REGIONAL MONITORING
PROGRAM FOR WATER QUALITY
IN SAN FRANCISCO BAY
sfei.org/rmp

2019 RMP Sport Fish Monitoring Sampling and Analysis Plan

Contribution #970

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1. Introduction

The Regional Monitoring Program for Water Quality in San Francisco Bay (RMP) monitors concentrations of contaminants in fish tissue as indicators of bioaccumulation of contaminants in the Bay. In 2019, the RMP will conduct its eighth round of sport fish monitoring by collecting sport fish samples from various locations in the Bay as a part of routine Status and Trends Monitoring. Add-ons to the routine Status and Trends sport fish monitoring design will include archiving for microplastics and fipronil, as well as additional collections of shiner surfperch in Priority Margin Unit areas (PMUs).

The target fish species are as follows (starred species are collected as bycatch only):

- Core Species:
    - White Croaker
    - Shiner Surfperch
    - White Sturgeon
    - Striped Bass
    - Halibut
    - Northern Anchovy
    - Jacksmelt
- Other Species:
    - Pacific Herring
    - Pacific Sardine
    - Staghorn Sculpin
    - Brown Rockfish
- Other Species (cont'd):
    - Blue Rockfish
    - Barred Surfperch*
    - Bat Rays
    - Rubberlip Perch*
    - Black Perch*
    - Cabezon*
    - Pacific Sanddab*
    - Diamond Turbot*
    - Petrale Sole*
    - Starry Flounder*
    - Monkeyface Prickleback
    - Largemouth Bass
    - Common Carp

The fish samples will be collected by staff with the Marine Pollution Studies Laboratory at Moss Landing Marine Labs (MPSL-MLML); they will also procure all necessary fish collection permits. Fish tissue will be processed by staff at the MPSL-DFW. Thereafter, samples will be analyzed for mercury, polychlorinated biphenyls (PCBs), polybrominated diphenyl ethers (PBDEs), dioxins, per- and polyfluoroalkyl substances (PFAS), fipronil, and selenium by the laboratories at MPSL-DFW, SGS-AXYS, Eurofins CalScience LLC, and Brooks Applied Laboratories (BAL). The results of this work will be included in the sport fish monitoring final report that will be prepared by RMP staff by the end of 2020.

The purpose of this Sampling and Analysis Plan is to clearly document the sampling design, methods, and responsibilities; and to facilitate coordination among project partners.

2. Key Personnel and Approvals

Table 1. Key Personnel and Approvals of Sampling and Analysis Plan

Contact	Role	Phone/Email	Plan Approval Date
Jay Davis	RMP Lead Scientist	jay@sfei.org 510-746-7368	12/1/19
Melissa Foley	RMP Manager	melissaf@sfei.org 510-746-7345	5/10/19
Don Yee	RMP QA Officer	don@sfei.org 510-7467369	
Amy Franz	RMP Data Manager	amy@sfei.org 510-746-7394	6/13/19
Meg Sedlak	Senior Program Manager	megs@sfei.org	6/4/19
Diana Lin	Environmental Scientist	diana@sfei.org 510-746-7385	6/4/19
Nina Buzby	Environmental Analyst	ninab@sfei.org 510-746-7393	Primary author
Marco Sigala	MPSL-MLML Program Manager	msigala@mlml.calstate.edu 831-771-4173 Marine Pollution Studies Lab 7544 Sandholdt Road Moss Landing, CA 95039	6/2/19
Autumn Bonnema	MPSL-DFW Associate Program Director	bonnema@mlml.calstate.edu 831-771-4175 Marine Pollution Studies Lab 7544 Sandholdt Road Moss Landing, CA 95039	6/5/19
Sean Campbell	SGS-AXYS Project Manager	scampbell@sgs.com 250-655-5834 SGS-AXYS Analytical 2045 Mills Road Sidney, BC, Canada V8L 5X2	5/29/19
Lydia Greaves & Lauren Blaiwes	Brooks Applied Labs Client Services Manager & Project Manager	lydia@brooksapplied.com Lauren@brooksapplied.com 18804 North Creek Parkway, Suite 100, Bothell, WA 98011	11/22/19

Carla Lee Hollowell	Project Manager, Marine Chemistry	CarlaHollowell@eurofinsus.com 714-895-5494 Eurofins Calscience, LLC 7440 Lincoln Way Garden Grove, CA 92841	11/27/19
Rebecca Pugh	NIST Project Manager	Rebecca.Pugh@noaa.gov 843-460-9864 NIST Hollings Marine Laboratory 331 Ft. Johnson Rd. Charleston, SC 29412	6/12/19
Wes Heim	MPSL-DFW Program Director	wheim@mlml.calstate.edu 831-771-4459 Marine Pollution Studies Lab 7544 Sandholdt Road Moss Landing, CA 95039	6/19/19

3. Sampling Design

The target species, as well as their target collection and composite information, are listed in Table 2. Species are chosen for their ability to serve as indicator species and the need for data to support inclusion in the Bay fish consumption advisory. Species will be collected from various regions in the Bay during summer 2019 (see Section 4 for details). The minimum target fish sizes, numbers in composites, and target number of fish for the 2019 study are also shown in Table 2.

