Draft
Quality Assurance Project Plan

for

# CENTRAL COAST LONG-TERM ENVIRONMENTAL ASSESSMENT NETWORK

Revised May 2024

# Group A: Project Management

## 1. TITLE AND APPROVAL SHEETS

Quality Assurance Project Plan

For

PROJECT NAME: Central Coast Long-term Environmental Assessment Network

Version 9.0
Date May 2024

NAME OF RESPONSIBLE ORGANIZATION Central Coast Long-term Environmental Assessment Network

## APPROVAL SIGNATURES

CCLEAN:

| Title: | Name: | Signature: | Date: |
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| Technical Advisor | Dane Hardin | | |
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REGIONAL BOARD:

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| CCLEAN organics and conventional analyses | Misty Mercier (Physis Environmental Laboratories; Physis) | 714-602-5320 | 9 |
| CCLEAN nutrient samples | Steve Cunningham (Moss Landing Marine Laboratories) | 916-730-9299 | 9 |
| City of Watsonville Contact, analysis of TSS samples | Bryan Condy (City of Watsonville) | 831-768-3179 | 9 |
| Benthic Analysis | Jim Oakden (Coastal Conservation and Research; CCR) | 831-479-0277 | 9 |
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# 4. PROJECT/TASK ORGANIZATION

### 4.1. Involved parties and roles.

The Central Coast Long-term Environmental Assessment Network (CCLEAN) is a long-term monitoring program designed to help municipal agencies and resource managers protect the quality of ocean waters in the Monterey Bay area. Begun in 2001, CCLEAN determines the sources, amounts and effects of contaminants reaching ocean waters. If the kinds and amounts of contaminants measured are impairing ocean waters, the information provided by CCLEAN will enable resource managers to implement corrective actions. CCLEAN is supported by the City of Santa Cruz, the City of Scotts Valley, the City of Watsonville, Moss Landing Power Plant, Monterey One Water, and Carmel Area Wastewater District, under the auspices of the Central Coast Regional Water Quality Control Board.

The parties involved in implementation of CCLEAN and their responsibilities are shown in Table 1.

Table 1. CCLEAN personnel responsibilities.

| Name | Organizational Affiliation | Title | Contact Information (Telephone number, email address) |
|---|---|---|---|
| Aroon Melwani | AMS | CCLEAN Program Director | 831-917-9243 amelwani@amarine.com |
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### 4.2. Quality Assurance Program Plan (QAPP)

This QAPP consists of the systems and plans necessary to provide adequate confidence that the studies and projects that CCLEAN sponsors and/or executes will meet program and study objectives satisfactorily and efficiently. The goal of quality assurance (QA) is to ensure that monitoring, research, and analytical activities are performed in a controlled manner, and maintained according to sound and defensible technical specifications, quality practices that ensure valid and retrievable data. QA also includes the quality control, which comprises all those actions necessary to verify the characteristic features of program elements and the resulting data.

### 4.3. Persons responsible for QAPP management

The Program Director of CCLEAN is responsible for maintaining and updating the QAPP, with the assistance of the CCLEAN QA Officer. Because the Program Director and QAO are not involved in data collection activities, as these data are instead generated by others, including program participants and subcontractors (see Section 4.4), the CCLEAN Program Director and QA Officer are largely independent of the entities generating the data.

The maintenance activities include:

a) Ensuring in concert with the CCLEAN lead agency and chairperson that contracting laboratories implement QA elements consistent with CCLEAN study and program objectives;
b) Coordinating QA elements relevant to CCLEAN projects and studies with contracting laboratories;
c) Overview of relevant quality assurance implementation plans relating to CCLEAN projects by contributing agencies;
d) Verify that QA requirements have been considered in conceptual stages of study plans; assure that project and study costs account for quality assurance and quality control;
e) Ensure that corrective actions consistent with CCLEAN QAPP are taken for all flags and other quality control defects; and
f) Provide an annual QA audit for the review of CCLEAN steering committee prior to finalizing annual and/or project reports.

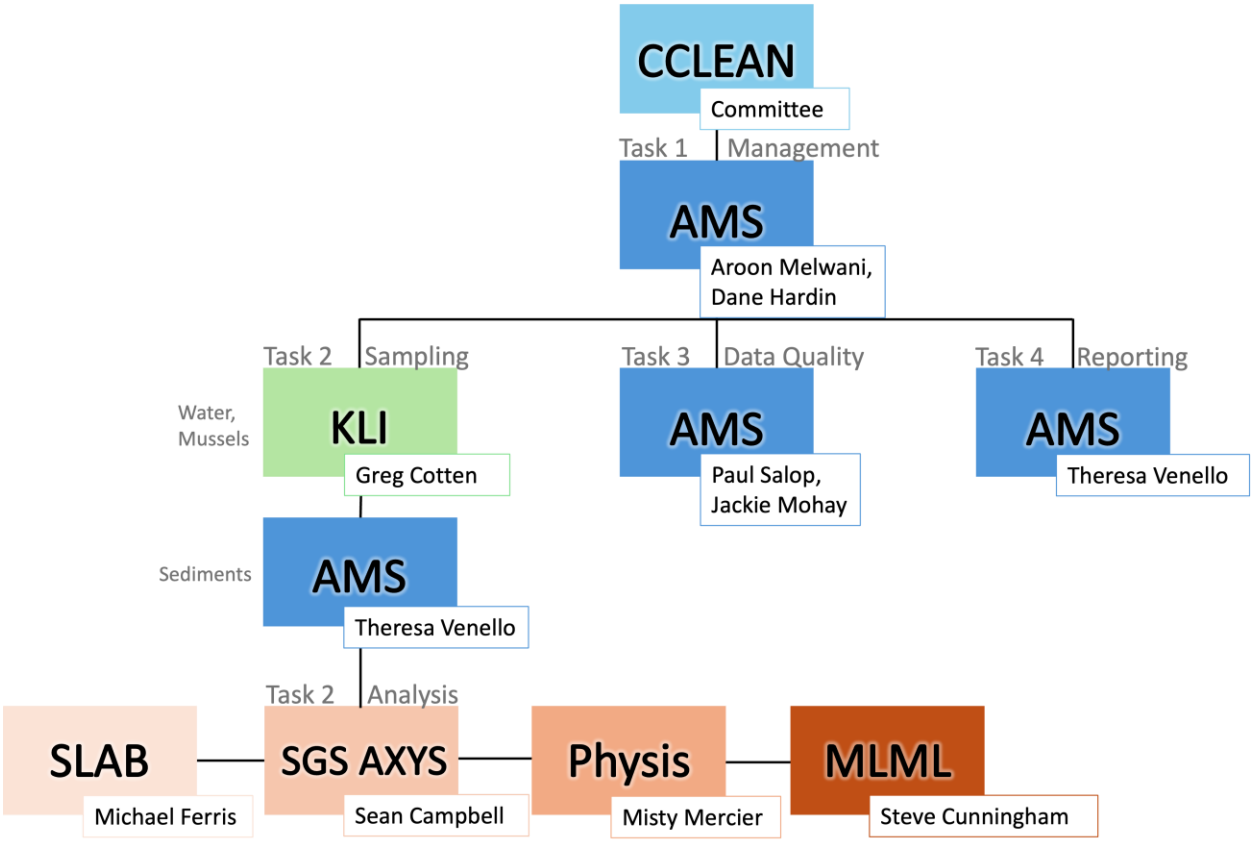
CCLEAN Base Program Contractors are responsible for:

a) Developing and implementing QA programs consistent with CCLEAN study objectives contracted to their organizations;
b) Preparing and producing QA audits of CCLEAN studies contracted to their organizations, when requested by CCLEAN;
c) Performing all corrective actions as indicated by CCLEAN's QAPP for studies and projects under their respective contracts.

### 4.4. Organizational chart and responsibilities

The organizational chart for the CCLEAN program is shown in Figure 1.

Figure 1. Organizational Chart for CCLEAN



# 5. PROBLEM DEFINITION/BACKGROUND

## 5.1. Problem statement

The complexity of environmental issues affecting ocean waters today have led to a general agreement that their protection is only possible by implementing regional approaches to monitoring and resource management. Ocean waters are affected by point-source discharges, storm runoff, rivers, discharges from ships, and aerial deposition. At the same time, many marine resources are diminishing under pressure from increasing usage. In the late 1990s, multiple agencies in the Monterey Bay area began working toward implementation of a regional approach to monitoring watersheds and marine waters.

The Central Coast Long-term Environmental Assessment Network (CCLEAN) is a long-term monitoring program designed by program participants through a commitment to environmental stewardship in order to fulfill several regulatory objectives.

CCLEAN is currently funded by the City of Santa Cruz, the City of Scotts Valley, the City of Watsonville, Moss Landing Power Plant, Monterey One Water, and Carmel Area Wastewater District, under the direction of the Central Coast Regional Water Quality Control Board (hereafter Water Board). CCLEAN fulfills a significant component of the subscribing agencies' compliance to their NPDES monitoring commitments. In addition, it represents a significant portion of their contributions to their communities' efforts at sustainability of their coastal environments. CCLEAN is also the current mechanism by which the Water Board fulfills part of its obligations under a monitoring framework developed to provide an ecosystem-based Water Quality Protection Program for the Monterey Bay National Marine Sanctuary (the Sanctuary). The monitoring framework evolved to fulfill the Water Board's obligations to the Management Plan for the Sanctuary. The Sanctuary's Management Plan includes a Memorandum of Agreement among eight federal, state, and regional agencies (including the Water Board). The Water Board's framework for partial fulfillment of this Water Quality Protection Program is the Central Coast Ambient Monitoring Program (CCAMP). This multidisciplinary program includes sampling in watersheds that flow into coastal regions, in estuarine coastal confluences, and at coastal sites. The goal of CCAMP is to "collect, assess, and disseminate scientifically based water quality information to aid decision-makers and the public in maintaining, restoring, and enhancing water quality and associated beneficial uses." CCLEAN provides the initial ocean component of CCAMP. CCLEAN has been underway since 2001 and its QAPP is being revised to incorporate recent program changes, and to retain consistency with the Water Board Surface Water Ambient Monitoring Program (SWAMP) requirements for data comparability.

