

February 1, 2022

Mr. Patrick Pulupa, Executive Officer Central Valley Regional Water Quality Control Board 11020 Sun Center Drive, #200 Rancho Cordova, CA 95670

Sent via electronic mail to Patrick.Pulupa@waterboards.ca.gov

RE: SUBMITTAL OF DELTA REGIONAL MONITORING PROGRAM ANNUAL REPORT PER RESOLUTION R5-2021-0054

Dear Mr. Pulupa,

Please find attached the Delta Regional Monitoring Program's (DRMP) 2020-2021 Annual Report, as required by Resolution R5-2021-0054, Item 5 of Attachment A. During the reporting period of fiscal year (FY) 2020 - 2021, the Delta RMP underwent a transition in structure, governance, and management. However, despite these and continued challenges due to COVID-19 and its associated restrictions and other restrictions, the DRMP maintained a robust program.

We appreciate working with your staff to present this report in a format that meets Central Valley Regional Water Quality Control Board (Regional Water Board) needs.

As required by the Resolution, the 2020-2021 Annual Report summarizes all monitoring projects or studies conducted during fiscal year 2020-2021. The report includes a list of all publicly available datasets (including data and metadata), explanations for why any aspect of the Monitoring Workplan was not completed, and any deviations from the Monitoring Workplan, Data Management Plan, or the QAPP.

The Annual Report includes two quality assurance sections, one for data managed by the DRMP and one where data is not managed by the DRMP. The Annual Report identifies and describes all Quality Assurance Project Plan (QAPP) deviations and any other project deviations that impacted the quality of the DRMP data to ensure data are of known and documented quality. This section also includes: a list and description of all deviations to the QAPP; the corrective action(s) taken to address the deviation(s); a description of how the Delta RMP monitors the effectiveness of any corrective actions and ensures any deviations do not occur frequently in the future; a summary of dataset completeness, precision, and accuracy; a list and description Mr. Patrick Pulupa RE: Delta RMP Annual Report Submittal February 1, 2022

of sample comparisons or tests that did not meet minimum test acceptability criteria for analyses or were considered invalid; results for all analyses completed during the reporting period and comparison of results to previous year's observations, if applicable; and, a list of monitoring data (and associated metadata) that do not meet predetermined quality control measures and measurement quality objectives.

The FY 20-21 Annual Report is included below. Additionally, four files (Attachment A – D) are attached separately as Excel workbooks and transmitted in the email with this letter.

If you have any questions regarding the report, please do not hesitate to reach out to Melissa Turner, the DRMP's Program Director at <u>mturner@mljenvironmental.com</u> or by phone at (530) 756-5200, or to me at <u>eofficer@cvcwa.org</u> or at (530) 268-1338.

Sincerely,

Debbie Webster

Debbie Webster, President Delta Regional Monitoring Program

Attached Separately: Attachment A DRMP CEC Yr1 Data Attachment B DRMP Hg Yr5 EDDs Attachment C DRMP CUP FY20-21 Data Attachment D DRMP SEP Microcystis Study Data

cc: via email

Adam Laputz - CVRWQCB Meredith Howard – CVRWQCB Selina Cole - CVRWQCB Melissa Turner – DRMP Program Director Jennifer Glenn – MLJ Environmental Lisa McCrink – MLJ Environmental DRMP Board of Directors



Delta RMP Annual Report

Fiscal Year July 1, 2020 – June 30, 2021

Submitted February 1, 2022 To the Central Valley Regional Water Quality Control Board

TABLE OF CONTENTS

1	Intr	oduction	5
2	Pro	gress of FY 20-21 Monitoring Projects	8
	2.1	Summary of Public Datasets	8
	2.2	Delta RMP Monitoring	
	2.2.	1 Mercury Study	12
	2.2.	2 Nutrients Studies	13
	2.2.	3 Pesticides and Toxicity Multi-Year Study	17
	2.2.	4 Constituents of Emerging Concern	18
	2.3	Deviations and Corrective Actions	
	2.3.	1 Summary of Deviations from Delta RMP QAPP	
	2.3.	2 Summary of Deviations from Delta RMP CEC QAPP	
3	Qua	ality Assurance – Data Managed by the Delta RMP	
	3.1	Constituents of Emerging Concern	
	3.1.	1 CEC Year 1 Monitoring Results	
	3.2	Pesticides and Aquatic Toxicity	
	3.2.	1 Current Use Pesticides	
	3.2.	2 Aquatic Toxicity	56
4	Qua	ality Assurance – Data Not Managed by the Delta RMP	
	4.1	Mercury Monitoring	58
	4.2	Nutrients	59
	4.2.	1 Cyanotoxin Monitoring in the Delta, USGS, and DWR	
	4.2.	2 Source Tracking of Cyanotoxin Blooms in the Delta, Bend Genetics, and	
	CVF	RWQCB	59

LIST OF TABLES

Table 1. Quality assurance assessment requirements of Board Resolution R5-2021-0054	6
Table 2. Publicly available datasets on CEDEN under the Program Code Delta RMP	10
Table 3. Publicly available datasets not on CEDEN.	11
Table 4. Microcystis study sampling dates.	15
Table 5. Summary of QAPP deviation forms submitted during FY 20-21.	22
Table 6. Precision measurement acceptability for Year 1 CEC Pilot Study.	30
Table 7. Blank sample acceptability for Year 1 CEC Pilot Study	31
Table 8. Spike sample acceptability for Year 1 CEC Pilot Study	31
Table 9. Surrogate recovery acceptability for Year 1 CEC Pilot Study	32
Table 10. Precision measurement acceptability for pesticide samples collected during FY 20-	21.
	35
Table 11. Blank sample acceptability for pesticide samples collected during FY 20-21	42



LIST OF FIGURES

Figure 1. Overview of progress of Delta RMP projects and studies during FY 20-21
Figure 2. Summary of monitoring events in relation to study periods occurring during FY 20-21
for all monitoring sectors

LIST OF APPENDICES

Appendix I – Mercury Monitoring Cruise Reports Appendix II – Microcystis Study Report Appendix III – Current Use Pesticides USGS Field and Chemistry Report Appendix IV – CEC Year 1 Data Report

LIST OF ATTACHMENTS

- Attachment A Constituents of Emerging Concern Year 1 Data
- Attachment B Mercury Electronic Data Deliverables for Year 5 Data
- Attachment C Current Use Pesticides and Toxicity Data for Fiscal Year 20-21
- Attachment D Microcystis Study Data

LIST OF ACRONYMS

AMS	Applied Marine Sciences
AQT	Advanced Query Tool
ASC	Aquatic Science Center
ASTM	American Society for Testing and Materials
CDEC	California Data Exchange Center
CEC	Constituent of Emerging Concern
CEDEN	California Environmental Data Exchange Network
CHABs	Cyanobacteria Harmful Algal Blooms
CRM	Certified Reference Material
CUP	Current Use Pesticides
CV RDC	Central Valley Regional Data Center
CVRWQCB	Central Valley Regional Water Quality Control Board
Delta RMP	Delta Regional Monitoring Program



5 5 4 4	
DFW	Department of Fish and Wildlife
DOC	Dissolved Organic Carbon
DWR	Department of Water Resources
EDD	Electronic Data Deliverable
ELISA	Enzyme-linked Immunoassay
EPA	Environmental Protection Agency
EVR	Effluent Valve Replacement
FY 19-20	Fiscal Year 2019-2020
FY 20-21	Fiscal Year 2020-2021
GC/MS	
	Gas Chromatography Mass Spectrometry
HAB	Harmful Algal Bloom
LC/MS/MS	Liquid Chromatography Mass Spectrometry - Mass Spectrometer
MDL	Method Detection Limit
MeHg	Methylmercury
MLML	Moss Landing Marine Laboratories
MPSL	Marine Pollution Studies Lab
MQO	Measurement Quality Objectives
MTL	Monitoring Trigger Level
NWIS	National Water Information System
NWQL	National Water Quality Laboratory
OCRL	Organic Chemistry Research Laboratory
PBDE	Polybrominated Diphenyl Ethers
PER	Pacific EcoRisk
PFAS	Per- and Polyfluoroalkyl Substances
PFOA	Perfluorooctanoic acid
PFOS	Perfluorooctanesulfonic acid
PPCP	Pharmaceuticals and Personal Care Product
QA	Quality Assurance
QAPP	Quality Assurance Project Plan
QC	Quality Control
qPCR	Quantitative Polymerase Chain Reaction
RL	Reporting Limit
RMA	Risk Management Agency
SC	Steering Committee
SEP	Supplemental Environmental Project
SFEI	San Francisco Estuary Institute
SM	Standard Methods
SOP	
	Standard Operating Procedure
SPATT	Solid Phase Adsorption Toxin Tracking
SRINCS	Sacramento River Nutrient Change Study
SRWTP	Sacramento Regional Wastewater Treatment Plant
SSC	Suspended Sediment Concentration
SWAMP	State Board Surface Water Ambient Monitoring Program
SWRCB	State Water Resource Control Board
TAC	Technical Advisory Committee
TIE	Toxicity Identification Evaluation
TMDL	Total Maximum Daily Load
TSS	Total Suspended Solids
57	



3 Delta RMP Annual Report for FY 20-21

USGS	United States Geological Survey
VSS	Volatile Suspended Solids
WY	Water Year
YSI	Yellow Springs Instrument



1 INTRODUCTION

This Annual Report is being submitted to the Central Valley Regional Water Quality Control Board (Regional Board or CVRWQCB) in accordance with Resolution R5-2021-0054 which was adopted October 15, 2021. The Annual Report documents the status of monitoring and special studies conducted by the Delta Regional Monitoring Program (Delta RMP) during the 2020-2021 Fiscal Year (FY 20-21), spanning from July 1, 2020, through June 30, 2021. Work conducted during this period was based on two Workplans developed and approved by the Steering Committee (SC) for the FY 20-21: Draft Workplan and Budget for the First Quarter of the 2020-2021 Fiscal Year (approved on June 25, 2020, <u>Delta RMP FY 20-21 Q1 Workplan</u>) and Detailed Workplan and Budget for Quarters 2-4 of the 2020-2021 Fiscal Year (approved on January 26, 2021, <u>Delta RMP FY 20-21 Q2-4 Workplan</u>).

The Delta RMP underwent a transition in structure and governance during FY 20-21. During this time the management responsibilities of the former implementing entity, the Aquatic Science Center (ASC) were transferred to Melissa Turner of MLJ Environmental, who began serving as Interim Program Manager in December of 2020. Given the magnitude and rapid pace of these changes, the FY 20-21 Workplan was developed in two phases with the first phase covering quarter 1 of the fiscal year and the second phase covering quarters 2-4.

Monitoring during FY 20-21 occurred across four monitoring sectors and is described in the second phase <u>Workplan (Q2-4)</u>:

- Mercury
- Nutrients
 - o Microcystis
 - United States Geological Survey (USGS) / Department of Water Resources (DWR) Cyanobacteria Study
- Pesticides and Aquatic Toxicity
- Constituents of Emerging Concern (CECs)

The status of each planned monitoring project is outlined below. A **Summary of Public Datasets**, **Deviations and Corrective Actions**, and the status of all projects and studies conducting **Delta RMP Monitoring** is provided below in **Progress of FY 20-21 Monitoring Projects**. Quality assurance assessments for each project and study are provided in the **Quality Assurance** sections according to the requirements outlined in **Table 1**. An overview of the progress of monitoring events, data acquisition, and reports for each of the Delta RMP projects and studies during FY 20-21 is summarized in **Figure 1**.



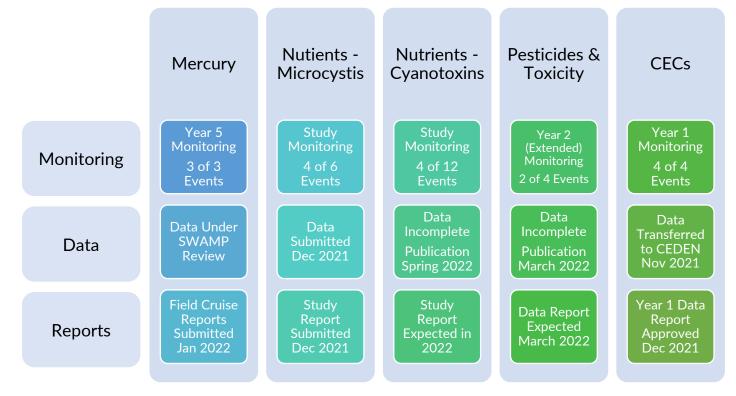
Annual Report Requirement from Resolution (Attachment A, 5 a-vii)	SECTION NUMBER	SECTION HEADER
Summarize all monitoring projects or studies conducted during the prior fiscal year.	2.2	Delta RMP Monitoring
Explanation for why any aspect of the Monitoring Workplan was not completed.	2.2	Delta RMP Monitoring
List of all publicly available datasets (including data and metadata).	2.1	Summary of Public Datasets
Deviations from the Monitoring Workplan, Data Management Plan, and Quality Assurance Project Plan (QAPP).	2.2, 2.3	Delta RMP Monitoring, Deviations and Corrective Actions
	3	Quality Assurance – Data Managed by the Delta RMP
Quality Assurance Section	4	Quality Assurance – Data Not Managed by the Delta RMP
List and description of all deviations to the QAPP.	2.3	Deviations and Corrective Actions
Corrective action(s) taken to address the deviation(s)	2.3	Deviations and Corrective Actions
Description of how the Delta RMP monitors the effectiveness of any corrective actions and ensure any deviations do not occur frequently in the future.	2.3	Deviations and Corrective Actions
Summary of dataset completeness.	3.1.1, 3.2.1.1, 3.2.2.1	Quality Control Sample Completeness
Summary of dataset precision.	3.1.1.2, 3.2.1.2, 3.2.2.2	Acceptability of Precisior Measurements
Summary of dataset accuracy.	3.1.1.3, 3.2.1.3, 3.2.2.3	Acceptability of Accuracy Measurements
List and description of sample comparisons or tests that did not meet minimum test acceptability criteria for analyses or were considered invalid.	3.1.1.4, 3.2.1.4, 3.2.2.4	Invalid Data
Results for all analyses completed during the reporting period and comparison of results to previous year's observations, if applicable.	Attachments A-D	NA
List of monitoring data (and associated metadata) that do not meet predetermined quality control measures and measurement quality objectives.	Attachments A-D	NA

Table 1. Quality assurance assessment requirements of Board Resolution R5-2021-0054.



Figure 1. Overview of progress of Delta RMP projects and studies during FY 20-21.

Not all studies start and end within a fiscal year; the number of events listed indicates the number of events completed in the fiscal year.





2 PROGRESS OF FY 20-21 MONITORING PROJECTS

2.1 SUMMARY OF PUBLIC DATASETS

A summary of datasets collected during FY 20-21 that have been published to an approved public database are outlined in **Table 2** for data in the California Environmental Data Exchange Network (CEDEN) and in **Table 3** for data in other publicly available databases such as National Water Information System (NWIS).

For the FY 20-21, three of the five monitoring sectors have datasets transferred to CEDEN:

- Mercury
- Pesticides and Aquatic
- CECs

Of these three projects, the Year 1 data set for CECs has been successfully transferred from the Central Valley Regional Data Center (CV RDC) to CEDEN. Data were transferred upon approval by the CEC Technical Advisory Committee (CEC TAC) in November 2021¹. Though all Year 1 data were successfully transferred to CEDEN, not all results are available through the CEDEN Advanced Query Tool (AQT) due to updates to database vocabulary regarding isotope dilution methods that are pending finalization by State Water Resource Control Board (SWRCB or State Board) staff. Finalized CEC data transferred to CEDEN are included in Attachment A.

Mercury data are submitted to the State Board Surface Water Ambient Monitoring Program (SWAMP) staff by Moss Landing Marine Laboratories (MLML) as CEDEN comparable Electronic Data Deliverable (EDDs). Once received, data undergo a final review and are uploaded to CEDEN by SWAMP staff. Mercury results were submitted to the State Board in June of 2021; as of January 17, 2022, the FY20-21 mercury data were still under review by SWAMP. Mercury project data will be available on CEDEN once this review is complete. For this Annual Report, the original mercury CEDEN EDDs submitted to SWAMP are included as Attachment B.

Current use pesticides (CUP) and aquatic toxicity project data are processed and evaluated on a water year (WY) basis. The dataset for the 2021 WY is anticipated to be verified and transferred to CEDEN in March of 2022. The transfer of these data to CEDEN is anticipated to coincide with a Data Report which evaluates the 2021 WY dataset in its entirety. Attachment C includes the CUP and aquatic toxicity data for the events sampled in the FY 20-21. Toxicity laboratory reports are available on the <u>DRMP Droplet</u> site which is accessible to Regional Board staff and TAC members.

¹ On October 28, 2021, the DRMP BOD approved the October 27, 2021 SC recommendation to expedite the data approval process. The BOD directed the CV RDC to transfer the CEC Year 1 data to CEDEN once the CEC TAC reviewed and approve the data.



Of the two nutrient studies taking place in FY 20-21, the Microcystis study conducted by Bend Genetics and the Regional Board has a complete dataset that is available for publication. These data have been submitted to Regional and State Board staff in tabular form (Attachment D); however, transfer to CEDEN is not yet possible because the database currently lacks the configuration for storing the results of quantitative polymerase chain reaction (qPCR) analyses. Efforts are ongoing at State Board to generate guidance for accurate and consistent storage of similar data generated by studies of harmful algal blooms (HABs) in CEDEN; at this time the final dataset is included as Attachment D of this report.



Parent Project Name	PARENT PROJECT CODE	PROJECT NAME	PROJECT CODE	Agency	SAMPLE PERIOD	Status
		2020 Delta RMP Current Use Pesticides	20DRMP5CUP	USGS	10/1/2020 - 9/30/2021	Data loaded into the CVRDC: all field data, 4 of 4 events for toxicity, 2 of 4 events for chemistry. Expected to have data in CEDEN in 2022.
Delta RMP -		2019 Delta RMP Current Use Pesticides	19DRMP5CUP	USGS	10/1/2019 - 9/30/2020	Available on CEDEN.
Current Use Pesticides ¹	DRMP_CUP	2018 Delta RMP Current Use Pesticides	18DRMP5CUP	USGS	10/1/2018 - 9/30/2019	Available on CEDEN.
		2016 Delta RMP Current Use Pesticides	16DRMP5CUP	USGS	7/1/2016 - 6/30/2017	Available on CEDEN.
		2015 Delta RMP Current Use Pesticides	15DRMP5CUP	USGS	7/1/2015 - 6/30/2016	Available on CEDEN.
Delta RMP - Constituents of Emerging Concern	DRMP_CEC	2020 Delta RMP Constituents of Emerging Concern	20DRMP5CEC	SFEI	7/1/2020 - 6/30/2021	Available on CEDEN. ²
		2020 Delta RMP Mercury	20DRMP5Hg	MPSL-DFW	7/1/2020 - 6/30/2021	Data finalization underway; project is being managed by SWRCB.
		2019 Delta RMP Mercury	19DRMP5Hg	MPSL-DFW	7/1/2019 - 6/30/2020	Loaded into the SFEI RDC; pending transfer to CEDEN.
Delta RMP - Mercury	DRMP_Hg	2018 Delta RMP Mercury	18DRMP5Hg	MPSL-DFW	7/1/2018 - 6/30/2019	Available on CEDEN.
		2017 Delta RMP Mercury	17DRMP5Hg	MPSL-DFW	7/1/2017 - 6/30/2018	Available on CEDEN.
		2016 Delta RMP Mercury 16DRMP5Hg MPSL-DFW		MPSL-DFW	7/1/2016 - 6/30/2017	Available on CEDEN.
Delta RMP -	DRMP PAT	2016 Delta RMP Pathogens	16DRMP5PAT	SFEI	4/1/2016 - 3/31/2017	Loaded into the SFEI RDC; pending transfer to CEDEN.
Pathogens	DRIVIP_PAT	2015 Delta RMP Pathogens	15DRMP5PAT	SFEI	4/1/2015 - 3/31/2016	Available on CEDEN.

Table 2. Publicly available datasets on CEDEN under the Program Code Delta RMP.

¹The Current Use Pesticides Parent Project Code includes data for pesticides, aquatic toxicity, copper, and ancillary parameters.

²Year 1 CEC Pilot Study data were transferred to CEDEN from the CV RDC on 11/27/2021; however, due to pending CEDEN vocabulary approvals based on guidance from State Board, 107 analytes are not yet available for public viewing through the Advance Query Tool.



The results of the cyanotoxin study conducted by the USGS and California DWR is not yet complete and ready for publication. Once these data are received and finalized, they will be uploaded to a combination of USGS and DWR public databases. The whole water sample analysis results generated by this study will be uploaded to NWIS under the USGS site numbers identified in **Table 3**. These results, along with those generated by the analyses of the Solid Phase Adsorption Toxin Tracking (SPATT) samples, will be published to the USGS ScienceBase; data are expected to be publicly available in Spring of 2022.

Continuous data collected are available through NWIS for the stations managed by USGS (LIB and MDM). Continuous data collected at stations managed by DWR (P8, RRI, and C10A) are available through the California Data Exchange Center (CDEC).

STUDY	LOCATION	Түре	SITE CODE	USGS SITE NUMBERS	Sample Period	Status	
			LIB	11455315			
			MDM	11312676		Data	
	NWIS Web	Whole Water	P8	375841121225601	3/1/2021 -	Publication in 2022	
	Interface ¹	Cyanotoxin Results	RRI	375747121215401	2/1/2022		
USGS/DWR			C10A	374045121155200			
Cyanobacteria Study	USGS ScienceBase ²	Whole Water and SPATT Sampler Cyanotoxin Results	NA	NA	3/1/2021 - 2/1/2022	Anticipated publication of provisional results in Spring 2022	

Table 3. Publicly available datasets not on CEDEN.

¹NWIS Web Interface is located: <u>https://nwis.waterdata.usgs.gov/usa/nwis/qwdata</u>

²USGS ScienceBase is located: <u>https://www.sciencebase.gov/catalog/</u>



2.2 DELTA RMP MONITORING

During the FY 20-21, monitoring and reporting activities occurred for mercury, nutrients, pesticides and aquatic toxicity, and CECs. **Figure 2** is an overview of the monitoring events that occurred during FY 20-21 relative to the monitoring design study period. Below is a description of the monitoring studies and associated activities that occurred during the FY 20-21.

2.2.1 Mercury Study

Fiscal year 2020-21 mercury monitoring evaluated mercury cycling in Delta water, and the uptake of methylmercury (MeHg) into fish. This year completed the fifth year of this project to support annual monitoring of higher trophic level fish and correlated this information to mercury and MeHg water and sediment concentrations measured at co-located sites. This information is critical to implementing the Delta MeHg Total Maximum Daily Load (TMDL), providing calibration and validation data for a California DWR mercury model, and informing other management and regulatory decisions related to water quality improvement and ecosystem restoration in the Delta.

This monitoring has provided essential evidence for regulators implementing the TMDL and contributed to ongoing analytical work by DWR.

The DWR model was used to guide regulations and operational decisions related to farming, flood control, and wetland management. Regional Board staff used these data to inform the 2020 Delta Mercury Control program including Phase 2 potential modifications and options.

As outlined in the <u>FY 19-20 Workplan</u> (Attachment B), there were three main elements of the FY 20-21 mercury monitoring design:

- 1. **Subregional trends in bass** Continued annual monitoring of methylmercury in black bass ("black bass" includes largemouth, smallmouth, and spotted bass) at seven stations (distributed among the TMDL subregions) to firmly establish baseline concentrations and interannual variation in support of monitoring of long-term trends as a critical performance measure for the TMDL. The design from the initial phase was planned to continue unchanged in the next phase. This design was planned to be re-evaluated after completion of a 10-year period (2016-2025).
- 2. **Subregional trends in water** Monitoring of methylmercury in water at seven stations in three sampling events (August 2021, and March and April 2022) extended the time series, with a low-cost approach, for time periods that are representative of conditions in high-flow (March and April) and low-flow (August) regimes and that link to concentrations in prey fish and black bass. These data may also be valuable in verifying trends and patterns predicted by numerical models of methylmercury transport and cycling being developed for the Delta and Yolo Bypass by the California DWR. These models may allow testing of various land and water management scenarios.
- 3. **Restoration monitoring** In a new element added in FY19-20, annual monitoring of methylmercury in black bass and prey fish at new stations (five for black bass and eight for



prey fish) located near habitat restoration projects will continue to assess the subregional impact of the projects on impairment. The details of the design for the restoration monitoring (station locations, mix of black bass and prey fish stations) have been determined with input from restoration managers and Delta RMP Mercury Subcommittee members.

Annual sport fish sampling started in August 2016 and is currently ongoing. The indicator of primary interest is total mercury in muscle fillet of 350-mm largemouth bass (or similar predator species). Total mercury in muscle fillet is a close surrogate for the element's more toxic form, methylmercury. The seven sites sampled are located to represent different subareas of the Delta andare co-located with the water monitoring sites. Sport fish monitoring occurred in September 2020 at 7 core locations and 5 restoration locations. Water sampling was conducted during three events (September, March, and April) at seven sites that align with sport fish monitoring sites. Indicators of primary interest are concentrations of methylmercury and total mercury in water. Important ancillary parameters include chlorophyll-a, dissolved organic carbon (DOC), suspended sediment concentrations (SSC), total suspended solids (TSS), and volatile suspended solids (VSS). The prey fish monitoring that was scheduled in May 2021 was originally postponed and then eventually cancelled due to permitting issues. Prey fish monitoring will not be included in the FY 21/22 mercury monitoring due to Delta smelt concerns and sensitive habitat permit restrictions. Cruise reports for the monitoring events conducted during FY 20-21 were provided to the Delta RMP on January 18, 2022 and are included as **Appendix I**.

During the FY 20/21, the following reports were approved by the Delta RMP and are available on the website:

Davis, J., D. Yee, W. Heim, A. Bonnema, and B. Jakl. 2021. Methylmercury and Total Mercury in Fish and Water from the Sacramento-San Joaquin Delta: Year Two (August 2017 – June 2018). Delta Regional Monitoring Program.

Davis, J., J. Ross, D. Yee, W. Heim, A. Bonnema, and B. Jakl. 2021. Methylmercury and Total Mercury in Fish and Water from the Sacramento-San Joaquin Delta: Year Three (June 2018 – June 2019). Delta Regional Monitoring Program.

Davis, J., J. Ross and W. Heim. 2021. Mercury and Methylmercury in Fish and Water from the Sacramento-San Joaquin Delta: Interpretive Report on the First Three Years of Monitoring (August 2016 – October 2019) by the Delta Regional Monitoring Program. Delta Regional Monitoring Program.

2.2.2 Nutrients Studies

2.2.2.1 2016 Water Year Modeling Report

Progress continues for the Delta-Suisun Water Year 2016 Hydrodynamic Biogeochemical Modeling Project being conducted by ASC. The project is a continued synthesis and integration of existing data to characterize status and trends of nutrient-related parameters and planning future monitoring and data analysis work. In December 2020, the Delta RMP agreed to extend the due date for the deliverables of this project from March to August 2021. The report was



delayed another month and was received on September 30, 2021, for review by the Nutrient Technical Advisory Committee (Nutrient TAC).

2.2.2.2 Chlorophyll Sensor Intercalibration Report

The "Chlorophyll Sensor Intercalibration Study" was a joint project between the Delta RMP and the San Francisco Bay Nutrient Management Strategy group and was a multi-agency effort. The chlorophyll sensor intercalibration study is a significant first step toward ensuring improved sensor network coordination that will help make better use of existing data collection efforts by state and federal agencies. In 2018-19, Phase 2 was completed when sensors from 6 different agencies were deployed side-by-side for two weeks to compare measurements at different locations in the Delta. These deployments occurred during May, July, and August. The <u>final</u> report was approved by the Steering Committee on June 24, 2021:

Stumpner E.B., J. Yin, M. Heberger, J. Wu, A. Wong, and Saraceno, J.F. 2021. San Francisco Estuary Chlorophyll Sensor and Sample Analysis Intercomparison. Delta Regional Monitoring Program.

2.2.2.3 Sacramento River Nutrient Change Study (SRiNCS) Report

Sampling for the Sacramento River Nutrient Change Study Phase 1: Effluent Valve Replacement Hold was conducted in September 2019. This study was a collaborative effort between Regional San, Applied Marine Sciences (AMS), USGS, and San Francisco State University. This study tracked the effects of changes in nutrient loading resulting from a short-term wastewater hold at the Sacramento Regional Wastewater Treatment Plant (SRWTP). In the summer of 2019, scheduled wastewater effluent holds occurred during the Effluent Valve Replacement (EVR) project, part of the EchoWater upgrade at the SRWTP. During an EVR hold, no treated effluent entered the Sacramento River for a period of up to 48 hours. Based on prior USGS research, this should create a parcel of effluent-free river water over six miles long in the Sacramento River. The impacts of short-term changes in nutrient loading were tracked in parcels of water with and without effluent during movement downstream in the Sacramento River and nearby channels. The project consisted of a one week-long river sampling campaign, field measurements, laboratory analyses, numeric modeling, and reporting. The project used multiple methods, including boat-mounted, high frequency monitoring of nutrients and fluorescence; discrete sampling for analyses of water quality, phytoplankton and zooplankton abundances, clam biomass, and phytoplankton carbon uptake (to determine growth rates). Data and hydrodynamic modeling were used to evaluate the response of phytoplankton to a range of nutrient loads and forms, as well as factors of light, turbidity, water residence time, and grazing by zooplankton and clams. A modeling report by Risk Management Agency (RMA) (standalone deliverable for the SRINCS project) was distributed to the Delta RMP Nutrients Subcommittee for review in 2020. Full study results and a draft final report were originally due in November 2020 but have been extended to early 2022 due to delays as a result of COVID-19 and additional time needed for internal reviews prior to submitting to the Delta RMP Nutrient TAC.



2.2.2.4 Microcystis Study

Cyanobacteria harmful algal blooms (CHABs) are a rising ecological issue in the Delta. Some locations are more prone to CHABs, but it is unclear where CHABs originate. The Source Tracking of Cyanobacteria Blooms in the Sacramento-San Joaquin Delta (also referred to as the Microcystis Study) is focused on the knowledge gap of understanding where blooms of the common CHAB genus, *Microcystis*, originate in the Delta. The project's primary hypothesis is that there are specific areas, where flows and tidal velocity are low, that contain high concentrations of benthic resting cells (*Microcystis* cells that overwinter at the sediment surface). These benthic resting cells ultimately recruit to the water column, grow into blooms at sites of overwintering, and are transported elsewhere in the Delta. It is also hypothesized that areas where CHABs are frequently observed and have higher flows and tidal velocities have relatively low-to-no benthic resting populations due to physical export from the system. This project was approved by the Delta RMP in August 2020 and is funded using Supplemental Environmental Project (SEP) funds obtained by the Regional Board as a result of enforcement actions.

The project began in November 2020. Water samples were collected during four events at 8 sites and sediment was collected during four events at 7-8 sites depending on the event. During November, there were issues collecting samples from the Clifton Forebay and San Joaquin River at Vernalis location. See **Table 4** for a list of sample dates.

Event	Day 1	Day 2	Day 3	Day 4	WATER	Sed	Sampling Notes
End of CHAB season	11/10/20	11/18/20	11/19/20	12/4/20		х	7 sites; Unable to collect at Clifton Forebay and Vernalis.
Very beginning of CHAB season	4/28/21	4/30/21	5/5/21	5/6/21		Х	8 sites; unable to collect at Vernalis
CHAB season (1)	6/4/21	6/7/21	6/9/21		Х	Х	8 sites
CHAB season (2)	6/29/21	6/30/21	7/2/21		Х	Х	8 sites
CHAB season (3)	7/14/21	7/15/21			Х		8 sites
CHAB season (4)	8/4/21	8/6/21			Х		8 sites

Table 4. Microcystis study sampling dates.

Molecular tools were used to analyze the samples including qPCR to quantify *Microcystis* and metagenomic sequencing of c-phycocyanin genes specific to cyanobacteria to develop unique genetic signatures or "fingerprints" of *Microcystis* assemblages in water and sediment samples. *Microcystis* source-tracking will be accomplished by comparing local sediment and water column abundances and strain profiles with adjacent sites across temporally relevant distances. Each molecular fingerprint will indicate the proportions of different strains of Microcystis in sediment



and water and changes in proportions of strains over time and space. Abundances of *Microcystis* resting cells and genetic characteristics of *Microcystis* in the water column and sediment will be used to test hypothesis about bloom origins. This work may ultimately be useful for identifying locations for implementation of focused CHAB management measures.

Dr. Ellen Preece, project lead, presented on the results from the study at the September 22, 2021 Delta RMP TAC. The final report was submitted to the Regional Board and the Delta RMP on December 31, 2021, and is attached as **Appendix II**. The report will be reviewed by the Nutrient TAC in early 2022.

2.2.2.5 USGS/DWR Cyanobacteria Study

The Delta RMP agreed to contribute funds to the following USGS/DWR monitoring effort, "Cyanotoxin Monitoring in the Delta: Leveraging existing USGS and DWR field efforts to identify cyanotoxin occurrence, duration and drivers" which included funds for the deployment of an additional instrument that monitors phytoplankton taxonomy continuously (bbe Fluoroprobe) at the Middle River station.

The study originally proposed to collect cyanotoxin data year-round (fall 2020 to fall 2021) from 4 stations in the Delta to enhance existing monitoring programs for flow, nutrients, water quality and phytoplankton, including HABs. Due to COVID-19 restrictions, sampling did not begin until March 2021. The Delta RMP funds will continue to fund this project through February 2022. The project includes measuring the presence of cyanotoxins with SPATT samplers and with discrete whole water sample collection at four locations: (1) Middle River at Middle River (MDM; USGS), (2) Liberty Island (LIB; USGS), (3) Vernalis (C10; DWR), and (4) Rough and Ready (P8; DWR). All stations measure flow and are equipped with Yellow Springs Instrument (YSI) EXOs field probes which measure water temperature, specific conductance, turbidity, pH, dissolved oxygen, and chlorophyll-a/blue-green algae. These stations also have a SUNA nitrate analyzer, except Rough and Ready.

The data will help identify linkages between environmental drivers (nutrients, flow, temperature) on HAB formation and cyanotoxin production, and can be used by managers and modelers to inform the design of future monitoring programs and to develop predictive models. The project will include online access to data and visualizations of spatial and temporal trends in cyanotoxins and associated data for use by managers and scientists. Findings will be presented at local conferences (e.g., Bay Delta, Interagency Ecological Program) and presented to the Delta RMP upon request. At the end of the project, a status and trend report that describes the approach and methods, summarizes any issues or lessons learned that occurred during data collection, provides tabular and/or graphical summaries of the spatial and temporal patterns in the data, evaluates the data quality, and relates study findings to the Delta RMP management questions will be provided. The report will also include comparison between the whole water and SPATT data and between the Liquid Chromatography Mass Spectrometry (LC/MS/MS) and Enzyme-linked Immunoassay (ELISA) data. The Delta RMP paid for 12 months of monitoring which will be completed in February 2022.



2.2.3 Pesticides and Toxicity Multi-Year Study

Water year 2021 (Oct 1, 2020 – Sept 30, 2021) was an extension of Year 2 of a multi-year study of current-use pesticides and aquatic toxicity in the Sacramento-San Joaquin Delta. A rotating basin monitoring design with monitoring at two fixed sites began in October 2018. The study design originally included a 4-year monitoring program covering six Delta sub-regions followed by an interpretive report will inform adaptive management and improve future monitoring. There were delays in continuing the Year 2 monitoring past March of 2020 due to delays in selecting a new toxicity laboratory. The Steering Committee decided to pause monitoring until the new toxicity laboratory was hired and to resume the Year 2 monitoring design in March 2021.

During that time, the Delta RMP solicited proposals for a new toxicity laboratory (previously the toxicity laboratory was the Aquatic Health Program Laboratory at UC Davis) and selected Pacific EcoRisk (PER) to perform toxicity analysis of the samples. Monitoring resumed in April 2021. There was a total of four events completed for the 2021 Water Year (October 2020 – September 2021), comprising Events 3 through 6 of the extended Year 2 monitoring.

Samples were analyzed for a suite of 174 CUP by the USGS Organic Chemistry Research Laboratory (OCRL). Compounds include fungicides, herbicides, insecticides, and their degradation products. In addition, crews measure field parameters (water temperature, pH, conductivity, dissolved oxygen, turbidity), and document conditions at the field site. The USGS National Water Quality Laboratory (NWQL) analyzes samples for copper and ancillary parameters (total nitrogen, total particulate carbon, particulate organic carbon, and dissolved organic carbon).

Pacific EcoRisk analyzed the toxicity of water samples for a suite of test organisms based on current Environmental Protection Agency (EPA) and SWAMP methods:

- *Ceriodaphnia dubia*, a daphnid or water flea (survival, reproduction) sensitive to organophosphate pesticides.
- Hyalella azteca, an aquatic invertebrate (survival) sensitive to pyrethroids
- *Selenastrum capricornutum* (also known as *Raphidocelis subcapitata*), a single-celled algae (growth) sensitive to herbicides.
- *Chironomus dilutes*, midge larvae (formerly *Chironomus tentans*) sensitive to fipronil and more sensitive in chronic exposures to imidacloprid than *C. dubia*.
- *Pimephales promelas* (growth, survival) chronic and acute effects on whole organism growth and survival.

The Delta RMP convened a Toxicity Identification Evaluation (TIE) Subcommittee in 2015 with the main responsibility of rapidly deciding, on a case-by-case basis, whether and how to allocate resources to conduct TIEs for samples exceeding a toxicity threshold (≥50% reduction in organism response relative to the lab control) and whether to conduct any follow-up analyses (e.g., additional TIE treatments, supporting analytical chemistry) with a sample where results may not clearly indicate a pesticide or class of contaminants causing toxicity. The TIE Subcommittee



was originally created to report results to the Delta RMP TAC. The Delta RMP reconvened the TIE Subcommittee in March 2021 with the charge that the subcommittee shall be lead and coordinated by the Delta RMP Program Manager along with the contracted toxicity laboratory and be composed of a representative from each of the following categories: agriculture, stormwater agencies, publicly owned treatment works, coordinated monitoring and regulatory agencies. There were four samples with TIEs performed during the 20/21 Water Year.

A USGS Field and Chemistry Report was provided to the Delta RMP on January 28, 2022, describing the samples collected for the 2021 Water Year and a summary of the pesticide results; this report is provided in **Appendix III**.

