are shown in Attachment 3.

### **Archiving Strategy**

MPSL will retain homogenate samples to allow for future reanalysis. Short-term archive samples will be stored at Schafer's Meats & Cold Storage in Oakland, CA. Long-term archive samples will be stored at the NIST facility in Charleston, SC. Archive samples will be taken from composite samples. The number and volume of archive samples of each species, as well as sample container, storage and transport information are included in Table 1 of Attachment 1. Container and sample handling and transfer information are included in Table 2 of Attachment 1.

Samples shipped to the archive facilities will be accompanied by COCs, which will include a unique entry for each archive container and include the following information: composite ID, station name, species, sample date (or year if fish from multiple sample dates are contained in the same composite), and container type and number.

Archives placed in short-term storage with AMS will be assigned a lot number when they are added to the freezer. The lot numbers, along with the information on the COCs, will be provided to SFEI by AMS in a spreadsheet.

Archives placed in long-term storage will require specialized labels for sample identification. NIST will send MPSL a label template containing the above-described fields. MPSL will provide NIST with completed template and NIST will send MPSL a set of pre-printed labels containing the following information: the barcode, fish species, composite ID (NIST calls this the "field ID"), year collected (with blank spaces where month/day can be written in) and aliquot ID. NIST will also provide pre-cleaned containers to MPSL.

NIST will add additional sample identifiers to the archive sample information provided by MPSL, and will provide this compiled archive data to SFEI in a spreadsheet. These additional sample identifiers will include an “Other ID #,” which is a longer version of the aliquot ID that includes the full site name, as well as a NIST assigned “Globally Unique Aliquot ID.”

MPSL will provide an electronic record of all archive sample data to SFEI, including at least the following information: composite ID, aliquot ID, container type, aliquot mass (including units and basis), and the location of storage (NIST or AMS).

### **Reporting**

SFEI will provide field staff with field sheets and COCs. MPSL will enter field collections information in SWAMP database format, and provide a copy of these records to SFEI. MPSL will provide a copy of composite IDs and information to SFEI in CEDEN database format, as well as a copy of COCs received from field staff and provided to the analytical labs.

SFEI will provide MPSL and AXYS with a Lab EDD template following the CEDEN database format. Analytical results will be entered in this Lab EDD format by each lab and provided to SFEI in digital format.

### **References**

SFEI. 2014. Quality Assurance Program Plan for The Regional Monitoring Program for Water Quality in San Francisco Bay. [http://www.sfei.org/sites/default/files/2014\\_RMP\\_QAPP\\_0.pdf](http://www.sfei.org/sites/default/files/2014_RMP_QAPP_0.pdf)

Surface Water Ambient Monitoring Program. 2014. Study of Lakes and Reservoirs with Low Concentration of Contaminants in Sport Fish - Final Quality Assurance Program Plan. [http://www.waterboards.ca.gov/water\\_issues/programs/swamp/docs/lakes\\_study/bog\\_low\\_conc\\_qapp\\_final.pdf](http://www.waterboards.ca.gov/water_issues/programs/swamp/docs/lakes_study/bog_low_conc_qapp_final.pdf)

## Attachment 1 - Artesian Slough Sport Fish Archive Protocol

Sample Handling: Ryan Mayfield, City of San Jose; Autumn Bonnema, MLML  
 Samples are shipped by SJSCWRF to MLML for dissection and homogenization.

**Table 1. Sport Fish Archiving Strategy**

Composites	# of containers for each composite	Tissue mass per container (g wet wt)	Container	Storage Purpose	Storage Location	Storage Temperature	Mass needed for each composite (g wet wt)
Striped bass 6 composites	3	15	22 ml Teflon vial <sup>a</sup>	Long-term time trends	NIST	-150 °C	120
	3	15	60 ml glass jar <sup>b</sup>	Time trends, CECs, QA/QC	AMS/Schaeffers	-18 °C	
	2	15	30 ml PP jar <sup>c</sup>	Time trends, CECs, QA/QC	AMS/Schaeffers	-18 °C	
Carp 2 composites	3	15	22 ml Teflon vial <sup>a</sup>	Long-term time trends	NIST	-150 °C	144