As outlined in Table 2, select species should be kept as bycatch if encountered during collections. These include barred surfperch, rubberlip perch, black perch, cabezon, pacific sanddab, diamond turbot, petrale sole, and starry flounder. Bycatch collections will initially be kept with MPSL-MLML and possibly transferred to AMS for RMP short-term archiving at Schaefer's Frozen Storage in Oakland, CA. The decision to keep bycatch for short-term archives will be determined closer to the time of transfer. It should be noted that if any collected largemouth bass do not fall within the target size range (larger or smaller), they should be kept as bycatch.

Table 2. 2019 Sport Fish and Collection Plan

	Minimum Target Size (mm TL[1])	Number of Bay Regions	Total Composites	Fish per Composite	Total Number of Fish
White Croaker[2]	200	Wherever available	10	5	50
Shiner Surfperch	100	9	36	20	720

White Sturgeon	1016 (FL)	3	4	3	12
Striped Bass[4]	450	3	9	3	27
Halibut	558	3	3	3	9
Northern Anchovy[2]	115	Wherever available	6	20	120
Jacksmelt	210	5	5	10	50
Pacific Herring[3]	170	3	6	20	120
Pacific Sardine	200	3	6	10	60
Staghorn Sculpin	150	3	3	10	30
Brown Rockfish[2]		Wherever available	3	5	15
Blue Rockfish[2]		Wherever available	3	5	15
Barred Surfperch	130	bycatch		5	
Bat Rays	Small - 458 Large - 610	3	6	5	30
Rubberlip Perch	180	bycatch		5	
Black Perch	150	bycatch		5	
Cabezon	381	bycatch		5	
Pacific Sanddab	190	bycatch		10	
Diamond Turbot	165	bycatch		10	
Petrale Sole	330	bycatch		5	
Starry Flounder	365	bycatch		5	
Monkeyface Prickleback	360	3	3	5	15
Largemouth Bass	305	2	2	5	10
Common Carp	450	2	2	5	10

[1]TL = Target length; FL = Fork length
[2]Fish will be collected wherever available in the Bay to meet sample needs
[3]Collect roe if available
[4]One additional fish from Central Bay and Artesian Slough will be collected for microplastic analysis (see Section 4)

4. Sampling Locations, Collection Methods and Documentation

Fish will be collected by MPSL-MLML during the summer of 2019 (late May to early September) using otter trawls, gillnets (multifilament, monofilament, and nylon), and hook-and-line sampling. MLML staff will procure all necessary fish collection permits. If any SFEI staff participate in field sampling, they must record all hours spent on the water in the navigable waters log. (https://docs.google.com/spreadsheets/d/1ePFtM07P8QKscu7GxNE8I0n1sTevAfrDW7wgyyIqRIU/edit#gid=0)

Sport fish will be collected from varying locations in San Francisco Bay depending on the species and analytes of interest (Tables 3a and 3b). Given that these are mobile organisms, collection locations are referred to by general areas. The rationale for the distribution of collection areas for each species is based on knowledge of fishing locations, where certain species are likely to be found in the Bay, as well as targeting areas where specific analytes are of interest (e.g., interest in microplastic levels in the wastewater-influenced Lower South Bay).