Within the framework of CCAMP, the goal of the CCLEAN program is to assist stakeholders in maintaining, restoring, and enhancing ocean water and sediment quality and associated beneficial uses in the Central Coast Region. The program's objective is to use high-quality data to address the following questions and objectives:

1. What are the status and long-term trends in the quality of ocean waters, sediments, and associated beneficial uses?
2. Do ocean waters and sediments comply with the California Ocean Plan and associated NPDES permits?
3. What are the major sources of contaminants to ocean waters?
4. What are the effects of wastewater discharges in ocean waters?
5. Manage the program adaptively to ensure cost effectiveness and response to emerging issues of concern to water quality managers.
6. Develop a long-term database on trends in the quality of ocean waters, sediments and associated beneficial uses.
7. Ensure that the database is compatible with other regional monitoring efforts and regulatory requirements.
8. Ensure that data are presented in ways that are understandable and relevant to the needs of stakeholders.

The questions lend themselves to hypothesis testing, which is the basis of program decision making, whenever possible. For example, determination of trends in contaminant concentrations in ocean waters, sediments and associated beneficial uses can be made by testing the null hypothesis to determine if no changes have occurred over time in the concentrations of contaminants or level of impairment by using either linear regression or a Seasonal Kendall Test. Specific examples of how the data will lead to outcomes and the applicable criteria for determining impairments are discussed in sections 5.2 and 5.3.

The CCLEAN program and decision-making process includes a commitment to adaptive management. This ensures the flexibility needed to add or delete program elements in response to previous findings or emerging concerns. For example, the CCLEAN Steering Committee implemented measurements of polybrominated diphenyl ethers (PBDEs) in 2006, screening for pyrethroids and fipronils in 2015, and funded a study of reproduction disrupting activity in wastewater in 2009, while reducing resources allocated to riverine monitoring.

**5.2. Decisions or outcomes**

Data sets from CCLEAN are made available for scientific research, regulatory purposes, and public awareness. Examples of how the data will be used by CCLEAN are as follows:

- Trend analysis - Data may be used to investigate seasonal, annual, and long-term patterns in pollutants entering ocean waters by testing with linear regression or Seasonal Kendall Test.
- Objectives and Guidelines - Data may be used to evaluate the status of ocean waters, sediments, and fish and shellfish tissues, and whether they achieve various water, sediment, and tissue quality guidelines.
- Integrated Contaminant Measurements - Tissue contaminants and benthic community data may be used to determine time-averaged trends in contaminant concentrations and their effects, and for comparison with other trend data.

- Data may be used to assess the relative contributions of point and nonpoint sources of pollutants to Monterey Bay.
- Impairment of beneficial uses can be determined by comparing the number of exceedances to statistical criteria established by the State of California for listing water bodies on the California State Water Resources Control Board (SWRCB) Total Maximum Daily Load (TMDL) 303(d) list of Water Quality Limited Segments.

**5.3. Water quality or other criteria**

Data generated through CCLEAN will be used to determine whether ocean waters and sediments are in compliance with the California Ocean Plan, satisfy some of the NPDES receiving water monitoring and reporting requirements of Program participants, and inform the ongoing TMDL development process. Regulatory criteria and comparative data used by the program include the following:

- Water - California Ocean Plan and Basin Plan standards, California Toxics Rule values
- Sediment – National Oceanic and Atmospheric Administration (NOAA) Effects Range Low and Median, California Sediment Quality Objective (when available), San Francisco Bay comparative data
- Tissue – California State Mussel Watch elevated data levels (for the 85$^{th}$ and 95$^{th}$ percentiles (EDL 85 and 95), US Food and Drug Administration (USFDA) alert levels, USEPA recreational and subsistence fisher screening values, and California Office of Environmental Health Hazard Assessment (OEHHA) screening values, Bodega Head and San Francisco Bay comparative data.

# 6. PROJECT/TASK DESCRIPTION

### 6.1. Work statement and produced products

The CCLEAN monitoring program is designed to 1) determine the major sources of contaminants that are affecting beneficial uses in nearshore Monterey Bay waters, 2) estimate the loads of those contaminants, and 3) determine the potential effects of those contaminants. To achieve these goals, CCLEAN measures contaminants in wastewater influent, effluent, ocean waters, mussels, sediments, and benthic communities. Section 1 of the CCLEAN Monitoring Plan (CCLEAN 2024) describes the sites that are currently being sampled in the Program.

All monitoring data collected are submitted to the California Environmental Data Exchange Network (CEDEN) and available via the CEDEN portal (http://ceden.waterboards.ca.gov/AdvancedQueryTool). Results are synthesized into reports available online at http://www.cclean.org.

### 6.2. Constituents monitored and measurement techniques

The CCLEAN program involves multiple sampling components and measurement techniques (Table 2). Constituents monitored and the methods used are described in detail in Section 3 of the CCLEAN Monitoring Plan (CCLEAN 2024).

Table 2. Overview of sample types and collection techniques.

| Sample Type | Sampling Method |
|---|---|
| Influent, Effluent, and River Sampling | Flow-proportioned solid-phase extraction and grab samples. |
| Receiving Water Sampling | Grab sample |
| Mussel Sampling | Hand collected |
| Influent, Effluent, and River Sampling | Grab samples |
| Sediment Sampling | Van Veen sediment grab |
| Ocean Sampling | Time-integrated solid-phase extraction and grab samples |

### 6.3. Project schedule

Project schedules for the CCLEAN program are shown in Section 1 of the CCLEAN Monitoring Plan (CCLEAN 2024). CCLEAN reports are submitted annually to the Water Board by March 31 for the previous July–June period. As CCLEAN data are used for permit compliance, raw data for influent and effluent samples are made available to dischargers by January 31 for the previous July-June period.

### 6.4. Geographical setting

CCLEAN sampling sites span the Monterey Bay area from Scott Creek in the north to Carmel Bay in the south as illustrated in Figure 2.

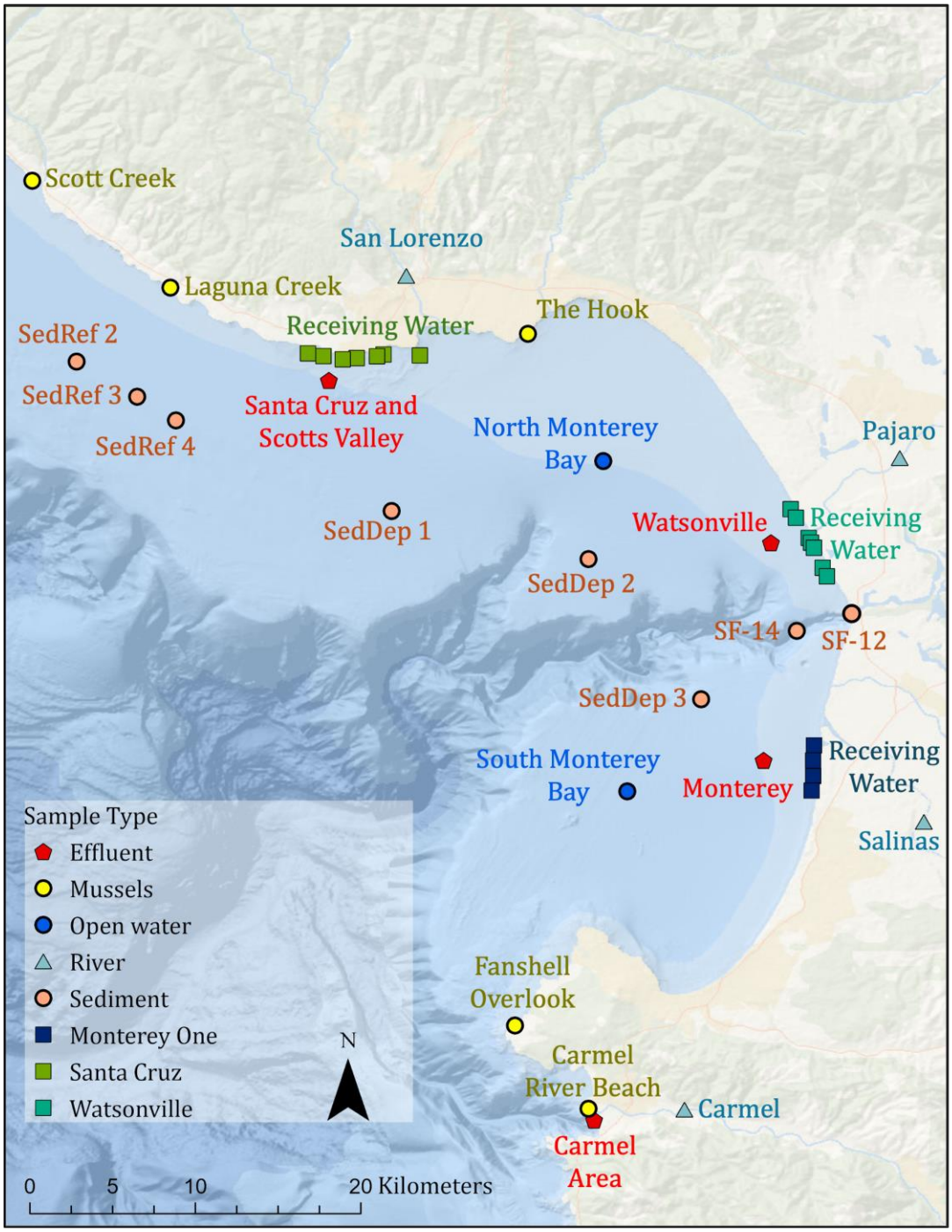



Figure 2. Map of all CCLEAN Monitoring Sites

# 7. QUALITY OBJECTIVES AND CRITERIA FOR MEASUREMENT DATA

The quantitative measurements that estimate the true value or concentration of a physical or chemical property always involve some level of uncertainty. The uncertainty associated with a measurement generally results from one or more of several areas: (1) natural variability of a sample; (2) sample handling conditions and operations; (3) spatial and temporal variation; and (4) variations in collection or analytical procedures. Stringent QA and QC procedures are essential for obtaining unbiased, precise, and representative measurements and for maintaining the integrity of the sample during collection, handling, and analysis, as well and for measuring elements of variability that cannot be controlled. Stringent procedures also must be applied to data management to assure that accuracy of the data are maintained. Further information on quality control can be found in Section 7 of the CCLEAN Monitoring Plan (CCLEAN 2024).