	3	8	10 ml PP cryovial <sup>d</sup>	Long-term time trends	NIST	-150 °C	
	3	15	60 ml glass jar <sup>b</sup>	Time trends, CECs, QA/QC	AMS/Schaeffers	-18 °C	
	2	15	30 ml PP jar <sup>c</sup>	Time trends, CECs, QA/QC	AMS/Schaeffers	-18 °C	
Largemouth bass 1 composite	3	15	60 ml glass jar <sup>b</sup>	Time trends, CECs, QA/QC	AMS/Schaeffers	-18 °C	45

PE = polyethylene; PP = polypropylene; CECs = contaminants of emerging concern; QA/QC = quality assurance/quality control

a = Pre-cleaned/PC class jars, Teflon-lined lid, supplied by ESS Vial (Oakland, CA). Shipped to NIST. Pre-cleaned and pre-labeled by NIST and sent to MLML.

b = Pre-cleaned jars will be provided by MLML but not pre-cleaned by MLML directly. 2 empty jars will be shipped to the storage facility and stored with the samples to serve as blanks in future reanalyses.

c = PP jars, linerless lid, supplied by Fisher Scientific. Pre-cleaned by MLML using routine procedures for metals (striped bass, largemouth bass). MLML will add a MeOH rinse for storage of samples for PFCs (carp).

d = Pre-cleaned and pre-labeled by NIST and sent to MLML.

**Table 2. Sport Fish Archiving Process**

<b>Container</b>	<b>Supply and Cleaning Chain</b>	<b>Sample Preparation Site</b>	<b>Sample Shipping and Storage<sup>1</sup></b>
22 ml Teflon vial	SFEI orders and ships to NIST. NIST cleans, labels and ships to MLML.	MLML fills container in the lab.	MLML ships to NIST Charleston.
10 ml PP cryovial	NIST currently has vials on-site. SFEI requests the number of vials to be cleaned and NIST cleans and ships to MLML.	MLML fills container in the lab.	MLML ships to NIST Charleston.

60 ml glass jar	MLML currently has jars on site.	MLML fills container in the lab.	MLML ships to AMS. AMS drops off at Schaeffers Oakland. 2 empty bottles will be shipped to AMS to serve as blanks for future analyses.
30 ml PP jar	MLML currently has jars on site. MLML cleans and fills.	MLML fills container in the lab.	MLML ships to AMS. AMS drops off at Schaeffers Oakland.

<sup>1</sup>Sample shipper is responsible for ensuring that samples are maintained at the appropriate temperature during transport. Any deviation should be noted and reported to SFEI.

## Attachment 2 - Principal Contacts

Name	Affiliation	Function	Phone	Email	Shipping Address
Phil Trowbridge	SFEI	RMP Program Manager	510-746-7345	philt@sfei.org	--
Amy Franz	SFEI	Archive database coordinator	510-746-7394	amy@sfei.org	--
Ryan Mayfield	SJSCRWF <sup>1</sup>	Fish sample collection coordinator. Field data collection and sample handling, storage and transport	408-635-4033	ryan.mayfield@sanjoseca.gov	--
Autumn Bonnema	MLML <sup>2</sup>	Container cleaning, sample dissection and homogenization. Lipid, moisture, Hg, PCB, and PBDE analyses. Allocation of tissue samples for short and long term storage archives	831-771-4175	bonnema@mlml.calstate.edu	Moss Landing, CA 95039 AXYS Analytical Services 2045 Mills Road West Sidney, BC V8L 5X2
Kalai Pillay	AXYS	PFC analyses and container cleaning.	250-655-5832	kpillay@axys.com	AXYS Analytical Services 2045 Mills Road West Sidney, BC V8L 5X2
Paul Salop	AMS	Coordinate RMP short term storage archives	925-373-7142	salop@amarine.com	Applied Marine Sciences 4749 Bennett Dr., Ste. L Livermore, CA 94551
Rebecca Pugh	NIST	Coordinate RMP long term storage archives	843-762-8952	rebecca.pugh@noaa.gov	National Institute of Standards and Technology Hollings Marine Laboratory 331 Ft. Johnson Rd. Charleston, SC 29412

1-SJSCRWF will be collaborating with UC Davis to collect fish. SJSCRWF will be responsible for field data and sample handling, storage, and transport to MLML.