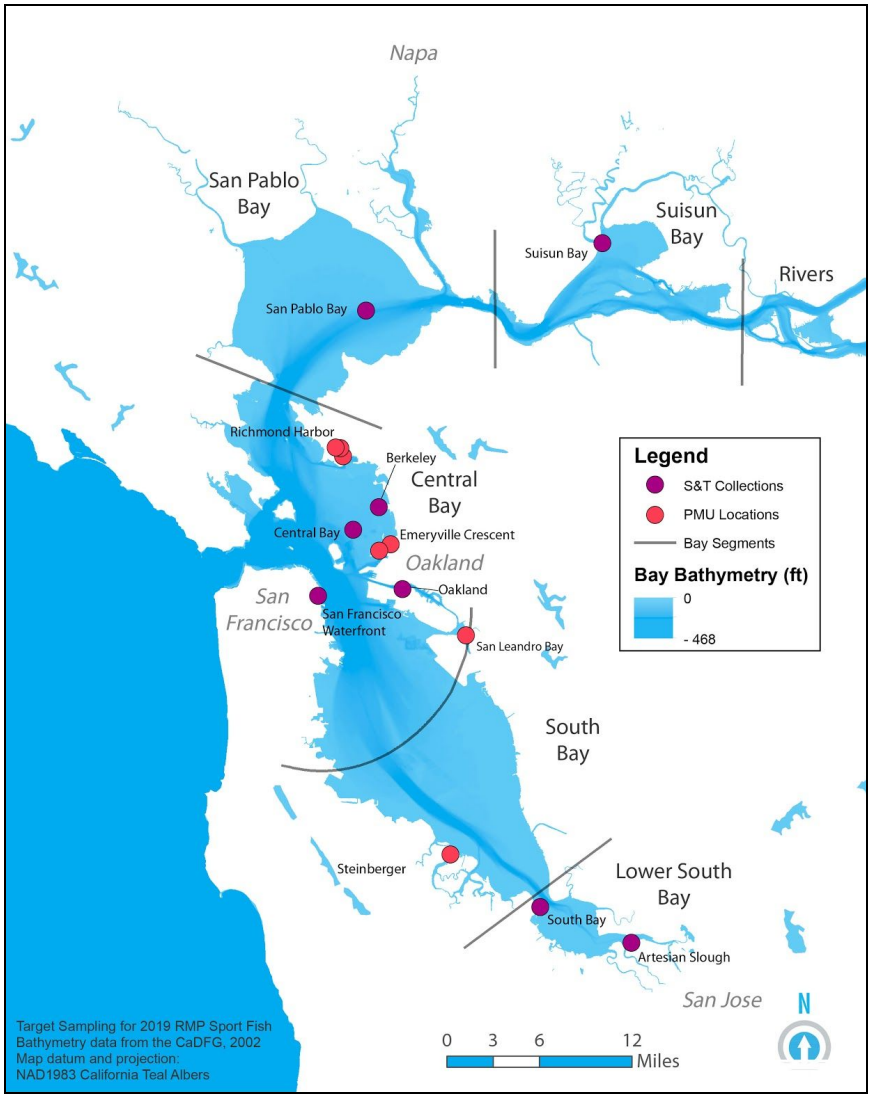

Figure 1. Map of San Francisco Bay sport fish sampling locations.

In addition to the RMP Status and Trends collections, three additional add-on efforts will be incorporated into collection efforts:

1. Fish digestive tracts for microplastics analysis will be archived from fish collected from multiple locations. To support the microplastic study, one additional striped bass (individual fish) will be collected from both Central Bay and Artesian Slough (Table 3c).
2. Fipronil analyses will be conducted on fish collected at multiple Bay locations (Table 3c).
3. Additional shiner surfperch will be collected from Priority Margin Units (PMUs) (Table 3b) for PCB analysis. These collection locations are shown as red circles in Figure 1 and will

involve collection efforts that take place in shallower, nearshore regions. Detailed satellite images of the intended PMU locations are shown in Figure 2.

Figure 2. Shiner Surfperch Priority Margin Unit Sampling Locations (Detailed View)

Coordinates of actual sampling locations will be recorded from GPS readings and reported on field sheets provided by SFEI (Attachment 1). Other notable information will also be recorded on these sheets such as sampling method, device, depth, and a description of the location. Any anomalies from original sampling objectives should be added to the field records and recorded on a list of deviations.

Total length and total fish mass will be measured and recorded in the field; with the exception of smaller fish, for which total body mass will be measured and recorded at the MPSL-DFW laboratory (Section 5). Fish to be archived for microplastic analysis should be weighed and measured prior to freezing when possible. When possible, sex (shiner surfperch and striped bass), parasites, body anomalies, and any other notable factors should be recorded on the field and or lab sheets. The target sizes of each species are outlined in Table 2. However, largemouth bass should be kept as bycatch even if they do not meet the target size.

At the end of the fish collection season, a cruise report will be submitted to SFEI that details weekly synopses of sampling efforts and provides figures for fishing locations and catch results.

Table 3a. 2019 Sport Fish Composites by Location

Station Name	South Bay	Oakland	San Francisco	Berkeley/ Central Bay	San Pablo Bay	Artesian Slough	Suisun Bay
CEDEN Station Code	*204STHBAY*	*203OAKLND*	*203SANFRN*	*203BRKLEY 203CENTRL*	*203SNPBLO*	*ARTSLGH*	*207SUISUN*
White Croaker	wherever available						
Shiner Surfperch[1]	3	3	3	3	3		
White Sturgeon	1			1	1		1
Striped Bass[2]				3	3	3	
Halibut			1	1	1		
Anchovy	wherever available						
Jacksmelt	1	1	1	1	1		
Pacific Herring	2			2	2		
Pacific Sardine	2			2	2		
Staghorn Sculpin	1			1	1		
Brown Rockfish	wherever available						
Blue Rockfish	wherever available						
Bat Rays	2			2	2		
Monkeyface Prickleback		1	1	1			
Largemouth Bass						2	
Common Carp						2	

[1]Additional collections will be made for PCB PMU margins work (see Table 3b for details)
[2]One additional striped bass will be collected from each of Central Bay and Artesian Slough for microplastic analysis (amounting to 10 total fish, 3 composites from the area)

Table 3b. Margins Shiner Surfperch PCB Collection Plan

Station Name	San Leandro	Richmond	Emeryville Crescent	Steinberger Slough
CEDEN Station Code	2RMPSLB	2RMPRH	203EMRYVL	204STNBGR
Composites *(20 fish each)*	3	9	6	3
Total Fish	60	180	120	60

Table 3c. Microplastic and Fipronil Design

	San Francisco Waterfront	Berkeley/ Central Bay	San Pablo Bay	Artesian Slough	Steinberger Slough (PMU)	San Leandro (PMU)	South Bay
Microplastic (individual fish)							

Shiner Surfperch	10	10	10			10	10
Striped Bass		10		10			
Fipronil (composite samples)							
Shiner Surfperch			2		2	2	
Striped Bass		2		2			
Common Carp				2			
Halibut		1					

5. Sample Handling, Processing, and Shipment to Laboratories

Field Handling

All largemouth bass, brown rockfish, and striped bass should be kept as whole fish prior to processing for individual mercury analysis. Pacific herring will also stay whole for later roe collection. Striped bass at the two locations (Central Bay and Artesian Slough) should be kept as whole fish to allow for preservation of the gastrointestinal (GI) tracts for microplastic analysis. At selected locations (Table 3c) 10 shiner surfperch from each site should also be retained for GI microplastic analysis (kept as whole fish).