A report titled, "Analysis and Interpretation of Pesticides and Toxicity Monitoring Data in the Sacramento-San Joaquin Delta" included the Delta RMP's first two years of current use pesticides and toxicity data and other data available in the Delta from 2011-2016. The report and database were finalized in 2021 and reviewed by the Pesticide TAC Subcommittee and the TAC. The TAC had significant concerns regarding the data compiled in the database, the analysis, and the conclusions which were documented in a memo to the Steering Committee. The Steering Committee approved payment of the final invoice of the contract; however, due to concerns documented by the TAC and included in the June 24, 2021 Steering Committee Package, the Steering Committee determined that the report not be posted on the Delta RMP website and this memo be provided to stakeholder as a synopsis of the concerns raised with the technical aspects of the Report and the concerns of making the Report available to the public.

2.2.4 Constituents of Emerging Concern

During the FY 20-21, the Delta RMP initiated the July 2018 Central Valley Pilot Study for Monitoring Constituents of Emerging Concern Work Plan. In October 2019, the Steering Committee approved funding to complete Year 1 of the study. The CEC Year 1 project was partially paid for using SEP funding received by the Regional Board from enforcement funds. SEP funds were used to pay for the clam sampling, additional water and sediment sampling, and chemical laboratory analysis. The CEC Year 1 Quality Assurance Project Plan (QAPP) was approved by the Steering Committee in 2020 and sample collection began in September 2020.

Monitoring in September included monitoring of water (9 sites), sediment (3 sites), fish (4 sites), and clams (6 sites). Monitoring also occurred in January (first flush event), April (dry event) and June (dry event). The monitoring design includes two storm events; however, due to a lack of rain, the April event ended up being a dry event. In addition, due to COVID-19 restrictions, only some of the sites were sampled in January. ASC was able to collect water samples at three locations. The DWR was responsible for collecting the other water samples by boat; but due to COVID-19 restrictions, DWR staff were unable to complete the sampling. It was agreed by the CEC Subcommittee that it would be better to have some samples from this runoff event rather than no samples. Samplers from DWR were able to collect samples for the remaining two events (April and June).



The data from Year 1 were reviewed and assessed by ASC; all results have been shared with the Regional Board and were uploaded into the CV RDC in November 2021. Data were finalized and transferred to CEDEN in November 2021.

Aquatic Science Center provided a Year 1 Data Report to the CEC TAC who recommended review by the Steering Committee for approval by the BOD. The <u>CEC Year 1 Data Report</u> was approved by the BOD on December 16, 2021 and is included as **Appendix IV**:

Weaver M. and D. Yee. 2021. Pilot Study of Constituents of Emerging Concern in the Sacramento-San Joaquin Delta Year 1 Data Report. Delta Regional Monitoring Program.



	2	2019									0 2							20	021								
	October	November	December	January	February	March	April	Мау	June	July	August	September	October	November	December	January	February	March	April	May	June	July	August	Spetember	October	November	December
Delta RMP Monitoring					19-							•,		F	- - - Y 20	0-21									1-22		
Mercury	3			١	∕ear ₄	4						1			Yea	ar 5		2	3	X			1	Yea	ar 6		
Nutrients - Microcystis														1				Stuc	ly Pe 2		3/4	5	6				
Nutrients - Cyanotoxins									HF									1	2	St 3	udy 4	Peri 5	od 6	7	8	9	10
Pesticides & Toxicity			1		20 V 2					Ext	tende	d Yea	ar 2			20)21 W	٧Y	3		4		5	6		′ear 3 22 W	
CECs			-						_			1			Yea	ar 1 2			3		4			Yea	ar 2 1/2		
												1				2			- 3		4				1/2		

Figure 2. Summary of monitoring events in relation to study periods occurring during FY 20-21 for all monitoring sectors.



20 Delta RMP Annual Report for FY 20-21 February 1, 2022

2.3 DEVIATIONS AND CORRECTIVE ACTIONS

The process to track deviations using the Delta RMP deviation forms was first implemented in 2019 by ASC. Under Resolution R5-2021-0054, all procedures that constitute a deviation from the associated approved QAPP must be approved by the CVRWQCB prior to implementation. Where deviations occur due to unanticipated circumstances and prior approval is not possible, the Delta RMP must notify the CVRWQCB Quality Assurance (QA) Representative within seven calendar days of becoming aware of the deviation. The Resolution was adopted in mid-October, 2021, after the FY 20-21 was completed and therefore deviations reported in this section may not adhere to the timelines and process of notification as outlined within the Resolution.

Deviations from approved QAPPs are documented via deviation forms which include the following:

- A description of the deviation that occurred
- Reason for the deviation
- Impact on the present and completed work
- Corrective actions taken as a result, by when and by whom

The deviation forms generated during FY 20-21, the associated corrective actions, and any resolutions are summarized below in **Table 5**.



DEVIATION NUMBER	STATUS	DEVIATION DATE	MONITORING SECTOR	TITLE	DESCRIPTION	CORRECTIVE ACTIONS	RESOLUTION
	· · · · · · · · · · · · · · · · · · ·				Delta RMP QAPP v5	<u>.</u>	
2020-02	Created, pending final review	8/10/2020	Mercury	Extension of 19-20 Workplan/ QAPP to Cover Sept 2020 Fish Collection.	The Steering Committee approved an extension of the 19/20 Workplan and the associated 19/20 QAPP to cover sampling of mercury in black bass and in water in September 2020; the extension is needed due to a backlog of activities delaying the approval of the FY20/21 Workplan and QAPP.	N/A	N/A
2020-09	Created, pending final review	4/2/2021	Mercury	Chlorophyll a Samples Out of Hold Time March 2020	DRMP March 2020 chlorophyl-a samples were analyzed outside of hold time.	The results were flagged with the appropriate CEDEN QA code to indicate the hold time violation	Appropriate Data Qualification
2020-10	Created, pending final review	4/2/2021	Pesticides and toxicity	USGS Did Not Meet Planned QA Frequencies	Not all of the planned field QA samples were collected, due to the final three events being cancelled	USGS will modify the sampling design in future years to collect QA samples more proportionally to the field samples collected at each event to reduce the impact of event cancelations on QA sample completeness.	Initial evaluation of 2021 Field Quality Control (QC) Frequency indicates new procedures are effective; complete assessment to occur on entire 2021 WY dataset.
2020-17	Created, pending final review	4/2/2021	Pesticides	FY19-20 QAPP Frequency of Lab Blanks and Replicates	The FY19-20 QAPP specifies a "one per batch or 20" frequency for lab blanks and lab reps, which may be ambiguous. If interpreted on a batchwise basis it didn't/doesn't occur for some analytes. Some analytes reported by NWQL did not conform with general State Board expectations that lab QC samples should occur at a frequency of the greater of 1 per batch or 1 per 20 field samples for all analytes.	Clarify laboratory control procedures with NWQL. Incorporate additional sample collections in the 2021 WY to ensure volume is provided for additional QC samples.	QAPP Revised Assessment of laboratory QC sample frequencies to be conducted on 2021 WY data once received from the laboratory.

Table 5. Summary of QAPP deviation forms submitted during FY 20-21.



DEVIATION NUMBER	STATUS	DEVIATION DATE	MONITORING SECTOR	TITLE	Description	Corrective Actions	RESOLUTION
					Delta RMP CEC QAPP v1		
2020-03	Created, pending final review	4/2/2021	CECs	CEC Fish sampled in September instead of May 2020	Fish were sampled for the Delta RMP CEC project in September 2020 instead of May 2020.	Update the CEC QAPP so that fish sampling for the CEC Pilot Study will happen at the same time as the fish collection for Mercury. Update to occur prior to the 21/22 monitoring.	QAPP Revised
2020-04	Created, pending final review	4/2/2021	CECs	Sediment Sampled at 519AMNDVY Instead of 519SWPDCP	Sediment sampling site location deviation due to similarly named and located sites and a StationCode/StationName inconsistency in QAPP Table 10.1. SPoT sampled sediment at the 519AMNDVY station and their actual Lat/Longs are very close to the target Lat/Longs listed in the QAPP.	Update CEC QAPP sediment station from 519SWPDCP to 519AMNDVY in Table 10.1. Update the StationName and Lat/Long values for 519SWPDCP and 519AMNDVY to match the CEDEN values.	QAPP Revised
2020-05	Created, pending final review	4/2/2021	CECs	Coordinates in QAPP Do Not Match CEDEN Coordinates	Latitudes and longitudes listed in QAPP Table 10.1 do not match CEDEN latitudes and longitudes for	Update CEC QAPP Table 10.1 with the correct latitudes and longitudes for the StationCodes listed in the CEC QAPP.	QAPP Revised
2020-06	Created, pending final review	4/2/2021	CECs	Field and Habitat Parameter Deviations from QAPP Expectations	Collection of field measurements and habitat parameters by several field crews is inconsistent with the language in the QAPP: -Fish sampling by MPSL-DFW did not include field WQ measurements -Sediment sampling by SPOT did not include field WQ measurements and the habitat observations differed from QAPP due to the use of a non-SWAMP field data sheet	ASC QA Officer recommends any future versions of CEC QAPP updates language to make explicit that the requirement to collect field WQ measurements is only applicable to water samples	QAPP Revised



DEVIATION NUMBER	STATUS	DEVIATION DATE	Monitoring Sector	TITLE	DESCRIPTION	Corrective Actions	RESOLUTION
2020-07	Created, pending final review	3/4/2020	CECs	Inclusion of Additional Analytes	Inclusion of additional Polybrominated Diphenyl Ethers (PBDE) congeners for fish, bivalves, and sediment; additional Per- and Polyfluorinated Substances (PFAS) compounds for fish and sediment; and additional Pharmaceuticals and Personal Care Product (PPCP) compounds in water reported.	Update CEC QAPP to include these additional analytes (assuming the same laboratories will be used in Year 2)	QAPP Revised
2020-08	Created, pending final review	10/16/2020	CECs	Clams Not Collected at Station San Joaquin River at Airport Way	AMS did not sample clams at the San Joaquin River at Airport Way station (541SJ501) during the 20/21 monitoring year.	AMS to collect samples at 541SJ501 by hand if necessary for future events. AMS can move further from the target site on the SJR to collect clams if needed. Revise QAPP to reflect additional sampling method and technique for collecting clams.	QAPP Revised
2020-11	Created, pending final review	10/16/20	CECs	Insufficient Clam Tissue Collected at 519SWPDCP and 510SACC3A	followed the CEC QAPP guidelines for clam collection (20 clams per	Paul Salop and Tim Mussen to determine a length to weight ratio for clam shell size so that it can be used as a proxy for clam tissue mass. Update CEC QAPP with new collection instructions for clams to ensure sufficient mass is collected.	QAPP Revised
2020-12	Created, pending final review	4/2/2021	CECs	Update SSC Method by Weck Labs in QAPP	SSC method was not updated in the QAPP after the lab selection was finalized, so the QAPP at time of approval did not include the correct lab method.	Update QAPP to reference the correct method for SSC analysis. (from SM2540D to ASTM D3977M).	QAPP Revised



DEVIATION NUMBER	STATUS	DEVIATION DATE	Monitoring Sector	TITLE	DESCRIPTION	Corrective Actions	RESOLUTION
2020-13	Created, pending final review	4/2/2021	CECs	Insufficient Sample Volumes Collected for a Subset of Weck Lab's Analysis	collected to run all of the required QC samples for SSC for Event 1 (September 2020) and 2 (January 2021); PPCP samples did not have	San Francisco Estuary Institute (SFEI) will collect additional sample volume for events 3 & 4. This was added to the field sampling guide. QAPP will be updated to indicate the additional volume required, per the field sampling guide.	QAPP Revised
2020-14	Created, pending final review	4/2/2021	CECs	No Lab Replicate Performed on Vista PFAS Samples in Jan 2021	being clear. Therefore, a lab	ASC will provide more explicit instructions and labeling, regarding what QA sample(s) any extra sample volume provided to the labs is intended for, on the COCs provided to the labs.	QAPP Revised Unspiked laboratory duplicates no longer required on whole bottle extractions. All Replicates provided to Vista in Year 2 will be field replicates.
2020-15	Created, pending final review	4/2/2021	CECs	PPCP Analysis by Weck Labs Reports Method Detection Limit (MDL) and Reporting Limit (RL) Higher than QAPP	All PPCP analytes were reported by Weck with MDL and RL levels above what were listed in the QAPP; estrone and 17-b-estradiol have MDLs above the Monitoring Trigger Level (MTL) listed in the QAPP. QAPP MDL and RL values were taken from the original Weck quote, but Weck revised their MDLs and RLs upwards significantly prior to finalizing lab selection. CEC TAC approved used of elevated limits.	Update table 7.3 to list the accurate RLs and MDLs. Review the utility of estrone and 17-b-estradiol as target analytes for the study.	QAPP Revised. Review of the utility of the results as compared to the intent of the Pilot Study would occur upon completion of the entire three-year study.



DEVIATION NUMBER	STATUS	DEVIATION DATE	Monitoring Sector	TITLE	DESCRIPTION	Corrective Actions	RESOLUTION
2020-16	Created, pending final review	4/2/2021	CECs	Three of Eight Sites were Sampled During Event 2.	For the first flush sampling event (Event 2, January 27t, 2021) three of the eight total sites were sampled for water. ASC was the only field entity that could collect samples at that time; DWR was unable to sample the remaining five sites which required boat access due to restrictions from COVID-19 Shelter in Place restrictions.		Year 2 implementation involves contracting with different agencies to serve as backup field sampling crews. While shelter in place orders are difficult to anticipate, increased planning and logistics coordination are intended to prevent missed samples in Year 2.
2020-18	Draft in Progress	Not Applicable ¹	CECs	Station Updates to CEC Sites	Reconciliation of Year 1 sample collections locations and CEDEN StationCodes. Related to 2020-04 and 2020-05	Update Table 10.1 of the CEC QAPP and StationCodes reported in Year 1 data to be in agreement.	QAPP Revised. Year 1 data revised prior to publication.
2020-19	Under Regional Board Review	12/13/2021	CECs	Lab Blank Contamination Flagging	Address lab blank contamination reported in Year 1 data and add additional flagging QA Code of FI to environmental results.	Environmental results associated with lab blanks not meeting the Measurement Quality Objective (MQO) should receive an Fl QA Code prior to data going to CEDEN. Remind Laboratories of notification expectations and corrective actions for lab blank contamination. Amend CEC Year 2 QAPP DM Standard Operating Procedure (SOP) for updated BRs on Fl code.	Year 1 data were revised to include the FI code prior to publication on CEDEN. Data management staff will continue to communicate EDD revisions as needed according to data verification procedures. Year 2 QAPP has been amended to reflect additional data management procedures (Amendment approved 12/21/2021).

¹The discovery that the station codes, station names, and target latitude / longitudes did not match was not discovered on a specific date but during the preparation of sampling and QAPP updates for Year 2 monitoring. Year 1 station information was updated to reflect the accurate information in the CV RDC.



2.3.1 Summary of Deviations from Delta RMP QAPP

2.3.1.1 Mercury Monitoring

Two of the four deviations to the Delta RMP QAPP occurred for the mercury monitoring project. Both deviations were caused by isolated incidents. The first deviation (2020-02) is associated with the extension of the previous QAPP to include a fall fish monitoring event that occurred in the next fiscal year. The deviation had no associated corrective actions as it was an ad hoc extension of the Fiscal Year 2019-2020 (FY 19-20) workplan and QAPP due to timing constraints. No adverse impacts on mercury data generated during the affected event resulted from this deviation. The second deviation associated with mercury monitoring (2020-09) was the result of laboratory conditions caused by the COVID-19 pandemic; the data were flagged accordingly, and no further assessment of corrective actions is necessary.

2.3.1.2 Current Use Pesticides and Aquatic Toxicity

Two of the four deviations to the Delta RMP QAPP were associated with current use pesticides and aquatic toxicity. Deviation 2020-10 was the result of unanticipated changes to the monitoring schedule that resulted in fewer annual field quality control (QC) samples than were required by the QAPP. The corrective action was to modify the QC sample planning such that field QC is collected at a rate more proportional to environmental samples throughout the year. The field QC samples for the current water year indicate that the corrective action has effectively resolved the original problem, with pesticide field blanks and duplicates being collected at a rate of 6% of the samples (**Section 3.2.1**) and toxicity field duplicates at a rate of 6% of samples (**Section 3.2.2.1**) from Events 3 and 4. A complete assessment of the annual field QC frequency for the 2021 WY will be provided in the QA Report to be included with the 2021 WY CUP Data Report.

Deviation 2020-17 occurred due to miscommunications between project managers and laboratory staff at the USGS NWQL resulting in laboratory QC samples being performed at a frequency less than what was intended in the QAPP. The QAPP has been revised for the 2022 WY monitoring to explicitly state the QC sample requirements for each of the analyses run by the NWQL; QAPP revisions for version 7 of the Delta RMP QAPP are under review and pending approval by the CVRWQCB. Additional communication with the NWQL and additional sample collection procedures were also put in place to prevent this issue from occurring in the future. An assessment of these corrective actions will be performed pending submission of the 2021 WY CUP data to the Delta RMP.

2.3.2 Summary of Deviations from Delta RMP CEC QAPP

2.3.2.1 Constituents of Emerging Concern

Monitoring for CECs in FY 20-21 was the first year of the three-year monitoring design outlined in the Pilot Study Workplan. Ten of the 14 deviations occurred due to circumstances that became known once implementation of the study design had begun. The primary corrective



action associated with the deviations was to update the QAPP to more accurately reflect the implementation of the project. All ten deviations were resolved by the revisions made to the CEC QAPP for Year 2 monitoring. The CEC QAPP for the FY 21-22 (version 2 or v2) was approved on October 12, 2021.

Deviation 2020-14 occurred due to miscommunication between the laboratory and project manager regarding the intended use of sample replicates provided to the laboratory. The revisions made to the CEC QAPP for Year 2 removed the requirement of laboratory duplicates where a whole bottle extraction is required for analysis.

Deviation 2020-15 resulted in an update to the CEC QAPP v2, with an additional corrective action of evaluating the usefulness of the results given the original intent of the CEC Pilot Study Workplan. The CEC TAC will continue to review the data as it is received and processed and use this information to develop the monitoring design for Year 3. An assessment or data useability will occur when the entire three-year study is complete; future monitoring will continue according to the target analytes prescribed in the CEC Pilot Study Workplan and will utilize the detection limits that can be achieved by the laboratory.

Deviation 2020-16 was regarding missed sample collection due to the COVID-19 pandemic. While the sampling constraints that led to this deviation were the result of an isolated incident for which no specific corrective action could be made, steps have been taken to prevent similar scenarios in the future. Backup sampling crews from two additional contractors are now on call to supplement the sampling effort in the event that a large number of samples need to be collected in a short time period (such as a large storm event) or when unforeseen circumstances prevent the primary sampling crews from being able to mobilize. The intent of having supplementary sampling crews and resources on standby is to prevent circumstances similar to those leading to deviation form 2020-16 from happening in the future. The successful mobilization of backup crews during a large storm event in October 2021 for the Year 2 monitoring indicates that this constraint is being successfully resolved for continued CEC monitoring.

Deviation 2020-19 was associated with discrepancies in data flagging rules implemented by ASC. During review of Year 1 data, questions regarding the application of data flags for samples associated with method blank contamination resulted in a desire to bring the Year 1 data flagging more in line with SWAMP procedures. This specifically resulted in the addition of the "FI" QA Code, which is used by SWAMP projects but was previously not a flag applied according to Delta RMP data management procedures implemented by ASC. Deviation 2020-19 was generated to document the discrepancy between the finalized Year 1 data and the data management SOPs for Year 1 data validation. The current Delta RMP data management procedures have been updated to stipulate the use of the "FI" code for all data processed by CV RDC staff; the CEC QAPP for Year 2 monitoring was updated in an amendment approved on December 21, 2021.



3 QUALITY ASSURANCE – DATA MANAGED BY THE DELTA RMP

3.1 CONSTITUENTS OF EMERGING CONCERN

The CEC Year 1 Data Report includes a QA Report which evaluates the acceptability of the data collected in FY 20-21 for the CEC Pilot Study (**Appendix IV**). A summary of completeness, precision, and accuracy is provided below.

3.1.1 CEC Year 1 Monitoring Results

3.1.1.1 Quality Control Sample Completeness

Of the CEC samples planned for the Year 1 monitoring, 86% (343 of 397) were collected and analyzed by the laboratories. Missed samples were due to a lack of bivalves obtained from a single site using the prescribed trawling procedures in October 2020 (four constituents not analyzed) and missed water samples due to COVID-19 sampling constraints in January 2021 (five sites with ten constituents not analyzed). See **Constituents of Emerging Concern** for more information regarding sampling constraints during the FY 20-21 monitoring.

Field QC sample requirements are outlined in the CEC Year 1 QAPP (v1). The requirements differ by matrix:

- Water samples require both field duplicates and field blanks,
- Sediment samples require only field duplicates, and
- Tissue samples require neither field duplicates nor field blanks.

Where required, field QC samples must be collected at a minimum frequency of 5%. For the Year 1 monitoring, field blanks comprised 8% (21 of 270) and field duplicates comprised 11% (29 of 270) of the water sample results received. Field blanks comprised 33% (7 of 21) of the sediment results analyzed.

Laboratory QC sample requirements are a combination of method blanks, laboratory duplicates, matrix spikes, and laboratory control spikes and are method/analyte specific. Laboratory QC are required at a frequency of 1 in 20 samples or one per batch. Analytical batches met QC sample requirements for 85% (23 of 27) of the FY 20-21 analyses. Of the 27 batches, one batch for method ASTM D3977M was missing a laboratory duplicate, two batches for method EPA 1694M were missing matrix spike samples, and one batch for method EPA 537M was missing both a laboratory duplicate and matrix spikes. Analyte-specific QC completeness is addressed in the CEC Year 1 Data Report provided in **Appendix IV**.



3.1.1.2 Acceptability of Precision Measurements

Precision is measured by a combination of field and laboratory duplicate samples including matrix spike duplicates and laboratory spike duplicates. Precision acceptability is summarized below in **Table 6**. Samples that did not meet acceptability criteria were flagged with one or more of the following CEDEN QACodes: "VFDP" or "VIL".

Method	LABORATORY	Matrix	Analyte	TOTAL DUPLICATE SAMPLES	DUPLICATE SAMPLES WITHIN LIMIT	Acceptability Met (%)
ASTM D3977M	Weck	Water	Suspended Sediment Concentration	5	5	100
AXYS MLA-033 Rev 06	SGS AXYS	Sediment	Moisture	3	3	100
AXYS MLA-033 Rev 06	SGS AXYS	Sediment	PBDE 047	3	3	100
AXYS MLA-033 Rev 06	SGS AXYS	Sediment	PBDE 099	3	3	100
AXYS MLA-033 Rev 06	SGS AXYS	Tissue	Lipid	1	1	100
AXYS MLA-033 Rev 06	SGS AXYS	Tissue	Moisture	1	1	100
AXYS MLA-033 Rev 06	SGS AXYS	Tissue	PBDE 047	1	0	0
AXYS MLA-033 Rev 06	SGS AXYS	Tissue	PBDE 099	1	1	100
EPA 1694M	Weck	Water	Bisphenol A	13	10	77
EPA 1694M	Weck	Water	Diclofenac	13	13	100
EPA 1694M	Weck	Water	Estradiol, 17beta-	13	13	100
EPA 1694M	Weck	Water	Estrone	13	13	100
EPA 1694M	Weck	Water	lbuprofen	13	13	100
EPA 1694M	Weck	Water	Triclosan	13	13	100
EPA 537M	Vista	Water	Perfluorooctanesulfonic acid (PFOS)	10	10	100
EPA 537M	Vista	Water	Perfluorooctanoic acid (PFOA)	10	10	100
EPA 625.1M	Physis	Water	Galaxolide	16	14	87.5
EPA 9060M	Weck	Sediment	Total Organic Carbon	4	4	100
SGS AXYS MLA-110 Rev 02	SGS AXYS	Sediment	Moisture	3	3	100
SGS AXYS MLA-110 Rev 02	SGS AXYS	Sediment	Perfluorooctanesulfonate	3	3	100
SGS AXYS MLA-110 Rev 02	SGS AXYS	Sediment	Perfluorooctanoate	3	3	100
SGS AXYS MLA-110 Rev 02	SGS AXYS	Tissue	Perfluorooctanesulfonate	1	1	100
SGS AXYS MLA-110 Rev 02	SGS AXYS	Tissue	Perfluorooctanoate	1	1	100
			Total	147	141	95.9

Table 6. Precision measurement acceptability for Year 1 CEC Pilot Study.



3.1.1.3 Acceptability of Accuracy Measurements

Accuracy and bias in the field and laboratory are measured through a combination of negative and positive control samples. Blank sample acceptability is summarized in **Table 7**. Blank samples that did not meet acceptability criteria were flagged with "IP", "VIP", or "VIPF". Laboratory spike sample acceptability is summarized in **Table 8**. Spike samples that did not meet acceptability were flagged with "EUM", "VEUM", or "VGB". Surrogate recoveries are summarized in **Table 9**. All surrogate recoveries met acceptability criteria.

Метнор	LABORATORY	Matrix	ANALYTE	Total Blank Samples	Samples WITHIN LIMITS	Acceptabilit y Met (%)
ASTM D3977M	Weck	Water	Suspended Sediment Concentration	6	5	83.3
AXYS MLA-033 Rev 06	SGS AXYS	Sediment	PBDE 047	1	0	0
AXYS MLA-033 Rev 06	SGS AXYS	Sediment	PBDE 099	1	0	0
AXYS MLA-033 Rev 06	SGS AXYS	Tissue	PBDE 047	1	1	100
AXYS MLA-033 Rev 06	SGS AXYS	Tissue	PBDE 099	1	0	0
EPA 1694M	Weck	Water	Bisphenol A	6	1	16.7
EPA 1694M	Weck	Water	Diclofenac	6	6	100
EPA 1694M	Weck	Water	Estradiol, 17beta-	6	6	100
EPA 1694M	Weck	Water	Estrone	6	6	100
EPA 1694M	Weck	Water	Ibuprofen	6	6	100
EPA 1694M	Weck	Water	Triclosan	6	6	100
EPA 537M	Vista	Water	Perfluorooctanesulfonic acid (PFOS)	7	7	100
EPA 537M	Vista	Water	Perfluorooctanoic acid (PFOA)	7	7	100
EPA 625.1M	Physis	Water	Galaxolide	7	1	14.2
EPA 9060M	Weck	Sediment	Total Organic Carbon	3	3	100
SGS AXYS MLA-110 Rev 02	SGS AXYS	Sediment	Perfluorooctanesulfonate	1	1	100
SGS AXYS MLA-110 Rev 02	SGS AXYS	Sediment	Perfluorooctanoate	1	1	100
SGS AXYS MLA-110 Rev 02	SGS AXYS	Tissue	Perfluorooctanesulfonate	1	0	0
SGS AXYS MLA-110 Rev 02	SGS AXYS	Tissue	Perfluorooctanoate	1	1	100
			Total	74	58	78.4

Table 7. Blank sample acceptability for Year 1 CEC Pilot Study.

Table 8. Spike sample acceptability for Year 1 CEC Pilot Study.

Метнор	LABORATORY	Matrix	Analyte	Total Spiked Samples	Samples WITHIN LIMITS	Acceptabilit y Met (%)
ASTM D3977M	Weck	Water	Suspended Sediment Concentration	4	4	100
AXYS MLA-033 Rev 06	SGS AXYS	Sediment	Moisture	2	2	100
AXYS MLA-033 Rev 06	SGS AXYS	Sediment	PBDE 047	3	3	100
AXYS MLA-033 Rev 06	SGS AXYS	Sediment	PBDE 099	3	3	100



31 Delta RMP Annual Report for FY 20-21

Метнор	LABORATORY	Matrix	Analyte	Total Spiked Samples	Samples WITHIN LIMITS	Acceptabilit y Met (%)
AXYS MLA-033 Rev 06	SGS AXYS	Tissue	Lipid	2	2	100
AXYS MLA-033 Rev 06	SGS AXYS	Tissue	Moisture	2	2	100
AXYS MLA-033 Rev 06	SGS AXYS	Tissue	PBDE 047	3	2	66.7
AXYS MLA-033 Rev 06	SGS AXYS	Tissue	PBDE 099	3	3	100
EPA 1694M	Weck	Water	Bisphenol A	14	8	57.1
EPA 1694M	Weck	Water	Diclofenac	14	14	100
EPA 1694M	Weck	Water	Estradiol, 17beta-	14	14	100
EPA 1694M	Weck	Water	Estrone	14	14	100
EPA 1694M	Weck	Water	lbuprofen	14	13	92.9
EPA 1694M	Weck	Water	Triclosan	14	14	100
EPA 537M	Vista	Water	Perfluorooctanesulfonic acid (PFOS)	13	13	100
EPA 537M	Vista	Water	Perfluorooctanoic acid (PFOA)	13	13	100
EPA 625.1M	Physis	Water	Galaxolide	18	12	66.7
EPA 9060M	Weck	Sediment	Total Organic Carbon	7	7	100
SGS AXYS MLA-110 Rev 02	SGS AXYS	Sediment	Moisture	2	2	100
SGS AXYS MLA-110 Rev 02	SGS AXYS	Sediment	Perfluorooctanesulfonate	3	3	100
SGS AXYS MLA-110 Rev 02	SGS AXYS	Sediment	Perfluorooctanoate	3	3	100
SGS AXYS MLA-110 Rev 02	SGS AXYS	Tissue	Perfluorooctanesulfonate	3	3	100
SGS AXYS MLA-110 Rev 02	SGS AXYS	Tissue	Perfluorooctanoate	3	3	100
			Total	171	157	91.8

Table 9. Surrogate recovery acceptability for Year 1 CEC Pilot Study.

Метнор	LABORATORY	Matrix	Analyte	Total Surrogate Samples	Samples Within Limits	Acceptability Met (%)
AXYS MLA-033 Rev 06	SGS AXYS	Sediment	PBDE 047 (Surrogate)	9	9	100
AXYS MLA-033 Rev 06	SGS AXYS	Sediment	PBDE 099 (Surrogate)	9	9	100
AXYS MLA-033 Rev 06	SGS AXYS	Tissue	PBDE 047 (Surrogate)	13	13	100
AXYS MLA-033 Rev 06	SGS AXYS	Tissue	PBDE 099 (Surrogate)	13	13	100
EPA 537M	Vista	Water	Perfluorooctanesulfonic acid-13C8 (Surrogate)	53	53	100
EPA 537M	Vista	Water	Perfluorooctanoic acid- 13C2 (Surrogate)	53	53	100
EPA 625.1M	Physis	Water	Dichlorobenzene-d4, 1,4- (Surrogate)	59	59	100
SGS AXYS MLA-110 Rev 02	SGS AXYS	Sediment	Perfluorooctanesulfonate 080 (Surrogate)	9	9	100
SGS AXYS MLA-110 Rev 02	SGS AXYS	Sediment	Perfluorooctanoate (Surrogate)	9	9	100
SGS AXYS MLA-110 Rev 02	SGS AXYS	Tissue	Perfluorooctanesulfonate 080 (Surrogate)	8	8	100
SGS AXYS MLA-110 Rev 02	SGS AXYS	Tissue	Perfluorooctanoate (Surrogate)	8	8	100
			Total	243	243	100



3.1.1.4 Invalid Data

There were no invalid CEC results analyzed during FY 20-21; all results have been flagged according to QAPP criteria.



33 Delta RMP Annual Report for FY 20-21 February 1, 2022

3.2 PESTICIDES AND AQUATIC TOXICITY

Current use pesticides and associated aquatic toxicity monitoring are conducted on a water year basis (October 1 through September 30). At the time of this report, not all results from the previous water year were finalized. The data evaluations below are based on the samples collected during FY 20-21. The samples collected during FY 20-21 were for the extended Year 2 of the monitoring design; the first two events were collected in FY 19-20 and monitoring was paused due to a delay in selecting a new toxicity laboratory (see **Pesticides and Toxicity Multi-Year Study**). Samples collected for the CUP and toxicity analysis during FY 20-21 includes two sampling events:

- Event 3, occurring on April 28 and 29, 2021
- Event 4, occurring June 15 and 16, 2021

During these two events, samples were collected by USGS sampling crews for pesticide analysis at the USGS OCRL, copper and ancillary parameters analysis at the USGS NWQL, and toxicity testing by PER. A USGS Field and Chemistry Report was provided to the Delta RMP on January 28, 2022, describing the samples collected for the water year and a summary of the pesticide results (**Appendix III**). Data have been received in a CEDEN comparable EDD format from PER and USGS OCRL. The USGS NWQL has experienced delays in finalizing results due to COVID-19 affecting staff availability. The CV RDC has not received the USGS NWQL data in a CEDEN comparable EDD; results are expected in February 2022. Results associated with the CUP monitoring in FY 20-21 received as of January 17, 2022, are summarized in the sections below.

The USGS Field and Chemistry Report will be incorporated into a CUP Data Report for the 2021 WY (including a QA Report) which will evaluate all samples collected for the 2021 WY, include an assessment of all quality assurance and quality control procedures, and summarize results.

A summary of completeness, precision, and accuracy measures for the events occurring during FY 20-21 is provided for CUP and toxicity in the following tables:

- 1. Current Use Pesticides: Table 10, Table 11, Table 12, and Table 13
- 2. Aquatic Toxicity: Table 14 and Table 15

3.2.1 Current Use Pesticides

3.2.1.1 Quality Control Sample Completeness

Of the samples planned for CUP monitoring during FY 20-21, 100% (4,624 of 4,624) were collected and analyzed by USGS OCRL. See **Pesticides and Toxicity Multi-Year Study** for more information regarding sampling during the FY 20-21.

The Delta RMP QAPP (v6.4) requires that field duplicates and field blanks be collected with associated pesticide analyses at an annual rate of 5%. Though the annual requirement cannot yet be fully assessed, field blanks comprised 6% (289 of 4,624) and field duplicates comprised 6% (289 of 4,624) of samples collected during FY 20-21 for analysis by USGS OCRL.



Laboratory QC sample requirements are a combination of method blanks, laboratory duplicates, matrix spikes, and laboratory control spikes and are method/analyte specific. Laboratory QC are required at a frequency of 1 in 20 samples or one per batch. Analytical batches met QC sample requirements for 63% (5 of 8) of the FY 20-21 analyses. Of the 8 batches, three batches analyzed for pesticides were missing matrix spike samples. A comprehensive assessment of the QC completeness for the entire Water Year will be addressed in the Data Report to be drafted when they dataset is complete.

3.2.1.2 Acceptability of Precision Measurements

Precision is measured by a combination of field and laboratory duplicate samples including matrix spike duplicates and/or laboratory duplicates. Precision acceptability is summarized below in **Table 10**. Pesticide samples that did not meet acceptability criteria were flagged with the CEDEN QACode "FDP".

				lei peseieide sampie			
Метнод	LABORATORY	Matri X	Fractions	Analyte	Total Duplicate Samples	DUPLICATE Samples WITHIN LIMIT	Acceptability Met (%)
USGS- OCRL_LC/MS/MS Sanders_2018	USGS OCRL	Water	Dissolved	Acetamiprid	4	4	100
USGS- OCRL_LC/MS/MS Sanders_2018	USGS OCRL	Water	Dissolved	Carbendazim	4	4	100
USGS- OCRL_LC/MS/MS Sanders_2018	USGS OCRL	Water	Dissolved	Carboxin	4	4	100
USGS- OCRL_LC/MS/MS Sanders_2018	USGS OCRL	Water	Dissolved	Chlorantraniliprole	4	4	100
USGS- OCRL_LC/MS/MS Sanders_2018	USGS OCRL	Water	Dissolved	Clothianidin	4	4	100
USGS- OCRL_LC/MS/MS Sanders_2018	USGS OCRL	Water	Dissolved	Cyantraniliprole	4	4	100
USGS- OCRL_LC/MS/MS Sanders_2018	USGS OCRL	Water	Dissolved	Cyazofamid	4	4	100
USGS- OCRL_LC/MS/MS Sanders_2018	USGS OCRL	Water	Dissolved	Cymoxanil	4	4	100
USGS- OCRL_LC/MS/MS Sanders_2018	USGS OCRL	Water	Dissolved	Desthio- prothioconazole	4	4	100
USGS- OCRL_LC/MS/MS Sanders_2018	USGS OCRL	Water	Dissolved ¹	Dichlorobenzenamine, 3,4-	4	4	100
USGS- OCRL_LC/MS/MS Sanders_2018	USGS OCRL	Water	Dissolved	Dichlorophenyl Urea, 3,4-	4	4	100

Table 10. Precision measurement acceptability for pesticide samples collected during FY 20-21.