2-Moss Landing Marine Laboratories is affiliated with the California Department of Fish and Wildlife's Water Pollution Control Laboratory (WPCL) through the San Jose State University Research Foundation. MLML will be responsible for transferring samples for analysis to WPCL for PCB and PBDE analyses.

## Attachment 3 - Reportable Parameters Lists - Organics

Table 1. Polychlorinated Biphenyls Analyte List

Polychlorinated Biphenyls (PCBs) <i>IUPAC numbers listed. Sums calculated by SFEI.</i> <i>*Congeners included in the Sum of 40 PCBs (SFEI).</i> <sup>1</sup> Coplanar PCBs			
PCB 008*	PCB 066*	PCB 128*	PCB 177*
PCB 018*	PCB 070*	PCB 137	PCB 180* <sup>1</sup>
PCB 019	PCB 074*	PCB 138*	PCB 187*
PCB 027	PCB 077 <sup>1</sup>	PCB 141*	PCB 189 <sup>1</sup>
PCB 028*	PCB 087*	PCB 146	PCB 194*
PCB 029	PCB 095*	PCB 149*	PCB 195*
PCB 031*	PCB 097*	PCB 151*	PCB 198
PCB 033*	PCB 099*	PCB 153*	PCB 199
PCB 044*	PCB 101*	PCB 156* <sup>1</sup>	PCB 200
PCB 049*	PCB 105* <sup>1</sup>	PCB 157 <sup>1</sup>	PCB 201*
PCB 052*	PCB 110*	PCB 158*	PCB 203*
PCB 056*	PCB 114 <sup>1</sup>	PCB 169 <sup>1</sup>	PCB 206
PCB 060*	PCB 118* <sup>1</sup>	PCB 170* <sup>1</sup>	PCB 209
PCB 064	PCB 126 <sup>1</sup>	PCB 174*	

**Table 2. Polybrominated Diphenyl Ethers Analyte List**

<b>Polybrominated Diphenyl Ethers (PBDEs)</b> <i>IUPAC number listed.</i>			
PBDE 007	PBDE 035	PBDE 105	PBDE 183
PBDE 008	PBDE 037	PBDE 116	PBDE 190
PBDE 010	PBDE 047	PBDE 119	PBDE 197
PBDE 011	PBDE 049	PBDE 120	PBDE 203
PBDE 012	PBDE 051	PBDE 126	PBDE 204
PBDE 013	PBDE 066	PBDE 128	PBDE 205
PBDE 015	PBDE 071	PBDE 138	PBDE 206
PBDE 017	PBDE 075	PBDE 140	PBDE 207
PBDE 025	PBDE 077	PBDE 153	PBDE 208
PBDE 028	PBDE 079	PBDE 154	PBDE 209
PBDE 030	PBDE 085	PBDE 155	
PBDE 032	PBDE 099	PBDE 166	
PBDE 033	PBDE 100	PBDE 181	

**Table 3. Polyfluorinated Compounds Analyte List**

<b>Perfluorinated Compounds</b> <i>Sums calculated by SFEI.</i>
<b>Carboxylic Acids</b>
Perfluorobutanoate (PFBA)
Perfluoropentanoate (PFPeA)
Perfluorohexanoate (PFHxA)
Perfluoroheptanoate (PFHpA)
Perfluorooctanoate (PFOA)
Perfluorononanoate (PFNA)
Perfluorodecanoate (PFDA)
Perfluoroundecanoate (PFUnA)
Perfluorododecanoate (PFDoA)
<b>Sulfonic Acids</b>
Perfluorobutanesulfonate (PFBS)
Perfluorohexanesulfonate (PFHxS)
Perfluorooctanesulfonate (PFOS)
Perfluorooctane sulfonamide (PFOSA)