Individual white sturgeon will be analyzed for selenium from four locations (Section 6, Table 4). For sturgeon, the posterior third of the fish will be processed in the field, making sure to retain a section that allows the collection of fillets from the epaxial and caudal regions, as well as tissue for general composite samples (Figure 3). If gut content analysis for selenium is a matrix of interest (it is not for 2019), sturgeon guts should be removed in the field and then preserved by adding a syringe of formalin inside the gut.

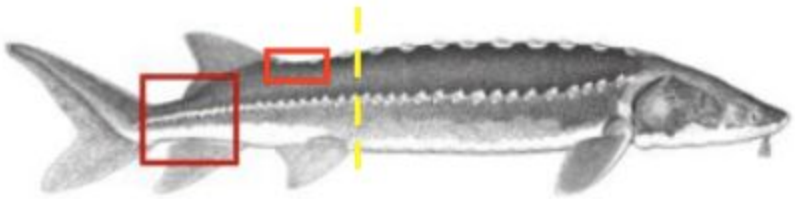

Figure 3. White sturgeon field processing diagram. Red boxes indicate fillet regions, yellow line is a guide for field processing efforts.

Upon collection in the field, some fish will be tagged with a unique ID while others (smaller species) will be associated with a general unique ID until processing in the lab. However, smaller shiner surfperch collected for microplastic analysis will be tagged and given a unique ID in the field. All fish are then wrapped in aluminum foil, placed in a clean labeled bag, transported on dry ice to the MPSL-DFW facilities, and placed in a -20°C freezer until dissection and compositing. Frozen tissue samples have a 12 month hold time from the date of collection. If a hold time violation occurs, data will be flagged appropriately in the final write up.

Field sheets will be digitized and converted to the CEDEN database format by MPSL-MLML staff. MPSL-MLML will provide SFEI with a cruise report and completed field sheets by April 30, 2020.

Processing

MPSL-DFW will measure the total length and weight of all small fish that were not measured in the field, then process samples to create the necessary individual and composite samples for laboratory analyses. Composites will be representative of multiple fish, details are outlined in Section 6. In general, fish will be dissected skin-off, and only the fillet muscle tissue will be used for analysis. Several species (shiner surfperch, jacksmelt, staghorn sculpin, northern anchovy, Pacific sardine, and Pacific herring) that are too small to be filleted will be processed whole but with head, tail, and viscera removed.

Every sample will be analyzed for percent moisture. Composite samples of species that will be analyzed for organics will also be analyzed for percent lipid. Selenium and mercury analysis of select species will be conducted on samples of individual fish; other analyses will be conducted on composite samples. Additionally, the digestive tracts of striped bass and shiner surfperch will be archived for microplastic analysis and stored at Schaefer's Cold Storage in Oakland, CA. Microplastic dissection details (Rochman et al. 2015) are outlined in Section 6. If fully formed roe are found in pacific herring they will either be extruded into sample containers and homogenized, if the roe are imbedded in gonad tissue both the gonad and roe will be dissected and homogenized together. The number of composites per analyte can also be found in Section 6. The species associated with each analyte is outlined in Table 5.

Shipping

MPSL-DFW will conduct some analyses onsite and ship appropriate subsamples to other labs for analysis. SGS-AXYS will receive composite samples for PCB, PBDE, PFAS, and dioxin analysis. BAL will receive composite and individual samples for selenium analysis. Eurofins CalScience will receive composite samples for fipronil analysis. The University of Toronto will receive shiner surfperch and striped bass digestive tracts for microplastic analysis (see Table 5 for total composites per lab). MPSL-DFW will also ship archive samples; long-term archives will go directly to NIST, and short-term archives will be sent to Applied Marine Sciences (AMS). Shipping details including addresses, required temperature, and travel time are outlined in Attachment 2.

MPSL-DFW will be responsible for ensuring that all samples are maintained at required temperatures 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 and until disposal is approved by SFEI.

6. Fish Dissection and Compositing

MPSL-DFW will dissect and homogenize fish tissue samples according to method MPSL-105 (Attachment 3). Composites will include equal masses from each individual fish in the composite and from fish within the same size class. The length of the smallest fish in the composite should be no less than 75% of the length of the largest fish. Once all sampling processing is complete, MPSL-DFW will provide SFEI with a record of the compositing information in a CEDEN tissue template.

Unless otherwise noted, all samples will be processed as muscle fillets with the skin off. Special attention will be paid to this aspect of the work to prevent varying concentrations based on lipid content, as was experienced during the 2014 RMP Sport Fish sampling efforts. As stated in Section 5, composite samples for organics will be analyzed for percent lipid.

Special dissection steps will be taken with each individual white sturgeon collected. Previous RMP selenium work has involved sample collection from two areas on sturgeon - epaxial and caudal. To compare the tissue from these areas and inform future comparisons to the historic time series, skinless fillets will be taken from two areas on each fish. The location and size of the fillet for both regions are outlined in Table 4 below.