Data Quality Objectives (DQOs) are established to ensure that data collected are sufficient and of adequate quality for the intended use. DQOs include both quantitative and qualitative assessment of the acceptability of data. The qualitative goals include representativeness and comparability, and the quantitative goals include completeness, sensitivity (detection and quantization limits), precision, accuracy, and contamination.

CCLEAN DQOs are driven by the program's commitment to SWAMP data compatibility; and to specific project and study objectives. And because data sets generated by CCLEAN are used in more than one type of analysis, data quality objectives must be rigorous enough to address those analyses with the most stringent detection limits and the greatest needs for accuracy. For example, estimating loads based upon 30-day flow proportioned samples requires modest accuracies and detection limits, whereas comparing measured concentrations to California Toxics Rule or California Ocean Plan objectives requires detection limits at least as low as the applicable objectives. Moreover, many of the compounds being measured by CCLEAN are found in very low concentrations and comparably low detection limits are necessary to give reasonable confidence that undetected compounds are not present.

Data quality objectives for this project are specified in Tables Table 3 through Table 13 and will consist of the following:

### 7.1. Comparability

Comparability is the degree to which data can be compared directly to other relevant studies. All data collection through implementation of CCLEAN will also be performed in a manner so that data are comparable with California Surface Water Ambient Monitoring Program (SWAMP) protocols.

### 7.2. Representativeness

The representativeness of data is the ability of the sampling locations and the sampling procedures to adequately represent the true condition of the sample sites. CCLEAN samples are collected to represent concentrations and loads of contaminants at different locations and the

effects of time on those concentrations and loads (i.e., long-term or seasonal patterns). As such, CCLEAN sampling activities are designed to maximize both spatial and temporal representativeness. Spatial representativeness of effluent loads is ensured by sampling all the major wastewater discharges in the program area. In addition, a single influent sample is also collected at the City of Watsonville for comparison to the relevant effluent loads. Sediment and mussel samples are collected randomly from fixed locations to represent areas distant from and close to sources of contaminants. Because of limited resources, temporal representativeness for effluent and ocean waters is achieved by sampling in the dry season and wet season in order to capture the minimum and maximum effects of rainfall on discharges of contaminants to the ocean. While logistical considerations require that sampling be scheduled well ahead of time, representativeness of wet-season and dry-season periods is improved by using a 30-day sampling period for effluent and ocean waters. The influent sample at the City of Watsonville is also collected using 30-sampling period, but only during the dry season. Sediment samples are collected in the early fall each year to represent the maximum annual diversity of benthic organisms before winter storms disrupt bottom sediments. Mussel samples are collected in the wet season to represent the maximum likely accumulation of contaminants from winter runoff. Both sediment and mussel samples tend to integrate their exposure to contaminants over time preceding sample collection and those samples represent the antecedent period.

In addition to the above elements of the study design, sample representativeness is ensured by proper collection and handling procedures (see table 18). These procedures minimize sample degradation by use of preservatives, cooling and/or keeping the samples in darkness so that analytical results represent the original sample matrix as much as possible.

### 7.3. Accuracy

Accuracy describes the degree of agreement between a measurement (or the average of measurements of the same quantity) and an acceptable reference or true value. The “true” values of the parameters measured in the Program are unknown and the overall accuracy (including representativeness) cannot be assessed. Control limit criteria are therefore based on “relative accuracy”, which is evaluated for each analysis of the Certified Reference Material (CRM) or Laboratory Control Material (LCM) by comparison of a given laboratory’s values to the “true” or “accepted” values. For CCLEAN, the “accepted” values are defined as the 95% confidence intervals of the mean.

For CCLEAN analyses, analytical accuracy, characterized through the use of reference samples and laboratory matrix spikes in the laboratory operation, is considered acceptable for the overall accuracy of the Program. Accuracy is expressed as percent recovery for reference materials:

% Recovery = MV / EV

Where: MV = the measured value
EV= the true expected (reference) value.

For matrix spikes, recovery is calculated from the original sample result, the expected value (EV = native + spike concentration), and the measured value with the spike (MV):

% Recovery = [(MV – NV) / SV] x 100%

Where: MV = the measured value of the spiked sample
NV= the native, unspiked result
SV= the spike concentration added

Surrogate standards are also spiked into samples for some analytical methods and used to correct for losses in the analytical process. Although recoveries on surrogates are to be reported, control limits for surrogates are method and laboratory specific. Where applicable, data will be reported as surrogate-corrected values and flagged accordingly.

**7.4. Precision**

Precision is used to measure the degree of mutual agreement among individual measurements of the same property under prescribed similar conditions. Overall precision usually refers to the degree of agreement for the entire sampling, operational, and analysis system. It is derived from reanalysis of individual samples (laboratory replicates) or multiple collocated samples (field replicates) analyzed on equivalent instruments and expressed as the relative percent difference (RPD) or relative standard deviation (RSD). Analytical precision can be determined from duplicate analyses of field samples, laboratory matrix spikes, and/or reference material samples. The analytical precision of duplicate measurements of samples or spikes will serve as the overall precision for CCLEAN.

Analytical precision is expressed as the RPD for duplicate measurements.

RPD = ABS([X1 - X2] / [(X1 + X2) / 2])

Where: X1 = the first sample result
X2 = the duplicate sample result.

In cases where more than one replicate is measured from a single sample or taken from a given site (on a scale presumed to be homogenous), rather than deriving RPDs for each pairwise combination, RSD can instead be calculated:

RSD = [stdev (X,,X2 ,..XN)] / [average (X,, X2 , ..XN)]

Where: X1 = the first sample result
XN = each successive sample result

If the laboratory-reported RPD (or RSD) exceeds the target for over 30% of the parameters in an analysis, the analysis is rerun. If after rerunning the analysis, RPD (or RSD) for a substantial number of analytes still exceeds the target, the problem is further investigated to identify

whether potential problems originate in field sampling or laboratory handling and analysis. Additional corrective actions including flagging of data or reanalysis of samples are taken where possible and as needed.

In cases where there is insufficient field sample to analyze both lab duplicates and matrix spike duplicates, a duplicate of the unspiked sample is generally preferred, due to the possibility of spiking too high, resulting in precision measurement for a concentration range not found in typical samples. Analyzing a laboratory replicate for a field sample different from that used for matrix spikes can alleviate a problem of insufficient sample material. In extreme cases where there is sufficient material for only a single analysis of each sample from the Program, other samples such as blank spikes, reference materials, or samples from another project may be used to evaluate analytical precision, again with caveats on the relevance of evaluations for samples with much higher concentrations.

### 7.5. Completeness

Completeness is defined as the percentage of valid data collected and analyzed compared to the total expected to being obtained under normal operating conditions. Overall completeness accounts for both sampling (in the field) and analysis (in the laboratory). Valid samples include those for analytes in which the concentration is determined to be below detection limits. Completeness is expressed as overall completeness for a given parameter for each CCLEAN component. Under ideal circumstances, the objective is to collect 100% of all field samples desired, with successful laboratory analyses on 100% of measurements (including QC samples). However, circumstances surrounding sample collections and subsequent laboratory analysis are influenced by numerous factors, including weather, shipping damage or delays, sampling crew or lab analyst error, and QC samples failing DQOs. An overall completeness of greater than 90% is considered acceptable for the Program.

### 7.6. Sensitivity

Different indicators of the sensitivity of an analytical method to measure a target parameter are often used including instrument detection limits (IDLs), method detection limits (MDLs), and reporting limits (RLs). Each of these indicators is described below:

The IDL is the lowest concentration of analyte that an analytical instrument can detect that is statistically different from the response obtained from the background instrumental noise. The IDL indicates the absolute sensitivity of the analytical technique or instrument. It is established by adding the analyte to reagent blank water or solvent to give a concentration within a few times the estimated IDL and by calculating the standard deviation for seven or more replicate measurements. The IDL should be determined at least on a quarterly basis for all analyses, or more frequently as specified by laboratory SOPs. For some analytical methods, IDL is dynamically determined through analysis of the background noise during each analytical run.

The MDL is the lowest concentration of analyte in distilled water, solvent, or another appropriate clean matrix that a method can detect reliably and that is statistically different from a blank carried through the complete method, including extraction and pretreatment of

the sample. The MDL is specified based on replicate analyses of seven or more measurements with a specified confidence level and defined as three times the standard deviation of replicate analyses of a sample that is 1 to 5 times the estimated detection limit for the analyte of concern. The MDL should be determined at a minimum on an annual basis.

The RL, or practical quantification limit (PQL), is the lowest level at which measurements become quantitatively meaningful and which are achievable on a routine day-to-day basis. The RL is typically set as approximately three to four times the MDL or ten times the IDL, or may be defined as the concentration of the minimum calibration point (expressed in concentration units equivalent to those for field samples). Analytical measurements above the MDL but below the RL should be reported as measured, but may be qualified by the laboratory as estimated or detected but not quantified (DNQ).

For CCLEAN, the MDL is the measurement of primary interest as typical analyses, especially for POPs, involve lowest detection limit analyses possible. However, as not all analyses support calculation of statistically-derived MDLs and QA review for SWAMP comparability relies on RLs instead, data quality assessment of sensitivity will compare analytical sensitivities against SWAMP recommended targets, where available. As of the revision date of this QAPP, SWAMP RL targets are only available for a subset of analyses in aqueous samples, and are specified as multiple orders of magnitude above CCLEAN-identified MDLs for POPs (Table 20).

### 7.7. Contamination

Collected samples may inadvertently be contaminated with target analytes at many points in the sampling and analytical process, from the materials shipped for field sampling, to the air supply in the analytical laboratory. Blank samples evaluated at multiple points in the process chain help assure that pollutants measured in samples actually originated from the target matrix in the sampled environment and are not artifacts of the collection or analytical process.