Delta RMP Annual Report for FY 20-21

35

Method	LABORATORY	Matri X	FRACTIONS	Analyte	Total Duplicate Samples	DUPLICATE SAMPLES WITHIN LIMIT	Acceptability Met (%)
USGS- OCRL_LC/MS/MS Sanders_2018	USGS OCRL	Water	Dissolved	Dichlorophenyl-3- methyl Urea, 3,4-	4	4	100
USGS- OCRL_LC/MS/MS Sanders_2018	USGS OCRL	Water	Dissolved	Dinotefuran	4	4	100
USGS- OCRL_LC/MS/MS Sanders_2018	USGS OCRL	Water	Dissolved	Diuron	4	4	100
USGS- OCRL_LC/MS/MS Sanders_2018	USGS OCRL	Water	Dissolved	Ethaboxam	4	4	100
USGS- OCRL_LC/MS/MS Sanders_2018	USGS OCRL	Water	Dissolved	Flonicamid	4	4	100
USGS- OCRL_LC/MS/MS Sanders_2018	USGS OCRL	Water	Dissolved	Flupyradifurone	4	4	100
USGS- OCRL_LC/MS/MS Sanders_2018	USGS OCRL	Water	Dissolved	Fluridone	4	4	100
USGS- OCRL_LC/MS/MS Sanders_2018	USGS OCRL	Water	Dissolved	Imidacloprid	4	4	100
USGS- OCRL_LC/MS/MS Sanders_2018	USGS OCRL	Water	Dissolved	Imidacloprid urea	4	4	100
USGS- OCRL_LC/MS/MS Sanders_2018	USGS OCRL	Water	Dissolved	Mandipropamid	4	4	100
USGS- OCRL_LC/MS/MS Sanders_2018	USGS OCRL	Water	Dissolved	Methoxyfenozide	4	4	100
USGS- OCRL_LC/MS/MS Sanders_2018	USGS OCRL	Water	Dissolved	Oryzalin	4	4	100
USGS- OCRL_LC/MS/MS Sanders_2018	USGS OCRL	Water	Dissolved	Oxathiapiprolin	4	4	100
USGS- OCRL_LC/MS/MS Sanders_2018	USGS OCRL	Water	Dissolved	Penoxsulam	4	4	100
USGS- OCRL_LC/MS/MS Sanders_2018	USGS OCRL	Water	Dissolved	Penthiopyrad	4	4	100
USGS- OCRL_LC/MS/MS Sanders_2018	USGS OCRL	Water	Dissolved	Sulfoxaflor	4	4	100
USGS- OCRL_LC/MS/MS Sanders_2018	USGS OCRL	Water	Dissolved	Tebufenozide	4	4	100



Method	LABORATORY	Matri X	Fractions	ANALYTE	Total Duplicate Samples	DUPLICATE SAMPLES WITHIN LIMIT	Acceptability Met (%)
USGS- OCRL_LC/MS/MS Sanders_2018	USGS OCRL	Water	Dissolved	Thiabendazole	4	4	100
USGS- OCRL_LC/MS/MS Sanders_2018	USGS OCRL	Water	Dissolved	Thiacloprid	4	4	100
USGS- OCRL_LC/MS/MS Sanders_2018	USGS OCRL	Water	Dissolved	Thiamethoxam	4	4	100
USGS- OCRL_LC/MS/MS Sanders_2018	USGS OCRL	Water	Dissolved	Thiamethoxam Degradate (CGA- 355190)	4	4	100
USGS- OCRL_LC/MS/MS Sanders_2018	USGS OCRL	Water	Dissolved	Thiamethoxam Degradate (NOA- 407475)	4	4	100
USGS- OCRL_LC/MS/MS Sanders_2018	USGS OCRL	Water	Dissolved	Tolfenpyrad	4	4	100
USGS- OCRL_LC/MS/MS Sanders_2018	USGS OCRL	Water	Dissolved	Tricyclazole	4	4	100
USGS-OCRL_GC/MS Sanders_2018	USGS OCRL	Water	Dissolved, Particulate	Acibenzolar-S-methyl	8	8	100
USGS-OCRL_GC/MS Sanders_2018	USGS OCRL	Water	Dissolved, Particulate	Allethrin	8	8	100
USGS-OCRL_GC/MS Sanders_2018	USGS OCRL	Water	Dissolved, Particulate	Atrazine	8	8	100
USGS-OCRL_GC/MS Sanders_2018	USGS OCRL	Water	Dissolved, Particulate	Azoxystrobin	8	8	100
USGS-OCRL_GC/MS Sanders_2018	USGS OCRL	Water	Dissolved, Particulate	Benfluralin	8	8	100
USGS-OCRL_GC/MS Sanders_2018	USGS OCRL	Water	Dissolved, Particulate	Benzovindiflupyr	8	8	100
USGS-OCRL_GC/MS Sanders_2018	USGS OCRL	Water	Dissolved, Particulate	Bifenthrin	8	8	100
USGS-OCRL_GC/MS Sanders_2018	USGS OCRL	Water	Dissolved, Particulate	Boscalid	8	8	100
USGS-OCRL_GC/MS Sanders_2018	USGS OCRL	Water	Dissolved, Particulate	Butralin	8	8	100
USGS-OCRL_GC/MS Sanders_2018	USGS OCRL	Water	Dissolved, Particulate	Captan	8	8	100
USGS-OCRL_GC/MS Sanders_2018	USGS OCRL	Water	Dissolved, Particulate	Carbaryl	8	8	100
USGS-OCRL_GC/MS Sanders_2018	USGS OCRL	Water	Dissolved, Particulate	Carbofuran	8	8	100
USGS-OCRL_GC/MS Sanders_2018	USGS OCRL	Water	Dissolved, Particulate	Chlorfenapyr	8	8	100
USGS-OCRL_GC/MS Sanders_2018	USGS OCRL	Water	Dissolved, Particulate	Chloro-N- (ethoxymethyl)-N-(2- ethyl-6- methylphenyl)acetamid e, 2-	8	8	100



Method	LABORATORY	Matri X	Fractions	Analyte	Total Duplicate Samples	DUPLICATE Samples WITHIN LIMIT	Acceptability Met (%)
USGS-OCRL_GC/MS Sanders_2018	USGS OCRL	Water	Dissolved, Particulate	Chlorothalonil	8	8	100
USGS-OCRL_GC/MS Sanders_2018	USGS OCRL	Water	Dissolved, Particulate	Chlorpyrifos	8	8	100
USGS-OCRL_GC/MS Sanders_2018	USGS OCRL	Water	Dissolved, Particulate	Chlorpyrifos oxon	8	8	100
USGS-OCRL_GC/MS Sanders_2018	USGS OCRL	Water	Particulate	Clomazone	8	8	100
USGS-OCRL_GC/MS Sanders_2018	USGS OCRL	Water	Dissolved, Particulate	Coumaphos	8	8	100
USGS-OCRL_GC/MS Sanders_2018	USGS OCRL	Water	Dissolved, Particulate	Cycloate	8	8	100
USGS-OCRL_GC/MS Sanders_2018	USGS OCRL	Water	Dissolved, Particulate	Cyfluthrin, Total	8	8	100
USGS-OCRL_GC/MS Sanders_2018	USGS OCRL	Water	Dissolved, Particulate	Cyhalofop-butyl	8	8	100
USGS-OCRL_GC/MS Sanders_2018	USGS OCRL	Water	Dissolved, Particulate	Cyhalothrin	8	8	100
USGS-OCRL_GC/MS Sanders_2018	USGS OCRL	Water	Dissolved, Particulate	Cypermethrin, Total	8	8	100
USGS-OCRL_GC/MS Sanders_2018	USGS OCRL	Water	Dissolved, Particulate	Cyproconazole	8	8	100
USGS-OCRL_GC/MS Sanders_2018	USGS OCRL	Water	Dissolved, Particulate	Cyprodinil	8	8	100
USGS-OCRL_GC/MS Sanders_2018	USGS OCRL	Water	Dissolved, Particulate	Dacthal	8	8	100
USGS-OCRL_GC/MS Sanders_2018	USGS OCRL	Water	Dissolved, Particulate	DDD(p,p')	8	8	100
USGS-OCRL_GC/MS Sanders_2018	USGS OCRL	Water	Dissolved, Particulate	DDE(p,p')	8	8	100
USGS-OCRL_GC/MS Sanders_2018	USGS OCRL	Water	Dissolved, Particulate	DDT(p,p')	8	8	100
USGS-OCRL_GC/MS Sanders_2018	USGS OCRL	Water	Dissolved, Particulate	Deltamethrin	8	8	100
USGS-OCRL_GC/MS Sanders_2018	USGS OCRL	Water	Dissolved, Particulate	Diazinon	8	8	100
USGS-OCRL_GC/MS Sanders_2018	USGS OCRL	Water	Dissolved, Particulate	Diazoxon	8	8	100
USGS-OCRL_GC/MS Sanders_2018	USGS OCRL	Water	Dissolved, Particulate	Dichloroaniline, 3,5-	8	8	100
USGS-OCRL_GC/MS Sanders_2018	USGS OCRL	Water	Particulate ¹	Dichlorobenzenamine, 3,4-	4	4	100
USGS-OCRL_GC/MS Sanders_2018	USGS OCRL	Water	Dissolved, Particulate	Dichlorvos	8	8	100
USGS-OCRL_GC/MS Sanders_2018	USGS OCRL	Water	Dissolved, Particulate	Difenoconazole	8	8	100
USGS-OCRL_GC/MS Sanders_2018	USGS OCRL	Water	Dissolved, Particulate	Dimethomorph	8	8	100
USGS-OCRL_GC/MS Sanders_2018	USGS OCRL	Water	Dissolved, Particulate	Dithiopyr	8	8	100
USGS-OCRL_GC/MS Sanders_2018	USGS OCRL	Water	Dissolved, Particulate	EPTC	8	8	100



Method	LABORATORY	Matri X	Fractions	Analyte	Total Duplicate Samples	DUPLICATE Samples WITHIN LIMIT	Acceptability Met (%)
USGS-OCRL_GC/MS Sanders_2018	USGS OCRL	Water	Dissolved, Particulate	Esfenvalerate	8	8	100
USGS-OCRL_GC/MS Sanders_2018	USGS OCRL	Water	Dissolved, Particulate	Ethalfluralin	8	8	100
USGS-OCRL_GC/MS Sanders_2018	USGS OCRL	Water	Dissolved, Particulate	Ethofenprox	8	8	100
USGS-OCRL_GC/MS Sanders_2018	USGS OCRL	Water	Particulate	Etoxazole	8	8	100
USGS-OCRL_GC/MS Sanders_2018	USGS OCRL	Water	Dissolved, Particulate	Famoxadone	8	8	100
USGS-OCRL_GC/MS Sanders_2018	USGS OCRL	Water	Dissolved, Particulate	Fenamidone	8	8	100
USGS-OCRL_GC/MS Sanders_2018	USGS OCRL	Water	Dissolved, Particulate	Fenbuconazole	8	8	100
USGS-OCRL_GC/MS Sanders_2018	USGS OCRL	Water	Dissolved, Particulate	Fenhexamid	8	8	100
USGS-OCRL_GC/MS Sanders_2018	USGS OCRL	Water	Dissolved, Particulate	Fenpropathrin	8	8	100
USGS-OCRL_GC/MS Sanders_2018	USGS OCRL	Water	Dissolved, Particulate	Fenpyroximate	8	8	100
USGS-OCRL_GC/MS Sanders_2018	USGS OCRL	Water	Dissolved, Particulate	Fipronil	8	8	100
USGS-OCRL_GC/MS Sanders_2018	USGS OCRL	Water	Dissolved, Particulate	Fipronil Desulfinyl	8	8	100
USGS-OCRL_GC/MS Sanders_2018	USGS OCRL	Water	Dissolved, Particulate	Fipronil Desulfinyl Amide	8	8	100
USGS-OCRL_GC/MS Sanders_2018	USGS OCRL	Water	Dissolved, Particulate	Fipronil Sulfide	8	8	100
USGS-OCRL_GC/MS Sanders_2018	USGS OCRL	Water	Dissolved, Particulate	Fipronil Sulfone	8	8	100
USGS-OCRL_GC/MS Sanders_2018	USGS OCRL	Water	Dissolved, Particulate	Fluazinam	8	8	100
USGS-OCRL_GC/MS Sanders_2018	USGS OCRL	Water	Dissolved, Particulate	Flubendiamide	8	8	100
USGS-OCRL_GC/MS Sanders_2018	USGS OCRL	Water	Dissolved, Particulate	Fludioxonil	8	8	100
USGS-OCRL_GC/MS Sanders_2018	USGS OCRL	Water	Dissolved, Particulate	Flufenacet	8	8	100
USGS-OCRL_GC/MS Sanders_2018	USGS OCRL	Water	Dissolved, Particulate	Flumetralin	8	8	100
USGS-OCRL_GC/MS Sanders_2018	USGS OCRL	Water	Dissolved, Particulate	Fluopicolide	8	8	100
USGS-OCRL_GC/MS Sanders_2018	USGS OCRL	Water	Dissolved, Particulate	Fluopyram	8	8	100
USGS-OCRL_GC/MS Sanders_2018	USGS OCRL	Water	Dissolved, Particulate	Fluoxastrobin	8	8	100
USGS-OCRL_GC/MS Sanders_2018	USGS OCRL	Water	Dissolved, Particulate	Flutolanil	8	8	100
USGS-OCRL_GC/MS Sanders_2018	USGS OCRL	Water	Dissolved, Particulate	Flutriafol	8	8	100
USGS-OCRL_GC/MS Sanders_2018	USGS OCRL	Water	Dissolved, Particulate	Fluxapyroxad	8	8	100



Method	LABORATORY	Matri X	FRACTIONS	Analyte	Total Duplicate Samples	DUPLICATE Samples WITHIN LIMIT	Acceptability Met (%)
USGS-OCRL_GC/MS Sanders_2018	USGS OCRL	Water	Dissolved, Particulate	Hexazinone	8	8	100
USGS-OCRL_GC/MS Sanders_2018	USGS OCRL	Water	Dissolved, Particulate	Imazalil	8	8	100
USGS-OCRL_GC/MS Sanders_2018	USGS OCRL	Water	Dissolved, Particulate	Indaziflam	8	8	100
USGS-OCRL_GC/MS Sanders_2018	USGS OCRL	Water	Dissolved, Particulate	Indoxacarb	8	8	100
USGS-OCRL_GC/MS Sanders_2018	USGS OCRL	Water	Dissolved, Particulate	Ipconazole	8	8	100
USGS-OCRL_GC/MS Sanders_2018	USGS OCRL	Water	Dissolved, Particulate	Iprodione	8	8	100
USGS-OCRL_GC/MS Sanders_2018	USGS OCRL	Water	Dissolved, Particulate	lsofetamid	8	8	100
USGS-OCRL_GC/MS Sanders_2018	USGS OCRL	Water	Dissolved, Particulate	Kresoxim-methyl	8	8	100
USGS-OCRL_GC/MS Sanders_2018	USGS OCRL	Water	Dissolved, Particulate	Malaoxon	8	8	100
USGS-OCRL_GC/MS Sanders_2018	USGS OCRL	Water	Dissolved, Particulate	Malathion	8	8	100
USGS-OCRL_GC/MS Sanders_2018	USGS OCRL	Water	Dissolved, Particulate	Metalaxyl	8	8	100
USGS-OCRL_GC/MS Sanders_2018	USGS OCRL	Water	Dissolved, Particulate	Metconazole	8	8	100
USGS-OCRL_GC/MS Sanders_2018	USGS OCRL	Water	Dissolved, Particulate	Methoprene	8	8	100
USGS-OCRL_GC/MS Sanders_2018	USGS OCRL	Water	Dissolved, Particulate	Metolachlor	8	8	100
USGS-OCRL_GC/MS Sanders_2018	USGS OCRL	Water	Dissolved, Particulate	Myclobutanil	8	8	100
USGS-OCRL_GC/MS Sanders_2018	USGS OCRL	Water	Dissolved, Particulate	Napropamide	8	8	100
USGS-OCRL_GC/MS Sanders_2018	USGS OCRL	Water	Dissolved, Particulate	Novaluron	8	8	100
USGS-OCRL_GC/MS Sanders_2018	USGS OCRL	Water	Dissolved, Particulate	Oxadiazon	8	8	100
USGS-OCRL_GC/MS Sanders_2018	USGS OCRL	Water	Dissolved, Particulate	Oxyfluorfen	8	8	100
USGS-OCRL_GC/MS Sanders_2018	USGS OCRL	Water	Dissolved, Particulate	Paclobutrazol	8	8	100
USGS-OCRL_GC/MS Sanders_2018	USGS OCRL	Water	Dissolved, Particulate	Parathion, Methyl	8	8	100
USGS-OCRL_GC/MS Sanders_2018	USGS OCRL	Water	Dissolved, Particulate	Pendimethalin	8	8	100
USGS-OCRL_GC/MS Sanders_2018	USGS OCRL	Water	Dissolved, Particulate	Pentachloroanisole	8	8	100
USGS-OCRL_GC/MS Sanders_2018	USGS OCRL	Water	Dissolved, Particulate	Pentachloronitrobenze ne	8	8	100
USGS-OCRL_GC/MS Sanders_2018	USGS OCRL	Water	Dissolved, Particulate	Permethrin, Total	8	8	100
USGS-OCRL_GC/MS Sanders_2018	USGS OCRL	Water	Dissolved, Particulate	Phenothrin	8	8	100



Method	LABORATORY	Matri X	Fractions	Analyte	Total Duplicate Samples	DUPLICATE Samples WITHIN LIMIT	Acceptability Met (%)
USGS-OCRL_GC/MS Sanders_2018	USGS OCRL	Water	Dissolved, Particulate	Phosmet	8	8	100
USGS-OCRL_GC/MS Sanders_2018	USGS OCRL	Water	Dissolved, Particulate	Picoxystrobin	8	8	100
USGS-OCRL_GC/MS Sanders_2018	USGS OCRL	Water	Dissolved, Particulate	Piperonyl Butoxide	8	8	100
USGS-OCRL_GC/MS Sanders_2018	USGS OCRL	Water	Dissolved, Particulate	Prodiamine	8	8	100
USGS-OCRL_GC/MS Sanders_2018	USGS OCRL	Water	Dissolved, Particulate	Prometon	8	8	100
USGS-OCRL_GC/MS Sanders_2018	USGS OCRL	Water	Dissolved, Particulate	Prometryn	8	8	100
USGS-OCRL_GC/MS Sanders_2018	USGS OCRL	Water	Dissolved, Particulate	Propanil	8	8	100
USGS-OCRL_GC/MS Sanders_2018	USGS OCRL	Water	Dissolved, Particulate	Propargite	8	8	100
USGS-OCRL_GC/MS Sanders_2018	USGS OCRL	Water	Dissolved, Particulate	Propiconazole	8	8	100
USGS-OCRL_GC/MS Sanders_2018	USGS OCRL	Water	Dissolved, Particulate	Propyzamide	8	8	100
USGS-OCRL_GC/MS Sanders_2018	USGS OCRL	Water	Dissolved, Particulate	Pyraclostrobin	8	8	100
USGS-OCRL_GC/MS Sanders_2018	USGS OCRL	Water	Dissolved, Particulate	Pyridaben	8	8	100
USGS-OCRL_GC/MS Sanders_2018	USGS OCRL	Water	Dissolved, Particulate	Pyrimethanil	8	8	100
USGS-OCRL_GC/MS Sanders_2018	USGS OCRL	Water	Dissolved, Particulate	Pyriproxyfen	8	8	100
USGS-OCRL_GC/MS Sanders_2018	USGS OCRL	Water	Dissolved, Particulate	Quinoxyfen	8	8	100
USGS-OCRL_GC/MS Sanders_2018	USGS OCRL	Water	Dissolved, Particulate	Resmethrin	8	8	100
USGS-OCRL_GC/MS Sanders_2018	USGS OCRL	Water	Dissolved, Particulate	Sedaxane	8	8	100
USGS-OCRL_GC/MS Sanders_2018	USGS OCRL	Water	Dissolved, Particulate	Simazine	8	8	100
USGS-OCRL_GC/MS Sanders_2018	USGS OCRL	Water	Dissolved, Particulate	Tebuconazole	8	8	100
USGS-OCRL_GC/MS Sanders_2018	USGS OCRL	Water	Dissolved, Particulate	Tebupirimfos	8	8	100
USGS-OCRL_GC/MS Sanders_2018	USGS OCRL	Water	Dissolved, Particulate	Tebupirimfos oxon	8	8	100
USGS-OCRL_GC/MS Sanders_2018	USGS OCRL	Water	Dissolved, Particulate	Tefluthrin	8	8	100
USGS-OCRL_GC/MS Sanders_2018	USGS OCRL	Water	Dissolved, Particulate	Tetraconazole	8	8	100
USGS-OCRL_GC/MS Sanders_2018	USGS OCRL	Water	Dissolved, Particulate	Tetramethrin	8	8	100
USGS-OCRL_GC/MS Sanders_2018	USGS OCRL	Water	Dissolved, Particulate	T-Fluvalinate	8	8	100
USGS-OCRL_GC/MS Sanders_2018	USGS OCRL	Water	Dissolved, Particulate	Thiobencarb	8	8	100



Method	LABORATORY	Matri X	Fractions	Analyte	Total Duplicate Samples	DUPLICATE SAMPLES WITHIN LIMIT	Acceptability Met (%)
USGS-OCRL_GC/MS Sanders_2018	USGS OCRL	Water	Dissolved, Particulate	Triadimefon	8	8	100
USGS-OCRL_GC/MS Sanders_2018	USGS OCRL	Water	Dissolved, Particulate	Triadimenol	8	8	100
USGS-OCRL_GC/MS Sanders_2018	USGS OCRL	Water	Dissolved, Particulate	Triallate	8	8	100
USGS-OCRL_GC/MS Sanders_2018	USGS OCRL	Water	Dissolved, Particulate	Tributyl Phosphorotrithioate, S,S,S-	8	8	100
USGS-OCRL_GC/MS Sanders_2018	USGS OCRL	Water	Dissolved, Particulate	Trifloxystrobin	8	8	100
USGS-OCRL_GC/MS Sanders_2018	USGS OCRL	Water	Dissolved, Particulate	Triflumizole	8	8	100
USGS-OCRL_GC/MS Sanders_2018	USGS OCRL	Water	Dissolved, Particulate	Trifluralin	8	8	100
USGS-OCRL_GC/MS Sanders_2018	USGS OCRL	Water	Dissolved, Particulate	Triticonazole	8	8	100
USGS-OCRL_GC/MS Sanders_2018	USGS OCRL	Water	Dissolved, Particulate	Zoxamide	8	8	100
EPA 160.2	USGS-PFRG- OCRL	Water	Particulate	Total Suspended Solids	3	0	0
		1155	1152	99.7%			

 13 ,4- Dichlorobenzenamine is analyzed on both instruments. The dissolved fraction is reported under the LC/MS/MS method due to lower detection limits; the particulate is reported under the GC/MS method.

3.2.1.3 Acceptability of Accuracy Measurements

Accuracy and bias in the field and laboratory are measured through a combination of negative and positive control samples. Blank sample acceptability is summarized in **Table 11**. All blank samples analyzed during FY 20-21 met acceptability criteria. Laboratory spike sample acceptability is summarized in **Table 12**. All spike samples analyzed during FY 20-21 met the percent recovery acceptability criteria. Surrogate recoveries are summarized in **Table 13**. All surrogate recoveries during FY 20-21 met acceptability criteria.

Table 11. Dialik sample acceptability for pesticide samples conected during 11 20-21.									
Метнор	LABORATORY	Matrix	Fractions	ANALYTE	Total Blank Samples	Samples WITHIN LIMITS	Acceptability Met (%)		
USGS-OCRL_LC/MS/MS Sanders_2018	USGS OCRL	Water	Dissolved	Acetamiprid	3	3	100		
USGS-OCRL_LC/MS/MS Sanders_2018	USGS OCRL	Water	Dissolved	Carbendazim	3	3	100		
USGS-OCRL_LC/MS/MS Sanders_2018	USGS OCRL	Water	Dissolved	Carboxin	3	3	100		
USGS-OCRL_LC/MS/MS Sanders_2018	USGS OCRL	Water	Dissolved	Chlorantraniliprole	3	3	100		
USGS-OCRL_LC/MS/MS Sanders_2018	USGS OCRL	Water	Dissolved	Clothianidin	3	3	100		

Table 11. Blank sample acceptability for pesticide samples collected during FY 20-21.



Метнор	LABORATORY	Matrix	Fractions	Analyte	Total Blank Samples	Samples Within Limits	Acceptability Met (%)
USGS-OCRL_LC/MS/MS Sanders_2018	USGS OCRL	Water	Dissolved	Cyantraniliprole	3	3	100
USGS-OCRL_LC/MS/MS Sanders_2018	USGS OCRL	Water	Dissolved	Cyazofamid	3	3	100
USGS-OCRL_LC/MS/MS Sanders_2018	USGS OCRL	Water	Dissolved	Cymoxanil	3	3	100
USGS-OCRL_LC/MS/MS Sanders_2018	USGS OCRL	Water	Dissolved	Desthio- prothioconazole	3	3	100
USGS-OCRL_LC/MS/MS Sanders_2018	USGS OCRL	Water	Dissolved ¹	Dichlorobenzenamine, 3,4-	3	3	100
USGS-OCRL_LC/MS/MS Sanders_2018	USGS OCRL	Water	Dissolved	Dichlorophenyl Urea, 3,4-	3	3	100
USGS-OCRL_LC/MS/MS Sanders_2018	USGS OCRL	Water	Dissolved	Dichlorophenyl-3- methyl Urea, 3,4-	3	3	100
USGS-OCRL_LC/MS/MS Sanders_2018	USGS OCRL	Water	Dissolved	Dinotefuran	3	3	100
USGS-OCRL_LC/MS/MS Sanders_2018	USGS OCRL	Water	Dissolved	Diuron	3	3	100
USGS-OCRL_LC/MS/MS Sanders_2018	USGS OCRL	Water	Dissolved	Ethaboxam	3	3	100
USGS-OCRL_LC/MS/MS Sanders_2018	USGS OCRL	Water	Dissolved	Flonicamid	3	3	100
USGS-OCRL_LC/MS/MS Sanders_2018	USGS OCRL	Water	Dissolved	Flupyradifurone	3	3	100
USGS-OCRL_LC/MS/MS Sanders_2018	USGS OCRL	Water	Dissolved	Fluridone	3	3	100
USGS-OCRL_LC/MS/MS Sanders_2018	USGS OCRL	Water	Dissolved	Imidacloprid	3	3	100
USGS-OCRL_LC/MS/MS Sanders_2018	USGS OCRL	Water	Dissolved	Imidacloprid urea	3	3	100
USGS-OCRL_LC/MS/MS Sanders_2018	USGS OCRL	Water	Dissolved	Mandipropamid	3	3	100
USGS-OCRL_LC/MS/MS Sanders_2018	USGS OCRL	Water	Dissolved	Methoxyfenozide	3	3	100
USGS-OCRL_LC/MS/MS Sanders_2018	USGS OCRL	Water	Dissolved	Oryzalin	3	3	100
USGS-OCRL_LC/MS/MS Sanders_2018	USGS OCRL	Water	Dissolved	Oxathiapiprolin	3	3	100
USGS-OCRL_LC/MS/MS Sanders_2018	USGS OCRL	Water	Dissolved	Penoxsulam	3	3	100
USGS-OCRL_LC/MS/MS Sanders_2018	USGS OCRL	Water	Dissolved	Penthiopyrad	3	3	100
USGS-OCRL_LC/MS/MS Sanders_2018	USGS OCRL	Water	Dissolved	Sulfoxaflor	3	3	100
USGS-OCRL_LC/MS/MS Sanders_2018	USGS OCRL	Water	Dissolved	Tebufenozide	3	3	100
USGS-OCRL_LC/MS/MS Sanders_2018	USGS OCRL	Water	Dissolved	Thiabendazole	3	3	100
USGS-OCRL_LC/MS/MS Sanders_2018	USGS OCRL	Water	Dissolved	Thiacloprid	3	3	100
USGS-OCRL_LC/MS/MS Sanders_2018	USGS OCRL	Water	Dissolved	Thiamethoxam	3	3	100



Method	LABORATORY	Matrix	Fractions	Analyte	Total Blank Samples	Samples WITHIN LIMITS	Acceptability Met (%)
USGS-OCRL_LC/MS/MS Sanders_2018	USGS OCRL	Water	Dissolved	Thiamethoxam Degradate (CGA- 355190)	3	3	100
USGS-OCRL_LC/MS/MS Sanders_2018	USGS OCRL	Water	Dissolved	Thiamethoxam Degradate (NOA- 407475)	3	3	100
USGS-OCRL_LC/MS/MS Sanders_2018	USGS OCRL	Water	Dissolved	Tolfenpyrad	3	3	100
USGS-OCRL_LC/MS/MS Sanders_2018	USGS OCRL	Water	Dissolved	Tricyclazole	3	3	100
USGS-OCRL_GC/MS Sanders_2018	USGS OCRL	Water	Dissolved, Particulate	Acibenzolar-S-methyl	6	6	100
USGS-OCRL_GC/MS Sanders_2018	USGS OCRL	Water	Dissolved, Particulate	Allethrin	6	6	100
USGS-OCRL_GC/MS Sanders_2018	USGS OCRL	Water	Dissolved, Particulate	Atrazine	6	6	100
USGS-OCRL_GC/MS Sanders_2018	USGS OCRL	Water	Dissolved, Particulate	Azoxystrobin	6	6	100
USGS-OCRL_GC/MS Sanders_2018	USGS OCRL	Water	Dissolved, Particulate	Benfluralin	6	6	100
USGS-OCRL_GC/MS Sanders_2018	USGS OCRL	Water	Dissolved, Particulate	Benzovindiflupyr	6	6	100
USGS-OCRL_GC/MS Sanders_2018	USGS OCRL	Water	Dissolved, Particulate	Bifenthrin	6	6	100
USGS-OCRL_GC/MS Sanders_2018	USGS OCRL	Water	Dissolved, Particulate	Boscalid	6	6	100
USGS-OCRL_GC/MS Sanders_2018	USGS OCRL	Water	Dissolved, Particulate	Butralin	6	6	100
USGS-OCRL_GC/MS Sanders_2018	USGS OCRL	Water	Dissolved, Particulate	Captan	6	6	100
USGS-OCRL_GC/MS Sanders_2018	USGS OCRL	Water	Dissolved, Particulate	Carbaryl	6	6	100
USGS-OCRL_GC/MS Sanders_2018	USGS OCRL	Water	Dissolved, Particulate	Carbofuran	6	6	100
USGS-OCRL_GC/MS Sanders_2018	USGS OCRL	Water	Dissolved, Particulate	Chlorfenapyr	6	6	100
USGS-OCRL_GC/MS Sanders_2018	USGS OCRL	Water	Dissolved, Particulate	Chloro-N- (ethoxymethyl)-N-(2- ethyl-6- methylphenyl)acetamide , 2-	6	6	100
USGS-OCRL_GC/MS Sanders_2018	USGS OCRL	Water	Dissolved, Particulate	Chlorothalonil	6	6	100
USGS-OCRL_GC/MS Sanders_2018	USGS OCRL	Water	Dissolved, Particulate	Chlorpyrifos	6	6	100
USGS-OCRL_GC/MS Sanders_2018	USGS OCRL	Water	Dissolved, Particulate	Chlorpyrifos oxon	6	6	100
USGS-OCRL_GC/MS Sanders_2018	USGS OCRL	Water	Dissolved, Particulate	Clomazone	6	6	100
USGS-OCRL_GC/MS Sanders_2018	USGS OCRL	Water	Dissolved, Particulate	Coumaphos	6	6	100
USGS-OCRL_GC/MS Sanders_2018	USGS OCRL	Water	Dissolved, Particulate	Cycloate	6	6	100



Метнор	LABORATORY	Matrix	Fractions	Analyte	Total Blank Samples	Samples WITHIN LIMITS	Acceptability Met (%)
USGS-OCRL_GC/MS Sanders_2018	USGS OCRL	Water	Dissolved, Particulate	Cyfluthrin, Total	6	6	100
USGS-OCRL_GC/MS Sanders_2018	USGS OCRL	Water	Dissolved, Particulate	Cyhalofop-butyl	6	6	100
USGS-OCRL_GC/MS Sanders_2018	USGS OCRL	Water	Dissolved, Particulate	Cyhalothrin	6	6	100
USGS-OCRL_GC/MS Sanders_2018	USGS OCRL	Water	Dissolved, Particulate	Cypermethrin, Total	6	6	100
USGS-OCRL_GC/MS Sanders_2018	USGS OCRL	Water	Dissolved, Particulate	Cyproconazole	6	6	100
USGS-OCRL_GC/MS Sanders_2018	USGS OCRL	Water	Dissolved, Particulate	Cyprodinil	6	6	100
USGS-OCRL_GC/MS Sanders_2018	USGS OCRL	Water	Dissolved, Particulate	Dacthal	6	6	100
USGS-OCRL_GC/MS Sanders_2018	USGS OCRL	Water	Dissolved, Particulate	DDD(p,p')	6	6	100
USGS-OCRL_GC/MS Sanders_2018	USGS OCRL	Water	Dissolved, Particulate	DDE(p,p')	6	6	100
USGS-OCRL_GC/MS Sanders_2018	USGS OCRL	Water	Dissolved, Particulate	DDT(p,p')	6	6	100
USGS-OCRL_GC/MS Sanders_2018	USGS OCRL	Water	Dissolved, Particulate	Deltamethrin	6	6	100
USGS-OCRL_GC/MS Sanders_2018	USGS OCRL	Water	Dissolved, Particulate	Diazinon	6	6	100
USGS-OCRL_GC/MS Sanders_2018	USGS OCRL	Water	Dissolved, Particulate	Diazoxon	6	6	100
USGS-OCRL_GC/MS Sanders_2018	USGS OCRL	Water	Dissolved, Particulate	Dichloroaniline, 3,5-	6	6	100
USGS-OCRL_GC/MS Sanders_2018	USGS OCRL	Water	Particulate ¹	Dichlorobenzenamine, 3,4-	3	3	100
USGS-OCRL_GC/MS Sanders_2018	USGS OCRL	Water	Dissolved, Particulate	Dichlorvos	6	6	100
USGS-OCRL_GC/MS Sanders_2018	USGS OCRL	Water	Dissolved, Particulate	Difenoconazole	6	6	100
USGS-OCRL_GC/MS Sanders_2018	USGS OCRL	Water	Dissolved, Particulate	Dimethomorph	6	6	100
USGS-OCRL_GC/MS Sanders_2018	USGS OCRL	Water	Dissolved, Particulate	Dithiopyr	6	6	100
USGS-OCRL_GC/MS Sanders_2018	USGS OCRL	Water	Dissolved, Particulate	EPTC	6	6	100
USGS-OCRL_GC/MS Sanders_2018	USGS OCRL	Water	Dissolved, Particulate	Esfenvalerate	6	6	100
USGS-OCRL_GC/MS Sanders_2018	USGS OCRL	Water	Dissolved, Particulate	Ethalfluralin	6	6	100
USGS-OCRL_GC/MS Sanders_2018	USGS OCRL	Water	Dissolved, Particulate	Ethofenprox	6	6	100
USGS-OCRL_GC/MS Sanders_2018	USGS OCRL	Water	Dissolved, Particulate	Etoxazole	6	6	100
USGS-OCRL_GC/MS Sanders_2018	USGS OCRL	Water	Dissolved, Particulate	Famoxadone	6	6	100
USGS-OCRL_GC/MS Sanders_2018	USGS OCRL	Water	Dissolved, Particulate	Fenamidone	6	6	100



Метнор	LABORATORY	Matrix	Fractions	ANALYTE	Total Blank Samples	Samples WITHIN LIMITS	Acceptability Met (%)
USGS-OCRL_GC/MS Sanders_2018	USGS OCRL	Water	Dissolved, Particulate	Fenbuconazole	6	6	100
USGS-OCRL_GC/MS Sanders_2018	USGS OCRL	Water	Dissolved, Particulate	Fenhexamid	6	6	100
USGS-OCRL_GC/MS Sanders_2018	USGS OCRL	Water	Dissolved, Particulate	Fenpropathrin	6	6	100
USGS-OCRL_GC/MS Sanders_2018	USGS OCRL	Water	Dissolved, Particulate	Fenpyroximate	6	6	100
USGS-OCRL_GC/MS Sanders_2018	USGS OCRL	Water	Dissolved, Particulate	Fipronil	6	6	100
USGS-OCRL_GC/MS Sanders_2018	USGS OCRL	Water	Dissolved, Particulate	Fipronil Desulfinyl	6	6	100
USGS-OCRL_GC/MS Sanders_2018	USGS OCRL	Water	Dissolved, Particulate	Fipronil Desulfinyl Amide	6	6	100
USGS-OCRL_GC/MS Sanders_2018	USGS OCRL	Water	Dissolved, Particulate	Fipronil Sulfide	6	6	100
USGS-OCRL_GC/MS Sanders_2018	USGS OCRL	Water	Dissolved, Particulate	Fipronil Sulfone	6	6	100
USGS-OCRL_GC/MS Sanders_2018	USGS OCRL	Water	Dissolved, Particulate	Fluazinam	6	6	100
USGS-OCRL_GC/MS Sanders_2018	USGS OCRL	Water	Dissolved, Particulate	Flubendiamide	6	6	100
USGS-OCRL_GC/MS Sanders_2018	USGS OCRL	Water	Dissolved, Particulate	Fludioxonil	6	6	100
USGS-OCRL_GC/MS Sanders_2018	USGS OCRL	Water	Dissolved, Particulate	Flufenacet	6	6	100
USGS-OCRL_GC/MS Sanders_2018	USGS OCRL	Water	Dissolved, Particulate	Flumetralin	6	6	100
USGS-OCRL_GC/MS Sanders_2018	USGS OCRL	Water	Dissolved, Particulate	Fluopicolide	6	6	100
USGS-OCRL_GC/MS Sanders_2018	USGS OCRL	Water	Dissolved, Particulate	Fluopyram	6	6	100
USGS-OCRL_GC/MS Sanders_2018	USGS OCRL	Water	Dissolved, Particulate	Fluoxastrobin	6	6	100
USGS-OCRL_GC/MS Sanders_2018	USGS OCRL	Water	Dissolved, Particulate	Flutolanil	6	6	100
USGS-OCRL_GC/MS Sanders_2018	USGS OCRL	Water	Dissolved, Particulate	Flutriafol	6	6	100
USGS-OCRL_GC/MS Sanders_2018	USGS OCRL	Water	Dissolved, Particulate	Fluxapyroxad	6	6	100
USGS-OCRL_GC/MS Sanders_2018	USGS OCRL	Water	Dissolved, Particulate	Hexazinone	6	6	100
USGS-OCRL_GC/MS Sanders_2018	USGS OCRL	Water	Dissolved, Particulate	Imazalil	6	6	100
USGS-OCRL_GC/MS Sanders_2018	USGS OCRL	Water	Dissolved, Particulate	Indaziflam	6	6	100
USGS-OCRL_GC/MS Sanders_2018	USGS OCRL	Water	Dissolved, Particulate	Indoxacarb	6	6	100
USGS-OCRL_GC/MS Sanders_2018	USGS OCRL	Water	Dissolved, Particulate	Ipconazole	6	6	100
USGS-OCRL_GC/MS Sanders_2018	USGS OCRL	Water	Dissolved, Particulate	Iprodione	6	6	100