Table 4. Selenium Sturgeon Dissection

	Caudal Region	Epaxial Region
Previous Sampling Events	2014 Status & Trends	2015-2017 Sturgeon Derby 2017 Muscle Plug Study
Fillet Collection Location	Fish section collected from behind the anal bent to the caudal keel	Fillet collected from the epaxial muscle, just in front of the dorsal fin
Tissue Sample Depth	Fillet sampled from a section with skin removed first, depth of skin removal varies	Depth of skin removal for fillet varies
Sample Size	Approximately 6 x 6 cm square, varies by fish	Approximately 3 x 5 cm
Location Graphic		

To dissect fish for microplastic analysis, methods from Rochman et al. 2015 should be followed. The entire gastrointestinal tract (from esophagus to anal pore) is the desired tissue. The gut should be placed in a pre-cleaned polypropylene container or a whirl pak bag and then frozen. The cleaning procedure for containers is first washing with soap and DI water followed by three

rinses with DI water. MPSL-DFW will purchase and clean the proper containers. To prevent contamination, tools should be rinsed with deionized (DI) water between individual samples, dissections should ideally be performed in a clean space or a clean cabinet. Laboratory staff should wear white cotton lab coats, avoid wearing synthetic clothing, and tie their hair back while dissecting samples meant for microplastic analysis.

If fully formed roe are found in pacific herring they will either be extruded into sample containers and homogenized, if the roe are imbedded in gonad tissue both the gonad and roe will be dissected and homogenized together.

Containers for composites and subsampling are dependent on analyses and are outlined in Table 5. Total container needs are based on target collection numbers. Pre-cleaned containers, if necessary, should be provided by the associated lab to MPSL-DFW before sample collections have finished (prior to 8/31/19).

For samples that will be analyzed for mercury, only Teflon or glass/quartz containers with Teflon-lined caps will be used to store and transport the samples. Remaining sample containers will be pre-cleaned and prepared by MPSL-DFW according to MPSL-101. The compositing lab (MPSL-DFW) and SGS-AXYS will ensure that Telfon materials are avoided when preparing and dealing with samples analyzed for PFAS.

Table 5. Analyte Composite totals and Container Types

	AXYS					MLML*		BAL*		Eurofins	UofT				
	Fish per composite	PCBs	PBDEs	Dioxins	PFAS	Hg Indiv	Hg Comp	Se Indiv	Se Comp	Fipronil	Archive for Microplastic (Indiv)	Long Term Archives		Short Term Archives	
Core RMP Species															
White Croaker	*5*	10		6	3		10		10			3	3	3	2
Shiner Surfperch	*20*	36	15	17	3		15		15	6	50	3		4	2
White Sturgeon	*3*	4			3	12		24**							
Striped Bass	*3*		3		3	28			9	2	30				
Halibut	*3*						3		3						
Northern Anchovy	*20*	6					6			4		3		3	2
Jacksmelt	*10*						5		5						
Other Species															
Pacific Herring	*20*	6					6								
Pacific Sardine	*10*	6					6								
Staghorn Sculpin	*10*	3					3								
Brown Rockfish	*5*	3				15									
Blue Rockfish	*5*	3					3								
Barred Surfperch	*5*														
Bat Rays	*5*	6					6								
Rubberlip Perch	*5*														
Black Perch	*5*														
Cabezon	*5*														
Pacific Sanddab	*10*														
Diamond Turbot	*10*														
Petrale Sole	*5*														
Starry Flounder	*5*														
Monkeyface Prickleback	*5*	3					3								
Largemouth Bass	*5*	2	2		2	10				2		3	2	3	2
Common Carp	*5*	2	2		2					2		3	2	3	2
	Total	93			16	65	66	0	42	16	80	15	7	16	10

Table 5. Analyte Composite totals and Container Types

	AXYS				MLML*		BAL*		Eurofins	UofT				
	Fish per composite	PCBs PBDEs Dioxins		PFAS	Hg Indiv	Hg Comp	Se Indiv	Se Comp	Fipronil	Archive for Microplastic (Indiv)	Long Term Archives		Short Term Archives	
	Minimum Mass (g)	15		3	N/A (fillets)	10	N/A (fillets)	10	20	N/A (GI tract)	23	10	70	35
	Sample Container	60mL amber glass jar (analytes stored together)		60mL HDPE bottle					4 oz glass jars	PP/PE container	22 mL teflon vial	10 mL PP cryovial	60 mL glass jar	30 mL PP jar
	Cleaning Needs	Jars will be pre-cleaned and sent directly to MPSL-MLML by SGS AXYS			Provided by MLML				Jars will be pre-cleaned and sent directly to MLML by Eurofins	Wash with soap and RO water. Rinse x3 with RO water			Provided by MLML	

*Percent moisture and percent lipids will be measured along with target analyte

**Sturgeon selenium analyses will be by individual fish and involve to fillet samples

Minimum tissue requirements for each composite were calculated based on the collection targets outlined in Table 2 and tissue mass needs for each analyte (Table 5). These values are also outlined by species and analyte in Attachment 4. Mass requirements may be exceeded or not fully met as tissue amounts are subject to actual collection totals and fish size. If possible amounts greater than the minimum mass should be provided to labs. Tissue requirements for sample archives have also been included in calculations. Archive containers should minimize headspace (i.e. 90-95% full container) to avoid sample contamination. Archive needs should be addressed following a hierarchy with long-term archives (22 mL teflon vials and 10 mL PP cryovial) as a first priority, followed by 60 mL glass jars for short-term archives, and, lastly, 30 mL PP jars also for short-term archives.