Method blanks (also called laboratory reagent blanks, extraction blanks, procedural blanks, or preparation blanks) are used to assess laboratory contamination during all stages of sample preparation and analysis. The method blank will be processed through the entire analytical procedure in a manner identical to the samples. Method blanks should be less than the RL or not exceed a concentration of 10% of the lowest reported sample concentration. A method blank concentration greater than the RL or 10% of the lowest reported sample concentration will require corrective action to identify and eliminate the source(s) of contamination before proceeding with sample analysis. If eliminating the blank contamination is not possible, all impacted analytes in the analytical batch shall be flagged. In addition, a detailed description of the likely contamination source(s) and the steps taken to eliminate/minimize the contaminants shall be included in narrative of the data report. If supporting data are presented demonstrating sufficient precision in blank measurement that the 99% confidence interval around the average blank value is less than RL or 10% of the lowest measured sample concentration, then the average blank value may be subtracted.

Equipment blanks are generated by the personnel responsible for cleaning sampling equipment. Equipment blanks must be analyzed before the equipment is shipped to the sampling site. In order to accommodate any necessary corrective action, equipment blank results should be available well in advance of the sampling event. To ensure that sampling equipment is contaminant-free, water known to be low in the target analyte(s) must be processed though the equipment as during sample collection. The specific type of water used for blanks is selected based on the information contained in the relevant sampling or analysis methods. The water must be collected in an appropriate sample container, preserved, and analyzed for the target analytes (in other words, treated as an actual sample). The inclusion of field blanks is dependent on the requirements specified in the relevant MQO tables, or in the sampling method or SOP. Typically, equipment blanks are collected when new equipment, equipment that has been cleaned after use at a contaminated site, or equipment that is not dedicated for surface water sampling is used.

A field blank is collected to assess potential sample contamination levels that occur during field sampling activities. Field blanks are taken to the field, transferred to the appropriate container, preserved (if required by the method), and treated the same as the corresponding sample type during the course of a sampling event. The inclusion of field blanks is dependent on the requirements specified in the relevant MQO tables or in the sampling method or SOP. Field blanks for other media and analytes should be conducted upon initiation of sampling. If field blank performance is acceptable, further collection and analysis of field blanks should be performed on an as-needed basis.

### 7.8. Field Replicates and Field Split Samples

Field replicates assess precision throughout the entire sampling and analysis process. These incorporate variation that may be encountered associated with sample heterogeneity, sample collection and handling, and analytical method. As part of the quality assurance program of CCLEAN, replicate or split samples will be collected for sediment and mussel samples for subsequent chemical analysis. Field duplicates will be submitted as blind samples to the analytical laboratory.

Field split samples may also be collected to assess variability, with a focus on the analytical process. The protocol for managing split samples is identical to that of field duplicates, but will involve analysis by external laboratory(ies). Field splits also will be collected and sent blind to additional laboratories selected to participate in the split sample analysis.

Table 3. Data quality objectives for laboratory analysis of nutrients (ammonia, nitrate, orthophosphate, silica, urea) in water. The completeness objective for CCLEAN field and laboratory samples is 95%. Program action limit for ammonia is that in the California Ocean Plan, Table 3. There are no applicable action limits for the other constituents in either influent, effluent, or ocean water.

| **Laboratory Quality Control** | **Frequency of Analysis** | **Measurement Quality Objective** |
|---|---|---|
| Calibration Standard | Daily per analytical method or manufacturer's specifications | Per analytical method or manufacturer's specifications |
| Calibration Verification | Per 10 samples | 90-110% recovery |
| Laboratory Blank | Per 10 samples or per analytical batch, whichever is more frequent | <RL for target analyte |
| Reference Material | Per 25 samples or per analytical batch, whichever is more frequent | 90-110% recovery |
| Matrix Spike | Per 25 samples or per analytical batch, whichever is more frequent | 80-120% recovery |
| Matrix Spike Duplicate | Per 20 samples or per analytical batch, whichever is more frequent | 80-120% recovery RPD<25% for duplicates |
| Laboratory Duplicate | Per 10 samples or per analytical batch, whichever is more frequent | RPD<10% (n/a if native concentration of either sample<RL) |
| **Field Quality Control** | **Frequency of Analysis** | **Measurement Quality Objective** |
| Field Duplicate | 5% of total project sample count | RPD<25% (n/a if native concentration of either sample<RL) |
| Field Blank, Travel Blank, Equipment Blank | Per method | <RL for target analyte |

Table 4. Data quality objectives for laboratory analysis of solids parameters (TSS) in water. The completeness objective for CCLEAN field and laboratory samples is 95%. Program action limit for TSS is that in the California Ocean Plan, Table 4.

| **Laboratory Quality Control** | **Frequency of Analysis** | **Measurement Quality Objective** |
|---|---|---|
| Laboratory Blank | Per 20 samples or per analytical batch, whichever is more frequent | <RL for target analyte |
| Laboratory Duplicate | Per 20 samples or per analytical batch, whichever is more frequent | RPD<25% (n/a if native concentration of either sample<RL) |
| **Field Quality Control** | **Frequency of Analysis** | **Measurement Quality Objective** |
| Field Duplicate | 5% of total project sample count | RPD<25% (n/a if native concentration of either sample<RL) |
| Field Blank, Equipment Blank | Per method | <RL for target analyte |

Table 5. Data quality objectives for laboratory analysis of synthetic organic compounds (PCBs, PAHs, PBDEs, dioxins, furans, and organochlorine pesticides) in water. The completeness objective for CCLEAN field and laboratory samples is 95%. Program action limits for POPs in influent and effluent are those in each participant's NPDES permit, as applicable. Program action limits for POPs in ocean water are those in the California Ocean Plan, Table 3, as applicable.

| **Laboratory Quality Control** | **Frequency of Analysis** | **Measurement Quality Objective** |
|---|---|---|
| Tuning[1] | Per analytical method | Per analytical method |
| Calibration | Initial method setup or when the calibration verification fails | · If RSD<20%, average RF may be used to quantitate; otherwise use equation of the curve<br>· First- or second-order curves only (not forced through the origin)<br>· Minimum of 5 points per curve (one of them at or below the RL) |
| Calibration Verification | Per 12 hours | Expected response or expected concentration ±30% |
| Laboratory Blank | Per 20 samples or per analytical batch, whichever is more frequent | <RL for target analyses |
| Laboratory Control Sample | Per 20 samples or per analytical batch, whichever is more frequent | Per laboratory procedure. |
| Matrix Spike[1] | Per 20 samples or per analytical batch, whichever is more frequent | 50-150% or based on historical laboratory control limits (average±3SD) |
| Matrix Spike Duplicate[1] | Per 20 samples or per analytical batch, whichever is more frequent | 50-150% or based on historical laboratory control limits (average±3SD); RPD<25% |
| Surrogate | Included in all samples and all QC samples | Refer to control limits listed in Appendix A. |
| Internal Standard | Included in all samples and all QC samples (as available) | Per laboratory procedure |

| Field Quality Control | Frequency of Analysis | Measurement Quality Objective |
|---|---|---|
| Field Duplicate | Not required – infeasible given sampling and analysis methods | Not applicable |
| Field Blank, Travel Blank, Equipment Blank | Not required – infeasible given sampling and analysis methods | Not applicable |

[1]Not applicable to isotope dilution methods

Table 6. Data quality objectives for laboratory analysis of fipronils, neonicotinoids, organophosphorus pesticides, carbamates, and phenolics in water. The completeness objective for CCLEAN field and laboratory samples is 95%. Program action limits for POPs in influent and effluent are those in each participant's NPDES permit, as applicable. Program action limits for POPs in ocean water are those in the California Ocean Plan, Table 3, as applicable.

| Laboratory Quality Control | Frequency of Analysis | Measurement Quality Objective |
|---|---|---|
| Tuning[2] | Per analytical method (See Physis SOP) | Per analytical method (See Physis SOP) |
| Calibration | Daily, or just prior to analysis; five or more standards spanning the sample result range[3], with the lowest standard at or below the RL | • Correlation coefficient (r2 >0.990) for linear and non-linear curves<br>• First- or second-order curves only (not forced through the origin)<br>• Minimum of 5 points per curve for linear fit or 6 points if quadratic fit (one of them at or below the RL) |
| Calibration Verification | Every 10 samples | • Expected response or expected concentration ±20%<br>• RF for SPCCs=initial calibration[2] |
| Laboratory Blank | Per 20 samples or per analytical batch, whichever is more frequent | <RL for target analytes |

| | | |
|---|---|---|
| Reference Material / Dup | Per 20 samples or per analytical batch (preferably blind) | 70-130% recovery if certified; otherwise, 50- 150% recovery; RPD<25% |
| Surrogate | Included in all samples and all QC samples as required by method | Based on historical laboratory control limits (50- 150% or better) |
| Internal Standard | Included in all samples and all QC samples (as available) | Per laboratory procedure |
| **Field Quality Control** | **Frequency of Analysis** | **Measurement Quality Objective** |
| Field Duplicate | 5% of total project sample count | RPD<25% (n/a if native concentration of either sample<RL) |
| Field Blank | Per method | <RL for target analyte |

[1]All detected analytes must be confirmed with a second column, second technique, or mass spectrometry.