Method	LABORATORY	Matrix	Fractions	Analyte	Total Blank Samples	Samples Within Limits	Acceptability Met (%)
USGS-OCRL_GC/MS Sanders_2018	USGS OCRL	Water	Dissolved, Particulate	lsofetamid	6	6	100
USGS-OCRL_GC/MS Sanders_2018	USGS OCRL	Water	Dissolved, Particulate	Kresoxim-methyl	6	6	100
USGS-OCRL_GC/MS Sanders_2018	USGS OCRL	Water	Dissolved, Particulate	Malaoxon	6	6	100
USGS-OCRL_GC/MS Sanders_2018	USGS OCRL	Water	Dissolved, Particulate	Malathion	6	6	100
USGS-OCRL_GC/MS Sanders_2018	USGS OCRL	Water	Dissolved, Particulate	Metalaxyl	6	6	100
USGS-OCRL_GC/MS Sanders_2018	USGS OCRL	Water	Dissolved, Particulate	Metconazole	6	6	100
USGS-OCRL_GC/MS Sanders_2018	USGS OCRL	Water	Dissolved, Particulate	Methoprene	6	6	100
USGS-OCRL_GC/MS Sanders_2018	USGS OCRL	Water	Dissolved, Particulate	Metolachlor	6	6	100
USGS-OCRL_GC/MS Sanders_2018	USGS OCRL	Water	Dissolved, Particulate	Myclobutanil	6	6	100
USGS-OCRL_GC/MS Sanders_2018	USGS OCRL	Water	Dissolved, Particulate	Napropamide	6	6	100
USGS-OCRL_GC/MS Sanders_2018	USGS OCRL	Water	Dissolved, Particulate	Novaluron	6	6	100
USGS-OCRL_GC/MS Sanders_2018	USGS OCRL	Water	Dissolved, Particulate	Oxadiazon	6	6	100
USGS-OCRL_GC/MS Sanders_2018	USGS OCRL	Water	Dissolved, Particulate	Oxyfluorfen	6	6	100
USGS-OCRL_GC/MS Sanders_2018	USGS OCRL	Water	Dissolved, Particulate	Paclobutrazol	6	6	100
USGS-OCRL_GC/MS Sanders_2018	USGS OCRL	Water	Dissolved, Particulate	Parathion, Methyl	6	6	100
USGS-OCRL_GC/MS Sanders_2018	USGS OCRL	Water	Dissolved, Particulate	Pendimethalin	6	6	100
USGS-OCRL_GC/MS Sanders_2018	USGS OCRL	Water	Dissolved, Particulate	Pentachloroanisole	6	6	100
USGS-OCRL_GC/MS Sanders_2018	USGS OCRL	Water	Dissolved, Particulate	Pentachloronitrobenzen e	6	6	100
USGS-OCRL_GC/MS Sanders_2018	USGS OCRL	Water	Dissolved, Particulate	Permethrin, Total	6	6	100
USGS-OCRL_GC/MS Sanders_2018	USGS OCRL	Water	Dissolved, Particulate	Phenothrin	6	6	100
USGS-OCRL_GC/MS Sanders_2018	USGS OCRL	Water	Dissolved, Particulate	Phosmet	6	6	100
USGS-OCRL_GC/MS Sanders_2018	USGS OCRL	Water	Dissolved, Particulate	Picoxystrobin	6	6	100
USGS-OCRL_GC/MS Sanders_2018	USGS OCRL	Water	Dissolved, Particulate	Piperonyl Butoxide	6	6	100
USGS-OCRL_GC/MS Sanders_2018	USGS OCRL	Water	Dissolved, Particulate	Prodiamine	6	6	100
USGS-OCRL_GC/MS Sanders_2018	USGS OCRL	Water	Dissolved, Particulate	Prometon	6	6	100
USGS-OCRL_GC/MS Sanders_2018	USGS OCRL	Water	Dissolved, Particulate	Prometryn	6	6	100



Метнор	LABORATORY	Matrix	Fractions	Analyte	Total Blank Samples	Samples WITHIN LIMITS	Acceptability Met (%)
USGS-OCRL_GC/MS Sanders_2018	USGS OCRL	Water	Dissolved, Particulate	Propanil	6	6	100
USGS-OCRL_GC/MS Sanders_2018	USGS OCRL	Water	Dissolved, Particulate	Propargite	6	6	100
USGS-OCRL_GC/MS Sanders_2018	USGS OCRL	Water	Dissolved, Particulate	Propiconazole	6	6	100
USGS-OCRL_GC/MS Sanders_2018	USGS OCRL	Water	Dissolved, Particulate	Propyzamide	6	6	100
USGS-OCRL_GC/MS Sanders_2018	USGS OCRL	Water	Dissolved, Particulate	Pyraclostrobin	6	6	100
USGS-OCRL_GC/MS Sanders_2018	USGS OCRL	Water	Dissolved, Particulate	Pyridaben	6	6	100
USGS-OCRL_GC/MS Sanders_2018	USGS OCRL	Water	Dissolved, Particulate	Pyrimethanil	6	6	100
USGS-OCRL_GC/MS Sanders_2018	USGS OCRL	Water	Dissolved, Particulate	Pyriproxyfen	6	6	100
USGS-OCRL_GC/MS Sanders_2018	USGS OCRL	Water	Dissolved, Particulate	Quinoxyfen	6	6	100
USGS-OCRL_GC/MS Sanders_2018	USGS OCRL	Water	Dissolved, Particulate	Resmethrin	6	6	100
USGS-OCRL_GC/MS Sanders_2018	USGS OCRL	Water	Dissolved, Particulate	Sedaxane	6	6	100
USGS-OCRL_GC/MS Sanders_2018	USGS OCRL	Water	Dissolved, Particulate	Simazine	6	6	100
USGS-OCRL_GC/MS Sanders_2018	USGS OCRL	Water	Dissolved, Particulate	Tebuconazole	6	6	100
USGS-OCRL_GC/MS Sanders_2018	USGS OCRL	Water	Dissolved, Particulate	Tebupirimfos	6	6	100
USGS-OCRL_GC/MS Sanders_2018	USGS OCRL	Water	Dissolved, Particulate	Tebupirimfos oxon	6	6	100
USGS-OCRL_GC/MS Sanders_2018	USGS OCRL	Water	Dissolved, Particulate	Tefluthrin	6	6	100
USGS-OCRL_GC/MS Sanders_2018	USGS OCRL	Water	Dissolved, Particulate	Tetraconazole	6	6	100
USGS-OCRL_GC/MS Sanders_2018	USGS OCRL	Water	Dissolved, Particulate	Tetramethrin	6	6	100
USGS-OCRL_GC/MS Sanders_2018	USGS OCRL	Water	Dissolved, Particulate	T-Fluvalinate	6	6	100
USGS-OCRL_GC/MS Sanders_2018	USGS OCRL	Water	Dissolved, Particulate	Thiobencarb	6	6	100
USGS-OCRL_GC/MS Sanders_2018	USGS OCRL	Water	Dissolved, Particulate	Triadimefon	6	6	100
USGS-OCRL_GC/MS Sanders_2018	USGS OCRL	Water	Dissolved, Particulate	Triadimenol	6	6	100
USGS-OCRL_GC/MS Sanders_2018	USGS OCRL	Water	Dissolved, Particulate	Triallate	6	6	100
USGS-OCRL_GC/MS Sanders_2018	USGS OCRL	Water	Dissolved, Particulate	Tributyl Phosphorotrithioate, S,S,S-	6	6	100
USGS-OCRL_GC/MS Sanders_2018	USGS OCRL	Water	Dissolved, Particulate	Trifloxystrobin	6	6	100
USGS-OCRL_GC/MS Sanders_2018	USGS OCRL	Water	Dissolved, Particulate	Triflumizole	6	6	100



Метнор	LABORATORY	Matrix	Fractions	ANALYTE	Total Blank Samples	Samples WITHIN LIMITS	Acceptability Met (%)
USGS-OCRL_GC/MS Sanders_2018	USGS OCRL	Water	Dissolved, Particulate	Trifluralin	6	6	100
USGS-OCRL_GC/MS Sanders_2018	USGS OCRL	Water	Dissolved, Particulate	Triticonazole	6	6	100
USGS-OCRL_GC/MS Sanders_2018	USGS OCRL	Water	Dissolved, Particulate	Zoxamide	6	6	100
EPA 160.2	USGS OCRL	Water	Particulate	Total Suspended Solids	3	3	100
				Total	867	867	100

 1 3,4- Dichlorobenzenamine is analyzed on both instruments. The dissolved fraction is reported under the LC/MS/MS method due to lower detection limits; the particulate is reported under the GC/MS method.

METHOD	-	Matrix		ANALYTE	Total Spiked	Samples WITHIN	Acceptability Met (%)
USGS-OCRL_LC/MS/MS Sanders_2018	USGS OCRL	Water	Dissolved	Acetamiprid	SAMPLES 4	Limits 4	100
USGS-OCRL_LC/MS/MS Sanders_2018	USGS OCRL	Water	Dissolved	Carbendazim	4	4	100
USGS-OCRL_LC/MS/MS Sanders_2018	USGS OCRL	Water	Dissolved	Carboxin	4	4	100
USGS-OCRL_LC/MS/MS Sanders_2018	USGS OCRL	Water	Dissolved	Chlorantraniliprole	4	4	100
USGS-OCRL_LC/MS/MS Sanders_2018	USGS OCRL	Water	Dissolved	Clothianidin	4	4	100
USGS-OCRL_LC/MS/MS Sanders_2018	USGS OCRL	Water	Dissolved	Cyantraniliprole	4	4	100
USGS-OCRL_LC/MS/MS Sanders_2018	USGS OCRL	Water	Dissolved	Cyazofamid	4	4	100
USGS-OCRL_LC/MS/MS Sanders_2018	USGS OCRL	Water	Dissolved	Cymoxanil	4	4	100
USGS-OCRL_LC/MS/MS Sanders_2018	USGS OCRL	Water	Dissolved	Desthio- prothioconazole	4	4	100
USGS-OCRL_LC/MS/MS Sanders_2018	USGS OCRL	Water	Dissolved ¹	Dichlorobenzenamine, 3,4-	4	4	100
USGS-OCRL_LC/MS/MS Sanders_2018	USGS OCRL	Water	Dissolved	Dichlorophenyl Urea, 3,4-	4	4	100
USGS-OCRL_LC/MS/MS Sanders_2018	USGS OCRL	Water	Dissolved	Dichlorophenyl-3- methyl Urea, 3,4-	4	4	100
USGS-OCRL_LC/MS/MS Sanders_2018	USGS OCRL	Water	Dissolved	Dinotefuran	4	4	100
USGS-OCRL_LC/MS/MS Sanders_2018	USGS OCRL	Water	Dissolved	Diuron	4	4	100
USGS-OCRL_LC/MS/MS Sanders_2018	USGS OCRL	Water	Dissolved	Ethaboxam	4	4	100
USGS-OCRL_LC/MS/MS Sanders_2018	USGS OCRL	Water	Dissolved	Flonicamid	4	4	100
USGS-OCRL_LC/MS/MS Sanders_2018	USGS OCRL	Water	Dissolved	Flupyradifurone	4	4	100
USGS-OCRL_LC/MS/MS Sanders_2018	USGS OCRL	Water	Dissolved	Fluridone	4	4	100

Table 12. Spike sample acceptability for pesticide samples collected during FY 20-21.



49 Delta RMP Annual Report for FY 20-21 February 1, 2022

METHOD	LABORATORY	Matrix	Fractions	Analyte	Total Spiked Samples	Samples WITHIN LIMITS	Acceptability Met (%)
USGS-OCRL_LC/MS/MS Sanders_2018	USGS OCRL	Water	Dissolved	Imidacloprid	4	4	100
USGS-OCRL_LC/MS/MS Sanders_2018	USGS OCRL	Water	Dissolved	Imidacloprid urea	4	4	100
USGS-OCRL_LC/MS/MS Sanders_2018	USGS OCRL	Water	Dissolved	Mandipropamid	4	4	100
USGS-OCRL_LC/MS/MS Sanders_2018	USGS OCRL	Water	Dissolved	Methoxyfenozide	4	4	100
USGS-OCRL_LC/MS/MS Sanders_2018	USGS OCRL	Water	Dissolved	Oryzalin	4	4	100
USGS-OCRL_LC/MS/MS Sanders_2018	USGS OCRL	Water	Dissolved	Oxathiapiprolin	4	4	100
USGS-OCRL_LC/MS/MS Sanders_2018	USGS OCRL	Water	Dissolved	Penoxsulam	4	4	100
USGS-OCRL_LC/MS/MS Sanders_2018	USGS OCRL	Water	Dissolved	Penthiopyrad	4	4	100
USGS-OCRL_LC/MS/MS Sanders_2018	USGS OCRL	Water	Dissolved	Sulfoxaflor	4	4	100
USGS-OCRL_LC/MS/MS Sanders_2018	USGS OCRL	Water	Dissolved	Tebufenozide	4	4	100
USGS-OCRL_LC/MS/MS Sanders_2018	USGS OCRL	Water	Dissolved	Thiabendazole	4	4	100
USGS-OCRL_LC/MS/MS Sanders_2018	USGS OCRL	Water	Dissolved	Thiacloprid	4	4	100
USGS-OCRL_LC/MS/MS Sanders_2018	USGS OCRL	Water	Dissolved	Thiamethoxam	4	4	100
USGS-OCRL_LC/MS/MS Sanders_2018	USGS OCRL	Water	Dissolved	Thiamethoxam Degradate (CGA- 355190)	4	4	100
USGS-OCRL_LC/MS/MS Sanders_2018	USGS OCRL	Water	Dissolved	Thiamethoxam Degradate (NOA- 407475)	4	4	100
USGS-OCRL_LC/MS/MS Sanders_2018	USGS OCRL	Water	Dissolved	Tolfenpyrad	4	4	100
USGS-OCRL_LC/MS/MS Sanders_2018	USGS OCRL	Water	Dissolved	Tricyclazole	4	4	100
USGS-OCRL_GC/MS Sanders_2018	USGS OCRL	Water	Dissolved, Particulate	Acibenzolar-S-methyl	8	8	100
USGS-OCRL_GC/MS Sanders_2018	USGS OCRL	Water	Dissolved, Particulate	Allethrin	8	8	100
USGS-OCRL_GC/MS Sanders_2018	USGS OCRL	Water	Dissolved, Particulate	Atrazine	8	8	100
USGS-OCRL_GC/MS Sanders_2018	USGS OCRL	Water	Dissolved, Particulate	Azoxystrobin	8	8	100
USGS-OCRL_GC/MS Sanders_2018	USGS OCRL	Water	Dissolved, Particulate	Benfluralin	8	8	100
USGS-OCRL_GC/MS Sanders_2018	USGS OCRL	Water	Dissolved, Particulate	Benzovindiflupyr	8	8	100
USGS-OCRL_GC/MS Sanders_2018	USGS OCRL	Water	Dissolved, Particulate	Bifenthrin	8	8	100
USGS-OCRL_GC/MS Sanders_2018	USGS OCRL	Water	Dissolved, Particulate	Boscalid	8	8	100



METHOD	LABORATORY	Matrix	FRACTIONS	ANALYTE	Total Spiked Samples	Samples WITHIN LIMITS	Acceptability Met (%)
USGS-OCRL_GC/MS Sanders_2018	USGS OCRL	Water	Dissolved, Particulate	Butralin	8	8	100
USGS-OCRL_GC/MS Sanders_2018	USGS OCRL	Water	Dissolved, Particulate	Captan	8	8	100
USGS-OCRL_GC/MS Sanders_2018	USGS OCRL	Water	Dissolved, Particulate	Carbaryl	8	8	100
USGS-OCRL_GC/MS Sanders_2018	USGS OCRL	Water	Dissolved, Particulate	Carbofuran	8	8	100
USGS-OCRL_GC/MS Sanders_2018	USGS OCRL	Water	Dissolved, Particulate	Chlorfenapyr	8	8	100
USGS-OCRL_GC/MS Sanders_2018	USGS OCRL	Water	Dissolved, Particulate	Chloro-N- (ethoxymethyl)-N-(2- ethyl-6- methylphenyl)acetamide , 2-	8	8	100
USGS-OCRL_GC/MS Sanders_2018	USGS OCRL	Water	Dissolved, Particulate	Chlorothalonil	8	8	100
USGS-OCRL_GC/MS Sanders_2018	USGS OCRL	Water	Dissolved, Particulate	Chlorpyrifos	8	8	100
USGS-OCRL_GC/MS Sanders_2018	USGS OCRL	Water	Dissolved, Particulate	Chlorpyrifos oxon	8	8	100
USGS-OCRL_GC/MS Sanders_2018	USGS OCRL	Water	Dissolved, Particulate	Clomazone	8	8	100
USGS-OCRL_GC/MS Sanders_2018	USGS OCRL	Water	Dissolved, Particulate	Coumaphos	8	8	100
USGS-OCRL_GC/MS Sanders_2018	USGS OCRL	Water	Dissolved, Particulate	Cycloate	8	8	100
USGS-OCRL_GC/MS Sanders_2018	USGS OCRL	Water	Dissolved, Particulate	Cyfluthrin, Total	8	8	100
USGS-OCRL_GC/MS Sanders_2018	USGS OCRL	Water	Dissolved, Particulate	Cyhalofop-butyl	8	8	100
USGS-OCRL_GC/MS Sanders_2018	USGS OCRL	Water	Dissolved, Particulate	Cyhalothrin	8	8	100
USGS-OCRL_GC/MS Sanders_2018	USGS OCRL	Water	Dissolved, Particulate	Cypermethrin, Total	8	8	100
USGS-OCRL_GC/MS Sanders_2018	USGS OCRL	Water	Dissolved, Particulate	Cyproconazole	8	8	100
USGS-OCRL_GC/MS Sanders_2018	USGS OCRL	Water	Dissolved, Particulate	Cyprodinil	8	8	100
USGS-OCRL_GC/MS Sanders_2018	USGS OCRL	Water	Dissolved, Particulate	Dacthal	8	8	100
USGS-OCRL_GC/MS Sanders_2018	USGS OCRL	Water	Dissolved, Particulate	DDD(p,p')	8	8	100
USGS-OCRL_GC/MS Sanders_2018	USGS OCRL	Water	Dissolved, Particulate	DDE(p,p')	8	8	100
USGS-OCRL_GC/MS Sanders_2018	USGS OCRL	Water	Dissolved, Particulate	DDT(p,p')	8	8	100
USGS-OCRL_GC/MS Sanders_2018	USGS OCRL	Water	Dissolved, Particulate	Deltamethrin	8	8	100
USGS-OCRL_GC/MS Sanders_2018	USGS OCRL	Water	Dissolved, Particulate	Diazinon	8	8	100
USGS-OCRL_GC/MS Sanders_2018	USGS OCRL	Water	Dissolved, Particulate	Diazoxon	8	8	100



METHOD	LABORATORY	Matrix	Fractions	ANALYTE	Total Spiked Samples	Samples WITHIN LIMITS	Acceptability Met (%)
USGS-OCRL_GC/MS Sanders_2018	USGS OCRL	Water	Dissolved, Particulate	Dichloroaniline, 3,5-	8	8	100
USGS-OCRL_GC/MS Sanders_2018	USGS OCRL	Water	Particulate ¹	Dichlorobenzenamine, 3,4-	4	4	100
USGS-OCRL_GC/MS Sanders_2018	USGS OCRL	Water	Dissolved, Particulate	Dichlorvos	8	8	100
USGS-OCRL_GC/MS Sanders_2018	USGS OCRL	Water	Dissolved, Particulate	Difenoconazole	8	8	100
USGS-OCRL_GC/MS Sanders_2018	USGS OCRL	Water	Dissolved, Particulate	Dimethomorph	8	8	100
USGS-OCRL_GC/MS Sanders_2018	USGS OCRL	Water	Dissolved, Particulate	Dithiopyr	8	8	100
USGS-OCRL_GC/MS Sanders_2018	USGS OCRL	Water	Dissolved, Particulate	EPTC	8	8	100
USGS-OCRL_GC/MS Sanders_2018	USGS OCRL	Water	Dissolved, Particulate	Esfenvalerate	8	8	100
USGS-OCRL_GC/MS Sanders_2018	USGS OCRL	Water	Dissolved, Particulate	Ethalfluralin	8	8	100
USGS-OCRL_GC/MS Sanders_2018	USGS OCRL	Water	Dissolved, Particulate	Ethofenprox	8	8	100
USGS-OCRL_GC/MS Sanders_2018	USGS OCRL	Water	Dissolved, Particulate	Etoxazole	8	8	100
USGS-OCRL_GC/MS Sanders_2018	USGS OCRL	Water	Dissolved, Particulate	Famoxadone	8	8	100
USGS-OCRL_GC/MS Sanders_2018	USGS OCRL	Water	Dissolved, Particulate	Fenamidone	8	8	100
USGS-OCRL_GC/MS Sanders_2018	USGS OCRL	Water	Dissolved, Particulate	Fenbuconazole	8	8	100
USGS-OCRL_GC/MS Sanders_2018	USGS OCRL	Water	Dissolved, Particulate	Fenhexamid	8	8	100
USGS-OCRL_GC/MS Sanders_2018	USGS OCRL	Water	Dissolved, Particulate	Fenpropathrin	8	8	100
USGS-OCRL_GC/MS Sanders_2018	USGS OCRL	Water	Dissolved, Particulate	Fenpyroximate	8	8	100
USGS-OCRL_GC/MS Sanders_2018	USGS OCRL	Water	Dissolved, Particulate	Fipronil	8	8	100
USGS-OCRL_GC/MS Sanders_2018	USGS OCRL	Water	Dissolved, Particulate	Fipronil Desulfinyl	8	8	100
USGS-OCRL_GC/MS Sanders_2018	USGS OCRL	Water	Dissolved, Particulate	Fipronil Desulfinyl Amide	8	8	100
USGS-OCRL_GC/MS Sanders_2018	USGS OCRL	Water	Dissolved, Particulate	Fipronil Sulfide	8	8	100
USGS-OCRL_GC/MS Sanders_2018	USGS OCRL	Water	Dissolved, Particulate	Fipronil Sulfone	8	8	100
USGS-OCRL_GC/MS Sanders_2018	USGS OCRL	Water	Dissolved, Particulate	Fluazinam	8	8	100
USGS-OCRL_GC/MS Sanders_2018	USGS OCRL	Water	Dissolved, Particulate	Flubendiamide	8	8	100
USGS-OCRL_GC/MS Sanders_2018	USGS OCRL	Water	Dissolved, Particulate	Fludioxonil	8	8	100
USGS-OCRL_GC/MS Sanders_2018	USGS OCRL	Water	Dissolved, Particulate	Flufenacet	8	8	100



METHOD	LABORATORY	Matrix	Fractions	Analyte	TOTAL SPIKED SAMPLES	Samples WITHIN LIMITS	Acceptability Met (%)
USGS-OCRL_GC/MS Sanders_2018	USGS OCRL	Water	Dissolved, Particulate	Flumetralin	8	8	100
USGS-OCRL_GC/MS Sanders_2018	USGS OCRL	Water	Dissolved, Particulate	Fluopicolide	8	8	100
USGS-OCRL_GC/MS Sanders_2018	USGS OCRL	Water	Dissolved, Particulate	Fluopyram	8	8	100
USGS-OCRL_GC/MS Sanders_2018	USGS OCRL	Water	Dissolved, Particulate	Fluoxastrobin	8	8	100
USGS-OCRL_GC/MS Sanders_2018	USGS OCRL	Water	Dissolved, Particulate	Flutolanil	8	8	100
USGS-OCRL_GC/MS Sanders_2018	USGS OCRL	Water	Dissolved, Particulate	Flutriafol	8	8	100
USGS-OCRL_GC/MS Sanders_2018	USGS OCRL	Water	Dissolved, Particulate	Fluxapyroxad	8	8	100
USGS-OCRL_GC/MS Sanders_2018	USGS OCRL	Water	Dissolved, Particulate	Hexazinone	8	8	100
USGS-OCRL_GC/MS Sanders_2018	USGS OCRL	Water	Dissolved, Particulate	Imazalil	8	8	100
USGS-OCRL_GC/MS Sanders_2018	USGS OCRL	Water	Dissolved, Particulate	Indaziflam	8	8	100
USGS-OCRL_GC/MS Sanders_2018	USGS OCRL	Water	Dissolved, Particulate	Indoxacarb	8	8	100
USGS-OCRL_GC/MS Sanders_2018	USGS OCRL	Water	Dissolved, Particulate	lpconazole	8	8	100
USGS-OCRL_GC/MS Sanders_2018	USGS OCRL	Water	Dissolved, Particulate	Iprodione	8	8	100
USGS-OCRL_GC/MS Sanders_2018	USGS OCRL	Water	Dissolved, Particulate	Isofetamid	8	8	100
USGS-OCRL_GC/MS Sanders_2018	USGS OCRL	Water	Dissolved, Particulate	Kresoxim-methyl	8	8	100
USGS-OCRL_GC/MS Sanders_2018	USGS OCRL	Water	Dissolved, Particulate	Malaoxon	8	8	100
USGS-OCRL_GC/MS Sanders_2018	USGS OCRL	Water	Dissolved, Particulate	Malathion	8	8	100
USGS-OCRL_GC/MS Sanders_2018	USGS OCRL	Water	Dissolved, Particulate	Metalaxyl	8	8	100
USGS-OCRL_GC/MS Sanders_2018	USGS OCRL	Water	Dissolved, Particulate	Metconazole	8	8	100
USGS-OCRL_GC/MS Sanders_2018	USGS OCRL	Water	Dissolved, Particulate	Methoprene	8	8	100
USGS-OCRL_GC/MS Sanders_2018	USGS OCRL	Water	Dissolved, Particulate	Metolachlor	8	8	100
USGS-OCRL_GC/MS Sanders_2018	USGS OCRL	Water	Dissolved, Particulate	Myclobutanil	8	8	100
USGS-OCRL_GC/MS Sanders_2018	USGS OCRL	Water	Dissolved, Particulate	Napropamide	8	8	100
USGS-OCRL_GC/MS Sanders_2018	USGS OCRL	Water	Dissolved, Particulate	Novaluron	8	8	100
USGS-OCRL_GC/MS Sanders_2018	USGS OCRL	Water	Dissolved, Particulate	Oxadiazon	8	8	100
USGS-OCRL_GC/MS Sanders_2018	USGS OCRL	Water	Dissolved, Particulate	Oxyfluorfen	8	8	100



METHOD	LABORATORY	Matrix	FRACTIONS	Analyte	Total Spiked Samples	Samples WITHIN LIMITS	Acceptability Met (%)
USGS-OCRL_GC/MS Sanders_2018	USGS OCRL	Water	Dissolved, Particulate	Paclobutrazol	8	8	100
USGS-OCRL_GC/MS Sanders_2018	USGS OCRL	Water	Dissolved, Particulate	Parathion, Methyl	8	8	100
USGS-OCRL_GC/MS Sanders_2018	USGS OCRL	Water	Dissolved, Particulate	Pendimethalin	8	8	100
USGS-OCRL_GC/MS Sanders_2018	USGS OCRL	Water	Dissolved, Particulate	Pentachloroanisole	8	8	100
USGS-OCRL_GC/MS Sanders_2018	USGS OCRL	Water	Dissolved, Particulate	Pentachloronitrobenzen e	8	8	100
USGS-OCRL_GC/MS Sanders_2018	USGS OCRL	Water	Dissolved, Particulate	Permethrin, Total	8	8	100
USGS-OCRL_GC/MS Sanders_2018	USGS OCRL	Water	Dissolved, Particulate	Phenothrin	8	8	100
USGS-OCRL_GC/MS Sanders_2018	USGS OCRL	Water	Dissolved, Particulate	Phosmet	8	8	100
USGS-OCRL_GC/MS Sanders_2018	USGS OCRL	Water	Dissolved, Particulate	Picoxystrobin	8	8	100
USGS-OCRL_GC/MS Sanders_2018	USGS OCRL	Water	Dissolved, Particulate	Piperonyl Butoxide	8	8	100
USGS-OCRL_GC/MS Sanders_2018	USGS OCRL	Water	Dissolved, Particulate	Prodiamine	8	8	100
USGS-OCRL_GC/MS Sanders_2018	USGS OCRL	Water	Dissolved, Particulate	Prometon	8	8	100
USGS-OCRL_GC/MS Sanders_2018	USGS OCRL	Water	Dissolved, Particulate	Prometryn	8	8	100
USGS-OCRL_GC/MS Sanders_2018	USGS OCRL	Water	Dissolved, Particulate	Propanil	8	8	100
USGS-OCRL_GC/MS Sanders_2018	USGS OCRL	Water	Dissolved, Particulate	Propargite	8	8	100
USGS-OCRL_GC/MS Sanders_2018	USGS OCRL	Water	Dissolved, Particulate	Propiconazole	8	8	100
USGS-OCRL_GC/MS Sanders_2018	USGS OCRL	Water	Dissolved, Particulate	Propyzamide	8	8	100
USGS-OCRL_GC/MS Sanders_2018	USGS OCRL	Water	Dissolved, Particulate	Pyraclostrobin	8	8	100
USGS-OCRL_GC/MS Sanders_2018	USGS OCRL	Water	Dissolved, Particulate	Pyridaben	8	8	100
USGS-OCRL_GC/MS Sanders_2018	USGS OCRL	Water	Dissolved, Particulate	Pyrimethanil	8	8	100
USGS-OCRL_GC/MS Sanders_2018	USGS OCRL	Water	Dissolved, Particulate	Pyriproxyfen	8	8	100
USGS-OCRL_GC/MS Sanders_2018	USGS OCRL	Water	Dissolved, Particulate	Quinoxyfen	8	8	100
USGS-OCRL_GC/MS Sanders_2018	USGS OCRL	Water	Dissolved, Particulate	Resmethrin	8	8	100
USGS-OCRL_GC/MS Sanders_2018	USGS OCRL	Water	Dissolved, Particulate	Sedaxane	8	8	100
USGS-OCRL_GC/MS Sanders_2018	USGS OCRL	Water	Dissolved, Particulate	Simazine	8	8	100
USGS-OCRL_GC/MS Sanders_2018	USGS OCRL	Water	Dissolved, Particulate	Tebuconazole	8	8	100



METHOD	LABORATORY	Matrix	Fractions	Analyte	Total Spiked Samples	Samples WITHIN LIMITS	Acceptability Met (%)
USGS-OCRL_GC/MS Sanders_2018	USGS OCRL	Water	Dissolved, Particulate	Tebupirimfos	8	8	100
USGS-OCRL_GC/MS Sanders_2018	USGS OCRL	Water	Dissolved, Particulate	Tebupirimfos oxon	8	8	100
USGS-OCRL_GC/MS Sanders_2018	USGS OCRL	Water	Dissolved, Particulate	Tefluthrin	8	8	100
USGS-OCRL_GC/MS Sanders_2018	USGS OCRL	Water	Dissolved, Particulate	Tetraconazole	8	8	100
USGS-OCRL_GC/MS Sanders_2018	USGS OCRL	Water	Dissolved, Particulate	Tetramethrin	8	8	100
USGS-OCRL_GC/MS Sanders_2018	USGS OCRL	Water	Dissolved, Particulate	T-Fluvalinate	8	8	100
USGS-OCRL_GC/MS Sanders_2018	USGS OCRL	Water	Dissolved, Particulate	Thiobencarb	8	8	100
USGS-OCRL_GC/MS Sanders_2018	USGS OCRL	Water	Dissolved, Particulate	Triadimefon	8	8	100
USGS-OCRL_GC/MS Sanders_2018	USGS OCRL	Water	Dissolved, Particulate	Triadimenol	8	8	100
USGS-OCRL_GC/MS Sanders_2018	USGS OCRL	Water	Dissolved, Particulate	Triallate	8	8	100
USGS-OCRL_GC/MS Sanders_2018	USGS OCRL	Water	Dissolved, Particulate	Tributyl Phosphorotrithioate, S,S,S-	8	8	100
USGS-OCRL_GC/MS Sanders_2018	USGS OCRL	Water	Dissolved, Particulate	Trifloxystrobin	8	8	100
USGS-OCRL_GC/MS Sanders_2018	USGS OCRL	Water	Dissolved, Particulate	Triflumizole	8	8	100
USGS-OCRL_GC/MS Sanders_2018	USGS OCRL	Water	Dissolved, Particulate	Trifluralin	8	8	100
USGS-OCRL_GC/MS Sanders_2018	USGS OCRL	Water	Dissolved, Particulate	Triticonazole	8	8	100
USGS-OCRL_GC/MS Sanders_2018	USGS OCRL	Water	Dissolved, Particulate	Zoxamide	8	8	100
				Total		1152	100

¹3,4- Dichlorobenzenamine is analyzed on both instruments. The dissolved fraction is reported under the Liquid Chromatography Mass Spectromety Mass Spectrometer (LC/MS/MS) method due to lower detection limits; the particulate is reported under the Gas Chromatography Mass Spectrometry (GC/MS) method.

	Table 13. Surrogate recovery	v acceptability for	pesticide sample	es collected during FY 20-21.
--	------------------------------	---------------------	------------------	-------------------------------

Method	LABORATORY	Matrix	Fractions	ANALYTE	Total Surrogate Samples	Surrogates within Limits	Acceptability Met (%)
USGS- OCRL_LC/MS/MS Sanders_2018	USGS OCRL	Water	Dissolved	Imidacloprid-d4 (Surrogate)	26	26	100
USGS- OCRL_LC/MS/MS Sanders_2018	USGS OCRL	Water	Dissolved	Monuron (Surrogate)	26	26	100
USGS-OCRL_GC/MS Sanders_2018	USGS OCRL	Water	Dissolved	Atrazine-13C3 (Surrogate)	26	26	100
USGS-OCRL_GC/MS Sanders_2018	USGS OCRL	Water	Particulate	DDE(p,p') (Surrogate)	26	26	100



Метнор	LABORATORY	Matrix	Fractions	ANALYTE	Total Surrogate Samples	Surrogates within Limits	Acceptability Met (%)
USGS-OCRL_GC/MS Sanders_2018	USGS OCRL	Water	Dissolved, Particulate	Fipronil-C13 (Surrogate)	52	52	100
USGS-OCRL_GC/MS Sanders_2018	USGS OCRL	Water	Particulate	Permethrin, cis- (Surrogate)	26	26	100
USGS-OCRL_GC/MS Sanders_2018	USGS OCRL	Water	Dissolved, Particulate	Trifluralin-d14 (Surrogate)	52	52	100
		234	234	100			

3.2.1.4 Invalid Data

All USGS OCRL results analyzed during FY 20-21 are considered valid and flagged according to QAPP criteria.

3.2.2 Aquatic Toxicity

3.2.2.1 Quality Control Sample Completeness

Of the samples planned for aquatic toxicity monitoring during FY 20-21, 100% (128 of 128) were collected by USGS and analyzed by PER for toxicity. See **Pesticides and Toxicity Multi-Year Study** for more information regarding sampling constraints during the FY 20-21.

The Delta RMP QAPP (v6.4) requires that field duplicates be collected with associated toxicity analyses at an annual rate of 5%. Though the annual requirement cannot yet be fully assessed, field duplicates comprised 6% (8 of 128) of results analyzed during FY 20-21.

Laboratory QC sample requirements entail the inclusion of a negative control sample with each batch. One hundred percent (32 of 32) of the batches analyzed during FY 20-21 met batch completeness requirements. A comprehensive assessment of the QC completeness for the entire Water Year will be addressed in the Data Report to be drafted when the dataset is complete.

3.2.2.2 Acceptability of Precision Measurements

Precision is measured by field duplicate samples, which are summarized below in **Table 14**; all field duplicate samples analyzed during FY 20-21 met acceptability criteria.

Метнор	Laboratory			Analyte	Ī	DUPLICATE SAMPLES	
EPA 821/R-02-013M	PER	Water	Growth ¹ , Survival	Chironomus dilutus	2	2	100
EPA 821/R-02-013	PER	Water	Survival, Reproduction ²	Ceriodaphnia dubia	2	2	100
EPA 821/R-02-013	PER	Water	Growth ³ , Survival	Pimephales promelas	2	2	100
EPA 821/R-02-013	PER	Water	Growth ⁴	Selenastrum capricornutum	1	1	100

Table 14. Precision measurement acceptability for toxicity samples collected during FY 20-21.



Метнор	Laboratory	Matrix	Endpoint	Analyte	TOTAL DUPLICATE SAMPLES	Duplicate Samples within Limit	Acceptability Met (%)
EPA 821/R-02-012M	EPA 821/R-02-012M PER Water Survival Hyale						100
	8	8	100				

¹Growth as the ash-free dry weight per surviving individual.

²Number of young per female.

³Biomass as wight per original individual.

⁴Total cell count.

3.2.2.3 Acceptability of Accuracy Measurements

Accuracy and bias in the laboratory are assessed through the use of negative control samples performed with each batch and reference toxicant tests performed periodically by the laboratory. The negative control sample results from FY 20-21 are summarized below in **Table 15**; all control samples generated during FY 20-21 met test acceptability criteria.

Table 13. Regative control sample acceptability for toxicity samples conected during 1120-21.											
Метнор	Labor atory	CONTROL	Matrix	Endpoint	OPOINT ANALYTE		Samples Within Limits	Acceptability Met (%)			
EPA 821/R-02- 013M	PER	Negative Control	Water	Growth ¹ , Survival	Chironomus dilutus	8	8	100			
EPA 821/R-02-013	PER	Negative Control	Water	Survival, Reproduction ²	Ceriodaphnia dubia	8	8	100			
EPA 821/R-02-013	PER	Salinity Control	Water	Survival, Reproduction ²	Ceriodaphnia dubia	4	4	100			
EPA 821/R-02-013	PER	Negative Control	Water	Growth ³ , Survival	Pimephales promelas	8	8	100			
EPA 821/R-02-013	PER	Negative Control	Water	Growth ⁴	Selenastrum capricornutum	4	4	100			
EPA 821/R-02- 012M	PER	Negative Control	Water	Survival	Hyalella azteca	4	4	100			
		36	36	100							

Table 15. Negative control sample acceptability for toxicity samples collected during FY 20-21.

¹Growth as the ash-free dry weight per surviving individual.

²Number of young per female.

3.2.2.4 Invalid Data

³Biomass as wight per original individual.

⁴Total cell count.

rotar cen count.

All aquatic toxicity results analyzed during FY 20-21 are considered valid and flagged according to QAPP criteria.



4 QUALITY ASSURANCE – DATA NOT MANAGED BY THE DELTA RMP

4.1 MERCURY MONITORING

Mercury monitoring for FY 20-21 was planned to take place over four events. The final event, scheduled for May of 2021, was intended to be prey fish monitoring at wetland restoration sites. Due to permitting constraints, prey fish monitoring was cancelled, and a deviation form was completed. Three of the four originally planned events were completed as planned in September 2020, March 2021, and April 2021. The Department of Fish and Wildlife would not issue permits to collect prey fish in areas of sensitive habitat for Delta smelt for the planned May 2021 event. Cruise reports were provided to the Delta RMP on January 18, 2022, describing the samples collected for the monitoring year (**Appendix I**).

The data generated during the three sampling events conducted during FY 20-21 have been processed and submitted to SWAMP for final data review and upload to CEDEN. These data are currently under review by SWRCB staff and not yet available to the public via the CEDEN AQT. The preliminary EDDs processed by MLML and provided to SWAMP are included in Attachment B to this report; these data are considered preliminary because they have not yet undergone a full SWAMP evaluation.