The minimum tissue required per composite for each lab is outlined in Table 6. MPSL-DFW and BAL will also be conducting Hg and Se analyses on individual fish, which requires 5 g of tissue. These mass requirements are not included in Table 6 totals as they will not be incorporated into homogenized composites. Again, these values may not reflect what labs actually receive but provide an estimate - the value will be finalized once collections have been completed.

It should be noted that both Table 6 and Attachment 4 do not include the University of Toronto's microplastic tissue needs because these analyses will occur only on fish digestive tracts, which will be provided whole, regardless of their total mass.

Table 6. Required Mass (g) per Composite, by Analytical Laboratory

	Archives	SGS AXYS[1]	MPSL-DFW[2]	BAL	Eurofins
White Croaker	138	18	10	10	0
Shiner Surfperch	128	18	10	10	20
White Sturgeon	0	18	10	20	0
Striped Bass	0	18	5	10	20
Halibut	0	0	10	10	20
Northern Anchovy	128	15	10	0	0
Jacksmelt	0	0	10	10	0
Pacific Herring	0	15	10	0	0
Pacific Sardine	0	15	10	0	0
Staghorn Sculpin	0	15	10	0	0
Brown Rockfish	0	15	0	0	0
Blue Rockfish	0	15	10	0	0
Bat Rays	0	15	10	0	0
Monkeyface Prickleback	0	15	10	0	0
Largemouth Bass	138	18	0	0	0
Common Carp	138	18	0	0	20

[1]Mass totals for SGS-AXYS samples include the required amount for integrated PCB, PBDE, and Dioxin analysis (15 g) as well as separate PCB analyses (3 g).
[2]Hg and Se analyses will also involve individual analysis, for which 5 g of tissue per fish should be collected.

7. Analytical Methods

The number of samples (individual or composite) of each species that will be analyzed for each target analyte is listed in Table 5. Lists of compounds that will be included in each organic parameter group (PCBs, PBDEs, PFAS, and dioxins) are shown in Attachment 5b.

Table 7 provides a summary of analytical methods and reporting units as well as an outline of what species and laboratories will be involved. Corresponding method detection limits (MDL) and reporting limits (RL) are outlined in Attachments 5a and 5b. Two additional dioxin analyses will be added to the samples from this study for the Surface Water Ambient Monitoring Program (SWAMP). These composites will be of shiner surfperch collected from Humboldt bay with 20 fish per sample.

As mentioned previously, all organic analyses should include percent moisture and percent lipid results. Duplicates and other QC sample results should be reported in the same units as initial tissue samples; for QC samples with no true mass (such as blanks), results should be reported as though the samples originated from digests or extracts of typical subsample masses used for analyses of field samples. This allows for comparison of equivalent concentration ranges across field and QC sample types. Reporting units for corresponding QC samples and materials are listed in Table 8.

Table 7. Laboratory Analysis Plan for 2019 RMP Sport Fish Samples

Parameter	Method	Lab Agency	Reporting Units	Species List
PCBs	MLA-010 *(EPA 1668 HRGC/HRMS, co- extracted by MLA-013)*	SGS AXYS	ng/g ww	• White Croaker • Shiner Surfperch • White Sturgeon • Anchovy • Pacific Herring • Pacific Sardine • Staghorn Sculpin • Brown Rockfish • Blue Rockfish • Bat Rays • Monkeyface Prickleback • Largemouth Bass • Common Carp
PBDEs	MLA-033 *(EPA 1614 HRGC/HRMS, co- extracted by MLA-013)*	SGS AXYS	ng/g ww	• White Sturgeon • Striped Bass • Largemouth Bass • Common Carp
Dioxins	MLA-017 *(EPA 1613B*	SGS AXYS	pg/g ww	• White Croaker • Shiner Surfperch • Shiner Surfperch (SWAMP)

	HRGC/HRMS, co-extracted by MLA-013)			
PFAS	MLA-110 *(LC-MS/MS, minimum batch size of 8)*	SGS AXYS	ng/g ww	• White Croaker • Shiner Surfperch • White Sturgeon • Striped Bass • Largemouth Bass • Common Carp
Hg - Indiv	EPA 7473	MPSL - MLML	ug/g ww	• Striped Bass • Brown Rockfish • Largemouth Bass
Hg - Comp	EPA 7473	MPSL - MLML	ug/g ww	• White Croaker • Shiner Surfperch • White Sturgeon • Striped Bass • Halibut • Anchovy • Jacksmelt • Pacific Herring • Pacific Sardine • Staghorn Sculpin • Blue Rockfish • Bat Rays • Monkeyface Prickleback
Se - Indiv	ICP-QQQ-MS, EPA 6020, Mod. with EPA 3050B digestion BAL-5000	BAL	ug/g dw	• White Sturgeon
Se- Comp	ICP-QQQ-MS, EPA 6020, Mod. with EPA 3050B digestion	BAL	ug/g dw	• White Croaker • Shiner Surfperch • Striped Bass • Halibut • Jacksmelt
Fipronil	EPA 8270D (M)/TQ/EI	Eurofins	ug/kg dw	• Shiner Surfperch • Striped Bass • Halibut • Common Carp
Microplastic	*Will only be archived*	University of Toronto	N/A	• Shiner Surfperch • Striped Bass