[2]Mass spectrometry only

Table 7. Data quality objectives for laboratory analysis of pyrethroids in water.[1] The completeness objective for CCLEAN field and laboratory samples is 95%. Program action limits for POPs in influent and effluent are those in each participant's NPDES permit, as applicable. Program action limits for POPs in ocean water are those in the California Ocean Plan, Table 3, as applicable.

| **Laboratory Quality Control** | **Frequency of Analysis** | **Measurement Quality Objective** |
|---|---|---|
| Tuning[2] | Per analytical method (See Physis SOP) | Per analytical method (See Physis SOP) |
| Calibration | Daily, or just prior to analysis; five or more standards spanning the sample result range[3], with the lowest standard at or below the RL | • Correlation coefficient (r2 >0.990) for linear and non-linear curves<br>• First- or second-order curves only (not forced through the origin)<br>• Minimum of 5 points per curve for linear fit or 6 points if quadratic fit (one of them at or below the RL) |
| Calibration Verification | Per 10 analytical samples[4] | • Expected response or expected concentration ±20%[5] |
| Laboratory Blank | Per 20 samples or per analytical batch, whichever is more frequent | <RL for target analytes |
| Reference Material | Per 20 samples or per analytical batch, whichever is more frequent | 50- 150% recovery |
| Matrix Spike | Per 20 samples or per analytical batch, whichever is more frequent | 50- 150% recovery |
| Matrix Spike Duplicate | Per 20 samples or per analytical batch, whichever is more frequent | 50- 150% recovery; RPD<35% |
| Surrogate[6] | Included in all samples and all QC samples | Based on historical laboratory control limits (50- 150% or better) |
| Internal Standard | Included in all samples and all QC samples (as available) | Per laboratory procedure |
| **Field Quality Control** | **Frequency of Analysis** | **Measurement Quality Objective** |
| Field Duplicate | 5% of total project sample count | RPD<35% (n/a if native concentration of either sample<RL) |

[1]All detected analytes must be confirmed with a second column, second technique, or mass spectrometry.
[2]Mass spectrometry only
[3]Sample results above the highest standard are to be diluted and re-analyzed
[4]Analytical samples include samples only and do not include clean-out or injection blanks
[5]Limit applies to a mid-level standard; low-level calibration checks near the reporting limit may have a wider range that is project – specific
[6]Laboratory historical limits for surrogate recovery must be submitted in the lab result comment section

Table 8. Data quality objectives for laboratory analysis of synthetic organic compounds (PFAS) in water. The completeness objective for CCLEAN field and laboratory samples is 95%. Program action limits for POPs in influent and effluent are those in each participant's NPDES permit, as applicable. Program action limits for POPs in ocean water are those in the California Ocean Plan, Table 3, as applicable.

| QC Parameter | Specification |
|---|---|
| MS Acquisition Rate | Minimum acquisition rate for every native analyte and labeled compound peak: At least 10 data points per peak. |
| Instrument Sensitivity | Run every 12 hours.<br>CAL A – S:N ≥ 3:1 for quantification ion.<br>CAL C – S:N ≥ 3:1 for quantification and confirmation ion. Native compound recoveries 70-130%. Ion ratios must be within 50-150% of the ratios determined from I-CAL CAL E. |
| Mass Calibration | Instrument must have a valid mass calibration following the manufacturer specified procedure prior to any sample analysis. The mass calibration is updated on an as-needed basis (e.g. QC failures, ion masses fall outside of the ±0.5 amu of the true value, major instrument maintenance, or if the instrument is moved.)<br>he entire range (bracketing all the masses of the target analytes) must be mass calibrated. The maximal allowed residual error is ≤ 0.1 Da for each mode with no more than two calibration points missed. |
| Mass Calibration Verification | Mass calibration must be verified after each mass calibration, prior to any sample analysis. Mass calibration must be performed per the instrument manufacturer's instructions. A mass calibration verification must be performed using standards whose mass range brackets the masses of interest (quantitative and qualitative ions). |
| Initial Calibration (I-CAL) | Run initially, and as required to maintain compliance with calibration verification and instrument sensitivity. The isotopically labeled analog of an analyte (surrogate standard) must be used for quantitation if commercially available (Isotope Dilution Quantitation). If a labeled analog is not commercially available, the surrogate standard with the closest retention time or chemical similarity to the native standard must be used for quantitation. |

| | |
|---|---|
| | NFDHA must pass Initial Calibration specifications for at least 5 calibration points.<br>Quantification is achieved by the constant RRF method. The I-CAL specifications (CAL B to CAL I) for the RRF are <20% RSD of mean RRFs and 70-130% recovery of analytes and surrogates at each concentration level from Cal C and above. For concentrations at or above the method LOQ, the total (branched and linear isomer) quantification ion response to the total (branched and linear isomer) confirmation ion response ratio must fall within ±50% (50-150%) of the ratio observed in the I-CAL, CAL E. CAL B to CAL I must meet a 3:1 S/N specification in the quantification ion and a 3:1 S/N in the confirmation ion. The A CAL (sensitivity CAL) must achieve 3:1 S/N for the quantification ion.<br>Refer to Section 4.1 for calculation of recovery standards.<br>The mean area count for each recovery standard is recorded and used to evaluate results for client sample analysis. There is no acceptance criteria associated with the mean recovery standard area data.<br>NFDHA: RRF RSD% must be ≤ 30% and must have 35-165% recovery for 5 calibration points. Not required to pass ion ratio specifications, but confirmation ion must be >3:1 S/N.<br>Peak Asymmetry, SGS AXYS guidance: 0.8-1.5 for PFBA and PFPeA measured in CAL E (mid cal point) at 10% of the peak height. If this is not achieved, perform instrument maintenance and re-run I-CAL. |
| Initial Calibration Verification (ICV): | After each Initial Calibration (I-CAL) and prior to sample analysis; analyze a second source standard (similar concentration to the CAL E); quantify against I-Cal, results must meet Cal/Ver accuracy specifications of 70% to 130%. Ion ratios must be within 50-150% of the ratios determined from I-CAL CAL E.<br>NFDHA: Native must recover between 50-170%, not required to pass ion ratio specifications but confirmation ion must be >3:1 S/N. |
| Retention Time (RT) window | Relative retention times (RRT) for linear and branched isomers vs. the surrogates are determined from the qualitative standard run every 12 hours (see Table 12). Maximum RRTs windows are 0.1 minutes. |
| Surrogate Standards | Must be added to every field sample, standard, blank, and QC sample. |

| | |
|---|---|
| | Recoveries of the surrogate standard analytes (EPA term for Surrogate Standard is Extracted Internal Standard-EIS; EPA term for Recovery Standard is Injection Internal Standard-IIS) are calculated by internal standard quantification against the IIS using an average RRF. Recovery criteria for surrogate standard analytes in instrument blanks and standards is 70% to 130%.<br>Recovery criteria for surrogate standard analytes in field samples and preparatory QC samples is 10% to 200%. Refer to Appendix A for OPR % recovery limits.<br>Requirement for DoD work: the lower acceptance limit for labeled (extracted external) surrogate standards in field samples, standards, blanks and QC samples is 20% recovery. For any labeled surrogate standard recoveries found to be below this specification for such samples, this will be addressed in the case narrative with client approval. |
| Recovery Standards | Must be added to every prepared field sample, standard, blank, and QC sample prior to instrumental analysis. Recovery standard analyte recovery is calculated by external standards by evaluation of the mean RF from the I-CAL with an SGS AXYS specification of 50% to 200%. Professional judgement applies.<br>Requirement for DoD work: non-extracted internal standard (or recovery standard) areas shall be greater than 30% of the average area of the calibration standards. |

| | |
|---|---|
| Calibration Verification (Cal/Ver or CCV) | CAL E. Prior to sample analysis and at the end of the analytical sequence, at least every 10 client samples, whichever occurs first. Quantify against I-CAL.<br>Native standard analyte and surrogate standard concentrations must be within ±30% of their true value. Recovery standard analyte concentrations must be within 50-200% of their true value. Ion ratios must be within 50-150% of the ratios determined from I-CAL CAL E. If the CCV criterion are not met, an instrument re-calibration is performed.<br>NFDHA: Native must recover between 50-170%, with a 10% allowance before batch reinjection is required. The confirmation ion must be >3:1 S/N.<br>Additional requirement for DoD: If the CCV criterion are not met, immediately analyze 2 additional CCVs. If both CCVs meet the criteria, samples may be reported without re-analysis. If either CCV exceed the criteria, or if two successive CCVs cannot be analyzed immediately following the failing CCV, corrective action must be taken. Once correction has been made and a CCV has been analyzed and met the criteria, all samples bracketed by the failing CCV must be re-analyzed.<br>For internal purposes monitor Peak Asymmetry for every Cal/Ver |
| Instrument Background | For DoD work an instrument blank containing surrogates is run immediately after every Initial Calibration (highest standard) and Calibration Verification and daily thereafter. The concentration of each analyte in the instrument blank must be ≤ ½ of the LOQ (C CAL). In this case the instrument blank can be run after CAL I. If any sample concentrations exceed the highest allowed standard and the sample(s) following exceed this acceptance criteria (>1/2 LOQ), they must be reanalyzed. The percent recoveries of both the surrogate and recovery standards in the instrument blank must be 50-200% of their true value. |
| Instrumental Carryover | The specification is 0.3 % carryover from the Cal Ver standard into following instrument blank or from any sample into the following injection. |
| Duplicate Samples | One per preparatory batch.<br>If conc. ≥ 5 times R.L., RPD ≤ 40%<br>If conc. < 5 times R.L., guideline RPD ≤ 100% |

| | |
|---|---|
| Ongoing Precision and Recovery (OPR or LCS) | One per preparatory batch Ongoing Precision and Recovery (OPR) or Laboratory Control Sample (LCS) is spiked at the same level as CAL E.<br>Refer to CCLEAN (2020) for OPR acceptance limits.<br><br>The ratio of the primary to secondary product ion responses in the total for branched and linear isomers must fall within ±50% of the same ratio observed in the mid-point initial calibration standard (CAL E). The ratio requirement does not apply where suitable (not detectable or inadequate S/N) secondary transitions are unavailable. |
| Method Blank (MB) | One per preparatory batch. No native standard can be detected > ½ LOQ or >1/10th the amount measured in field samples in the batch, whichever is greater. |
| Low-Level Ongoing Precision and Recovery (LLOPR) | One per preparatory batch. Spiked at 2X the LOQ (i.e. at 2X CAL C) and serves to verify the LOQ<br>Recovery criteria for native standards in the LOQ/LLOQ are set at 70-130% and must meet ratio specifications. |
| MS/MSD | One per preparatory batch. Native standard concentration must be spiked at concentrations ≥ LOQ and ≤ the mid-level calibration concentration.<br><br>MS/MSD recoveries are evaluated against the DOD specific acceptance ranges for OPRs listed in CCLEAN (2020), or against the MLA-110 OPR method recovery limits for analytes not listed in CCLEAN (2020). RPDs are evaluated against the DoD specific limit of ≤30%. |
| Bile Salts Interference check standard (TDCA) | Run after every I-CAL, regardless of the matrix to be analyzed, to establish that bile salts will not interfere with any PFOS isomers. Also run at least daily, following the instrument sensitivity check, prior to the analysis of tissue samples, There shall be at least one minute of separation between the TDCA check standard and the earliest eluter of PFOS (PFOS IV). The retention time of TDCA and PFOS may be handwritten on chromatograms for client reports. |

[1]Corrective actions stated in the current version of DoD QSM, Appendix B, Table B-15 must be utilized when QC parameter fails to meet the specification.