Mercury monitoring includes the collection of samples to be analyzed for total mercury in fish tissue (September only) and for mercury, methylmercury, and additional parameters in water (September, March, and April). Field QC sample requirements are outlined in the Delta RMP QAPP:

- Mercury and methylmercury in water require field duplicates, field blanks, and equipment blanks,
- Additional parameters in water require field duplicates and field blanks, and
- Tissue samples require neither field duplicates nor field blanks.

Where required, field QC samples must be collected at a frequency of 5% of annual environmental samples. A complete assessment of the field QC frequency will be conducted when data are finalized and available to the public.

Lab QC samples required by the QAPP are a combination of laboratory blanks, duplicates, matrix spikes, control spikes, and Certified Reference Materials (CRMs). A complete assessment of the precision, accuracy, and completeness given the acceptability criteria for each of these samples will be conducted once the data are finalized and available to the public.



4.2 NUTRIENTS

4.2.1 Cyanotoxin Monitoring in the Delta, USGS, and DWR

Data collection for the cyanotoxin study was to occur over a 12-month period. The collection began in March 2021. During FY 20-21, 4 of the 12 months of data were collected.

Quality assurance and QC procedures for these samples are conducted according to the individual quality assurance manuals and standard operating procedures maintained by USGS and DWR. Field QC sample collection follows the USGS and DWR quality assurance protocols for blanks and replicates. A minimum of one QC sample (e.g., blank, replicate) will be collected every 10 samples (10% of the total environmental samples). Quality control data will be reviewed by the project chief and QC failures are assessed by staff. Corrective actions are taken with either field or laboratory staff, as necessary.

Data generated by this study are still being analyzed by the laboratories and processed by USGS. Study data have not yet been provided to the Delta RMP. Once complete, whole water sample results will be made available on NWIS. Both whole water and SPATT sampler results will be made available via the USGS ScienceBase once processed and reviewed.

4.2.2 Source Tracking of Cyanotoxin Blooms in the Delta, Bend Genetics, and CVRWQCB

Field sampling began in November 2020 and concluded in July 2021 for the Microcystic study. A final report, *Mapping benthic overwintering Microcystis sp. within the Sacramento-San Joaquin Delta,* was provided to the CVRWQCB and Delta RMP on December 31, 2021. This report is included as **Appendix II**. The associated dataset discussed in this report is provided as Attachment D to this report. Data are not yet published to CEDEN and are pending SWRCB guidance on storing qPCR results.



APPENDIX I – MERCURY MONITORING CRUISE REPORTS



Delta RMP Annual Report for FY 20-21 February 1, 2022 Appendix 1: Cruise Report

Appendix 1 Cruise Report for the Delta Regional Monitoring Program (Delta RMP) Mercury Monitoring for Subregional Trends in Black Bass and Water

Year 5 FY20/21 Trend Work

Sampling Dates: September 08, 2020 – April 13, 2021

Prepared by Marine Pollution Studies Laboratory Staff (<u>MPSL-DFW</u>)

at Moss Landing Marine Laboratories; San Jose State University

Introduction

This report describes the sampling activities of the Delta Regional Monitoring Program (DRMP) in subareas of the Delta region of California. Sampling activities included the collection of fish tissue (black bass), and water samples with basic field parameters. Samples were collected by Marine Pollution Studies Laboratory (MPSL-DFW) at Moss Landing Marine Laboratories (MLML) staff.

1.0 Cruise Report

1.1 Objectives

The objectives were to collect fish and water samples that would provide spatial and temporal data to answer DRMP management and assessment questions. Black bass were collected annually using an electrofisher boat at seven (7) fixed stations selected for long-term monitoring. Sixteen (16) black bass were collected spanning a broad size range for each station. Each bass was analyzed individually for mercury.. The annual fish collection was paired with water collection at each of the seven stations.

Depth-integrated water samples were collected in the thalweg at seven (7) stations. These stations are strategically located to correlate with the fish monitoring and Delta water import and export locations. Chemical analyte groups for the water collection include: total Hg, dissolved Hg, total MeHg and dissolved MeHg. The following ancillary water parameters were collected to aid in interpretation of the MeHg data: chlorophyll *a*, dissolved organic carbon (DOC), total suspended solids (TSS), and volatile suspended solids (VSS).

1.2 MPSL Sampling personnel

Wesley Heim April Sjoboen Guimarães Autumn Bonnema Gary Ichikawa William Jakl Chris Beebe Scot Lucas Project Director Research Technician, Crew Lead Associate Project Director Project Assistant, Crew Lead Project Associate, Crew Lead Research Technician Research Technician

1.3 Authorization to collect samples

All sampling personnel are MPSL-DFW staff (San Jose State University Foundation and the California Department of Fish and Wildlife) contracted through the State of California Water Board SWAMP Program to conduct the sample collection activities listed herein.

1.4 Station selection

Based upon the recommendations of the Delta RMP Steering Committee and Technical Advisory Committee with representatives from the Central Valley Regional Water Quality Control Board, USEPA, California Department of Water Resources, the State and Federal Contractors Water Agency, and various discharger groups, stations were selected to represent key subareas of the Delta.

1.5 Summary of types of samples authorized to be collected

Up to sixteen (16) black bass individuals of the same species were collected using an electrofisher boat for each of the seven (7) stations. The sixteen individuals spanned a broad size range to support assessment of the length:mercury relationship and ANCOVA analysis. Upon collection, each fish was tagged with a unique ID that corresponded to the latitude/longitude where it was collected. Physical parameters were collected for each individual fish, which included: weight, total length, fork length, and presence of any abnormalities. Fish samples were stored on ice until returned to the laboratory. Large fish were partially dissected in the field using the following protocol: fish were placed on a cutting board covered with a clean plastic bag where the head, tail, and guts were removed using a clean (laboratory detergent, DI) cleaver. The sex of the fish was noted. The fish were then wrapped in tin foil, with the dull side inward, and double-bagged in zipper-closure bags with other fish from the same location. All equipment was re-cleaned between stations.

At the laboratory, samples were stored in a freezer until they were processed for authorized dissection and analysis.

A depth-integrated water sample was collected at seven (7) stations following MPSL-DFW SOP MPSL-111 Revision 2 using a bucket sampler (SWAMP Clean Water Team SOP 2.1.1.4) modified to

accommodate a trace metal cleaned 4L glass bottle (I-Chem Part # 145-4000) (MPSL-101). A new trace metal cleaned 4L glass bottle, tubing and filter were used for each site. In the thalweg, the bucket sampler with the 4L was lowered to 0.5m from the bottom to a maximum depth of 15m and raised through the water column at a sufficient rate so that the bottle was not completely filled upon retrieval, achieving a depth-integrated sample. Total samples were aliquoted into analyte-specific bottles by pouring. The 4L bottle was agitated between samples to maintain consistency. Filtered samples were collected by attaching a 0.45µm ground water filter to trace metal clean tubing and a peristaltic pump, and aliquoted into the analyte-specific bottle. At each water station, four analytes were collected: total Hg, filtered Hg, total MeHg and filtered MeHg. The following ancillary water samples were collected at each station to help interpretation of mercury data: chlorophyll *a*, DOC and TSS/VSS. DOC samples were acidified upon collection. All samples were stored on wet ice until returned to the laboratory.

At the laboratory, Hg and MeHg samples were acidified. MeHg, DOC and TSS/VSS samples were stored in a refrigerator and chlorophyll *a* samples were stored in a freezer until they were analyzed.

Basic field parameters (temperature, pH, specific conductance, salinity, dissolved oxygen concentration, dissolved oxygen saturation, and turbidity) along with station information (station depth, location, weather, hydromodifications and habitat) were also noted. All collections and sample processing for water and fish followed the Delta RMP QAPP.

1.6 Results

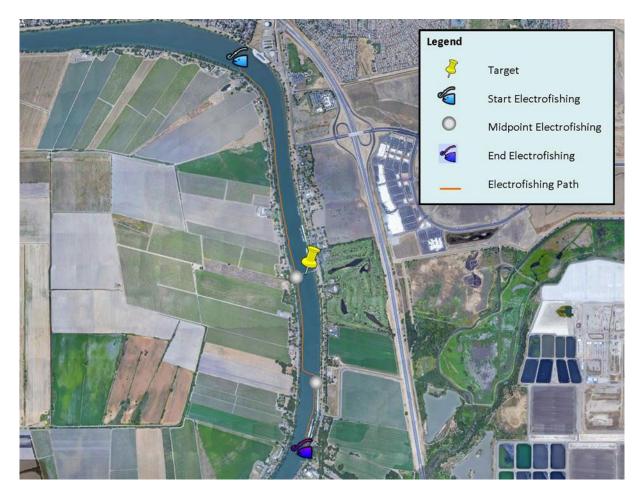
A detailed fish catch, fish total length, descriptions and maps of sample collection for all stations can be found below. Also included are the dates of the depth-integrated water sampling events. Table 1 indicates on which page collection details for each station can be found.

 Table 1. Delta RMP Collection Sites for Year 5 (FY20/21) Trend Work.

Station Code	Station Name	Page Number
510ST1317	Sacramento River at Freeport	<u>6</u>
510ADVLIM	Cache Slough at Liberty Island Mouth	<u>Z</u>
544ADVLM6	Lower Mokelumne River 6	<u>8</u>
544LILPSL	Little Potato Slough	<u>9</u>
207SRD10A	Sacramento River at Mallard Island	<u>10</u>
510ST1666	Sherman Island	<u>11</u>
544MDRBH4	Middle River at Borden Hwy	<u>12</u>
541SJC501	San Joaquin River at Vernalis/Airport	<u>13</u>

Sacramento River at Freeport (510ST1317)

Latitude: 38.45556 Longitude: -121.50189 Collection Method: Electroshock, depth-integrated grab Date(s) of Fish Collection: 09/09/2020 Date(s) of Water Collection: 09/09/2020, 03/10/2021, 04/12/2021 Samplers: April Sjoboen Guimarães, Autumn Bonnema, Gary Ichikawa, Scot Lucas

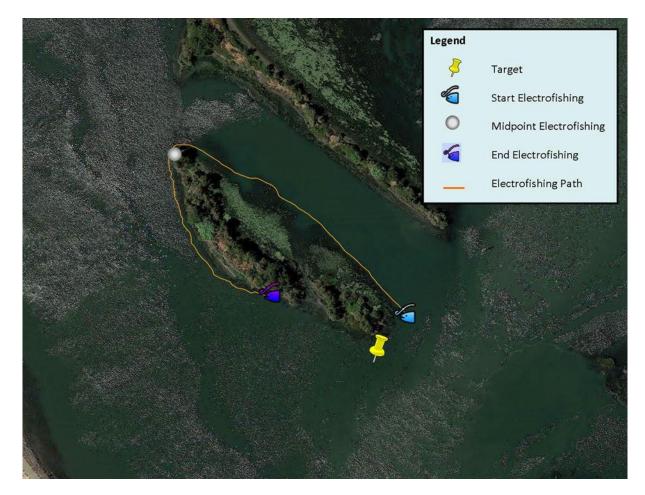


Largemouth Bass, TL (mm)											
202 208 231 252 272 294 320 337 350 352									352		
364 365 371 409 451 486											

Comments: The sampling vessel was launched from Stan's Yolo Marina in Sacramento, CA. Sixteen (16) Largemouth bass were sampled along the transect adjacent to the target station. Spotted bass were also present. All water collection was done in close proximity of the target station where the channel discharge was greatest.

Cache Slough at Liberty Island Mouth (510ADVLIM)

Latitude: 38.24213 Longitude: -121.68539 Collection Method: Electroshock, depth-integrated grab Date(s) of Fish Collection: 09/15/2020 Date(s) of Water Collection: 09/09/2020, 03/10/2021, 04/12/2021 Samplers: April Sjoboen Guimarães, Autumn Bonnema, Chris Beebe

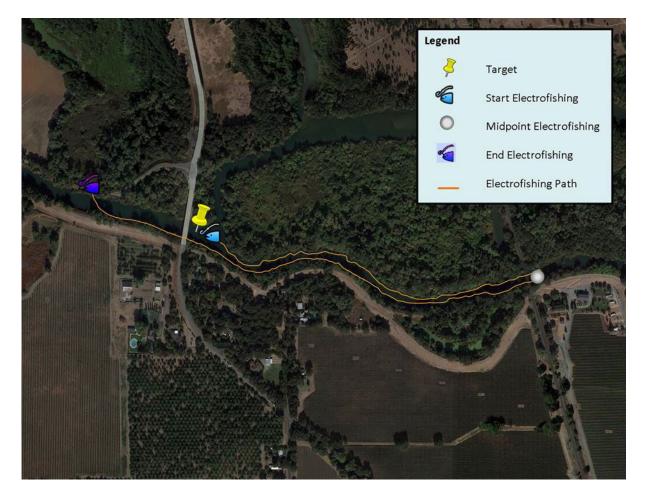


Largemouth Bass, TL (mm)											
219 222 238 284 284 292 312 335 343 346									346		
372											

Comments: The sampling vessel was launched from Arrowhead Marina in Clarksburg, CA. Sixteen (16) Largemouth bass were sampled along the transect adjacent to the target station. All water collection was done in close proximity of the target station where the channel discharge was greatest.

Lower Mokelumne River 6 (544ADVLM6)

Latitude: 38.25542 Longitude: -121.44006 Collection Method: Electroshock, depth-integrated grab Date(s) of Fish Collection: 09/09/2020 Date(s) of Water Collection: 09/08/2020, 03/10/2021, 04/12/2021 Samplers: April Sjoboen Guimarães, Autumn Bonnema, Gary Ichikawa, Scot Lucas



Largemouth Bass, TL (mm)											
216 221 226 257 286 295 307 331 350 355									355		
365 396 400 412 459 470											

Comments: The sampling vessel was launched from New Hope Landing in Walnut Grove, CA. Sixteen (16) Largemouth bass were sampled along the transect adjacent to the target station. All water collection was done in close proximity of the target station where the channel discharge was greatest.

Little Potato Slough (544LILPSL)

Latitude: 38.09627 Longitude: -121.49602 Collection Method: Electroshock, depth-integrated grab Date(s) of Fish Collection: 09/10/2020 Date(s) of Water Collection: 09/08/2020, 03/10/2021, 04/12/2021 Samplers: April Sjoboen Guimarães, Autumn Bonnema, Gary Ichikawa, Scot Lucas



Largemouth Bass, TL (mm)											
205 225 228 255 280 287 320 325 339 344									344		
355	355 376 384 419 436 541										

Comments: The sampling vessel was launched from Tower Park Marina in Lodi, CA. Sixteen (16) Largemouth bass were sampled along the transect adjacent to the target station. All water collection was done in close proximity of the target station where the channel discharge was greatest.

Sacramento River at Mallard Island (207SRD10A)

Latitude: 38.04288 Longitude: -121.92011 Collection Method: Depth-integrated grab Date(s) of Water Collection: 09/09/2020, 03/11/2021, 04/13/2021 Samplers: April Sjoboen Guimarães, Autumn Bonnema



Comments: The sampling vessel was launched from Pittsburg Yacht Club in Pittsburg, CA. All water collection was done in close proximity of the target station where the channel discharge was greatest. The corresponding fish were collected from Sherman Island (510ST1666).

Sherman Island (510ST1666)

Latitude: 38.0431 Longitude: -121.80440 Collection Method: Electroshock Date(s) of Fish Collection: 09/21/2020 Samplers: William Jakl, Chris Beebe



Largemouth Bass, TL (mm)											
213 220 225 256 286 301 325 330 341 361											
371 389 394 462 501 524											

Comments: The sampling vessel was launched from Sherman Island County Park in Rio Vista, CA. Sixteen (16) Largemouth bass were sampled along the transect adjacent to the target station. Target coordinates are from a pre-existing station used as a reference point. This site was chosen to correspond with the water samples from Mallard Island (207SRD10A).

Middle River at Borden Hwy (544MDRBH4)

Latitude: 37.89083 Longitude: -121.48833 Collection Method: Electroshock, depth-integrated grab Date(s) of Fish Collection: 09/08/2020 Date(s) of Water Collection: 09/08/2020, 03/11/2021, 04/13/2021 Samplers: April Sjoboen Guimarães, Autumn Bonnema, Gary Ichikawa, Scot Lucas

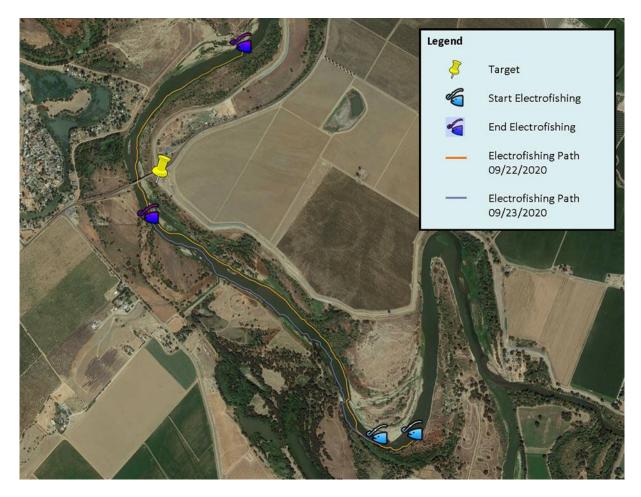


Largemouth Bass, TL (mm)											
209 218 245 251 255 272 308 316 326 367											
370 382 390 420 425 474											

Comments: The sampling vessel was launched from Discovery Bay Yacht Harbor in Discovery Bay, CA. Sixteen (16) Largemouth bass were sampled along the transect adjacent to the target station. All water collection was done in close proximity of the target station where the channel discharge was greatest.

San Joaquin River at Vernalis/Airport (541SJC501)

Latitude: 37.67556 Longitude: -121.26417 Collection Method: Electroshock, depth-integrated grab Date(s) of Fish Collection: 09/22/2020, 09/23/2020 Date(s) of Water Collection: 09/08/2020, 03/11/2021, 04/13/2021 Samplers: April Sjoboen Guimarães, Autumn Bonnema, William Jakl, Chris Beebe



Largemouth Bass, TL (mm)											
204 204 223 257 267 296 329 336 355 361											
363 379 390 426 439 578											

Comments: The electrofishing vessel was launched along the bank on 09/22/2020 and from Two Rivers RV Park in Manteca, CA on 09/23/2020. Sixteen (16) Largemouth bass were sampled along the transect adjacent to the target station. All water collection was done from the bridge as an integrated bucket grab in close proximity of the target station.

1.7 Discussion

A total of seven (7) stations were successfully sampled for fish tissue using a dedicated electrofishing vessel.

Seven (7) stations were successfully sampled for depth-integrated water samples and basic water parameters.

Appendix 2: Cruise Report

Appendix 2 Cruise Report for the Delta Regional Monitoring Program (Delta RMP) Mercury Restoration Monitoring for Black Bass

Year 5 FY20/21 Restoration Work

Sampling Dates: September 14, 2020 – September 16, 2020

Prepared by Marine Pollution Studies Laboratory Staff (<u>MPSL-DFW</u>) at Moss Landing Marine Laboratories; San Jose State University

Introduction

This report describes the sampling activities of the Delta Regional Monitoring Program (DRMP). This sampling effort focuses on monitoring the impacts of wetland restoration projects on accumulation of mercury in black bass in the Delta. Sampling activities included the collection of fish tissue (black bass) and basic field parameters.

Samples were collected by California Department of Fish and Wildlife (CDFW)/Marine Pollution Studies Laboratory (MPSL-DFW) at Moss Landing Marine Laboratories (MLML) staff.

1.0 Cruise Report

1.1 Objectives

The objectives were to collect fish samples from restoration or planned restoration wetlands in the Delta and analyze the samples for mercury concentration. The generated dataset will be used to support answers to DRMP management and assessment questions related to wetland restorations and mercury.

1.2 MPSL Sampling personnel

Wesley Heim April Sjoboen Guimarães Chris Beebe Project Director Research Technician, Crew Lead Research Technician

1.3 Authorization to collect samples

All sampling personnel are MPSL-DFW staff (San Jose State University Foundation and the California Department of Fish and Wildlife) contracted through the State of California Water Board SWAMP Program to conduct the sample collection activities listed herein.

1.4 Station selection

Based upon the recommendations of the Delta RMP Steering Committee and Technical Advisory Committee with representatives from the Central Valley Regional Water Quality Control Board, USEPA, California Department of Water Resources, the State and Federal Contractors Water Agency, and various discharger groups, stations were selected near restoration zones in the Delta.

1.5 Summary of types of samples authorized to be collected

Up to sixteen (16) black bass individuals of the same species were collected using an electrofisher for each of the five (5) stations. The sixteen individuals spanned a broad size range to support assessment of the length:mercury relationship and ANCOVA analysis. Upon collection, each fish collected was tagged with a unique ID that corresponded to the latitude/longitude where it was collected. Physical parameters were collected for each individual fish, which included: weight, total length, fork length, and presence of any abnormalities. Fish samples were stored on ice until returned to the laboratory. Large fish were partially dissected in the field using the following protocol: fish were placed on a cutting board covered with a clean plastic bag where the head, tail, and guts were removed using a clean (laboratory detergent, DI) cleaver. The sex of the fish was noted. The fish were then wrapped in tin foil, with the dull side inward, and double-bagged in zipper-closure bags with other fish from the same location. All equipment was re-cleaned between stations.

At the laboratory, samples were stored in a freezer until they were processed for authorized dissection and analysis.

Basic station information (station depth, location, weather, hydromodifications and habitat) were noted. All collections and sample processing for fish followed the Delta RMP QAPP.

1.6 Results

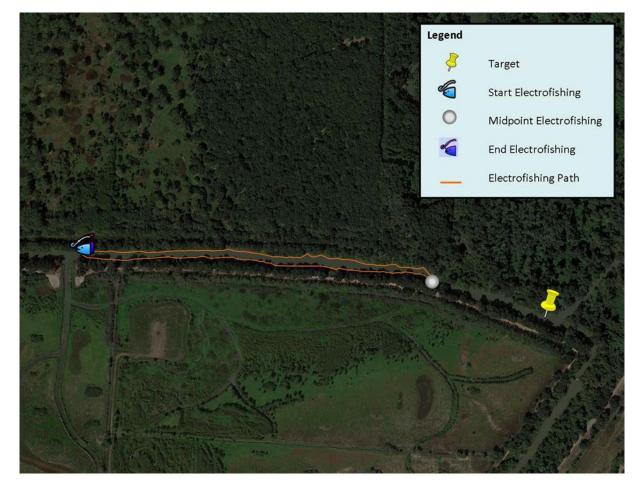
A detailed fish catch, fish total length, descriptions and maps of sample collection for all stations can be found below. Table 1 indicates on which page collection details for each station can be found.

 Table 1. Delta RMP Collection Sites for Year 5 (FY20/21) Restoration Work.

Station Code	Station Name	Page Number
544GZSLWC	Grizzly Slough - Westervelt - Cougar	<u>5</u>
544MCWILT	McCormack-Williamson Tract	<u>6</u>
510ST0787	Lindsey Slough	<u>7</u>
510TDNLHT	Yolo Flyway Farms	<u>8</u>
511XSSLIB	Lookout Slough	<u>9</u>

Grizzly Slough - Westervelt - Cougar (544GZSLWC)

Latitude: 38.25343 Longitude: -121.4069 Collection Method: Electroshock Date(s) of Fish Collection: 09/14/2020 Samplers: April Sjoboen Guimarães, Chris Beebe



	Largemouth Bass, TL (mm)											
205 212 238 249 261 291 325 354 363 385												
385 389 399 460 465 522												

Comments: The sampling vessel was launched from New Hope Landing in Walnut Grove, CA. Sixteen (16) Largemouth bass were sampled along the transect adjacent to the target station.

McCormack-Williamson Tract (544MCWILT)

Latitude: 38.2264 Longitude: -121.49144 Collection Method: Electroshock Date(s) of Fish Collection: 09/14/2020 Samplers: April Sjoboen Guimarães, Chris Beebe



Largemouth Bass, TL (mm)											
210 230 234 272 273 281 302 321 339 341											
369	371	384	409	491	569						

Comments: The sampling vessel was launched from New Hope Landing in Walnut Grove, CA. Sixteen (16) Largemouth bass were sampled along the transect adjacent to the target station.

Lindsey Slough (510ST0787)

Latitude: 38.25843 Longitude: -121.75801 Collection Method: Electroshock Date(s) of Fish Collection: 09/16/2020 Samplers: April Sjoboen Guimarães, Chris Beebe



	Largemouth Bass, TL (mm)											
207 212 240 269 290 300 313 332 336 340												
347	367	393	434	453	557							

Comments: The sampling vessel was launched from Arrowhead Marina in Clarksburg, CA. Sixteen (16) Largemouth bass were sampled along the transect adjacent to the target station.

Yolo Flyway Farms (510TDNLHT)

Latitude: 38.33842 Longitude: -121.64953 Collection Method: Electroshock Date(s) of Fish Collection: 09/15/2020 Samplers: April Sjoboen Guimarães, Chris Beebe



Largemouth Bass, TL (mm)											
234 234 252 279 291 306 345 349 349 372											
391 396 399 418 421 424											

Comments: The sampling vessel was launched from Arrowhead Marina in Clarksburg, CA. Sixteen (16) Largemouth bass were sampled along the transect adjacent to the target station.

Lookout Slough (511XSSLIB)

Latitude: 38.31038 Longitude: -121.69304 Collection Method: Electroshock Date(s) of Fish Collection: 09/16/2020 Samplers: April Sjoboen Guimarães, Chris Beebe



Largemouth Bass, TL (mm)											
199 202 216 260 273 296 317 339 342 344											
345 357 373 411 414 444											

Comments: The sampling vessel was launched from Arrowhead Marina in Clarksburg, CA. Sixteen (16) Largemouth bass were sampled along the transect adjacent to the target station.

1.7 Discussion

A total of five (5) stations were successfully sampled for fish tissue using a dedicated electrofishing vessel.

APPENDIX II – MICROCYSTIS STUDY REPORT



Delta RMP Annual Report for FY 20-21 February 1, 2022

Mapping benthic overwintering Microcystis sp. within the Sacramento-San Joaquin Delta

Ellen P. Preece¹, Timothy G. Otten², Janis Cooke³

¹Robertson-Bryan, Inc. 3100 Zinfandel Drive, St 300. Rancho Cordova, CA

²Bend Genetics, LLC. 87 Scripps Drive St 301. Sacramento, CA

³ Central Valley Regional Water Quality Control Board, 11020 Sun Center Drive, St 200. Rancho Cordova, CA

Introduction

Cyanobacteria harmful algal blooms (CHABs) have become a persistent seasonal problem throughout much of the San Francisco Bay (Bay)/Sacramento-San Joaquin Delta (Delta) since they were first documented in 1999 (Lehman et al. 2005). The Delta cyanobacteria community is generally dominated by *Microcystis*, but in high flow wet years *Aphanizomenon* and *Dolichospermum* also occasionally form blooms (Minoi 2011, Lehman et al. 2017, Kurobe et al. 2018). To date, the most frequently detected class of cyanotoxins in the Delta are microcystins with toxicity primarily attributed to *Microcystis* (Otten et al. 2017). *Microcystis* is widespread throughout much of the Delta. It presents atypically, instead of the paint-like scum usually associated with *Microcystis* blooms, the cells within the Delta tend to form large (up to 1 cm across) flakes that are dispersed throughout the photic zone. Dense surface scums tend to only be found in stagnant areas with little to no tidal velocity.

Several well studied environmental factors are recognized to contribute to *Microcystis* dominance within the Delta. First, phosphorus and nitrogen that fuel CHABs are commonly replete in the Delta (Jassby et al. 2008, Lehman et al. 2017). Second, the regional temperature threshold of ~19°C that promotes *Microcystis* growth in the Delta is typically exceeded by June (Lehman et al. 2017). Peak CHAB abundance occurs from July to September when water temperatures exceed 23°C (Lehman et al. 2013, CCHAB portal 2021). Third, there is sufficient water column irradiance and clarity to trigger the initial vertical migration of over-wintering *Microcystis* cells from bottom sediments (Lehman et al. 2013). Fourth, salinity in many areas of the Delta is below the 10 ppt threshold that enabbales *Microcystis* growth (Preece et al. 2017). Although *Microcystis* has been found at salinities up to 18 ppt in the Delta, it is likely to be stressed and not actively multiplying under these conditions (Lehman et al. 2005). Finally, there are numerous locations throughout the Delta that have long hydraulic residence times, low tidal or riverine velocity, and little water exchange with surrounding areas.

Although the relationship of these environmental factors to *Microcystis* presence are generally well understood (Lehman et al. 2013, 2017), another factor believed to be of critical importance revolves around the dynamics of overwintering benthic cells. Blooms that disappear from the water column are subject to a range of fates, including physical export, death or dormancy. Dormant cells will enter a vegetative state and sink out of the water column and into to the sediment. Overwintering vegetative *Microcystis* colonies remain photosynthetically active and reenter the water column through active resuspension when environmental factors provide favorable growth conditions or through passive wind-induce resuspension (Verspagen et al.

2005). Once established in a system, this overwintering strategy allows *Microcystis* cells to form recurring, seasonal blooms (Cai et al. 2021).

The *Microcystis* overwintering strategy was exemplified in Copco Reservoir (Klamath River; Northern California) where metagenomic analysis of *Microcystis* DNA from water and sediment samples revealed the presence of multiple *Microcystis* strains that exhibit boom and bust population cycles. There were generally only one or two strains present in the water column at any given time; however, all strains were detectable in the sediments (Otten 2016). When one strain was supplanted by another, the deposition of the receding strains to the sediment significantly augmented the standing stock of cells. Similar patterns have been shown to occur in Lake Erie where the recruitment of benthic overwintering *Microcystis* cells are at least partially responsible for initiating summer blooms (Kitchens et al. 2018). This recruitment cycle is likely a universal phenomenon, but the dynamics of overwintering—including loss rates—and its contribution to subsequent blooms has not been well studied in the Delta.

While Microcystis is recognized as a globally important CHAB genus capable of forming prolific blooms, it actually has a slower growth rate than most eukaryotic phytoplankton (Harke et al. 2016). Further, it will only outcompete eukaryotic phytoplankton if it is able to maintain its position near the air-water interface. Therefore, it grows best in warm, hydrologically stagnant waters, even though it can survive for a period of time in cool and turbulent flowing waters (Otten et al. 2015). In riverine ecosystems Microcystis cells likely exhibit low-to-no growth due to the high mixing and short residence times (Paerl and Otten 2013). Indeed, the Sacramento River which generates a majority of the Delta outflow (Lehman et al. 2020) has fewer CHAB reports than areas of the Delta that have lower flow velocity and turbulence such as the flooded islands (e.g., Mildred Island and Franks Tract), smaller rivers (e.g., Old River and Mokelumne River), and backwater sloughs (e.g., Discovery Bay, Stockton Waterfront, and Windmill Cove) (CCHAB Network 2021). These slower moving portions of the Delta tend to experience more frequent and persistent blooms than other Delta habitats (Otten et al. 2017, CCHAB Network 2021). Thus, it is possible that most *Microcystis* biomass observed throughout the Delta may originate from only a few key locations where site specific residence times are long and the Microcystis seedstock is most pronounced.

With no obvious upstream sources for *Microcystis* to enter into the Delta, the most likely source of summer blooms within the system is that they originate primarily from overwintering *Microcystis* seedstocks that recruit to the water column when conditions are favorable. Since only a fraction of the total benthic cells recruit to the surface, the expectation is that areas with higher concentrations of resting cells will recruit higher numbers of cells to the water column, and as a result, blooms will be more intense in these locations. Thus, we hypothesized that overwintering benthic *Microcystis* in a few specific Delta locations may be the primary sources for *Microcystis* blooms observed in distant— but hydrologically connected—locations throughout the Delta. At flooded island sites, we expected that dormant *Microcystis* cells that settled out of the water column in the late fall would eventually be purged from the area over the course of the winter due to these sites being shallow, well mixed, and continually flushed. By the following spring, we anticipated that there would be little *Microcystis* biomass remaining in sediments of the flooded island sites to serve as bloom inoculum, even though these sites are regarded as CHAB hotspots during the summer. Conversely, we anticipated that backwater sloughs would carry higher seed stocks into the spring and that these sites would serve as the

primary sources of *Microcystis* in the lower and central Delta. To our knowledge, this is the first study focused on elucidating the spatiotemporal origins of *Microcystis* blooms within the Delta.

Methods

Site Description

The Bay-Delta is the largest estuarine system on the U.S. Pacific Coast and contains the only inland Delta in the world. The Delta is formed by the Sacramento River that flows from the north where it converges with the San Joaquin River that flows from the south. These two rivers and multiple tributaries drain 40% of California's surface water, including the agriculturally-rich Central Valley and snowmelt from the Sierra Nevadas. From the convergence—near the town of Antioch, the Delta flows to the Pacific Ocean through Suisun Bay, San Pablo Bay, and San Francisco Bay (i.e., Central Bay).

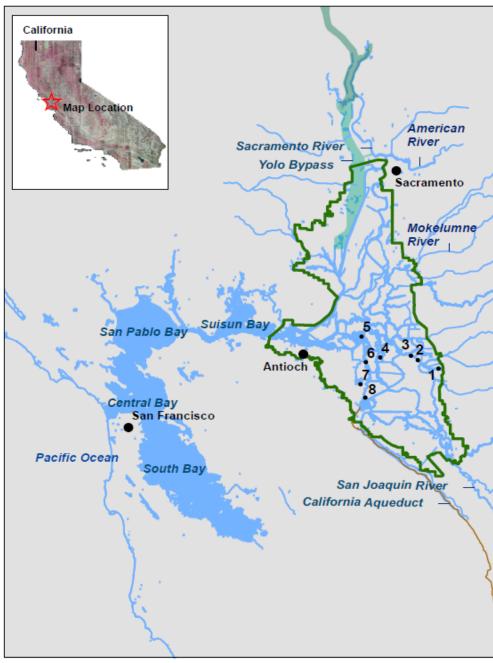
The Delta is comprised of shallow flooded islands, backwater sloughs, flowing rivers, managed canals, and static peripheral areas. Water depth throughout the Delta varies based on location. Depth in the flooded islands varies from two to six meters, while peripheral areas and canals range in depth from two to eight meters, and the San Joaquin River Channel has depths up to 13 meters. Tidal influence also varies based on location. Tidal velocities can range from a peak of 60 cm s^{-1} or more at locations such at Franks Tract and tends to decrease in locations more distal from the ocean. Delta flows exhibit strong seasonal variations due to the region's pronounced wet (November–April) and dry (May–October) seasons.

The hydrologically complex Delta is greatly influenced by the State and Federal Water Projects that draw water into a conveyance network (i.e., California aqueduct) that transfers water to the south for agricultural, residential, municipal and industrial use. For example, when water is drawn from the southern Delta through the conveyance network it can cause net negative streamflow in some locations of the Delta (Lehman et al. 2008, 2015). Another example is the operation of the Delta cross channel gates and temporary barriers that are used to alter flow pathways and to improve water quality and water supply reliability (Kimmerer et al. 2019).

Eight sites were sampled for this study. All of the sites are known to be impacted by CHABs and included: Stockton waterfront, Discovery Bay, Rancho del Rio, Buckley Cove, San Joaquin River near Windmill Cove, Frank's Tract, Mildred Island, and Old River @ Kings Island (**Figure 1**). We predicted that three of these sites (Discovery Bay, Stockton Waterfront, and Windmill Cove) would be where Delta *Microcystis* blooms would originate and where the densest blooms would occur based on past monitoring observations (CCHAB Network 2021). The remaining sites were expected to have low *Microcystis* seed stocks in the spring and were anticipated to experience delayed onset of detectable *Microcystis* within the water column.

Field Sampling

Field sampling began in November 2020 and concluded in July 2021. Sediment samples were collected from each site in November to establish the initial concentration of benthic *Microcystis* cells heading into winter. The November sampling event was chosen to coincide with the end of the bloom season when *Microcystis* cells were generally absent from the water. Samples were



collected the following spring to assess the benthic population prior to peak flushing from snowmelt. April was chosen with the expectation that there would be significant losses to the seed banks at each site due to death and physical export from resuspension and flushing during the wet winter months. Cells that still remained were expected to serve as the upcoming summer's bloom source.

Lateral transects (five sediment samples per sampling site) were conducted to assess site heterogeneity. Sediment samples were collected using a gravity sediment corer (NLA corer, Aquatic Research

Figure 1. Map of the Sacramento-San Joaquin Delta and San Francisco Estuary. Locations are indicated by number and are as follows: 1. Stockton Waterfront, 2. Buckley Cove, 3. Windmill Cove, 4. Mildred Island, 5. Franks Tract, 6. Old River @ Rancho Del Rio, 7. Discovery Bay, and 8. Old River @ Kings Island

Instruments, <u>http://www.aquaticresearch.com/gravity_slide_hammer_corers.htm</u>) fitted with a polycarbonate barrel. Samplers targeted a depth of 3 to 4 meters to collect sediment samples. However, due to varying site conditions some samples were collected at up to 7 meters depth. Cores were extruded in the field. The top 1 cm of sediment was placed into 50 ml centrifuge tubes and stored on ice during transport to the laboratory.

Based on prior studies in the Delta, June is typically when *Microcystis* is first observed in the water column (Lehman et al. 2017). Thus, four sampling events were conducted (2X in June/July) for water collection to capture entry of *Microcystis* into the water column from the sediment. Water samples were collected approximately 0.5 m below the surface using a pole or sub-surface sampling device (e.g, Kemmerer). Samples were placed into 500 mL amber glass bottles and stored on ice during transport to the laboratory. Water was taken from within a bloom if present, but surface scums were avoided if possible. Water was used for 1) QPCR to screen for total and toxin-producing *Microcystis*, 2) microcystin testing by enzyme linked immunosorbent assay (ELISA), and 3) amplicon metagenomics to identify strains (*work to be completed in summer 2022*).

At each site, dissolved oxygen, temperature, electrical conductivity, pH (YSI ProDSS sonde) and turbidity (Hach 2100Q meter) were measured. Visual observations of site conditions, local weather and the California Department of Water Resources HAB bloom visual index score were recorded during each sampling event.

Laboratory Procedures

Each sediment sample was freeze dried, then total DNA was extracted from a 0.25 g sub-sample (dry weight) using a Qiagen PowerLyzer PowerSoil DNA extraction kit. Sediment samples from the winter and spring transects were analyzed individually. Real-time QPCR was used to quantify total *Microcystis* based on single copy c-phycocyanin genes (*cpcB*; Otten et al. 2015) and total microcystin-producing *Microcystis* based on single copy microcystin synthetase E genes (*mcyE*; Sipari et al. 2010).