8. Quality Control Samples

Detailed analytical method and QA/QC protocols can be found in Elements 13 and 14 of the 2019 Quality Assurance Program Plan for the Regional Monitoring Program for Water Quality in San Francisco Bay (RMP 2018). Table 8 outlines expected QC samples for each analyte. Where the frequency is described as “1 per 20 or batch/set”, it indicates the minimum should be the higher frequency of the two (e.g., a lab batch of 10 samples would still require at least one lab blank; conversely, a reporting set of 35 field samples would include 2 or more CRMs even if they were all reported as a single dataset).

Archived samples will also be analyzed for analytes for which they had been previously reported. This is to evaluate the stability or consistency of samples and of the analytical methods used (whether the same lab is used as in the prior round or not). For the more abundant species collected (5 or more composites for a given analysis), a number of archive samples from prior sampling rounds equivalent to ~10% (rounded to the nearest whole number) of the new sample count will be reanalyzed. Ideally the results will be within the acceptable precision range for lab replicates if the lab is unchanged, or within the accepted range for accuracy if the lab is changed.

Table 8. Number of Field samples and Laboratory Quality Control Samples for 2019 Sport Fish Study

Laboratory	Parameter	Field Samples	Lab Blank	Lab Dupe	MS[1]	MSD[1]	CRM[1]	LCS[1,2]	Hist. Archive
Reporting units		*ug/g ww*	*ug/g ww*	*ug/g ww*	*ug/g ww*	*ug/g ww*	*ug/g ww* [5]	*ug/g ww* [5]	*ug/g ww*[6]
Eurofins	Fipronil and degradates	13	1 per 20 or set				1 per 20 or set		
BAL	Se - indiv	24	1 per 20 or batch				1 per 20 or set		
	Se - comp	48	1 per 20 or batch				1 per 20 or set		~10%
SGS-AXYS	PCBs	96	1 per 20 or batch			NA[3]	1 per 20 or set		~10%
	PBDEs	22	1 per 20 or batch			NA[3]	1 per 20 or set		~10%
	Dioxin	21	1 per 20 or batch			NA[3]	1 per 20 or set		~10%
	PFAS	16	1 per 20 or batch			NA[3]	NA		~10%
MLML	Hg - indiv	107	1 per 20 or batch				1 per 20 or set		
	Hg - comp	78	1 per 20 or batch				1 per 20 or set		~10%

1. MS=Matrix Spike; MSD=Matrix Spike Duplicate; CRM=Certified Reference Material; LCS = Lab Control Sample (e.g., OPR = ongoing precision & recovery or other lab internal recovery sample) .
2. LCS efforts should be conducted by the lab's SOPs for type and frequency; reporting is mandatory if MSs and CRMs or other natural matrix recovery samples are not possible/available (e.g., due to MS sample failure/insufficient material, or lack of certified reference materials for the given or similar matrix).
3. Matrix spike duplicates are not required for PCBs, PBDEs, dioxins and PFASs due to limits on sample mass. However, if concentrations in lab duplicate samples are expected to be non-detect or close to the method detection limit (<-3xMDL), then SFEI may request that MSDs (at 5-20xMDL) be prioritized (over unspiked lab duplicates) and performed on samples with extra mass to assess repeatability of laboratory methods.
4. Microplastic blank methods involve simulating the same procedures as used to apportion other samples and then providing the analytical lab with the resultant empty container.

5. CRM results and expected values should be reported in whatever basis (ww or dw) the material certificate provides (ww if both present). LCS results and expected values should be reported as an approximate equivalent concentration and basis for a “typical” analyzed subsample, e.g., if a typical analyzed sample mass is 1 g ww, 10 ng of target analyte in an LCS would be reported as 10 ng/g ww. This puts the LCS and field sample results on equivalent scales to allow evaluation as to whether the LCS is at a low/moderate/high concentration equivalent; LCSs at <20x average field sample concentrations are more relevant and thus preferred.
6. Historical archive samples are to be analyzed for species with 5 or more composites. Archived composites from prior sampling rounds are to be analyzed only for those species where sufficient counts for those analytes are achieved. ~10% indicates a count equal to 10% (rounded) of the new sample count for a given combination of species and analyte.

9. Samples for RMP Archives

As mentioned in Section 5, MPSL-DFW will retain samples until all analyses are complete, and analytical and QC results have been reviewed and approved by the SFEI Project Manager and QA Officer, and until the SFEI Project Manager approves of discarding samples. Short-term archive samples will be stored at Schafer’s Cold Storage in Oakland, CA. Long-term archive samples will be stored at the NIST Biorepository facility in Charleston, SC. Archives samples will be taken from composite samples.