Table 9. Data quality objectives for laboratory analysis of trace metals in water. The completeness objective for CCLEAN field and laboratory samples is 95%. Program action limits for inorganics in influent and effluent are those in each participant's NPDES permit, as applicable. Program action limits for inorganics in ocean water are those in the California Ocean Plan, Table 3, as applicable.

| **Laboratory Quality Control** | **Frequency of Analysis** | **Measurement Quality Objective** |
|---|---|---|
| Calibration Standard | Per analytical method or manufacturer's specifications | Per analytical method or manufacturer's specifications |
| Calibration Verification | Per 10 analytical samples | 80 – 120% |
| Laboratory Blank | Per 20 samples or per analytical batch, whichever is more frequent | <RL for target analytes |
| Reference Material | Per 20 samples or per analytical batch (preferably blind) | 75-125% recovery |
| Matrix Spike | Per 20 samples or per analytical batch, whichever is more frequent | 75-125% recovery |
| Matrix Spike Duplicate | Per 20 samples or per analytical batch, whichever is more frequent | 75-125% recovery, RPD<25% (n/a/ if native concentration of either sample <RL) |
| Laboratory Duplicate | Per 20 samples or per analytical batch, whichever is more frequent | RPD<25% (n/a/ if native concentration of either sample <RL) |
| Internal Standard | Included in all samples and all QC samples (as available) | Per laboratory procedure |
| **Field Quality Control** | **Frequency of Analysis** | **Measurement Quality Objective** |
| Field Duplicate | 5% of total project sample count | RPD<25% (n/a if native concentration of either sample<RL) |
| Field Blank, Eqpt Blank | Per method | <RL for target analyte |

Table 10. Data quality objectives for laboratory analysis of fecal indicator bacteria (enterococcus, total coliform, fecal coliform, and E. coli) in freshwater. The completeness objective for CCLEAN field and laboratory samples is 95%. Program action limits for FIB in waters are in the California Ocean Plan, Table 1 and Table 2.

| **Laboratory Quality Control** | **Frequency of Analysis** | **Measurement Quality Objective** |
|---|---|---|
| Sterility Checks | Per lot of dehydrated culture media as instructed in SM 9020B.4.i.5 and SM 9222D.1.a | No growth |
| | For non-sterile filters and pads per lot as instructed in SM 9020B.4.h.1.1 | No growth |
| | Membrane Filter Media, filters, buffered dilution water, rinse water, and all equipment per series of samples as instructed in SM 9020B.8.a.5 | No growth |
| | Multiple Tube Media, dilution water, and glassware as instructed in SM 9020B.8.a.5 | No growth |
| Laboratory Positive Control | Per new lot of dehydrated culture media for the following methods: Colilert, Colilert -18, Colisure, Enterolert, or other chromogenic/fluorogenic methods.<br>Per new lot of commercially-prepared culture media ampules for USEPA-approved fecal coliform and E. coli membrane filter methods (e.g. SM 9222, m-ColiBlue24, EPA 1603).<br>Per batch for laboratory-prepared culture media for USEPA-approved fecal coliform and E. coli membrane filter methods (e.g., SM 9222) | Positive response |
| Laboratory Negative Control | Per new lot of dehydrated culture media for the following methods: Colilert, Colilert -18, Colisure, Enterolert, or other chromogenic/fluorogenic methods.<br>Per new lot of commercially-prepared culture media ampules for USEPA-approved fecal coliform | Negative response |

| | | |
|---|---|---|
| | and E. coli membrane filter methods (e.g. SM 9222, m-ColiBlue24, EPA 1603).<br>Per batch for laboratory-prepared culture media for USEPA-approved fecal coliform and E. coli membrane filter methods (e.g., SM 9222) | |
| Laboratory Duplicate | Per 10 samples or per analytical batch, whichever is more frequent | Rlog ≤ 3.27 x R[1] Computation of R from duplicate laboratory sample analyses[1] |
| Laboratory Blank | Required only when samples are diluted; dilution water must be tested | No growth |
| **Field Quality Control** | **Frequency of Analysis** | **Measurement Quality Objective** |
| Field Duplicate | Not required | N/A |
| Field Blank | Not required | N/A |

[1] Method for determining precision as described in 2013 revisions to indicator bacteria analyses in fresh water for SWAMP QAPP (http://www.swrcb.ca.gov/water_issues/programs/swamp/mqo.shtml):

In order to determine precision for bacterial analysis, the following procedure (adapted from Standard Methods 9020 Section 8.b) will be used. Note: When determining the precision of bacterial analyses, it is important to distinguish between different matrices (drinking water, wastewater, ambient water). Duplicate results from different matrices must be kept separate when calculating precision.

In order to calculate the laboratory precision for bacterial analyses, the results from the preceding 15 positive samples of a specific type (matrix) are used to calculate a running mean. The results used to calculate the running mean must all correspond to the same quality control parameter, in this instance laboratory duplicates (as opposed to field duplicates). The results of different quality control parameters such as laboratory and field duplicates must not both be used to calculate a single running mean. Note: Field duplicates are not a current SWAMP requirement.

Step 1: Record the results from duplicate analyses (designated as $D_1$ and $D_2$).

Step 2: Calculate the logarithm (here designated as $L_1$ and $L_2$) of each duplicate result. Note: If either of the values D1 or D2 are less than 1, add 1 to both values before calculating the logarithms.

$$L_1 = \log D_1$$
$$L_2 = \log D_2$$

Step 3: Calculate the range of logarithms ($R_{log}$) for each pair of duplicates. $R_{log}$ is equal to the absolute value of the difference between the two numbers.

$$R_{log} = | L_1 - L_2 |$$

Step 4: Calculate the mean of $R_{log}$ (R) for the duplicates analyzed

$$R = (\sum R_{log})/n$$

Where

$\sum R_{log}$ = the sum of the ranges of logarithms calculated for each pair of duplicates
n = the number of pairs of duplicates (in this case, n = 15)

Step 5: Assess the precision of the duplicate analyses. In order for the laboratory to demonstrate an acceptable level of precision, the range of logarithms for a particular duplicate must be less than the mean of the range of logarithms multiplied by 3.27.

$$R_{log} \leq 3.27 \times R$$

Table 11. Data quality objectives for laboratory analysis of synthetic organic compounds (PCBs, PAHs, PBDEs, and organochlorine pesticides) in sediment and tissue. The completeness objective for CCLEAN field and laboratory samples is 95%. Program action limits for POPs in sediments are those in the NOAA sediment quality alert levels (ERLs). Program action limits for POPs in mussels are those in either the California Office of Environmental Health Hazard Assessment (OEHHA) or U.S. Food and Drug Administration, as appropriate.

| **Laboratory Quality Control** | **Frequency of Analysis** | **Measurement Quality Objective** |
|---|---|---|
| Tuning[1] | Per analytical method | Per analytical method |
| Calibration | Initial method setup or when the calibration verification fails | If RSD<20%, average RF may be used to quantitate; otherwise use equation of the curve<br>First- or second-order curves only (not forced through the origin)<br>Minimum of 5 points per curve (one of them at or below the RL) |
| Calibration Verification | Per 12 hours | Expected response or expected concentration ±30%<br>RF for SPCCs=initial calibration[1] |
| Laboratory Blank | Per 20 samples or per analytical batch, whichever is more frequent | <RL for target analytes |
| Reference Material | Per 20 samples or per analytical batch (preferably blind) | 70-130% recovery if certified; otherwise, 50- 150% recovery |
| Laboratory Control Sample | Per 20 samples or per analytical batch, whichever is more frequent | Per laboratory procedure. |
| Matrix Spike[1] | Per 20 samples or per analytical batch, whichever is more frequent | 50-150% or based on historical laboratory control limits (average±3SD) |
| Matrix Spike Duplicate[1] | Per 20 samples or per analytical batch, whichever is more frequent | 50-150% or based on historical laboratory control limits (average±3SD); RPD<25% |
| Surrogate | Included in all samples and all QC samples | Per method |
| Internal Standard | Included in all samples and all QC samples (as available) | Per laboratory procedure |
| **Field Quality Control** | **Frequency of Analysis** | **Measurement Quality Objective** |
| Field Duplicate | Not required – infeasible given sampling and analysis methods | RPD </= 25% |

| | | |
|---|---|---|
| Field Blank, Travel Blank, Equipment Blank | Not required – infeasible given sampling and analysis methods | Not applicable<RL for target analyte |

[1]For mass spectrometry only, not required for isotope dilution methods

Table 12. Data quality objectives for laboratory analysis of conventional parameters (total organic carbon and grain size) in sediment. The completeness objective for CCLEAN field and laboratory samples is 95%.

| **Laboratory Quality Control** | **Frequency of Analysis** | **Measurement Quality Objective** |
|---|---|---|
| Calibration Standard | Per analytical method or manufacturer's specifications | Per analytical method or manufacturer's specifications |
| Laboratory Blank | Total organic carbon only: one per analytical batch (n/a for other parameters) | <RL or <30% of lowest sample |
| Reference Material | Total organic carbon only: one per 20 samples or per analytical batch, whichever is more frequent (n/a for other parameters) | 80-120% recovery |
| Laboratory Duplicate | One per analytical batch | RPD<25% (n/a if native concentration of either sample<RL) |
| **Field Quality Control** | **Frequency of Analysis** | **Measurement Quality Objective** |
| Field Duplicate | Not required | Not applicable |
| Field Blank | Not required | Not applicable |

Table 13. Data quality objectives for laboratory analysis of pyrethroids, fipronils, and neonicotinoids in sediment. The completeness objective for CCLEAN field and laboratory samples is 95%.