Water samples were thoroughly mixed, then concentrated by vacuum filtration onto GF/F glass fiber filters (1.2 μ m, 25 mm dia.) for DNA extraction using a Qiagen PowerLyzer PowerSoil DNA extraction kit. Liquid aliquots (10 mL) were stored frozen for subsequent toxin testing. Water samples were subjected to three rounds of freeze-thawing to lyse the cells, then total microcystins were analyzed by ELISA. In total, there were 17 water samples analyzed by ELISA, and these data were compared with the QPCR gene copy estimates in the water column and used to relate molecular data with OEHHA/EPA public health monitoring action levels for cyanotoxins.

Statistical Analysis and Maps

Statistical analyses were performed with R software (Version 4.1.2). The data from November was log-transformed and its normality and homogeneity of variances were verified prior to statistical treatments with Shapiro-Wilkes and Levene's test. Mean differences in total *Microcystis* cell equivalents at the sampling locations in November were compared across sites using a one-way ANOVA with log-transformed data. Significant differences across locations in November were calculated using a post hoc Tukey test. Since the April data set did not satisfy normality, the differences between total *Microcystis* cell equivalents at the sampling locations were compared using the non-parametric Kruskal-Wallis one-way ANOVA test. Significant differences across locations were calculated using Dunn's post-hoc test with Holm's correction. Two-parameter linear regression was used to explore the relationship between *Microcystis* cell equivalents in the water column to microcystin concentrations in June and July. The significance threshold was set at p<0.05. Maps were generated in ArcMap 10.2.2. *Microcystis* cell equivalents

at each location were distributed into groups based on the ArcGIS quantile classification of November 2020 data.

Results

November 2020 and April 2021 transects showed high heterogeneity at each of the sampling locations suggesting that the cells were not evenly distributed when they settled out of the water column (**Figure 2** and **Figure 3**). Even with the high heterogeneity it was clear that there were significant differences in the mean number of cells across sites in November (**Table 1**). As shown in **Figure 4**, *Microcystis* cell abundances were greatest at Discovery Bay and Windmill Cove followed by a relatively high abundance of cells at Mildred Island and the Stockton Waterfront. In November Discovery Bay, Windmill Cove, Mildred Island, and the Stockton Waterfront had significantly higher concentrations of *Microcystis* cells than Old River @ Rancho Del Rio. Discovery Bay and Windmill Cove also had significant higher concentrations of *Microcystis* cells than Franks Tract and Buckley Cove while Mildred Island had significantly higher concentrations than Franks Tract, but not Buckley Cove.

	Buckley Cove	Discovery Bay	Franks Tract	Mildred Island	Old River @ Rancho Del Rio	Stockton Waterfront	Windmill Cove
Buckley Cove							
Discovery Bay	0.0188						
Franks Tract	0.9985	0.0053					
Mildred Island	0.0546	0.9992	0.0166				
Old River @ Rancho Del Rio	0.3610	0.0001	0.6773	0.0003			
Stockton Waterfront	0.2270	0.8996	0.0847	0.9908	0.0017		
Windmill Cove	0.0162	1.0000	0.0045	0.9983	0.0001	0.8760	

Table 1. Post-hoc Tukey adjusted p-values to compare significance differences in total *Microcystis* across sites in November. Bold values indicate significant differences at p<0.05

Total *Microcystis* cell equivalents in the surface sediment decreased from November to April by at least two orders of magnitude across the sites. Although cell equivalents were considerably lower in April, the highest spring abundances were observed at Discovery Bay and Windmill Cove (Figure 4). These locations had significantly higher cell equivalents than Old River @ Rancho Del Rio and Franks Tract (**Table 2**). Mildred Island also had significantly higher cell equivalents than Old River @ Rancho Del Rio (Table 2). There were no other statistical differences in *Microcystis* cell equivalents between sites in April.

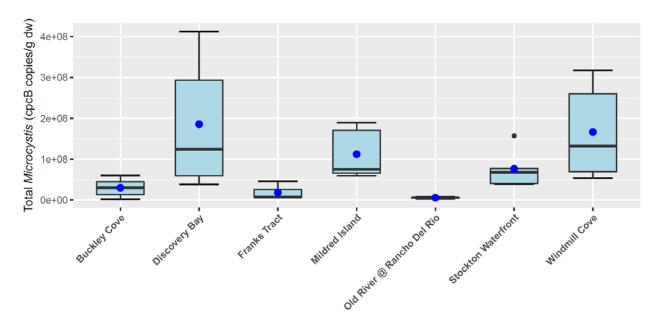


Figure 2. Boxplot showing variability in total *Microcystis* cell equivalents per gram dry sediment across transects (n = 5 replicates/site) in November 2020. Dark blue dots indicate the average *Microcystis* cell equivalents as measured by QPCR at each site.

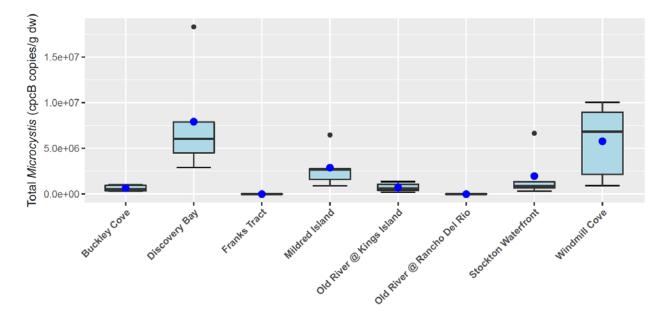


Figure 3. Boxplot showing variability in total *Microcystis* cell equivalents per gram dry sediment across transects (n = 5 replicates/site) in April 2021. Dark blue dots indicate the average *Microcystis* cell equivalents as measured by QPCR at each site.

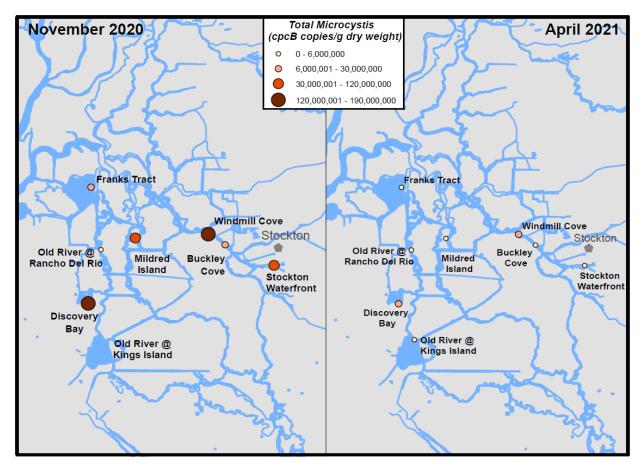


Figure 4. Total *Microcystis* cell equivalents per gram dry sediment measured in November 2020 and April 2021 (n=5 sediment samples per site and date). Old River @ Kings Island was not sampled in November 2020.

Table 2. Post-hoc Dunn's test with Holm's correction adjusted p-values to compare significance differences in total
Microcystis across sites in April 2021. Bold values indicate significant differences at p>0.05

	Buckley Cove	Discovery Bay	Franks Tract	Mildred Island	Old River @ Kings Island	Old River @ Rancho Del Rio	Stockton Waterfront	Windmill Cove
Buckley Cove								
Discovery Bay	0.4508							
Franks Tract	1.0000	0.0020						
Mildred Island	1.0000	1.0000	0.0519					
Old River @ Kings Island	1.0000	0.5546	1.0000	1.0000				
Old River @ Rancho Del Rio	1.0000	0.0020	1.0000	0.0497	1.0000			
Stockton Waterfront	1.0000	1.0000	0.5522	1.0000	1.0000	0.5259		
Windmill Cove	0.7858	1.0000	0.0064	1.0000	1.0000	0.0061	1.0000	

In November the proportion of toxigenic *Microcystis* to total *Microcystis* in the sediment ranged from approximately 6-14% depending on the sampling location except for Old River @ Rancho Del Rio (**Figure 5**). At this site toxic cells comprised approximately 60% of the *Microcystis* population. By April the proportion of toxic cells decreased even further. At Mildred Island there were no toxic cells present and at Buckley Cove the toxic cells comprised less than 0.04% of the total resting cells. At the other locations toxic cells comprised 3% or less of the total resting cells (**Figure 6**).

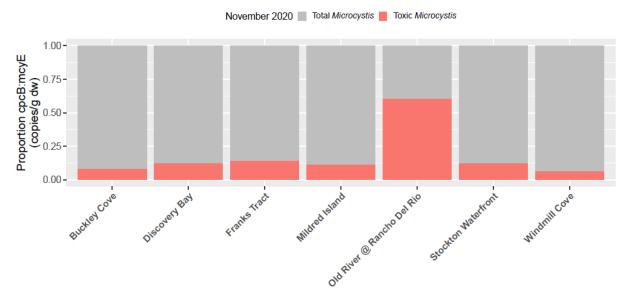


Figure 5. Proportion of toxigenic *Microcystis* (*mcyE*-possessing) relative to total *Microcystis* (*cpcB*-possessing) at each of the eight sampling sites in November 2020. Old River @ Kings Island was not sampled in November 2020.

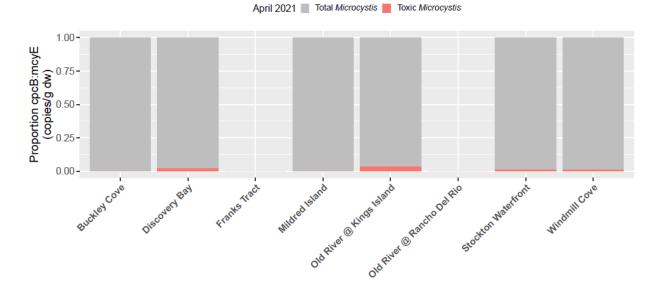


Figure 6. Proportion of toxic *Microcystis* cells as measured by mcyE to total *Microcystis* cells as measured by cpcB at each of the eight sampling sites in April 2021. No *Microcystis* cells were detected at Old River @ Rancho Del Rio or Franks Tract.

The relationship between total *Microcystis* cell equivalents in the water samples and total microcystin concentration in June and July was weak and not significant (**Figure 7**, p=0.288, n=15). Similarly, the relationship between toxic *Microcystis* cell equivalents in the water samples and total microcystin concentrations in June and July was also weak and not significant (**Figure 8**, p=0.944, n=15). No cpcB copies or microcystin were detected in two of the water samples, both from Franks Tract.

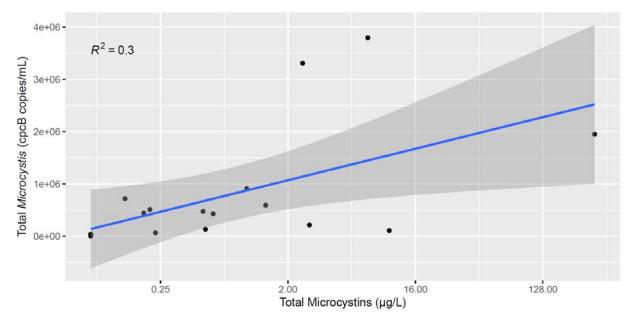


Figure 7. Linear regression between total *Microcystis* cell equivalents in the water and total microcystin concentrations in the water at the eight sampling sites in June and July. The shaded dark grey band represents the 95% confidence interval.

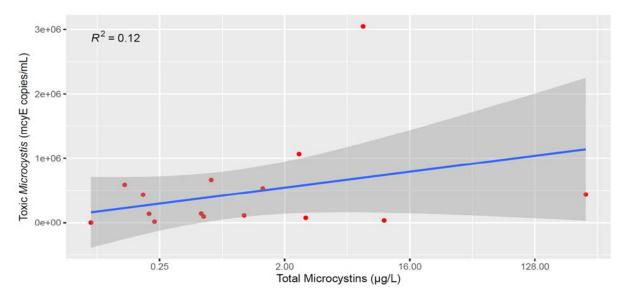


Figure 8. Linear regression between toxic *Microcystis* cell equivalents in the water and total microcystin concentrations in the water at the eight sampling sites in June and July. The shaded dark grey band represents the 95% confidence interval.

The highest microcystin measured in June and July was 298 μ g/L at Windmill Cove. This exceeded the OEHHA/EPA public health monitoring danger action level (i.e., 20 μ g/L; Figure 7). Interestingly, the locations with the next highest microcystin concentrations were where total *Microcystis* cell equivalents were non-detect or very low in April 2021. Microcystin at Buckley Cove on June 29, 2021 was 7.4 μ g/L while microcystin at Old River @ Rancho Del Rio on July 15, 2021 was 10.5 μ g/L. Both of these locations exceeded the warning action level of 6.0 μ g/L. Notably, Discovery Bay, Mildred Island, and the Stockton Waterfront had microcystin concentrations above the caution action level of 0.8 μ g/L, but were below the warning action level in June and July.

Based on visual observations of the water surface from the boat during the first June sampling event (i.e., June 4 – 6, 2021), *Microcystis* abundances were greatest at Discovery Bay and Stockton waterfront. By June 6, 2021, *Microcystis* cell abundance at Discovery Bay and Stockton waterfront were dense throughout the water column, but there was no surface scum present. *Microcystis* and other cyanobacteria cells were moderately abundant in the water column at Buckley Cove, Windmill Cove, and Old River @ Rancho Del Rio. No *Microcystis* was observed in the water column at Mildred Island or Franks Tract during the first sampling event, but were present by the second sampling event in late June. Visual observation scores using an index developed by Department of Water Resources provide semi-qualitative comparisons of abundance (**Table 3**).

Cyanobacteria Visual Scores ¹	June Event 1	June Event 2	July Event 1	July Event 2	
Buckley Cove	3	3	3	4	
Discovery Bay	4	4	3	3	
Franks Tract	1	2-3	3	4	
Mildred Island	1	3	3	3	
Old River @ Kings Island	1	3	3	3	
Old River @ Rancho Del Rio	2	3	3	3	
Stockton Waterfront	4	4	4	4-5	
Windmill Cove	3	3	4	4	
¹ Department of Water Resources cyanobacteria visual index score guidelines : 1 = absent; 2 = low abundance with widely scattered colonies; 3 = medium abundance with adjacent colonies; 4 = high abundance with contiguous colonies; 5 = very high					

Table 3. Visual cyanobacteria observations reported using California Department of Water Resources *Microcystis* scoring index

No cyanobacteria were observed in the water column during the April 2021 collection event. However, temperatures in the surface water and near the sediment were generally at or above the regional temperature threshold of 19°C that promotes *Microcystis* growth in the Delta (Lehman et al. 2017). By the first June event when *Microcystis* flakes were observed in the water column temperatures exceeded 21°C at all sampling sites (**Table 4**).

abundance with contiguous colonies forming mats or scum.

Sites	Spring Sediment Event 4/28/ thru 5/6 2021	Spring Sediment Event 4/28/ thru 5/6 2021	June 2021 - Bloom Event 1	June 2021 - Bloom Event 2
	surface water (°C)	water at depth 10 ft (or bottom if less than 10 ft) (°C)	surface water (°C)	surface water (°C)
Buckley Cove	21	19.9	24.7	26.5
Discovery Bay	19.5	18.6	22.9	27.8
Franks Tract	19.4	not recorded	22.6	23.9
Mildred Island	21	not recorded	23.6	25.0
Old River @ Kings Island	18.4	not recorded	21.1	24.7
Old River @ Rancho del Rio	19.5	19.3	22.3	25.2
Stockton waterfront	21.6	23	25	26.7
Windmill Cove	19.9	19.5	23.5	26.7

Table 4. Water temperature in April and June 2021 at the eight sampling sites.

Discussion

As expected, there was a large depletion of *Microcystis* cells at each of the study locations over the course of winter. These findings are consistent with other studies that have found that *Microcystis* colonies in the benthos reach a maximum in late fall and then decline throughout winter and spring (e.g. Tsujimaura et al. 2000, Brunberg and Blomqvist 2002, Verspagen et al. 2005, Kitchens et al. 2018). Studies of overwintering *Microcystis* cells have typically occurred in lakes where loss rates are attributed to mortality or transport to deeper portions of the lake (Burnberg and Blomqvist 2002, Kitchens et al. 2018). In our study it is not possible to conclude what percentage of the overwintering cells died or were transported to other location in the Delta during the winter period.

In April, there were no *Microcystis* cells detected in the sediment at Franks Tract or Old River @ Rancho Del Rio. Conversely, there was a relatively high concentration of *Microcystis* cells that remained in the sediment of Mildred Island in April. Both Franks Tract and Mildred Island are characterized as being more lacustrine-like areas of the Delta, but the levees surrounding Franks Tract are perforated with multiple breaches while the levee surrounding Mildred Island is only breached in a few locations (Lucas et al. 2002, Young et al. 2018). Although both locations are subject to winter flushing, the lower potential for hydrodynamic exchange at Mildred Island may explain the persistence of overwintering cells at this location relative to Franks Tract. Another explanation for cell persistence at Mildred Island in April may be related to the 2021 drought. The drought was severe enough to warrant construction of a temporary emergency drought barrier on the West False River to keep salinity from entering the freshwater portion of the Delta. Since droughts in the Delta are characterized by lower outflows, longer residence times, and less flushing (Lehman et al. 2020), it is plausible that the amount of overwintering cells at Mildred Island would be lower or non-detectable by April in wetter water years. This possibility will be further explored in year two of this study (*to be completed in summer 2022*).

A substantial *Microcystis* bloom was observed at Franks Tract in the summer of 2021, despite the absence of overwintering cells in April. There are several possible explanations for the source of this bloom. While not highly probable, it could have been seeded by an overwintering pelagic population (Tian et al. 2021). The November and April sampling dates were focused solely on the sediment, so it is not possible to ascertain if any overwintering pelagic cells were present at this location or at other sampling locations. Alternatively, the shallow margins of Franks Tract, which also were not sampled in this study, may also harbor benthic overwintering *Microcystis* cells. The most likely scenario is that Franks Tract was seeded with *Microcystis* cells in the water at Franks Tract in early June which suggests there was little to no *Microcystis* colonies in the area to seed the initial bloom. This possibility will be further probed once the metagenomic portion of the study is completed.

Another unexpected finding was the lower concentration of *Microcystis* cells at the Stockton Waterfront compared to those found at Discovery Bay. Both the Stockton Waterfront and Discovery Bay are notorious in the Delta for producing cyanobacteria scums and associated microcystins. The relatively low concentrations of benthic cells at the Stockton Waterfront compared to Discovery Bay may be an artifact of the former's benthic substrate. Blooms were characterized as severe in the water column near Stockton sediment collection site (i.e., near the Moreli Park boat ramp) which is located in the western portion of the waterway. However, there is substantial boat traffic that causes bottom disturbance at this location and there is hydrologic connectivity with the San Joaquin River. Indeed, samplers described the samples collected at this site as less depositional (i.e., hard bottomed) than the other sampling locations. Ongoing work for this study is focused on the eastern portion of the Waterfront to assess if that area has more persistent seedstock than the western portion of the waterway.

Microcystis populations are composed of a mix of toxic and non-toxic subpopulations (e.g., Rinta-Kanto et al. 2005, Otten et al. 2017). While various environmental factors have been linked to the proportion of toxic *Microcystis* in the water column (Park et al. 2017), less is known about factors that influence the proportion of toxic *Microcystis* in the sediment. The proportion of toxic cells in the sediment is likely to closely reflect antecedent bloom dynamics in the water column since there is no reason to believe toxic or nontoxic cells have higher survival rates in the sediment. In general, the proportion of toxic to total *Microcystis* cells in the sediment ranged from 6 to 14% in November and less than 3% in April. The exception to this was the Old River @ Rancho Del Rio where toxic cells comprised over 60% of the total cells in November.

Although it is unknown why Old River @ Rancho Del Rio had a higher ratio of toxic cells in the sediment compared to the other locations, it is likely that the most recent *Microcystis* populations in the water column were primarily comprised of toxic strains. Importantly, the contribution of toxigenic *Microcystis* to the greater Delta may be minimal considering that this location had the lowest amount of *Microcystis* observed across all sites. Our findings are comparable to the proportions of toxic strains have that have been reported in other studies where the proportion of toxic strains was <1% to 68% at 15 study sites in Lake Erie in November (Kitchens et al. 2018), 0 to 30% in Copco Reservoir, California in February (Otten et al. 2016), and 5.3 to 98% in Hoedong Reservoir, Korea during the October to December sampling period (Park et al. 2017). Further studies are needed to elucidate how the proportion of toxic *Microcystis* cells change in the sediment throughout the course of a bloom season and the contribution of these cells to the

Microcystis blooms in the water column. Importantly, the proportion of toxic *Microcystis* in the sediment does not necessarily reflect the potential toxigenicity that can occur within the water column since each strain will recruit according to its own physiological requirements and on its own schedule.

We observed a weak, but positive, relationship between microcystin and toxic *Microcystis* (as measured by *mcyE* genes) in the surface water in June and July. During this study toxigenic *Microcystis* was only a minor contributor to the total *Microcystis* community. However, when a similar approach was used to characterize *Microcystis* over a wider time period (2011–2012), toxigenic *Microcystis* was observed to comprise between 18–46% of the total *Microcystis* community with the Delta and peak toxigenicity tended to occur later in the summer (August-September) than was investigated here (Otten et al. 2017). Thus, our results may be related to the relatively low sample size (n=15) or the timing of our study. Samples were collected through November 2021 and they will be analyzed as part of the larger study which incorporates the amplicon metagenomic component. As such, these relationships will be revisited with the expanded time series.

Conclusions

A major impetus for this study was to determine if only a few, localized areas exhibited an outsized influence on *Microcystis* bloom formation throughout the Delta. Further research is necessary before this question can be fully addressed, but findings from our study suggest that there are likely multiple locations in the Delta that maintain high *Microcystis* seed stocks into the spring that could initiate blooms. Results from phase one of the study reported herein show that benthic resting cells are highest at Discovery Bay, followed by Windmill Cove, Stockton Waterfront and Mildred Island. These locations retained a relatively large number of overwintering cells into April and therefore have the most potential to promote *Microcystis* in the water column in summer 2021. However, Old River @ Rancho Del Rio, Franks Tract, and Buckley Cove had little-to-no standing stock to serve as bloom inoculum by April, suggesting that *Microcystis* at these locations are likely seeded from other upstream locations in the Delta.

The second phase of this project is currently underway and will be used to further elucidate the connection between blooms throughout the Delta once sample collection is completed in spring 2022. Amplicon-based metagenomics (16S-23S rRNA locus) using barcoded primers and the Illumina MiSeq platform will be utilized to characterize the cyanobacterial community and their relative abundance in the water and sediment samples collected from each site/date (Otten et al. 2017) over a two-year period. This metagenomic approach will provide us with a snapshot view of the relative abundance of each *Microcystis* strain that serves as a molecular fingerprint. We will compare these fingerprints to samples collected at the different locations and between the sediment and water samples in order to assess if *Microcystis* cells observed in the water column originate from local sediment stocks or more distal sources that had the highest resting seedstock (i.e., Discovery Bay, Windmill Cove, Mildred Island, and the Stockton Waterfront). Distinct profiles generated from each site is that the water column profile should match the benthic profile. If the water column profile is distinct from the benthic profile, then there is reason to believe that those cells originate from distal sources.

We are also repeating the November and April portions of the study to further investigate if there are annual differences in concentrations of overwintering cells at the study locations. December 2021 has already set a record for snowfall in the Sierra, so the spring flushing in 2022 may be more pronounced than was observed in 2021. Therefore, it may be possible to compare how different water year types affect the standing stock of cells. Finally, once final results are obtained and analyzed we will provide recommendations for future work on this topic with the ultimate goal of helping managers better understand locations that should receive targeted mitigation.

Acknowledgements

This project could not have been completed without the SEP funds, staff and boat time provided in-kind by the Central Valley Water Board. SEP funds provided the resources to analyze all of the samples reported within this document. Further, the funds provided the opportunity to collect and preserve all the samples for the amplicon metagenomics that are slated to be analyzed in early 2022.

References

American Public Health Association (APHA). 1998. Standard Methods for the Examination of Water and Wastewater. Twentieth edition. APHA.

Brunberg, A-K., Blomqvist, P. 2002. Benthic overwintering of *Microcystis* colonies under different environmental conditions. Journal of Plankton Research. 24(11):1247-1252.

Cai, P., Cai, Q, He, F., Huang, Y., Tian, C., Zingqiang, W., Wang, C., Xiao, B. 2021. Flexibility of *Microcystis* overwintering strategy in response to winter temperatures. Microorganisms. 9, 2278. https://doi.org/10.3390/microorganisms9112278.

California Cyanobacteria and Harmful Algal Bloom Network of the California Water Quality Monitoring Council. 2021. *HAB Incident Reports Map*. Available: https://mywaterquality.ca.gov/ habs/where/freshwater_events.html. Accessed: September 12, 2021.

Harke, M.J., Steffen, M.M., Gobler, C.J., Otten, T.G., Wilhelm, S.W., Wood, S.A., Paerl, H.W. 2016. A review of the global ecology, genomics, and biogeography of the toxic cyanobacterium, *Microcystis* spp. Harmful Algae. 54:4-20.

Jassby, A. D. 2008. Phytoplankton in the Upper San Francisco Estuary: Recent Biomass Trends, Their Causes and Their Trophic Significance. San Francisco Estuary and Watershed Science 6(1):Article 2.

Kitchens, C.M., Johengen, T.H., Davis, T.W. 2018. Establishing spatial and temporal patterns in *Microcystis* sediment seed stock viability and their relationship to subsequent bloom development in Western Lake Erie. PlosOne ps://doi.org/10.1371/journal.pone.0206821

Kurobe, T., Lehman, P.W., Hammock, B.G., Bolotaolo, M.B., Lesmeister, S., Teh, S.J. 2018. Biodiversity of cyanobacteria and other aquatic microorganisms across a freshwater to brackish water gradient determined by shotgun metagenomic sequencing analysis in the San Francisco Estuary, USA. PLoS ONE 13(9):e0203953. <u>https://doi.org/10.1371/journal.pone.0203953</u>

Lehman, P.W., Boyer, G., Hall, C., Waller, S., Gehrts, K. 2005. Distribution and toxicity of a new colonial Microcystis aeruginosa bloom in the San Francisco Estuary, California.

Lehman, P.W., Boyer, G., Satchwell, M., Waller, S. 2008. The influence of environmental conditions on the seasonal variation of Microcystis cell density and microcystins concentration in San Francisco Estuary. Hydrobiologia 600:187-204.

Lehman, P.W., Marr, K., Boyer, G.L., Acuna, S., Teh, S.J. 2013. Long-term trends and causal factors associated with Microcystis abundance and toxicity in San Francisco Estuary and implication for climate change impacts. Hydrobiologia 718:141-158.

Lehman, P.W., Kurobe, T., Lesmeister, S., Baxa, D., Tung, A., Teh, S.J. 2017. Impacts of the 2014 severe drought on the *Microcystis* bloom in San Francisco Estuary. Harmful Algae. 63:94–108.

Lehman, P. W., T. Kurobe, and S. J. Teh. 2020. Impact of Extreme Wet and Dry Years on the Persistence of *Microcystis* Harmful Algal Blooms in San Francisco Estuary. Quaternary International 2020. DOI:<u>10.1016/j.quaint.2019.12.003</u>.

Lucas, L.V., J.E. Cloern, J.K. Thompson, and N.E. Monsen. 2002. Functional variability of habitats within the Sacramento-San Joaquin Delta: restoration implications. Ecological Applications 12: 1528–1547.

Mioni, C., R. Kudela and D. Baxa. 2012. *Harmful Cyanobacteria Blooms and Their Toxins in Clear Lake and the Sacramento-San Joaquin Delta (California)*. Surface Water Ambient Monitoring Program Report 10-058-150. Prepared for the Central Valley Regional Water Quality Control Board, Rancho Cordova, CA.

Otten, T.G., Crosswell, J.R., Mackey, S., Dreher, T.W. 2015. Application of molecular tools for microbial source tracking and public health risk assessment of a Microcystis bloom traversing 300 km of the Klamath River. Harmful Algae 46:71-81.

Otten, T.G. 2016. Assessment of benthic, overwintering *Microcystis* in Copco Reservoir and its implication on summer bloom recruitment. *Technical Report*.

Otten, T.G., Paerl, H.W., Dreher, T.W., Kimmerer, W.J., Parker, A.E. 2017. The molecular ecology of Microcystis sp. blooms in the San Francisco Estuary. Environmental Microbiology 19(9):3619-3637.

Preece, E. P., M. Bryan, F. J. Hardy, and B. C. Moore. 2017. A Review of Microcystin Detections in Estuarine and Marine Waters: Environmental Implications and Human Health Risk. *Harmful Algae* 61:31–45.

Paerl, H.W., Otten, T.G. 2013. Harmful cyanobacterial blooms: causes, consequences and controls. Microbial Ecology. 65:995-1010.

Park, B.S., Li, Z., Kang, Y-H, Shin, H.H., Joo, J-H, Han, M-S. 2017. Distinct bloom dynamics of toxic and non-toxic *Microcystis* (cyanobacteria) subpopulation in Hoedong Reservoir (Korea). Microbial Ecology. 75:163-173.

Rinta-Kanto, J.M., Ouellette, A.J.A., Boyer, G.L., Twiss, M.R., Bridgeman, T.B., Wilhelm, S.W. 2005. Quantification of toxic *Microcystis* spp. during the 2003 and 2004 blooms in western Lake Erie using quantitative real-time PCR. Environmental Science and Technology. 39(11):4198-4205.

Sipari, H., Rantala-Ylinen, A., Jokela, J., Oksanen, I., Sivonen, K. 2010. Development of a Chip Assay and Quantitative PCR for Detecting Microcystin Synthetase E Gene Expression. Applied and Environmental Microbiology. 76(12):3797-3805.

Tian, H., Junjie, J., Chen, B., Lefebrve, D.D., Lougheed, S.C. 2021. Depth-Dependent spatiotemporal dynamics of overwintering pelagic *Microcystis* in a temperate water body. Microorganisms. 9(1718):1-17. <u>https://doi.org/10.3390/microorganisms9081718</u>

Tsujimura, S., Tsukada, H., Nakahara, H., Nakajima, T. and Nishino, M. (2000) Seasonal variations of *Microcystis* populations in sediments of Lake Biwa, Japan. Hydrobiologia, **434**, 183–192.

Verspagen, J.M., Snelder, E.O., Visser, P.M., Jöhnk, K.D., Ibelings, B.W., Mur, L.R., Huisman, J. 2005.Benthic–pelagic coupling in the population dynamics of the harmful cyanobacterium *Microcystis* Freshw. Biol., 50(5):854-867.

Wetzel, R.G., Likens, G.E. 2000. Limnological Analyses. Third Edition. Springer, New York.

Young, M.J., Feyrer, F., Colombano, D.D., Conrad, J.L., Sih, A. 2018. Fish-habitat relationships along the estuarine gradient of the Sacramento-San Joaquin Delta, California: Implications for habitat restoration. Estuaries and Coasts. 41:2389-2409.

APPENDIX III – CURRENT USE PESTICIDES USGS FIELD AND CHEMISTRY REPORT



Delta RMP Annual Report for FY 20-21 February 1, 2022 Delta Regional Monitoring Program: Pesticides and Toxicity Field and Laboratory Report for Water Year 2021

Introduction

This informal report provides a brief description of field activities for the collection of water samples for current-use pesticide and toxicity testing, and laboratory procedures and results for current-use pesticides undertaken as part of the Delta Regional Monitoring Program (DRMP). This report covers the third year of monitoring (water year 2021) under the revised current-use pesticides/toxicity monitoring design approved by the DRMP Steering Committee in 2018. Sampling was conducted for Events 1-3 out of a planned six events during water year 2020 at which point sampling ceased due to the lack of a contracted laboratory to perform toxicity testing and impacts due to the COVID-19 pandemic. Sampling resumed in April 2021 and a total of four events were sampled in water year 2021. These events represent Events 3-6 of the second year of sampling and the completion of sampling in the Sacramento River and Northeast Delta subregions (Figure 1).

Background

The current monitoring design is focused on understanding pesticide occurrence and toxicity within the Sacramento/San Joaquin Delta by sampling a large number of sites (36 per year), selected using a Generalized Random Tesselation Stratified (GRTS) approach. For logistical reasons this revised design divides the Delta up into 6 sub-regions based on water source, and only two adjacent sub-regions are sampled in one water year (Figure 1). For the two sub-regions sampled, one sub-region is sampled completely (24 GRTS sites) and the other sub-region is partially sampled (12 GRTS sites). The remaining 12 GRTS sites within the partially sampled sub-region are sampled in the following water year.

In addition to the GRTS sites, two Delta input sites sampled during the 2015-2017 DRMP monitoring, (Ulatis Creek at Brown Rd and San Joaquin River at Buckley Cove) continue to be sampled during the revised program. It was decided to continue sampling at the two fixed sites to provide long term monitoring data. Additionally, these sites were chosen because they generally had the highest concentrations of pesticides and the most instances of aquatic toxicity of the five sites sampled in 2015-2017.

Under the current monitoring design, samples are collected during 6 targeted events (2 fall/winter storms, spring runoff, and spring, summer, and fall irrigation period events). Samples are collected once per event at each of the 2 fixed sites and at 6 GRTS sites per event. A total of 48 environmental water samples are collected per year (24 in one completely sampled sub-region, 12 in the partially sampled sub-region, and 12 samples collected at the fixed sites).

Water samples are collected by USGS personnel following standard protocols and are analyzed for dissolved and sediment associated pesticides at the USGS Organic Chemistry Research Laboratory (OCRL), for copper, dissolved organic carbon (DOC), particulate organic carbon (POC), particulate inorganic carbon (PIC), total particulate carbon (TPC), and total particulate nitrogen (TPN) at the USGS National Water Quality Laboratory (NWQL), following the standard methods used in the first two years of sampling. Beginning in water year 2021 toxicity testing is being conducted by Pacific Ecorisk (PER) a private environmental consulting and testing firm.

In addition to the environmental water samples, an extensive suite of quality control (QC) samples is collected and analyzed by the OCRL and NWQL. Samples include field and laboratory blanks, field replicates and matrix spike and spike replicates. Numbers of QC samples and data quality objectives are documented in the project specific Quality Assurance Project Plan (QAPP).

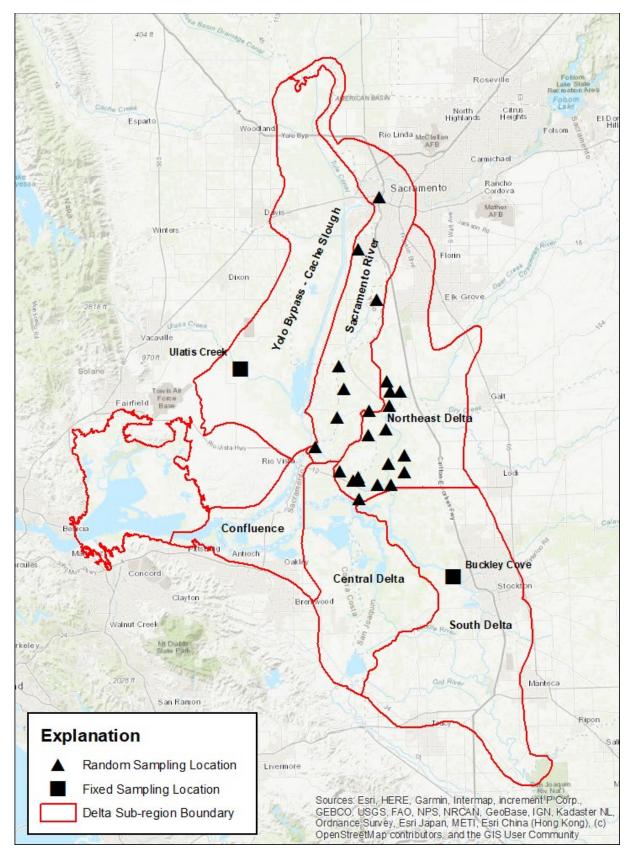


Figure 1. Map showing Delta sub-regions and fixed and GRTS sampling sites.

Field Activities

Sampling for Events 3-6 was conducted by personnel from the USGS California Water Science Center at sites shown in Figure 2 and following procedures described in the DRMP QAPP. Water samples were collected concurrently for analysis of current-use pesticides, copper, DOC, POC, TPN, TPC, and PIC, as well as for multispecies toxicity testing. All samples were collected as grab samples and all sites were accessed by boat with the exception of the fixed sampling station, Ulatis Creek at Browns Road. The study design approved by the Delta RMP called for grab samples because of the large volume of water required for collecting toxicity and pesticide samples concurrently. Samples were collected between the high and low tide, or on the ebb tide (for tidally influenced sites) by submerging narrow-mouthed bottles at mid-channel to a depth of 0.5 meters (m).

Pesticide samples were collected in pre-cleaned, baked amber-glass bottles and transported on ice to the USGS OCRL in Sacramento, California. Samples for analysis at the USGS NWQL (copper, DOC, POC, PIC, TPC, and TPN) were collected in Teflon bottles, processed at the USGS California Water Science Center, and shipped on ice to the NWQL. Sample collection and handling methods are described in more detail in De Parsia and others (2018 and 2019). Water samples for toxicity analyses were collected in pre-cleaned, 4-liter, amber-glass bottles provided by PER. Bottles were triple rinsed with native water on-site before sample collection. Ten bottles were collected at each site and transported on ice to the USGS California Water Science Center where they were picked up by a PER courier at the end of each sampling day.

Basic water-quality measurements (water temperature, specific conductance, dissolved oxygen, pH, and turbidity) were taken at a depth of 0.5m during each sample collection using a YSI EXO multi-parameter meter equipped with conductivity/temperature, dissolved oxygen, pH, and turbidity sensors. The meter was calibrated using appropriate procedures and standards before each sampling event as described in the USGS National Field Manual (U.S. Geological Survey, variously dated). Basic water-quality parameter data are shown in Table 1.

Event 3

This was the first sampling event following the discontinuance of sampling in March 2020. A revised QAPP was prepared for the DRMP pesticide and toxicity research program. Work going forward will follow the guidelines established in this version of the QAPP and the program design approved in 2018. Although the QAPP was still under review at the time of this sampling event, permission to sample was given by the interim program manager.