The number and volume of archive samples of each species, in addition to sample container type, are outlined in Table 5. Further details on storage, and transport information can be found in Attachment 6. Minimum mass required per archive can be found in Table 6. This archiving plan deviates slightly from the RMP Archiving strategy (Appendix 2 of SFEI, 2017) by including largemouth bass and common carp, but is historically consistent with archiving procedures for white croaker and shiner surfperch. Additionally any collections of bycatch species will be treated as short term archive samples and stored as whole fish.

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-month average if fish from multiple sample dates are contained in the same composite), and container type and number (if >1 archive per composite).

Staff at MPSL-DFW will fill the archive containers per the compositing plan outlined in Section 6. MPSL-DFW will ship long-archives directly to NIST in Charleston, SC and ship the short term archives to AMS who will be responsible for adding the short-term archives to Shaefer’s Cold Storage in Oakland, CA. Archives placed in short-term storage will be assigned a lot number and tagged for intended use when added to the freezer. The lot numbers along with the information on the COCs, will be provided to SFEI in a spreadsheet.

Archives in long-term storage will require specialized pre-printed barcoded labels. MPSL-DFW will send NIST a completed template (Attachment 7) of sample information that is needed on the label. Once the completed template is received, NIST will send MPSL-DFW a set of pre-printed

barcoded labels containing the following information: the barcode (this refers to the Globally Unique Aliquot ID created by the NIST sample tracking database), fish species, composite ID (NIST refers to this field as the “field ID”), year collected (with blank spaces where month/day can be written in) and aliquot ID. NIST will also provide pre-cleaned sampling containers to MPSL DFW, both of which should arrive at MPSL-MLML before 8/31/19. NIST will add additional sample identifiers to the archive sample information provided by MPSL DFW, and will provide all such information to SFEI in a spreadsheet. Additional sample identifiers are: “Other ID” (longer version of the aliquot ID including the full site name) and “Globally Unique Aliquot ID” (NIST assigned).

MPSL-DFW will provide an electronic record of all archive samples to SFEI including information on composite IDs, aliquot IDs, container type, aliquot mass (units and basis), as well as the location of archive storage (NIST or Schafer’s).

10. Reporting

MPSL-MLML will summarize the 2019 sport fish field sampling effort (in a Cruise Report) and total mercury concentrations for inclusion in the RMP technical report. The MPSL-MLML data summary shall include collection information for fish in a CEDEN EDD template. The template will include information such as station name, collection date, sample unique ID, species, length, weight, etc. Additionally, MPSL-MLML will provide mercury analysis results as well as recommendations, if any, to improve methods for future sport fish sampling.

Laboratory results from SGS AXYS, MPSL-DFW, Eurofins and BAL analyses will be provided to SFEI in SFEI provided templates with all associated quality assurance metadata as detailed in Table 8. The field sampling information for this study as discussed in Sections 4 and 5 will be included in the Cruise Report prepared by MPSL-MLML.

The results from this study will be reported to the TRC and Steering Committee, and may appear in the 2020 RMP Update report (October 2020).

References

Rochman, C. M.; Tahir, A.; Williams, S. L.; Baxa, D. V.; Lam, R.; Miller, J. T.; Teh, F.; Weroilangi, S.; Teh, S. J. 2015. Anthropogenic debris in seafood: Plastic debris and fibers from textiles in fish and bivalves sold for human consumption. “Anthropogenic Debris in Seafood: Plastic Debris and Fibers from Textiles in Fish and Bivalves Sold for Human Consumption.” Scientific Reports 5 (September): 14340. https://doi.org/10.1038/srep14340.

Yee, D.; Franz, A.; Wong, A.; Trowbridge, P. 2018. 2018 Quality Assurance Program Plan for the Regional Monitoring Program for Water Quality in San Francisco Bay. SFEI Contribution No. 890. San Francisco Estuary Institute : Richmond, CA. http://www.sfei.org/documents/2018-quality-assurance-program-plan-regional-monitoring-program-water-quality-san-francisco-bay.

Attachments

Attachment 1. Field Sheets
https://drive.google.com/open?id=1_oLZLUJI3EL-oc7QsuS3ObHfAqpWbE4y

Attachment 2. Shipping Details
https://docs.google.com/spreadsheets/d/1nt1bRwbOJZCaeX6-gqfFHFC6xHimEffMGyHEvL_E-sE/edit#gid=0

Attachment 3. Standard Operating Procedure for Fish Dissection and Processing, MPSL Method #105
https://drive.google.com/open?id=1Vl5IdEOn0U79PI5AJGTIESAG7TK1l1sh

Attachment 4. Compositing plan
https://docs.google.com/spreadsheets/d/13-rjsrPI27HvN97U3AKVlPJGXJf2lLmq33spUuFFvxw/edit#gid=0

Attachment 5a. 2019 Sport Fish Target Analytes & Report Limits Table
https://drive.google.com/open?id=1oY7CHzJdBAJNS97rp9PuXS53XbE-iBYt

Attachment 6. 2019 Sport Fish Archiving Protocol
https://docs.google.com/document/d/1_Vu9PhflWwHOcJpeGJzvWo8u0zMRf3CFtNJcTAFIbcU/edit

Attachment 7. NIST Archive Label Template
https://drive.google.com/open?id=1QIbPE7hxqU_nWJ1oJWZgMWIZ_vALrSqC