| **Laboratory Quality Control** | **Frequency of Analysis** | **Measurement Quality Objective** |
|---|---|---|
| Tuning[2] | Per analytical method (See Physis SOP) | Per analytical method (See Physis SOP) |
| Calibration | Daily, or just prior to analysis; five or more standards spanning the sample result range[3], with the lowest standard at or below the RL | Correlation coefficient (r2 >0.990) for linear and non-linear curves<br>First- or second-order curves only (not forced through the origin)<br>Minimum of 5 points per curve for linear fit or 6 points if quadratic fit (one of them at or below the RL) |
| Calibration Verification | Every 10 samples | Expected response or expected concentration ±20%<br>RF for SPCCs=initial calibration[2] |
| Laboratory Blank | Per 20 samples or per analytical batch, whichever is more frequent | <RL for target analytes |
| Reference Material / Dup | Per 20 samples or per analytical batch (preferably blind) | 70-130% recovery if certified; otherwise, 50- 150% recovery; RPD<25% |
| Surrogate | Included in all samples and all QC samples as required by method | Based on historical laboratory control limits (50- 150% or better) |
| Internal Standard | Included in all samples and all QC samples (as available) | Per laboratory procedure |
| **Field Quality Control** | **Frequency of Analysis** | **Measurement Quality Objective** |
| Field Duplicate | 5% of total project sample count | RPD<25% (n/a if native concentration of either sample<RL) |
| Field Blank | Per method | <RL for target analyte |

# 8. SPECIAL TRAINING NEEDS/CERTIFICATION

### 8.1. Specialized training or certifications

Personnel in any laboratory performing CCLEAN analyses will be well versed in good laboratory practices, including standard safety procedures. It is the responsibility of the particular analytical component project officer, laboratory manager, and/or supervisor to ensure that safety training is mandatory for all laboratory personnel. Each laboratory is responsible for maintaining a current safety manual in compliance with the Occupational Safety and Health Administration (OSHA), or equivalent state or local regulations. The safety manual will be readily available to laboratory personnel. Proper procedures for safe storage, handling, and disposal of chemicals will be followed at all times; each chemical will be treated as a potential health hazard and good laboratory practices will be implemented accordingly.

### 8.2. Training and certification documentation

Any laboratory performing analysis of bacteria in mussels shall be certified by the State of California Department of Health Services according to the USFDA Shellfish testing program to perform Shellfish meat and Shellfish Growing Waters microbiological testing.

### 8.3. Training personnel

Each field sampling contractor and analytical laboratory is responsible for training its personnel per relevant standard operating procedures. Periodic audits will be conducted of field sampling activities to confirm adherence to the CCLEAN QAPP.

# 9. DOCUMENTS AND RECORDS

Field sampling contractors will collect records for sample collection and will be responsible for developing sampling plans and sampling reports and delivering them to the Program Director. Samples sent to analytical laboratories will include Chain of Custody (COC) forms. Analytical laboratories will collect records for sample receipt and storage, analyses, and reporting.

### 9.1. Field Documentation

All field documentation will be stored, processed, and distributed according to Section 6.1 of the CCLEAN Monitoring Plan (CCLEAN 2024). Any updates to this QAPP will be distributed to all parties on the distribution list. Persons responsible for maintaining records for this project are shown in Table 14.

Table 14. Responsibilities for Record Collection and Maintenance.

| Name | Organizational Affiliation | Records | Retention (yrs after contract end) |
|---|---|---|---|
| Aroon Melwani | CCLEAN Program Director-AMS | Lab reports, sampling plans, sampling reports | 5 |
| Paul Salop | CCLEAN QAO-AMS | QA reviews/audits | 5 |
| Greg Cotten | KEI | Lab reports for influent, effluent, ocean, and mussel sampling; Field datasheets; COCs | 5 |
| Sean Campbell | SGS AXYS | Lab records for influent, effluent, ocean, mussel, and sediment POPs | 5 |
| Misty Mercier | Physis | Lab records for influent, effluent, and ocean water sampling | 5 |
| Jim Oakden | CCR | Field datasheets, lab records for benthic sampling | 5 |
| Michael Ferris | SLAB | Lab records for pathogens analysis | 5 |

The Project Director will oversee the actions of these persons and will arbitrate any issues relative to records retention and any decisions to discard records. Copies of all records will be maintained by the applicable field-sampling contractor or analytical laboratory for at least five years after project completion.

## 9.2. Laboratory Documentation

CCLEAN requires specific actions to be taken by contract laboratories, including requirements for data deliverables, quality control, and on-site archival of project-specific information. Each of these aspects is described below.

### *9.2.1. Data Reporting Format*

Each laboratory will deliver data in electronic formats to the Project Manager. Each will be responsible for storage and safekeeping of these records. The analytical laboratory will report the analytical data via an analytical report consisting of, at a minimum:

1. Letter of transmittal
2. Chain of custody information
3. Analytical results for field and quality control samples (in CEDEN template format)
4. Case narrative

Documentation for analytical data are kept on file at the laboratories, or may be submitted with analytical results. These may be reviewed during external audits of the Program, as needed. These records may include the analyst's comments on the condition of the sample and progress of the analysis, raw data, instrument printouts, and results of calibration and QC checks. Paper or electronic copies of all analytical data, field data forms and field notebooks, raw and condensed data for analysis performed on-site, and field instrument calibration notebooks are kept as part of the Program archives for a minimum period of eight years.

### *9.2.2. Other Laboratory QA/QC Documentation*

All laboratories will have the latest version of the CCLEAN QAPP in electronic format. In addition, the following documents and information from the laboratories will be current, and they will be available to all laboratory personnel participating in the processing of CCLEAN samples:

1. Laboratory QA plan: Clearly defines policies and protocols specific to a particular laboratory, including personnel responsibilities, laboratory acceptance criteria, and corrective actions to be applied to the affected analytical batches, qualification of data, and procedures for determining the acceptability of results.
2. Laboratory SOPs: Contain instructions for performing routine laboratory procedures, describing exactly how a method is implemented in the laboratory for a particular analytical procedure. Where published standard methods allow alternatives at various steps in the process, those approaches chosen by the laboratory in their implementation (either in general or in specific analytical batches) are to be noted in the data report, and any deviations from the standard method are to be noted and described.
3. Instrument performance information: Contains information on instrument baseline noise, calibration standard response, analytical precision and bias data, detection limits, scheduled maintenance, etc.

4. Control charts: Control charts are developed and maintained throughout the Program for all appropriate analyses and measurements for purposes of determining sources of an analytical problem or in monitoring an unstable process subject to drift. Control charts serve as internal evaluations of laboratory procedures and methodology and are helpful in identifying and correcting systematic error sources. Control limits for the laboratory quality control samples are ±3 standard deviations from the certified or theoretical concentration for any given analyte.

Records of all quality control data, maintained in a bound notebook at each workstation, are signed and dated by the analyst. Quality control data include documentation of standard calibrations, instrument maintenance and tests, and analyses of CRMs. Control charts of the data are generated by the analysts monthly or for analyses done infrequently, with each analysis batch. The laboratory quality assurance specialist will review all QA/QC records with each data submission, and will provide QA/QC reports to the Program Director with each batch of submitted sample data.

# Group B: Data Generation and Acquisition

## 10. SAMPLING PROCESS DESIGN

### 10.1. Sampling Design

CCLEAN measures inputs to the ocean of POPs, suspended sediments, nutrients and pathogens in water and effects in ocean waters by sampling wastewater influent, effluent, rivers, mussels, sediments, and benthic communities, and ocean waters using a judgmental design. The CCLEAN Monitoring Plan (CCLEAN 2024) provide details of the sites currently sampled (Section 1.2) and methods (Section 4) used in the current Program Year. In general, the types of samples to be collected each year are as follows:

Influent and Effluent

- 250-liter samples collected twice per year for the analysis of POPs, during 30-day periods in the wet season and in the dry season;
- Monthly samples collected for nutrients;
- Grabs collected for pyrethroids, fipronils, neonicotinoids, and PFAS twice per year

Rivers

- 250-liter sample collected twice per year for the analysis of POPs over a 30-day period in the wet and dry seasons
- Grab samples for nutrients collected twice per year
- Grabs collected for pyrethroids, fipronils, neonicotinoids, and PFAS twice per year

Ocean Water

- 250-liter samples collected twice per year for the analysis of POPs, during 30-day periods in the wet season and in the dry season
- Two grabs for nutrients and bacteria collected from each site at the beginning and end of the buoy deployment period

Mussels

- Annual collection of sample composites of 30–40 individuals for analysis of POPs and bacteria

Sediment

- Annual collection of bulk surface sediment samples for analysis of POPs, total organic carbon and particle size
- Collection of single replicates every five years for analysis of benthic infauna

Because CCLEAN has been sampling since 2001, access to all sites is well established, although ocean conditions sometimes limit access. The approximate schedules for sampling each program element are as follows:

Table 15. Program Element, Intervals, and Approximate Timing.

| Program Element | Season | Approximate Dates |
|---|---|---|
| Effluent | Wet Season | February - March |
| | Dry Season | June - November |
| | Monthly | July - June |
| Influent | Dry Season | June - November |
| Rivers | Wet Season | February - March |
| | Dry Season | June - November |
| Ocean Water | Wet Season | February - March |
| | Dry Season | June - November |
| Mussels | Wet Season | February - March |
| Sediment | Dry Season | September - October |

Samples for POP analysis will be shipped to the laboratory for analysis as soon after they are collected from the field as possible, although mussel tissues will be removed from the shells and homogenized before being shipped. Samples for bacteria and nutrient analyses will be delivered to the laboratory for analysis as soon as possible after being collected.

All the data collected by CCLEAN are used to achieve its objectives and there are no data that are collected for informational purposes only.

### 10.2. Sampling Uncertainty

There are multiple sources of potential sampling uncertainty associated with the implementation of CCLEAN, including: (1) measurement error; (2) natural (inherent) variability; (3) sample misrepresentation (or poor representativeness); and (4) sampling bias (statistical meaning). Measures incorporated to address these areas of uncertainty are discussed below:

1. Measurement error combines all sources of error related to the entire sampling and analysis process (i.e., to the measurement system). All aspects of dealing with uncertainty due to measurement error have been described elsewhere within this QAPP.