Water year 2021 was characterized by much below normal precipitation. Little to no rain occurred in the Sacramento and Delta region in either March or April 2021. As a result, Event 3_WY2021 can be considered a dry season/spring runoff event. Flow on area rivers was below normal (Figure 3). At the time of sampling some agricultural irrigation had been occurring for permanent crops like nuts and stone fruits, but most row crops and rice fields were still in the planting/preparation stage. A very minor precipitation event occurred on 4/25/21 with precipitation totals in the Sacramento and Delta area totaling roughly 0.1" or less.

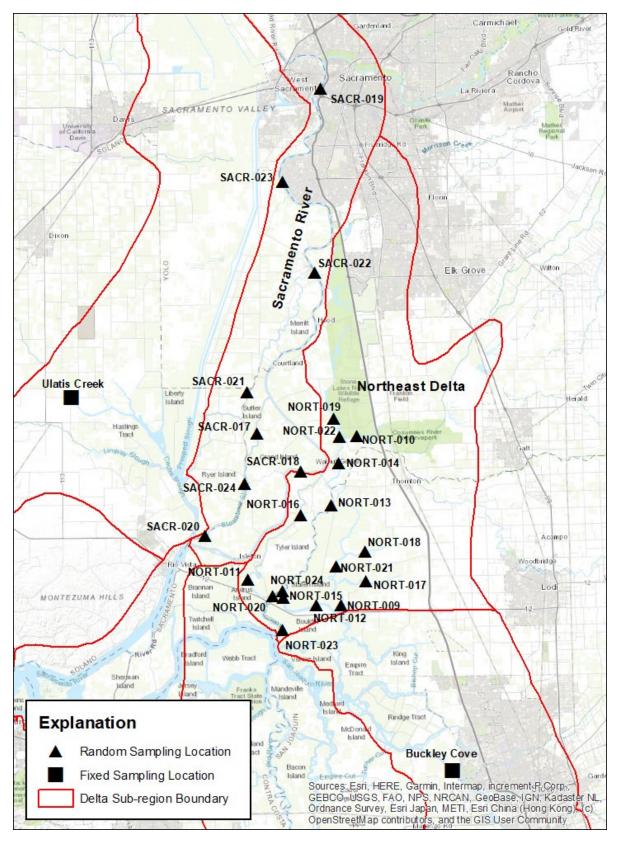


Figure 2. Map showing fixed and GRTS sites sampled in water year 2021.

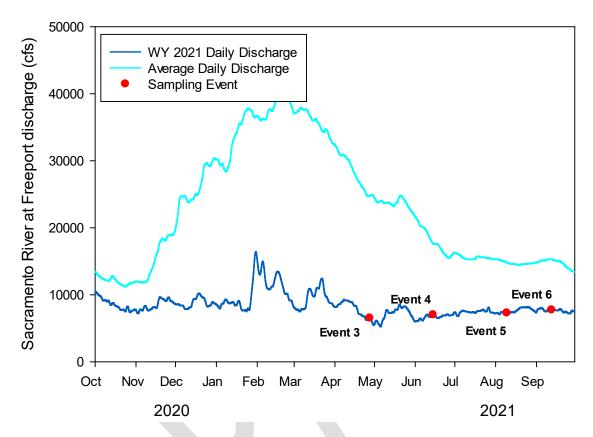


Figure 3. Water year 2021 discharge for the Sacramento River at Freeport, sampling event dates and Sacramento River at Freeport average discharge.

Sampling occurred over a 2-day span from April 28th to April 29th. On 04/28/2021 water samples were collected from Ulatis Creek by wading at 08:25. It was noted that the low flow channel had switched from the left bank and center of the channel to the right bank and center of the channel as it had been in previous years (Figure 4). Samples were collected by hand dipping bottles in the center of the channel at 0.3 m depth.



Figure 4. Flow conditions at Ulatis Creek.

Following sampling at Ulatis Creek, the full sampling crew met at the Rio Vista public boat ramp, launched the sampling boat, and proceeded on an approximately 30mi loop course to collect samples at SACR-017 and SACR-018. Samples were collected at 10:45 at SACR-017 on Steamboat Slough and at 11:45 at SACR-018 on the Sacramento River (Figure 5). The crew then returned to Rio Vista, pulled the boat and moved to Wimpy's Marina off Walnut Grove road in Walnut Grove. Sampling of site NORT-010 on Lost Slough occurred at 14:25 (Figure 6). Conditions were clear and warm with no precipitation. Samples were kept on wet ice and transported to the USGS California Water Science Center at the Sacramento State campus. Toxicity samples were picked up by Pacific Ecorisk personnel at approximately 18:00.



Figure 5. SACR-018 Sacramento River.



Figure 6. NORT-010 on Lost Slough.

On 04/29/2021 USGS personnel collected samples from the San Joaquin River near Buckley Cove, NORT-009, NORT-012, and NORT-011. The boat was launched from Ladd's Marina in Stockton at approximately 09:00 and samples were taken at Buckley Cove at 09:10. A toxicity replicate sample was collected at this site. The boat was then relaunched from B & W Resort Marina in Isleton to better access the remaining sites. NORT-009 was sampled at 11:25 on South Mokelumne River. The exact sampling location could not be reached due to blockage by aquatic vegetation (Figure 7). This vegetation looked dead and it is unknown if it had recently been sprayed with herbicide or if it was killed by winter temperatures. Samples were collected approximately 40 meters northwest of the target location. It was also noted while collecting samples at this site that two, spray-boom equipped helicopters flew overhead (less than 0.25 mi away). No spray was noted coming from the equipment, and the helicopters looked to be transiting from one location to another rather than making spraying passes. Additionally, agricultural disking was taking place on islands adjacent to the site and large volumes of dust were blowing around in the immediate area.



Figure 7. Vegetation covering site NORT-009.

NORT-012 was sampled at 11:55 (Figure 8) on the South Mokelumne River. Again, agricultural disking was taking place on islands adjacent to the site and some dust was blowing around in the immediate area. NORT-011 was sampled at 12:55 on Georgiana Slough (Figure 9). This site is close to numerous riverside residences and boat docks. All sites were sampled within acceptable distances from their respective target locations. Conditions were sunny and very warm. Samples were kept on wet ice and transported to the USGS California Water Science Center at Sacramento State campus. Toxicity samples were picked up by Pacific Ecorisk personnel at approximately 16:30.

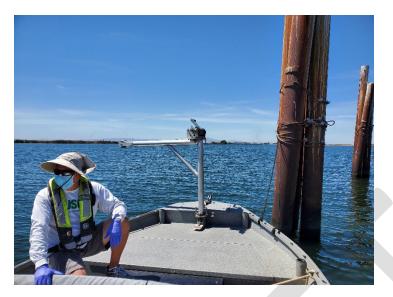


Figure 8. Preparing to sample at NORT-012 on the South Mokelumne River.



Figure 9. Approaching site NORT-011 on Georgiana Slough.

Event 4

This was the second sampling event of WY21 and is considered Event 4 of the second year of sampling under the current monitoring design. Samples were collected June 15th and 16th. This is considered an irrigation runoff sampling event. On 6/15/2021 water samples were collected from Ulatis Creek by wading at 08:35. (Figure 10). It was noted that flows seemed to be slightly higher than during the April sampling event. It was also noted that the water had a faint smell of treated wastewater and the water appeared cloudy. Dissolved oxygen was measured at 3.6 mg/L (Table 1). Samples were collected by hand dipping bottles in the center of the channel at a depth of 0.1 m.



Figure 10. Flow conditions at Ulatis Creek.

Following sampling at Ulatis Creek, the full sampling crew met at the Rio Vista public boat ramp, launched the sampling boat, and proceeded to sample SACR-020. Samples were collected at 10:00 on Steamboat Slough near the confluence with Cache Slough (Figure 11). The crew then returned to Rio Vista, pulled the boat, and moved to B&W Marina off Hwy 12. Sampling of site NORT-015 on the South Mokelumne River occurred at 11:45 (Figure 12) and at NORT-016 on Georgianna Slough at 13:30 (Figure 13). Conditions were clear and warm with no precipitation. Samples were kept on wet ice and transported to the USGS California Water Science Center at the Sacramento State campus. Toxicity samples were picked up by Pacific Ecorisk personnel at approximately 17:00.



Figure 11. SACR-020 Steamboat Slough.



Figure 12. NORT-015 on South Mokelumne River



Figure 13. NORT-016 Georgianna Slough

On 06/16/2021 USGS personnel collected samples from the San Joaquin River near Buckley Cove, NORT-013, NORT-014, and SACR-019. The boat was launched from Ladd's Marina in Stockton at approximately 08:25 and samples were taken at Buckley Cove at 08:35 (Figure 14.) The boat was then pulled and relaunched from Wimpy's Marina in near Walnut Grove. NORT-013 was sampled at 11:10 on North Mokelumne River (Figure 15). It was noted that agricultural harvesting or roadside mowing was taking place adjacent to the sampling site and quite a bit of grass/fine vegetation debris was blowing onto the surface of the water during sample collection.



Figure 14. Buckley Cove



Figure 15. NORT-013 North Mokelumne River

NORT-014 was sampled at 12:05 (Figure 16) on Snodgrass Slough. The boat and crew then returned to the marina, pulled the boat and drove to Miller Park in Sacramento. The boat was launched from Miller Park at approximately 14:00. Samples were collected at SACR-019 at 14:15 (Figure 17). At this point field personnel realized that the site names for SACR-020 and SACR-019 had been switched during the previous days sampling. Pacific Ecorisk personnel were immediately contacted by phone and notified of the mistake in bottle labeling. All sites were sampled within acceptable distances from their respective target locations. Conditions were sunny and very warm. Samples were kept on wet ice and transported to the USGS California Water Science Center at Sacramento State campus. Toxicity samples were picked up by Pacific Ecorisk personnel at approximately 16:30.



Figure 16. NORT-014 on Snodgrass Slough.



Figure 17. Approaching site SACR-019 on the Sacramento River.

Event 5

This was the third sampling event of WY21 and is considered Event 5 of the second year of sampling under the current monitoring design. Samples were collected August 10th and 11th. This is considered an irrigation runoff sampling event. Flows on area rivers were much below normal (Figure 3). Some agricultural land (rice, etc) was fallowed in the Sacramento Valley due to the drought, resulting in lower than normal flows in agricultural drainage water influenced waterways.

On 8/10/2021 USGS personnel launched the boat at New Hope Landing marina near Walnut Grove and proceeded to site NORT-019 on Snodgrass Slough (Figure 18). Sampling took place at 09:35 approximately 30 meters west of the target coordinates due to the presence of abundant aquatic vegetation at the target coordinates. The presence of bright green algae was also noted at the site and personnel donned protective equipment (shoulder length gloves, face masks and eye protection) during sampling (Figure 19).

The crew then traveled through the Delta Cross Channel into the Sacramento River and proceeded approximately 15 miles north to site SACR-022 located on the Sacramento River at Clarksburg. It was noted that a barge and crane were conducting levee excavation work approximately 400 meters upstream and that some woody debris was present at the site during sampling (Figure 20). Samples were collected at 11:00 at the target coordinates. The crew then motored back south, entered Sutter Slough, and proceeded to site SACR-021 where samples were collected at the target coordinates at 11:35 (Figure 21). The crew then returned to New Hope Landing Marina. At this point Jim Orlando and Matt Uychutin returned with the boat and samples collected so far to Sacramento while Matt de Parsia and Elisabeth Newman proceeded to Ulatis Creek to collect a sample there. Conditions at Ulatis Creek were similar to those encountered during the June sampling event with low water and the presence of much aquatic vegetation. Samples were collected at 14:25 by wading and hand dipping sample bottles (Figure 22). It was noted that as during the previous sampling event dissolved oxygen saturation was measured at a very low level (16.5%). Samples were kept on wet ice and transported to the USGS California Water Science Center at the Sacramento State campus. Toxicity samples were picked up by Pacific Ecorisk personnel at approximately 17:00.



Figure 18. NORT-019 Snodgrass Slough



Figure 19. Algae and aquatic vegetation at NORT-019



Figure 20. Barge and crane removing vegetation upstream of SACR-022



Figure 21. SACR-021 Sutter Slough



Figure 22. Ulatis Creek looking downstream

On 08/11/2021 USGS personnel collected samples from the San Joaquin River near Buckley Cove, NORT-017, NORT-08, and NORT-020. The boat was launched from Ladd's Marina in Stockton at approximately 09:00 and samples were taken at Buckley Cove at 09:20 (Figure 23). The presence of bright green algae was noted throughout the water column at the site and personnel donned protective equipment. The boat was then pulled and relaunched from B&W Marina. NORT-018 was sampled at 11:25 on Hog Slough (Figure 24). It was noted that agricultural drain water was being pumped into the waterway approximately 500 meters west of the sampling site (Figure 25).



Figure 23. Buckley Cove

NORT-017 was sampled at 12:05 on Sycamore Slough at the target coordinates. It was noted that aquatic vegetation in both Hog Slough and Sycamore Slough looked burnt down in spots and was likely recently sprayed with herbicides (Figure 26). The crew then proceeded to site NORT-020 at the confluence of the North and South Mokelumne Rivers. Samples (including a toxicity field replicate) were collected at 12:50 (Figure 27). Samples were kept on wet ice and transported to the USGS California Water Science Center at the Sacramento State campus. Toxicity samples were picked up by Pacific Ecorisk personnel at approximately 17:00.



Figure 24. NORT-018 Sycamore Slough (Ag drain in Fig 25 is in the background)



Figure 25. Agricultural drain water being pumped into the waterway near NORT-018



Figure 26. Burnt aquatic vegetation on Hog Slough



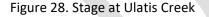
Figure 27. NORT-020 at the confluence of the North and South Mokelumne Rivers

Event 6

This was the fourth sampling event of WY21 and is considered Event 6 of the second year of sampling under the current monitoring design. Samples were collected September 13th and 14th. This is considered an irrigation runoff sampling event. A very minor rainfall event occurred on September 10th and 11th which produced generally less than 0.1" of at most Valley locations. Despite the rainfall no flow occurred on Arcade Creek in Sacramento and only a very minor rise in stage occurred on Ulatis Creek (Figure 28).

Ulatis Creek Level

All of the data and information on these plots is raw, unrefined, and has not yet been reviewed by qualified staff. The plots come directly from the monitoring equipment is subject to intermitten fluctuations, or spikes, which can cause invalid readings. These plots are issued once or twice a day and may not reflect current readings. The data and information is subject to change at any time for a variety of reasons. All times are Pacific Standard. Add one hour during daylight savings time. Top of Bank. Bottom of Bridge are for local reference only and do not indicate flood levels. Active Scripting must be enabled in your browser settings to view the charts. Ulatis Creek @ Leisure Town Road, Water Level (ft) Top of Bank = 14.7 ft, Bottom of Bridge = 14 ft 2021/09/08 16:00: 1UCLTR Stage: 0.93 Last Update: 9/10/2021 8:00 Value: 0 90958588 14 12 10 ŧ Stage -87 Sep 08 Sep 09 Sep 10 Sep



On 9/13/2021 USGS personnel sampled Ulatis Creek by wading at 08:25. Flows were low and much of the channel was choked with aquatic vegetation (Figure 29). Following sampling at Ulatis the full sampling crew met up at the Hogback Island Boat Launch on Steamboat Slough. While assembling at the boat ramp Jim Orlando spoke with Sacramento County Sheriff's deputies who were conducting several cannabis eradication operations by helicopter in the area (Figure 30). Deputies reported that there were numerous grow sites in the area on farmed islands, on the channel side of local levees, as well as on in-channel islands. They also reported seeing used pesticide containers at these sites on a routine basis. The boat was launched at approximately 10:00 and samples were collected at SACR-024 at 10:40 (Figure 31). The crew then pulled the boat and relaunched it from New Hope Landing Marina near Walnut Grove. Samples were collected from NORT-022 on Snodgrass Slough at 12:20 (Figure 32). The crew then pulled the boat once again and transported it to Garcia Bend Park in Sacramento where it was

relaunched. Sampling occurred at SACR-023 at 14:05 (Figure 33). Samples were kept on wet ice and transported to the USGS California Water Science Center at the Sacramento State campus. Toxicity samples were picked up by Pacific Ecorisk personnel at approximately 17:00



Figure 29. Ulatis Creek looking downstream.



Figure 30. Cannabis eradication operations by helicopter near Steamboat Slough on 9/13/2021



Figure 31. Sampling at SACR-024 on Steamboat Slough.



Figure 32. NORT-022 Snodgrass Slough



Figure 33. SACR-023 on the Sacramento River

On 09/14/2021 USGS personnel collected samples from the San Joaquin River near Buckley Cove, NORT-021, NORT-23, and NORT-024. The boat was launched from Ladd's Marina in Stockton at approximately 09:00 and samples were taken at Buckley Cove at 09:20 (Figure 34). The boat was then pulled and relaunched from B&W Marina. NORT-021 was sampled at 11:55 on the South Mokelumne River (Figure 35). NORT-024 was sampled at 12:40 on the North Mokelumne River near the confluence with the South Mokelumne River (Figure 36). The crew then proceeded to site NORT-023 on the Mokelumne River near the San Joaquin River confluence. Samples were collected at 13:20 (Figure 37). All samples were collected at the target coordinates. Samples were kept on wet ice and transported to the USGS California Water Science Center at the Sacramento State campus. Toxicity samples were picked up by Pacific Ecorisk personnel at approximately 17:00.



Figure 34. Buckley Cove



Figure 35. NORT-021 South Mokelumne River



Figure 36. NORT-024 North Mokelumne River



Figure 37. NORT-023 Mokelumne River

Analytical Methods

Pesticide concentrations in surface water were measured by the U.S. Geological Survey OCRL using two methods: (1) liquid chromatography/tandem mass spectrometry (LC/MS/MS) and (2) gas chromatography/mass spectrometry (GC/MS). Thirty-five compounds were analyzed using the LC/MS/MS method described in Hladik and Calhoun (2012) and 127 compounds were analyzed using the GC/MS methods described in Hladik and others (2008, 2009) and Hladik and McWayne (2012). Pesticide concentrations for 127 compounds in suspended sediment were measured by the OCRL using the GC/MS methods described in Hladik and others (2008, 2009) and Hladik and McWayne (2012). Dissolved organic carbon, DOC; particulate inorganic carbon, PIC; particulate organic carbon, POC; total particulate carbon, TPC; total particulate nitrogen, TPN; and copper analyses were performed by the U.S. Geological Survey National Water Quality Laboratory (NWQL). Dissolved organic carbon was analyzed at the NWQL using the method described in Open-File Report 92–480 (Brenton and Arnett, 1993). Particulate inorganic carbon, POC, TPC, and TPN were analyzed at the NWQL using U.S. Environmental Protection Agency (EPA) method 440.0 (Zimmermann and others, 1997). Copper was analyzed at the NWQL using the method described by Garbarino and others (2006). More detailed information on the sample processing and analytical methods employed along with method detection limits can be found in De Parsia and others (2018 and 2019).

Quality Control Methods and Results

Field blanks, field replicates, laboratory blanks, laboratory control samples, laboratory matrix spikes, and matrix-spike replicates were used to validate pesticide concentrations measured in water and in suspended sediments. Field replicates and blanks and laboratory blanks, replicates, matrix spikes and spike replicates were analyzed to validate results for analytes measured at the NWQL.

Quality control sample results for copper, DOC, POC, TN, PIC, and TOC are pending completion of sample analyses by the USGS NWQL.

Field Blanks

Four pesticide field blanks (two for analysis by GC/MS and two for analysis by LC/MS/MS) were collected to verify the cleanliness of pesticide sample collection and processing protocols. Filters from the two pesticide field blanks collected for analysis by GC/MS also were saved and analyzed as suspended-sediment field blanks. No pesticides were detected in any of the pesticide field blanks.

Field Replicates

Four pesticide field-replicate samples (two for analysis by GC/MS and two for analysis by LC/MS/MS) were analyzed to test the reproducibility of results based on field-sampling methods. There were 15 detections of pesticides in the sample pairs analyzed. Results from the environmental and field-replicate pairs satisfied the QAPP requirement of less than 25 percent relative percent difference (RPD) between environmental samples and their field-replicate pairs. The correlation of pesticide detections between

the paired environmental and replicate samples was 100 percent. Suspended sediments were also analyzed in the two GC/MS field replicate samples and no pesticides were detected in these samples.

Laboratory Blanks

Laboratory blank samples were prepared at the USGS OCRL and analyzed for each analytical method and laboratory sample batch containing Delta RMP field samples for all four sampling events. No pesticides were detected in any of the laboratory blanks.

Laboratory Replicates

Laboratory replicate samples were prepared at the USGS OCRL and analyzed for each analytical method and laboratory sample batch containing Delta RMP field samples for all four sampling events. There were 55 sample pairs where pesticides were detected. Results from all environmental and laboratoryreplicate pairs satisfied the QAPP requirement of less than 25 percent relative percent difference (RPD) between environmental samples and their laboratory-replicate pairs. The correlation of pesticide detections between the paired environmental and laboratory replicate samples was 100 percent. Suspended sediments were also analyzed in the four GC/MS laboratory replicate samples and no pesticides were detected in these samples.

Laboratory Control Samples

Laboratory control samples were prepared at the USGS OCRL and analyzed for each analytical method and laboratory sample batch containing Delta RMP field samples for all four sampling events. All analytes in each sample met the QAPP requirement for recovery of 70% to 130%.

Laboratory Matrix Spikes

Four pesticide matrix-spike samples (two for analysis by GC/MS and two for analysis by LC/MS/MS) and four corresponding pesticide matrix-spike replicate samples were collected to assess pesticide recovery, degradation, sorption, and potential interferences caused by the sampling matrix. Filters from the two GC/MS pesticide matrix spike and the two matrix spike replicates also were saved and analyzed as suspended-sediment matrix spike and matrix spike replicates. All matrix-spike samples met the QAPP objective of 70–130 percent recovery of pesticide matrix-spike compounds, and less than 25% RPD between matrix spike and matrix-spike replicate pairs.

Pesticide Surrogate Compounds

To assess the efficiency of water-sample extraction analytical methods, ${}^{13}C_3$ -atrazine, ${}^{13}C$ -fipronil and d₁₄-trifluralin (GC/MS), and monuron and d₄-imidacloprid (LC/MS/MS) were used as recovery surrogates and added to all extracts. To assess the efficiency of filter-sample extraction, d₁₄-trifluralin, ${}^{13}C_{12}$ -p,p'-DDE, and ${}^{13}C_6$ -cis-permethrin were used as recovery surrogates for extracts. All samples satisfied the QAPP requirement of 70–130 percent recovery of surrogate compounds.

Environmental Sample Results

A total of 32 environmental samples were analyzed for dissolved pesticides by the USGS OCRL during the 2021 water year. During this period a total of 49 pesticides were detected in the dissolved phase (13 fungicides, 17 herbicides, 18 insecticides and the synergist piperonyl butoxide). Each of the 32 samples analyzed contained mixtures of from 4 to 27 pesticides per sample. Frequently detected pesticides

included azoxystrobin, and methoxyfenozide (100% of samples); 3,4-DCA, (91%), imidacloprid (66%), and fluridone and metolachlor (59%). Maximum concentrations ranged from below method detection limits to 3,710 ng/L (metolachlor).

A total of 32 environmental samples were analyzed for suspended sediment associated (particulate) pesticides by the USGS OCRL. During this period three pesticides were detected on suspended sediments. The pesticides detected included bifenthrin (2 detections), cyhalothrin (1 detection), and metolachlor (1 detection).

Ten of the 32 samples contained at least one pesticide with a concentration above an EPA aquatic life benchmark. Bifenthrin was detected above its chronic invertebrate benchmark of 1.3 ng/L in the Event 3 sample collected at NORT-009 and in the Event 4 Buckley Cove and Ulatis Creek samples. Cyhalothrin was detected in the Event 4 Ulatis Creek sample at 25.3 ng/L (acute fish toxicity benchmark is 14.5 ng/L). Imidacloprid was detected above its chronic invertebrate benchmark of 10.0 ng/L in the Event 3 SACR-017 sample and the Event 5 and 6 Ulatis Creek samples. Dichlorvos was detected above its chronic invertebrate toxicity benchmark of 5.8 ng/L in two Event 6 samples (NORT-021 and SACR-023). Metolachlor was detected above its chronic invertebrate benchmark of 1,000 ng/L in the Event 3 Ulatis Creek sample. Diuron was detected above its recently (2021) lowered vascular plant acute toxicity benchmark of 130 ng/L in the Event 3 Buckley Cove sample and the Event 5 Ulatis Creek sample.

A total of 32 environmental samples were analyzed for copper, DOC, TPC, TPN, PIC, POC, and DOC by the USGS NWQL. Final sample results for copper, DOC, POC, TN, PIC, and TOC are pending completion of sample analyses by the USGS NWQL.

All analytical and field parameter results are available for download through the USGS NWIS (https://nwis.waterdata.usgs.gov/ca/nwis/qwdata) or CEDEN databases (<u>https://ceden.waterboards.ca.gov/AdvancedQueryTool</u>) using the sampling event and station identification information found in Table 1.

References Cited

Brenton, R.W., and Arnett, T.L., 1993, Methods of analysis by the U.S. Geological Survey National Water Quality Laboratory—Determination of dissolved organic carbon by UV-promoted persulfate oxidation and infrared spectrometry: U.S. Geological Survey Open-File Report 92–480, 12 p., https://doi.org/10.3133/ofr92480

De Parsia, M., Orlando, J.L., McWayne, M.M., and Hladik, M.L., 2018, Pesticide inputs to the Sacramento– San Joaquin Delta, 2015–16: Results from the Delta Regional Monitoring Program: U.S. Geological Survey Data Series 1089, 49 p., <u>https://doi.org/10.3133/ds1089</u>

De Parsia, M., Woodward, E.E., Orlando, J.L., and Hladik, M.L., 2019, Pesticide mixtures in the Sacramento–San Joaquin Delta, 2016–17: Results from year 2 of the Delta Regional Monitoring Program: U.S. Geological Survey Data Series 1120, 33 p., https://doi.org/10.3133/ds1120.

Garbarino, J.R., Kanagy, L.K., and Cree, M.E., 2006, Determination of elements in natural-water, biota, sediment, and soil samples using collision/reaction cell inductively coupled plasma-mass spectrometry: U.S. Geological Survey Techniques and Methods 5–B1, 88 p., <u>https://doi.org/10.3133/tm5B1</u>.

Hladik, M.L., and Calhoun, D.L., 2012, Analysis of the herbicide diuron, three diuron degradates, and six neonicotinoid insecticides in water—Method details and application to two Georgia streams: U.S. Geological Survey Scientific Investigations Report 2012–5206, 10 p., https://doi.org/10.3133/sir20125206.

Hladik, M.L., and McWayne, M.M., 2012, Methods of analysis—Determination of pesticides in sediment using gas chromatography/mass spectrometry: U.S. Geological Survey Techniques and Methods 5–C3, 18 p., <u>https://doi.org/10.3133/tm5C3</u>.

Hladik, M.L., Smalling, K.L., and Kuivila, K.M., 2008, A multi-residue method for the analysis of pesticides and pesticide degradates in water using HLB solid-phase extraction and gas chromatography-ion trap mass spectrometry: Bulletin of Environmental Contamination and Toxicology, v. 80, p. 139–144, https://www.researchgate.net/publication/5655709 A Multi-

Residue Method for the Analysis of Pesticides and Pesticide Degradates in Water Using HLB Soli d-Phase Extraction and Gas Chromatography-Ion Trap Mass Spectrometry.

Hladik, M.L., Smalling, K.L., and Kuivila, K.M., 2009, Methods of analysis—Determination of pyrethroid insecticides in water and sediment using gas chromatography/mass spectrometry: U.S. Geological Survey Techniques and Methods 5–C2, 18 p., <u>https://doi.org/10.3133/tm5C2</u>.

U.S. Geological Survey, variously dated, National field manual for the collection of water-quality data (version 7): U.S. Geological Survey Techniques and Methods, book 9, chaps. A1–A10, accessed April 5, 2013, at http://water.usgs.gov/owq/FieldManual/.

Zimmermann, C.F., Keefe, C.W., and Bashe, J., 1997, Method 440.0—Determination of carbon and nitrogen in sediments and particulates of estuarine/coastal waters using elemental analysis: Cincinnati, Ohio, U.S. Environmental Protection Agency, Revision 1.4, sec. 11.4.2, 10 p., <u>https://cfpub.epa.gov/si/si_public_file_download.cfm?p_download_id=525245</u>.

Event	CEDEN Code	USGS Site Name	USGS Site Number	Latitude	Longitude	Date	Time	Air Temperature ℃	Water Temperature ℃	Ph	DO (mg/L)	DO (%)	Specific Conductance (microSiemen s/cm)	Salinity	Turbidity (UFU)
3	544LSAC13	SAN JOAQUIN R A BUCKLEY COVE NR STOCKTON CA	375831121223701	37.97528	-121.37694	4/29/2021	9:10	18.6	19.6	7.5	7.8	84.8	755	0.37	2.1
3	511ULCABR	ULATIS C A BROWNS RD NR ELMIRA CA	11455261	38.30667	-121.79361	4/28/2021	8:25	14.9	16.8	7.6	3.3	34.3	805	0.39	6.2
3	NORT-009	DELTA RMP NORT-009	380720121295401	38.12235	-121.49829	4/29/2021	11:25	24.7	18.6	7.8	9.7	103.6	191	0.1	1.3
3	NORT-010	DELTA RMP NORT-010	381612121283901	38.26999	-121.47745	4/28/2021	14:25	26.9	20.0	IM	8.9	98.0	140	0.06	5.2
3	NORT-011	DELTA RMP NORT-011	380845121360201	38.14596	-121.60069	4/29/2021	12:55	30.1	19.2	7.5	8.3	89.2	168	0.08	2.3
3	NORT-012	DELTA RMP NORT-012	380722121313101	38.1228	-121.52521	4/29/2021	11:55	27.3	19.3	8.4	10.9	118.0	173	0.08	1.3
3	SACR-017	DELTA RMP SACR-017	381627121351901	38.27415	-121.58859	4/28/2021	10:45	19.5	18.5	7.7	8.2	86.9	161	0.08	2.7
3	SACR-018	DELTA RMP SACR-018	381423121322401	38.23966	-121.53999	4/28/2021	11:45	24.4	18.8	7.8	8.2	88.0	165	0.08	3.4
4	544LSAC13	SAN JOAQUIN R A BUCKLEY COVE NR STOCKTON CA	375831121223701	37.97528	-121.37694	6/16/2021	8:35	20.9	23.3	7.7	7.3	85.9	633	0.31	2.2
4	511ULCABR	ULATIS C A BROWNS RD NR ELMIRA CA	11455261	38.30667	-121.79361	6/15/2021	8:25	20.7	19.0	7.7	3.6	39.0	835	0.41	24.5
4	NORT-013	DELTA RMP NORT-013	381235121302601	38.20981	-121.50713	6/16/2021	11:10	25.0	23.1	8.1	8.5	99.1	176	0.08	2.9
4	NORT-014	DELTA RMP NORT-014	381449121295401	38.24697	-121.49829	6/16/2021	12:05	31.2	23.8	8.1	8.6	102.1	171	0.08	3.2
4	NORT-015	DELTA RMP NORT-015	380747121334201	38.12969	-121.56176	6/15/2021	11:45	28.5	22.8	8.0	8.4	96.9	176	0.08	1.3
4	NORT-016	DELTA RMP NORT-016	381206121322901	38.20163	-121.54138	6/15/2021	13:30	29.6	23.0	7.8	8.4	97.7	141	0.07	0.8
4	SACR-019	DELTA RMP SACR-019	383431121304201	38.57538	-121.51169	6/16/2021	14:15	32.1	24.0	7.9	8.3	98.2	123	0.06	1.0
4	SACR-020	DELTA RMP SACR-020	381105121385301	38.1846	-121.64806	6/15/2021	10:00	21.5	21.7	8.0	8.1	91.9	197	0.09	3.4
5	544LSAC13	SAN JOAQUIN RA BUCKLEY COVE NR STOCKTON CA	375831121223701	37.97528	-121.37694	8/11/2021	9:20	21.49	25.04	7.41	7.07	85.7	276.8	0.13	2.6
5	511ULCABR	ULATIS C A BROWNS RD NR ELMIRA CA	11455261	38.30667	-121.79361	8/10/2021	14:25	30.81	21.02	7.38	1.46	16.5	901	0.44	5.64
5	NORT-017	DELTA RMP NORT-017	380834121281301	38.14276	-121.47036	8/11/2021	12:05	26.01	25.1	7.87	7.86	95.3	237.5	0.11	0.29
5	NORT-018	DELTA RMP NORT-018	381008121281301	38.16881	-121.47039	8/11/2021	11:25	23.96	24.84	7.52	7.8	94.6	323	0.15	1.65
5	NORT-019	DELTA RMP NORT-019	381710121301101	38.28613	-121.50318	8/10/2021	9:35	20.16	24.71	7.78	8.04	96.7	149.8	0.07	3.21
5	NORT-020	DELTA RMP NORT-020	380751121342701	38.13087	-121.57406	8/11/2021	12:50	31.49	24.7	7.96	8.55	103.1	145.2	0.07	1.27
5	SACR-021	DELTA RMP SACR-021	381837121355501	38.31035	-121.59847	8/10/2021	11:45	26.55	23.58	7.68	8.08	95.9	150	0.07	1.08
5	SACR-022	DELTA RMP SACR-022	382451121311701	38.41424	-121.52147	8/10/2021	11:00	26.9	24.65	7.59	7.94	93.8	160.3	0.07	1.28
6	544LSAC13	SAN JOAQUIN R A BUCKLEY COVE NR STOCKTON CA	375831121223701	37.97528	-121.37694	9/14/2021	9:20	23.54	25.22	7.43	7.59	92.3	537	0.26	2.89
6	511ULCABR	ULATIS C A BROWNS RD NR ELMIRA CA	11455261	38.30667	-121.79361	9/13/2021	8:25	19.76	20.97	7.41	0.44	4.8	789	0.39	4.01
6	NORT-021	DELTA RMP NORT-021	380922121301101	38.15614	-121.50311	9/14/2021	11:55	26.99	24.51	7.67	7.42	88.9	235	0.11	1.07
6	NORT-022	DELTA RMP NORT-022	381611121294701	38.26963	-121.49641	9/13/2021	12:20	29	24.5	7.66	8.04	96.5	199.6	0.09	1.87
6	NORT-023	DELTA RMP NORT-023	380604121334701	38.10115	-121.56298	9/14/2021	13:20	26.12	25.14	8.63	11.64	141.5	214.3	0.1	33.34
6	NORT-024	DELTA RMP NORT-024	380806121334701	38.13515	-121.5631	9/14/2021	12:40	25.21	24.36	7.75	7.82	93.6	198.8	0.09	1.05
6	SACR-023	DELTA RMP SACR-023	382939121332101	38.49416	-121.55587	9/13/2021	14:05	32.89	24.97	8.01	8.98	108.5	187.7	0.09	1.53
6	SACR-024	DELTA RMP SACR-024	381347121361201	38.2297	-121.60339	9/13/2021	10:40	23.19	24.04	7.77	8.32	99.4	199.2	0.09	0.56
										· · · · ·				-	

APPENDIX IV – CEC YEAR 1 DATA REPORT



Delta RMP Annual Report for FY 20-21 February 1, 2022



Pilot Study of Constituents of Emerging Concern in the Sacramento-San Joaquin Delta Year 1 Data Report

Authors:

Michael Weaver and Don Yee: San Francisco Estuary Institute – Aquatic Science Center

October 2021





Table of Contents

Table of Contents	2
List of Appendices	4
List of Figures	4
List of Tables	4
Summary	5
Introduction	5
This Report	6
Monitoring Description	6
Water	7
Sediment	8
Fish	8
Bivalve	9
Methods	11
Sample Collection	12
Water	12
Sediment	12
Fish	13
Bivalve	13
Sample Preparation and Analytical Methods	13
Water	13
Sediment	14
Fish Tissue	15
Bivalve Tissue	15
Quality Assurance	17
Field and Analytical Completeness	17
Precision and Accuracy for Field and Laboratory QC	17
Blank Contamination	17
Corrective Actions	18

Results	18
Water	18
Sediment	19
Fish	20
Bivalve	21
References	22

List of Appendices

Appendix 1:	Fish Cruise Report
11	1

- Appendix 2: <u>Bivalve Cruise Report</u>
- Appendix 3: <u>Quality Assurance Review</u>
- Appendix 4: <u>Deviation Forms</u>
- Appendix 5: <u>Water Data Tables</u>
- Appendix 6: <u>Sediment Data Tables</u>
- Appendix 7: Fish Data Tables
- Appendix 8: <u>Bivalve Data Tables</u>

List of Figures

Figure 1 Map of sampling locations

11

List of Tables

Table 1	Sampling station code, name, latitude, longitude, and collection dates.	9
Table 2	Sample collection, preparation and analysis methods and agencies for water and sediment samples	16
Table 3	Sample collection, preparation and analysis methods and agencies for tissue samples	16

Suggested Citation:

Weaver, M. and D. Yee. 2021. Pilot Study of Constituents of Emerging Concern in the Sacramento-San Joaquin Delta, Year 1 Data Report. Delta Regional Monitoring Program.

Summary

This report documents the first year results from a pilot study for the monitoring of Constituents of Emerging Concern (CECs) in the Sacramento-San Joaquin River Delta (the Delta). A suite of CECs recommended for monitoring by a State Water Resources Control Board guidance document were analyzed in water, sediment and tissue samples obtained from the Delta. Many of the primary target compounds in the water matrix were frequently not detected, but the few that were measured generally appeared to be in a concentration range similar to those reported in the literature for other water bodies (examples in the text). For the polybrominated diphenyl ethers (PBDEs) and perfluoroalkyl and polyfluoroalkyl substances (PFAS) measured in sediment and tissue, results were also in a similar concentration range to those found in other water bodies such as San Francisco Bay. The relative abundance of individual compounds in these analyte groups also were also similar to data from other studies. In sediment, primary target PBDEs 047 and 099 were detected in all samples, but at <1 ng/g dw, while 209 (the most degradation-resistant, and dominant in the "deca" formulation that was banned last, but a secondary PBDE analyte with high RPDs (125-175%) in replicates that exceed the MQO of <35%) was most abundant in 2 of 3 samples, while tissue samples primarily had PBDE 047 and 099, which have chemical properties conducive to bioaccumulation. Of the PFAS, PFOS was detected at the highest concentrations. These data provide a baseline for comparison to other regions in California and beyond, and to track potential trends in environmental concentrations and exposure, with management restrictions or changing use patterns for these chemicals.

Introduction

A pilot study for the monitoring of Constituents of Emerging Concern (CECs) in the Sacramento-San Joaquin River Delta (the Delta) by the Delta Regional Monitoring Program (Delta RMP) was conducted beginning in 2020. This pilot study (Larry Walker Associates 2018) was designed by Larry Walker Associates, an entity representing Delta RMP stakeholders, based on the State Water Resources Control Board design guidance (Tadesse 2016) to better understand methods of evaluating ambient concentrations and sources of Constituents of Emerging Concern (CECs) in different Central Valley surface water scenarios.

The stated goals for the study in the statewide guidance document from the State Board (Tadesse 2016) are:

"This statewide pilot study implements the second phase of the recommendation which is to gather data to determine the occurrence and biological impacts of CEC. The result of this pilot study will help the State Water Board to develop a statewide CEC monitoring strategy and control action."

"The objective of the CEC statewide pilot study monitoring plan is to generate statewide data to inform Water Board managers of the status and trends of CECs

in water. The plan is designed to narrow the data gap among regions by producing comparable CEC data throughout the state."

The responsible agency for the first year of the surface water monitoring program was the San Francisco Estuary Institute-Aquatic Science Center (SFEI-ASC), acting as the implementing entity to the Delta RMP. The pilot study's Quality Assurance Project Plan (QAPP), version 1.0 (Heberger et al. 2020), developed by SFEI-ASC, describes how the project was to be managed, organized and implemented in year one. Deviations from the plans and procedures outlined in that QAPP that occurred during the implementation of the project are documented in this report (<u>Appendix 4</u>).

This Report

This data report presents the methods and results for the first year of CEC monitoring by the Delta Regional Monitoring Program. In 2020, the Delta RMP initiated CEC monitoring of water, sediment, fish and bivalves. Fish were collected in September 2020 from four stations and analyzed for PFAS (PFOS and PFOA) and PBDEs. Clams were collected in October 2020 from five stations and analyzed for PBDEs. Sediment was collected in August and September 2020 from three stations and analyzed for PFAS (PFOS and PFOA) and PBDEs (and ancillary parameters). Quarterly sampling of PFAS (PFOS and PFOA), Pharmaceuticals and Personal Care Products (PPCPs) (including estrone, 17-beta-estradiol, ibuprofen, diclofenac, triclosan, and bisphenol A), galaxolide, and ancillary parameters in water, at eight sites, began in September 2020, with further sampling conducted in January, April, and June 2021.

Monitoring Description

Water

Water samples were collected four times between September 2020 and June 2021. There were eight water collection sites (see Figure 1) that were planned to be sampled at each sampling event (three sites by SFEI-ASC and five sites by Department of Water Resources - Municipal Water Quality Investigations (DWR-MWQI)). However, due to COVID-19 related restrictions, DWR-MWQI did not sample during the second sampling event (in January 2021), so samples were only collected at three sites by SFEI-ASC during that event.

SFEI-ASC collected water samples at Sacramento River at Elkhorn Boat Launch Facility (519SUT108), Dry Creek at Roseville Wastewater Treatment Plant (WWTP) (519DRYCRK) and Old Alamo Creek at Lewis Road (511SOL011) during the September, January, April and June sampling events. DWR-MWQI collected water samples at American River at Discovery Park (519AMNDVY), Sacramento River at Freeport (510ST1301), Sacramento River at Hood Monitoring Station Platform (510SACC3A), San Joaquin River at Buckley Cove (544LSAC13) and San Joaquin River at Airport Way near Vernalis (541SJC501), during the September, April and June sampling events. Further details on sampling stations and dates are listed in Table 1.

At each site and event where water sampling occurred, samples were collected for every planned water analysis (PFOS and PFOA, galaxolide, PPCPs, and SSC), field water quality measurements (dissolved oxygen, pH, specific conductivity, temperature, and turbidity) were taken, and habitat observations were recorded. QC samples were also collected as required by the project QAPP (see <u>Appendix 3</u> for additional details). Details on water sample collection methods are described in the Methods section of this report.

The water sampling monitoring design called for four sampling events on a schedule (listed in QAPP table 10.2) beginning with a summer (dry season) event, followed by a late summer/early fall event, a first flush event and spring storm event. The order of these events was shifted, due to a late start in sampling, so water collections began with an early fall event in 2020 and ended with a dry season event in summer 2021. Additionally, due to a lack of rainfall in spring 2021, the spring sampling event was a dry event rather than a spring storm event.

Some deviations from the StationCodes listed in the QAPP occurred during water sampling due to a) QAPP latitudes and longitudes not matching the CEDEN coordinates for the stations and b) CEDEN stations in the same vicinity sharing near-identical station names (see deviation forms 2020-04 and 2020-05 in <u>Appendix 4</u> for more details). As a result of this, for the American River at Discovery Park site listed in the QAPP, water was sampled at the station with CEDEN StationCode 519AMNDVY (not 519SWPDCP), for the Dry Creek u/s of WWTP site, water was sampled at 519DRYCRK (not 519LSAC12), and for the Sacramento River at Veterans Bridge site, water was sampled at 519SWPVTB). (The determination of the most appropriate StationCode to use was made by MLJ Environmental, based on which CEDEN station most closely

matched the actual coordinates sampled at, with a preference to have consistent StationCodes used among the different project matrices, wherever possible.)

Sediment

Sediment samples were collected in August and September 2020, concurrently with a State Water Resources Control Board - Surface Water Ambient Monitoring Program -Stream Pollution Trends Monitoring Program (SWRCB-SWAMP-SPoT) sediment cruise and SFEI-ASC's first event of water sampling for this project. Sediment was collected at three locations (two sites by SFEI-ASC and one site by SWRCB-SWAMP-SPoT). These locations were a subset of the water sample collection sites (see Figure 1).

SFEI-ASC collected sediment samples at Dry Creek at Roseville WWTP (519DRYCRK) and Old Alamo Creek at Lewis Road (511SOL011), in September 2020. SWRCB-SWAMP-SPoT collected sediment samples at American River at Discovery Park (519AMNDVY) in August 2020. Further details on sampling stations and dates are listed in Table 1.

At each site where sediment sampling occurred, samples were collected for every planned sediment analysis (PFOS and PFOA, PBDEs, TOC), field water quality measurements (dissolved oxygen, pH, specific conductivity, temperature and turbidity) were taken, and habitat observations were recorded. QC samples were also collected as required by the project QAPP (see <u>Appendix 3</u> for additional details). Details on sediment sample collection methods are described in the Methods section of this report.

As with water sampling, some deviations from the StationCodes listed in the QAPP occurred during sediment sampling due to a) QAPP latitudes and longitudes not matching the CEDEN coordinates for the stations and b) CEDEN stations in the same vicinity sharing near-identical station names (see deviation forms 2020-04 and 2020-05 in <u>Appendix 4</u> for more details). As a result of this, for the American River at Discovery Park site listed in the QAPP, sediment was sampled at the station with CEDEN StationCode 519AMNDVY (not 519SWPDCP), and for the Dry Creek u/s of WWTP site, sediment was sampled at 519DRYCRK (not 519LSAC12).

Fish

Fish samples were collected from four stations in the Delta (Figure 1). Fish samples were collected at a subset of the eight water sample collection sites (though in two instances, different stations in the same vicinity of the water collection sites were sampled). Fish collections were completed in September 2020. Details on sampling stations and dates are listed in Table 1 and in greater detail in the cruise report (<u>Appendix 1</u>).

For two fish sampling stations, there were deviations from the Station Codes listed in the QAPP, due to multiple CEDEN stations in the same vicinity sharing near-identical station names (see deviation form 2020-04 in <u>Appendix 4</u>). As a result of this, for the Sacramento River at Veterans Bridge site listed in the QAPP, fish were sampled at 519ST1309 (not 519SWPVTB), and for the Sacramento River at Freeport site, fish were sampled at 510ST1317 (not 510ST1301).

Bivalve

Sampling of *Corbicula fluminea* clams was planned at six sites in the San Francisco Bay-Delta in October 2020. At one site, San Joaquin River at Airport Way near Vernalis, sampling was attempted, but no clams were collected due to the site being inaccessible (see deviation form 2020-08 in <u>Appendix 4</u>), so clam samples were only collected at 5 sites (Figure 1). Details on sampling stations and dates are listed in Table 1 and in greater detail in the cruise report (<u>Appendix 2</u>).

As with water sampling, some deviations from the StationCodes listed in the QAPP occurred during clam sampling due to a) QAPP latitudes and longitudes not matching the CEDEN coordinates for the stations and b) CEDEN stations in the same vicinity sharing near-identical station names (see deviation forms 2020-04 and 2020-05 in <u>Appendix 4</u> for more details). As a result of this, for the American River at Discovery Park site listed in the QAPP, clams were sampled at the station with CEDEN StationCode 519AMNDVY (not 519SWPDCP), and for the Sacramento River at Veterans Bridge site, clams were sampled at 519SUT108 (not 519SWPVTB).

CEDEN Station Code	CEDEN Station Name	CEDEN Target Latitude	CEDEN Target Longitude	Fish Collection Dates	Bivalve Collection Dates	Sediment Collection Dates	Water Collection Dates
510SACC3A	Sacramento River at Hood Monitoring Station Platform	38.36771	-121.5205	-	10/15/2020	-	9/29/2020, 4/13/2021, 6/15/2021
510ST1301	Sacramento River at Freeport, CA	38.45555	-121.50194	-	10/15/2020	-	9/29/2020, 4/13/2021, 6/15/2021
510ST1317	Sacramento River/Freeport	38.4556	-121.5019	9/9/2020	-	-	-
511SOL011	Old Alamo Creek at Lewis Road	38.34643	-121.89702	-	-	9/30/2020	9/30/2020, 1/27/2021, 4/14/2021, 6/16/2021
519AMNDVY ¹	American River at Discovery Park	38.60094	-121.5055	-	10/15/2020	8/19/2020	9/29/2020, 4/13/2021, 6/15/2021
519DRYCRK ²	Dry Creek at Roseville WWTP	38.734098	-121.3144446	-	-	9/30/2020	9/30/2020, 1/27/2021, 4/14/2021, 6/16/2021

Table 1 Sampling station code, name, latitude, longitude, and collection dates.

¹Water and bivalve samples originally recorded with Station Code 519SWPDCP.

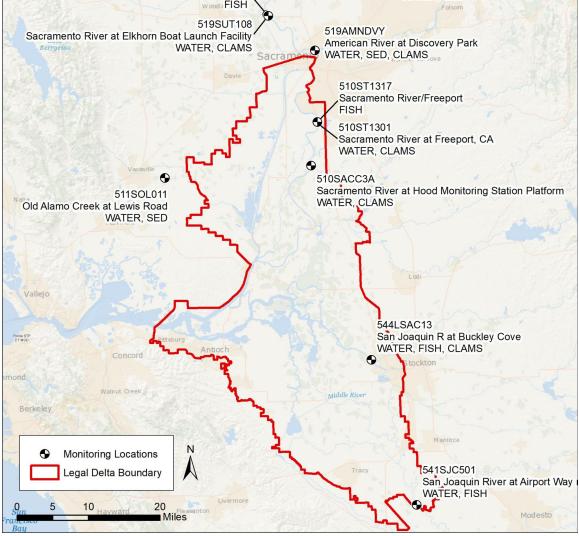
² Water and sediment samples originally recorded with Station Code 519LSAC12.

519ST1309	Sacramento River at Veterans Bridge-03SWSBIO	38.67468	-121.62751	9/9/2020	-	-	-
519SUT108 ³	Sacramento River at Elkhorn Boat Launch Facility	38.67245	-121.625	-	10/15/2020	-	9/30/2020, 1/27/2021, 4/14/2021, 6/16/2021
541SJC501	San Joaquin River at Airport Way near Vernalis	37.67555556	-121.2641667	9/22/2020	-	-	9/30/2020, 4/14/2021, 6/16/2021
544LSAC13	San Joaquin R at Buckley Cove	37.971833	-121.373619	9/8/2020	10/16/2020	-	9/30/2020, 4/14/2021, 6/16/2021

³ Water and bivalve samples originally recorded with Station Code 519SWPVTB.



Figure 1 Map of sampling locations. Labels show the CEDEN station code, station name, and



Methods

Sample Collection

Water

DWR-MWQI water sampling was conducted by a modified version of the "Direct Dip Method" described in the DWR Standard Operating Procedure (SOP) document "Collection of Water Quality Samples for Laboratory Analysis." A clean empty bottle for each station was attached to a pole. The empty bottle was quickly submerged below the water surface, facing upstream, and allowed to fill, avoiding contact with the bottom sediment, or any debris or surface scum. The filled bottle was then removed from the water, and the contents poured into clean sample bottles which were then capped. The process was repeated until all the needed samples were collected. Samples were labeled and placed in an ice chest for transport back to the laboratory of the collection agency. Samples were then packed on ice and shipped, or delivered directly, with Chain of Custody forms (CoCs) to the respective analytical laboratories. SFEI-ASC water sampling was conducted by direct submersion of bottles by hand, in alignment with DWR protocols, and separately packed and shipped, or delivered directly, with CoCs to the respective analytical laboratories.

Handheld portable YSI instruments were taken to the field and used to measure the following ancillary water column parameters: temperature, pH, dissolved oxygen concentration and percent saturation, specific conductivity, and turbidity at each site and event.

Sediment

Sediment sampling conducted by SWRCB-SWAMP-SPoT and SFEI-ASC was performed by using shallow polycarbonate cores and scoops together to remove the top 5 cm of sediment from each site. A single core at each site could not provide sufficient material to perform all analyses, so several cores were collected at regular intervals along the reach of a site until sufficient material for all analyses was obtained. For PFOA and PFOS, core contents were scooped directly into sample jars. For PBDEs and TOC, core grabs were composited in a container before subsampling into separate jars for the respective analyses. Samples were kept chilled in an ice chest for return to each collection agency's laboratory, where they were packed chilled on ice and shipped to the analytical laboratory with CoCs. Upon receipt at the analytical laboratory, samples were kept frozen until extraction and analysis.

Handheld portable YSI instruments were used to measure the following ancillary water quality parameters in the field: temperature, pH, dissolved oxygen concentration and percent saturation, specific conductivity, and turbidity. These parameters were measured at each site and event, with the exception of American River at Discovery Park, where dissolved oxygen percent saturation and turbidity were not measured by SWRCB-SWAMP-SPoT (see deviation form 2020-06 in <u>Appendix 4</u>). The CEC QAPP requested ("if possible") measurement of porewater pH, which was not done for any of the sites. Porewater pH may be useful for understanding speciation and partitioning behavior for some CECs, but PBDEs have no acid-base forms, and PFOS and PFOA are

affected only at low pH (<3), which occurs rarely in natural sediment. Future sediment pore water pH measurement may be unnecessary for these specific CECs.

Fish

Fish sampling was conducted by Marine Pollution Studies Laboratory (MPSL-DFW), described briefly here, and in further detail in <u>Appendix 1</u>. Fish (channel catfish, largemouth bass, Sacramento sucker) were collected from stations by electrofishing. At each location, five or more fish, of one or two of the target species, were collected. Upon collection, each fish collected was tagged with a unique ID. Physical parameters measured for each individual fish included: weight, total length, fork length, and presence of any abnormalities. Large fish were partially dissected in the field at the dock; fish were placed on a cutting board covered with a clean plastic bag where the head, tail, and entrails were removed using a clean cleaver. Fish samples were stored on dry ice for the duration of transport to MPSL-DFW at Moss Landing Marine Labs (MLML) in Moss Landing, CA. At MPSL-DFW samples were stored in a -30 °C freezer until processed for authorized dissection, composited and shipped to SGS-AXYS for analysis (as described in the Sample Preparation and Analytical Methods section).

Bivalve

Collection of resident *Corbicula fluminea* was conducted by Applied Marine Sciences (AMS) at 5 locations, with a sixth planned site not sampled due to inaccessibility by boat. Samples were collected using a stainless steel clam dredge towed behind a research boat proceeding slowly upcurrent within the target sampling area. If clams were present in the dredge cage, they were dumped into a pre-cleaned cooler. Live clams were selected and rinsed to remove adhered sediments, then placed into a second pre-cleaned cooler for temporary storage. The dredging process was repeated until a sufficient number and volume of clams was collected to support all analyses, but for two sites the masses collected were not sufficient to do the analyses at the targeted detection limits; affected results include the following comment "MDL elevated due to limited sample mass collected" (Deviation Form 2020-11). Each collected clam had its length, width, and weight recorded, and was then sorted into an approximate size class. Clams were randomly assigned to groups for each sample, with approximately the same proportion of each size class as found overall within the site. Samples were shipped frozen with their CoCs to the analytical lab.

For the bivalve collections, handheld portable YSI instruments were taken to the field to record water quality parameters of temperature, pH, dissolved oxygen concentration and percent saturation, specific conductivity, and turbidity at each site.

Sample Preparation and Analytical Methods

Water

Vista analyzed samples for PFAS (PFOA and PFOS) in water using Vista SOP 49 Rev. 22, a lab modification of EPA Method 537 for determination of PFAS in Drinking Water by

Solid Phase Extraction and Liquid Chromatography/Tandem Mass Spectrometry (LC/MS/MS). Target analytes were loaded by passing the collected samples, spiked with internal standards, through a solid phase extraction cartridge, which was then eluted with solvent. The extract was concentrated to a reduced final volume, and the final extract analyzed on the LC/MS/MS system.

Vista subcontracted measurement of galaxolide in aqueous samples to Physis, which used a lab modification of EPA 625.1 (Base/Neutrals and Acids by GC/MS) for analysis. In the EPA method, a measured volume of sample is serially extracted with methylene chloride at pH 11 - 13 and again at a pH less than 2 using a separatory funnel or continuous liquid/liquid extractor. The extract is concentrated to a reduced volume, and analyzed by GC/MS. Qualitative identification of an analyte is made using the retention time and the relative abundance of two or more characteristic masses (m/z's), and quantified using an internal standard technique.

Weck analyzed water samples using their internal SOP ORG111.R4.0, for Determination of Endocrine Disrupting Compounds, Pharmaceuticals, and Personal Care Products. The method is a variant of EPA Method 1694. Solid phase extraction (SPE) was used for aqueous samples, with the extract quantified by liquid chromatography and electrospray ionization tandem mass spectrometry (LC- ESI/MS/MS) or atmospheric pressure chemical ionization tandem mass spectrometry (LC-APCI/MS/MS). Isotopic dilution was used as an attempt to account for effects from the analytical process and matrix interferences.

Weck also analyzed water samples for suspended sediment concentration (SSC) using a method derived from ASTM D3977. Suspended solids are separated from water samples, dried, and weighed.

Sediment

SGS AXYS received sediment samples for CECs, which were frozen after receipt for storage until analysis. After samples were removed from frozen storage, they were thawed, and samples were homogenized following SGS AXYS SOP SLA-013 Rev. 10 "Procedures for Homogenization of Solids and Tissues". Samples were homogenized within their containers to minimize contamination, then aliquots of appropriate size removed for analysis.

SGS AXYS analyzed sediment samples for PBDEs using AXYS method MLA-033 Rev. 06 "Analytical Method For The Determination Of Brominated Diphenyl Ethers (BDE) And Other Brominated Flame Retardants (BFR)", a lab modification of EPA Method 1614A. Samples were spiked with ¹³C-labelled surrogate standards before analysis, then solvent extracted. The extracts were cleaned up by column chromatography, reduced to a final extract, and analyzed by high-resolution gas chromatography with high-resolution mass spectrometric detection (HRGC-HRMS).

SGS AXYS analyzed sediment samples for PFAS using AXYS method MLA-110 Rev. 02 "Analytical Procedure for the Analysis of Per- and Polyfluoroalkyl Substances (PFAS) in Aqueous Samples, Solids, Tissues, AFFF Products and Solvent Extracts by LC-MS/MS." After spiking with isotopically labeled surrogate standards samples were solvent extracted and cleaned up by Solid Phase Extraction (SPE). The extracts were then analyzed by liquid chromatography/mass spectrometry (LC-MS/MS). Final sample concentrations were determined by isotope dilution/internal standard quantification.

Weck analyzed sediment samples for TOC using a modified version of EPA Method 9060. Organic carbon is measured using a carbonaceous analyzer. This instrument converts the organic carbon in a sample to carbon dioxide (CO_2) which is then measured by a detector.

Fish Tissue

MPSL generated fish tissue composites from the collected fish. Fish selected for analysis (of the collected species, only Sacramento sucker and channel catfish were chosen) were dissected skin-off, with only the fillet muscle tissue used to generate composite samples to send to the analytical laboratory. Fish tissue samples were shipped with their CoCs in coolers with ice packs to SGS AXYS.

Upon receipt of the chilled fish composites at the analytical laboratory SGS AXYS, samples were frozen and stored in the dark in clean amber glass jars with screw caps at -20°C prior to analysis. After composite samples were removed from frozen storage at SGS AXYS, they were thawed and processed using the same SOPs for homogenization (SOP SLA-013 Rev. 10) and analysis of PBDEs (MLA-033 Rev. 06) and PFAS (MLA-110 Rev. 02) as used for sediment samples.

Bivalve Tissue

SGS AXYS received whole bivalves shipped frozen. Bivalves were removed from their shells and homogenized following the SOP SLA-013 Rev. 10. The SOP specifies various alternatives for homogenization depending on the sample material and size; due to the small mass of bivalve tissue in the samples, samples were manually homogenized using lab scissors and forceps to minimize material loss. Following homogenization, samples were analyzed for PBDEs using MLA-033 Rev. 06.

Parameter Group	Collection Agencies	Lab Agency	Collection Method	Preparation/ Preservation	Digest Extract Method	Analytical Method
Sediment PBDE	SFEI, SWRCB-SWAMP-SPoT	SGS AXYS	Sed_Core	LabFrozen	AXYS MLA-033 Rev 06	AXYS MLA-033 Rev 06
Sediment PFAS	SFEI, SWRCB-SWAMP-SPoT	SGS AXYS	Sed_Core	LabFrozen	SGS AXYS MLA-110 Rev 02	SGS AXYS MLA-110 Rev 02
Sediment TOC	SFEI, SWRCB-SWAMP-SPoT	WKL	Sed_Core	None	None	EPA 9060M
Water Galaxolide	DWR-MWQI, SFEI	Physis	Water_Grab	None	EPA 625	EPA 625.1M
Water PFAS	DWR-MWQI, SFEI	VAL	Water_Grab	None	EPA 537M	EPA 537M
Water PPCPs	DWR-MWQI, SFEI	WKL	Water_Grab	FieldAcidified	EPA 3535	EPA 1694M
Water SSC	DWR-MWQI, SFEI	WKL	Water_Grab	None	None	ASTM D3977M

Table 2Sample collection, preparation, and analysis methods and agencies for water and
sediment samples

Table 3Sample collection, preparation, and analysis methods and agencies for tissue
samples

Parameter Group	Collection Agency	Compositing Agency	Lab Agency		Preparation/ Preservation	Digest Extract Method	Analytical Method
Bivalve PBDE	AMS-CA	SGS AXYS	SGS AXYS	Trawl	FieldFrozen, LabFrozen		AXYS MLA-033 Rev 06
Fish PBDE	MPSL-DFW	MPSL-DFW	SGS AXYS	Shock	Skin off, LabFrozen		AXYS MLA-033 Rev 06
Fish PFAS	MPSL-DFW	MPSL-DFW	SGS AXYS	Shock	Skin off, LabFrozen	SGS AXYS MLA-110 Rev 02	SGS AXYS MLA-110 Rev 02

Quality Assurance

Additional details of the quality assurance review of the data for this CEC study are provided in <u>Appendix 3</u>. In that review, individual QC samples that failed MQOs were flagged using CEDEN QACodes. This section provides a high level summary of that review.

Field and Analytical Completeness

In the first water sampling event, completeness issues were primarily insufficient counts of lab QC samples for PPCPs due to insufficient material collected. For the second water event, only 3 of 8 planned sites were sampled, as one team could not sample due to COVID-19 restrictions. For clam sampling one site was inaccessible and was not sampled. For sediment TOC analysis, although a matrix spike/matrix spike duplicate (MS/MSD) pair was reported as one measure of lab precision, no unspiked lab replicate was reported. Several fish tissue results for N-methyl perfluorooctane sulfonamide ethanol(N-MeFOSE) and N-ethyl perfluorooctane sulfonamide ethanol (N-EtFOSE) were flagged as not quantitative and not reported (rejected) by the lab due to poor surrogate recoveries (<8% recovery). The remaining desired field and lab QC samples were successfully collected and analyzed.

Precision and Accuracy for Field and Laboratory QC

Recoveries for water galaxolide MS/MSDs ranged 104% to 264%, many over the 50-150 target. Although LCS samples met recovery targets, there were numerous very high MS recoveries, which were flagged. No deviations were found for PFAS recovery or precision in water samples. In PPCP LCS samples, BPA was recovered up to 4x of its expected value, and an ibuprofen LCS had 163% recovery. BPA and iopromide also had a few MS/MSD recoveries outside of the target 50-50% range. Thus, although BPA may be among the most often detected PPCPs, its quantitation may be uncertain.

In sediment samples, PBDE recoveries in LCS and MS samples met the target 70-130% recovery range. RPDs for PBDE 209 also exceeded the MQO of <35% in replicate samples from 519DRYCRK (125%) and replicate analyses of sediment from 511SOL011 (175%). Of the PFAS, Perfluoro-3,6-dioxaheptanoate had one MS recovery of 182% and one LCS at 141%, exceeding the 70-130% target, which were flagged. No deviations from TOC recovery or precision targets were found.

Tissue PBDE recoveries in LCS and MS samples were all within the target 70-130% recovery range. Of the PFAS, Methyl-perfluorooctanesulfonamidoethanol, N- also had 326% recovery in one LCS and was flagged. Some MS recoveries were high for 5:3 Fluorotelomer Carboxylic Acid (up to 194%), and 7:3 Fluorotelomer Carboxylic Acid (152%), and low for Perfluorododecanesulfonate (27%), outside the 50-150% target range, and were flagged in those samples. MS/MSD precision RPDs were above the target 25% for PBDE 209 (39%) and Ethyl-perfluorooctanesulfonamidoethanol, N- (46%).

However, the recoveries and precision on the most abundant PBDE compounds and PFOS had no deviations.

Blank Contamination

Blank contamination was encountered for a number of CECs. For water samples, Galaxolide was detected in 3 of 4 blanks (maximum 145 ng/L), a similar magnitude as sites with lower concentration samples, but the highest field sample concentrations were much higher than in blanks. PFOA and PFOS were not detected in blank water samples. Of the PPCPs, Bisphenol A, had blank concentrations similar to those in many field samples, with concentrations up to 180 ng/L. One field blank had measured SSC of 21 mg/L and was flagged.

PBDEs 047, 099, 100, and 154 were found in the sediment blank and flagged in that sample, but field sample concentrations averaged more than 100x higher so were likely minimally impacted. None of the PFAS were detected in the sediment blank.

For the tissue blank, PBDEs 047, 099, 100, 154, and 209 were found. PFOS, undecanoate, and tridecanoate, were also detected and flagged in the blank. Blank contamination likely impacted the results in all species for PBDE 209, and Sacramento sucker for PBDE 099, as the blanks accounted for more than ¹/₃ of the concentrations in the field samples. The Perfluoroundecanoate blank was also over ¹/₃ the field sample result for one Sacramento sucker and one channel catfish sample, so those results may be noticeably impacted.

Corrective Actions

After the first water collection event in which insufficient material to generate lab QC samples, field and lab procedures were altered so sufficient material was available for subsequent events. Other deviations such as variable recovery and precision occurred sporadically and are generally difficult to reproduce consistently to diagnose causes, so no specific corrective actions were identified. Similarly the blank contamination found for chemicals such as BPA and PBDEs, are compounds commonly found in many products, so their sources are difficult to fully identify and eliminate in both lab and field environments.

Results

All analytical and field parameter results are available for download through the CEDEN database (https://ceden.waterboards.ca.gov/AdvancedQueryTool) using the sampling event and station identification information found in Table 1.

Water

<u>Appendix 5</u> presents a tabulation of results for all of the parameters measured in water samples.

With quarterly water collections at eight sites (three of the sites sampled four times and five of the sites sampled three times), each target water analyte was analyzed in 27 field samples (not including QA samples).

There were no detections of Triclosan, Diclofenac, Estrone, or Estradiol, 17beta- in any of the water samples collected. Ibuprofen was detected at three of the eight sites, and in 5 of 27 samples overall, with concentrations ranging from 13 ng/L to 80 ng/L. Bisphenol A was detected at every site, and in 15 of 27 samples overall, with concentrations ranging from 12 ng/L to 330 ng/L. However, the lowest concentration samples were in a similar range as seen for lab blanks, so those concentrations are uncertain. Results for 7 additional secondary PPCP parameters were also reported for the water samples, as part of the suite of analytes included in the analytical method. Concentrations detected for these secondary PPCP parameters are listed along with the primary target analytes in Appendix 5. The reported detections were generally in a similar range as reported in the literature for other freshwater bodies: salicylic acid in some rivers were over 200 ng/L (https://pubs.acs.org/doi/10.1021/acs.chemrev.8b00299), similar to the maximum in this study of ~500 ng/L; a compilation of naproxen in various worldwide freshwater bodies (https://doi.org/10.1007/s00253-019-10343-x) generally reported concentrations around 1ug/L or lower; some reported ibuprofen data

(<u>https://doi.org/10.1016/j.heliyon.2020.e04087</u>) were mostly <1ug/L, so results here often were a similar order of magnitude..

Galaxolide was detected at every site, and in every water sample collected, with concentrations ranging from 55.6 ng/L to 47100 ng/L. The four highest concentrations were all detected at Old Alamo Creek at Lewis Road, with the lowest concentration detected at that site being 33900 ng/L. Galaxolide lab and field blanks ranged up to 145 ng/L, so blank contamination may have impacted some of the lower concentration sites, but were negligible compared to the Old Alamo Creek results. A study in Toronto (DOI: 10.1039/C8EM00341F) with measurements from creeks had concentrations <1000 ng/L, and wastewater effluents >10000 ng/L, so the Old Alamo Creek results are consistent with wastewater influence.

PFOS (*reported as Perfluorooctanesulfonic acid*) was quantified (above the Reporting Limit) for water at three of eight sites, and in 11 of 27 water samples overall, and detected but not quantified (below the Reporting Limit) in one sample at one other site. Quantified concentrations of PFOS ranged from 2.35 ng/L to 11.7 ng/L. PFOA (*reported as Perfluorooctanoic acid*) was quantified (above the Reporting Limit) for water at two of eight sites, and in 8 of 27 water samples overall, and detected but not quantified (below the Reporting Limit) in four samples at two other sites. Quantified concentrations of PFOA ranged from 2.21 ng/L to 10.3 ng/L. San Francisco Bay concentrations reported by the Bay RMP (downloaded from cd3.sfei.org) were in a similar range: PFOS averaged 6 ng/L and PFOA 15 ng/L.

Suspended sediment concentration (SSC) was measured as an ancillary parameter, and was detected in 15 of 27 water samples, at 6 of 8 sites, in concentrations ranging from 5 mg/L dw to 64 mg/L.

The following ranges in field water quality parameters were measured in Delta surface water over the 4 sampling events: temperature = 6.81-25.72 °C; pH = 6.7-9.2; dissolved

oxygen = 3.79-22.18 mg/L; dissolved oxygen = 46.6-177 % saturation; specific conductivity = $0.11-1091 \mu$ S/cm; turbidity = 0-7.6 FNU and 2.7-49.6 NTU. Field habitat observations were also recorded at each sample site and event, and are available for download through the CEDEN database.

Sediment

<u>Appendix 6</u> presents a tabulation of results for all of the parameters measured in sediment.

PBDE 047 was detected at all three sites sampled for sediment, in concentrations ranging from 0.0153 ng/g dw to 0.721 ng/g dw. PBDE 099 was detected at all three sites sampled for sediment, in concentrations ranging from 0.0165 ng/g dw to 0.561 ng/g dw. For both PBDE 047 and PBDE 099, the highest concentrations were detected at Old Alamo Creek at Lewis Road and the lowest at American River at Discovery Park.

Results for six additional secondary PBDE parameters were also reported for the sediment samples, as part of the suite of analytes included in the analytical method (PBDE 028/33, PBDE 100, PBDE 153, PBDE 154, PBDE 183, PBDE 209). Concentrations detected for these secondary PBDE parameters are listed along with the primary target analytes in <u>Appendix 6</u>. The reported PBDE concentrations are in a similar range as reported for San Francisco Bay (cd3.sfei.org), with individual PBDE congeners typically < 1ng/g dw in sediment.

PFOS (reported as Perfluorooctanesulfonate) was detected but not quantified (below the Reporting Limit) at one of the three sites sampled, and not detected at the other two sites. PFOA (reported as Perfluorooctanoate) was not detected in any of the sediment samples collected. The concentrations were lower than in San Francisco Bay, where the maximum detected PFOS concentration was <4 ng/g dw in sediment.

Results for 38 additional secondaryPFAS parameters were also reported for the sediment samples, as part of the suite of analytes included in the analytical method. Concentrations detected for these secondaryPBDE parameters are listed along with the primary target analytes in <u>Appendix 6</u>.

Total organic carbon (TOC) was measured as an ancillary parameter, with concentrations ranging from 474 mg/Kg dw to 4560 mg/Kg dw at the three sediment sites.

The following ranges in ancillary field water quality parameters were measured in Delta surface water during sediment sampling: temperature = $19.82-25.72^{\circ}$ C; pH = 7.41-8.4; dissolved oxygen = 3.79-8.51 mg/L; dissolved oxygen = 46.6-75.8% saturation; specific conductivity = $0.112-65.9 \mu$ S/cm; turbidity = 4.5-6.4 NTU. Neither turbidity or dissolved oxygen (% saturation) were recorded at the site and event where SWRCB-SWAMP-SPoT collected sediment. Field habitat observations were also recorded at each sample site and event, and are available for download through the CEDEN database.

Fish

<u>Appendix 7</u> presents a tabulation of results for all of the parameters measured in fish.

PBDE 047 was detected at all four sites sampled, in concentrations ranging from 1.62 ng/g dw to 55.5 ng/g dw. PBDE 099 was detected at three of four sites sampled, in concentrations ranging from 0.0125 ng/g dw to 2.87 ng/g dw. The reported concentrations are generally comparable to those in fish from San Francisco Bay (cd3.sfei.org) with a maximum PBDE 047 of 27 ng/g ww, and maximum PBDE 099 of 1.2 ng/g ww for Shiner Surfperch (in the period 2000-2019).

Results for six additional secondaryPBDE parameters were also reported for the fish samples, as part of the suite of analytes included in the analytical method (PBDE 028/33, PBDE 100, PBDE 153, PBDE 154, PBDE 183, PBDE 209). Concentrations detected for these secondaryPBDE parameters are listed along with the primary target analytes in <u>Appendix 7</u>.

PFOS (*reported as perfluorooctanesulfonate*) was quantified (above the Reporting Limit) for fish from three of four sites sampled, and detected but not quantified (below the Reporting Limit) for the fourth site. Quantified concentrations of PFOS ranged from 3.72 ng/g dw to 7.99 ng/g dw. PFOA (*reported as Perfluorooctanoate*) was not detected in any of the fish samples collected. PFOS concentrations in San Francisco Bay fish were higher, averaging up to 10 ng/g ww in some fish species; bioaccumulation will differ by species, but results appear to be a similar order of magnitude.

Results for 38 additional secondary PFAS parameters were also reported for the fish samples, as part of the suite of analytes included in the analytical method. Concentrations detected for these secondary PBDE parameters are listed along with the primary target analytes in <u>Appendix 7</u>.

Ancillary field water quality parameters were not measured in Delta surface water during fish sampling (see deviation form 2020-06 in Appendix 4). Field habitat observations were recorded at each sample site and event, and are available for download through the CEDEN database.

Bivalve

<u>Appendix 8</u> presents a tabulation of results for all of the parameters measured in clams.

PBDE 047 was detected at all five sites sampled, in concentrations ranging from 7.51 ng/g dw to 131 ng/g dw. PBDE 099 was detected at all five sites sampled, in concentrations ranging from 1.65 ng/g dw to 70.9 ng/g dw. For both PBDE 047 and PBDE 099, the highest concentrations were detected at Sacramento River at Hood Monitoring Station Platform and the lowest at Sacramento River at Elkhorn Boat Launch Facility.

Results for six secondary PBDE parameters were also reported for the clam samples, as part of the suite of analytes included in the analytical method (PBDE 028/33, PBDE 100,

PBDE 153, PBDE 154, PBDE 183, PBDE 209). Concentrations detected for these secondary PBDE parameters are listed along with the primary target analytes in <u>Appendix 8</u>.

The following ranges in ancillary field water quality parameters were measured in Delta surface water during clam sampling: temperature = 17.5-21.5°C; pH = 6.96-8.06; dissolved oxygen = 7.04-9.58 mg/L; dissolved oxygen = 79.9-101.9% saturation; specific conductivity = 57.4-620 µS/cm; turbidity = 0.02-4.9 FNU. Field habitat observations were also recorded at each sample site and event, and are available for download through the CEDEN database.

References

- Heberger, M., D. Yee, J. Yin, M. Weaver, A. Wong, A. Gilbreath, and A. Franz. 2020. Pilot Study of Constituents of Emerging Concern in the Sacramento-San Joaquin Delta, Quality Assurance Project Plan, version 1.0. Delta Regional Monitoring Program.
- Larry Walker Associates. 2018. Central Valley Pilot Study for Monitoring Constituents of Emerging Concern (CECs) Work Plan. Larry Walker Associates, Davis, CA. https://www.sfei.org/sites/default/files/programs/drmp/drmp-wq/drmp_cec_pilot_st udy.pdf
- Tadesse, Dawit. 2016. Constituents of Emerging Concern (CECs): Statewide Pilot Study Monitoring Plan. State Water Resources Control Board. <u>https://www.waterboards.ca.gov/water_issues/programs/swamp/cec_aquatic/docs/oi</u> <u>ma_sw_cec_mon_plan.pdf.</u>
- Marine Pollution Studies Laboratory Staff. 2020. Appendix 1 Cruise Report for the Delta Regional Monitoring Program (DRMP) Pilot Study Work Plan Monitoring For Constituents of Emerging Concern. Marine Pollution Studies Laboratory at Moss Landing Marine Laboratories, San Jose State University.
- Applied Marine Sciences. 2020. Appendix 2 Cruise Report 2020 Delta RMP Clam Sampling. Applied Marine Sciences, Livermore, CA.