2. Natural (inherent) variability occurs in any environment monitored, and is often much wider than the measurement error. This inherent variability will be taken into consideration when interpreting results of the various lines of inquiry.

3. Sample misrepresentation happens at the level of an individual sample or field measurement where an individual sample collected is a poor representative for overall conditions encountered. To address this situation, CCLEAN has been developing and

implementing a number of QA-related measures, including training and auditing of field crews to ensure their proper implementation.

4. Sampling bias relates to the sampling design employed and whether the appropriate statistical design is employed to allow for appropriate understanding of environmental conditions. Potential sources of bias include sampling and analytical methods. In the case of sampling, bias is controlled by using prescribed methods to provide repeatable results. For example, if samples are collected in a systematic way that targets specific types of organisms (e.g., mussels of a certain size), and there is inconsistency in the types of organisms collected in each sampling effort, bias is introduced, insofar as analytical measurements might vary according to organism type. This type of bias also could occur if different sieve mesh sizes were used each time for removing benthic infauna from sediment. These potential sources of bias are controlled by always collecting mussels of approximately the same size from all locations and by using a standardized sieve mesh size for processing all benthic samples. Sampling bias can also be introduced by using sampling methods that do not effectively collect certain types of analytes. For example, the in situ solid-phase extraction method used by CCLEAN for sampling POPs does not adequately sample highly polar compounds. This type of bias is controlled by only analyzing non-polar compounds.

5. Analytical bias is introduced if measurement methods are either more or less accurate under different ambient conditions or if they inherently misrepresent the actual concentration of an analyte. Applying Quality Control limits to measurements of reference performance spikes and laboratory spikes helps control the former type of analytical bias in water samples for analysis of POPs. Control of this type of bias in other samples is done primarily through examination throughout the analytical process for interferences due to matrix effects. Bias due to inherent misrepresentation of analyte concentrations is controlled by requiring analysis of certified reference materials, laboratory reference materials or standards.

# 11. SAMPLING METHODS

The CCLEAN program comprises multiple sampling components. A brief summary of each is provided below. The CCLEAN Monitoring Plan (CCLEAN 2024) provides further details of the sampling methods, which will utilize several field sampling SOPs developed for the CCLEAN Program. Additionally, a Sampling Plan for each field sampling effort is prepared by the Field Program Manager and submitted to the Program Director two weeks prior to sampling that provides information on sampling dates, procedures, and personnel involved. Any problems that occur during sampling are reported immediately to the Program Director by the respective Field Program Manager and corrective actions are taken, when possible. A Sampling Report is submitted within two weeks following the completion of sampling that provides information on actual sampling dates, duration of sampling efforts, unusual conditions or problems encountered and corrective actions taken.

## 11.1. Wastewater Influent and Effluent Sampling

Effluent sampling includes collection of 30-day flow proportioned samples twice per year (i.e., in the wet season and in the dry season) for analysis of POPs. A single, dry season influent sample is also collected at the City of Watsonville using 30-day flow proportioned methods. The objective of this sampling component is to estimate the loads to Monterey Bay of POPs from City of Watsonville influent and effluent from all major sources. Annual loads of POPs are estimated by calculating the average daily load during each sampling period (average flow multiplied by concentration) and multiplying the average load from both sampling periods by 365.

CCLEAN employs an in situ solid-phase extraction process for sampling POPs in influent and effluent that captures contaminants in both the particulate and dissolved phases. This method is discussed in greater detail in Section 11.1.1. The constituents measured in influent and effluent by CCLEAN are shown in Table 20. All of these POPs are in the California Ocean Plan Table 3, except the PAHs biphenyl, naphthalene, 1-methylnaphthalene, 2-methylnaphthalene, 2,6-dimehtylnaphthalene, 2,3,5-trimethylnaphthalene, acenaphthene, dibenzothiophene, 1-methylphenanthrene, fluoranthene, benzo(e)pyrene and perylene, and PFCs and PBDEs. The California Ocean Plan Table 3 constituents not measured in influent and effluent by CCLEAN are shown in Table 21.

### *11.1.1. Solid-Phase Extraction Sampling*

The collection of 30-day flow proportioned samples of influent and effluent is accomplished by KEI using programmable ISCO 3700 samplers. The CCLEAN Monitoring Plan (CCLEAN 2024) details the specific details of the sampling. Dry-season influent and effluent samples are collected with the ISCO equipment during the months of July–November and wet-season effluent samples are collected during the months of December–June. An equipment blank sample is collected for each sampling period by pumping ultra-pure water through the equipment.

The SOPs that apply to this sampling task are as follows:

- KLI –CCL-2006002-02 (28 Oct. 2008) for CCLEAN Solid-Phase Extraction Column and Glass Fiber Filter Handling Procedures and
- KLI –CCL-2019001-01 (17 April 2019) for CCLEAN Teflon7 Sample Tubing, Silicon Peristaltic Tubing, Silicon Tubing, Teflon7 Check Valve, Stainless Steel Glass Fiber Filter Canister, and Ocean Micropump Cleaning Procedures.

These SOPs from Kinnetic Environmental, Inc. are proprietary and are available for examination at the Program Director's office in Santa Cruz, CA.

#### *11.1.2. Grabs by Plant Personnel*

Influent and effluent grab samples for are collected by personnel of the program participants and analyzed for various constituents. Nutrients are analyzed in participant's laboratories or contract laboratories, fipronils, neonicotinoids, and pyrethroids are analyzed in contract laboratories. The objective of this sampling component is to estimate the loads to Monterey Bay of nutrients, fipronil, neonicotinoids, and pyrethroids from City of Watsonville influent and effluent from all major sources. All grabs are taken from the influent and effluent stream at the point where samples are collected for the regular influent/effluent monitoring required under each NPDES permit. Annual loads of these constituents are estimated by calculating the load on each sampling date (flow multiplied by concentration) and multiplying the average load among all samples by 365.

The SOPs that apply for collection of grab samples are based on EPA-approved methods and are on file at each wastewater treatment plant.

### 11.2. Receiving Water Sampling

Receiving water sampling consists of monthly or more frequent sampling for pathogen indicators at stations along the 30-foot contour near the wastewater discharges of CAWD, Santa Cruz, Watsonville, and Monterey One Water. Measurements are made for total coliform, fecal coliform, and Enterococcus bacteria. Locations of receiving water monitoring sites for each agency are described in Section 4 of the CCLEAN Monitoring Plan (CCLEAN 2024).

The SOPs that apply for collection of receiving water samples are based on EPA-approved methods and are on file at each wastewater treatment plant.

### 11.3. Mussel Sampling

Mussel sampling consists of collecting mussels once a year during the wet season for analysis of POPs and bacteria. Section 4 of the CCLEAN Monitoring Plan (CCLEAN 2024) outlines the detailed methods employed. The objective of this program element is to determine the extent to which humans and sea otters might be exposed to POPs and pathogens from consumed components of the food web. Mussel sampling will be performed by KEI, with POP analyses analyzed by SGS AXYS and bacteria analyzed by Sonoma County Public Health Lab. Mussel collection and processing will be consistent with the California Department of Fish and Wildlife's most recent SOP.

The SOPs that apply to this sampling task are as follows:

- Department of Fish and Wildlife's Standard Operating Procedures (DFG SOP 102).
- KLI –CCL-2006003-01 for Collection and Processing of Mussels.

This proprietary SOP is available for examination at the Program Director's office in Santa Cruz, CA.

### 11.4. Sediment Sampling

The objectives of this program component are to measure concentrations of POPs in sediments where the sediments are most likely to be deposited after washing off the land and out of rivers, and the effects of POPs on benthic infauna. Section 4 of the CCLEAN Monitoring Plan (CCLEAN 2024) details the coordinates and depths of sampling sites where sediment samples are currently collected. Sampling is conducted by AMS, with support from other consultants. Benthic infauna are analyzed by Coastal Conservation and Research, POPs are analyzed by SGS AXYS, and total organic carbon (TOC) and grain size are analyzed by Physis.

The SOP that applies to this sampling task is:

- CCLEAN Sediment Sampling and Analysis Plan (e.g., AMS 2023)

### 11.5. Ocean Water Sampling

The objective of this program component is to determine the status and trends of contaminants in nearshore waters of Monterey Bay and whether ocean waters comply with the California Ocean Plan.

Ocean buoys are deployed twice per year for 30-day periods at a site in northern Monterey Bay and at a site in southern Monterey Bay. Section 4 of the CCLEAN Monitoring Plan (CCLEAN 2024) details the coordinates of sampling sites where ocean samples are currently collected. The buoys contain sampling equipment that collects time-integrated samples of POPs using the same particle filters and columns packed with XAD-2 resin as used in the wastewater sampling. Duplicate grabs are collected from each site at buoy deployment and buoy retrieval for analysis of total coliform, fecal coliform, enterococcus, $NO_3$-N, $NH_3$-N, urea-N, and O-$PO_4$, $SiO_2$ and TSS.

The SOPs that apply to this sampling task are as follows:

- KLI –CCL-2006002-02 (28 Oct. 2008) for CCLEAN Solid-Phase Extraction Column and Glass Fiber Filter Handling Procedures and
- KLI –CCL-2019001-01 (17 April 2019) for CCLEAN Teflon7 Sample Tubing, Silicon Peristaltic Tubing, Silicon Tubing, Teflon7 Check Valve, Stainless Steel Glass Fiber Filter Canister, and Ocean Micropump Cleaning Procedures.

These SOPs from Kinnetic Environmental, Inc. are proprietary and are available for examination at the Program Director's office in Santa Cruz, CA. Collection of bacteria, nutrient and TSS samples are according to EPA-approved protocols.

### 11.6. River Sampling

Rivers discharging into Monterey Bay have been found to contribute significant loads of pollutants to ocean waters. The objective of this program components is to quantify the concentrations of pollutants and the annual loads entering Monterey Bay from river discharges. Section 4 of the CCLEAN Monitoring Plan (CCLEAN 2024) details the coordinates of sampling sites where river samples are currently collected.

The SOPs that apply to this sampling task are as follows:

- KLI –CCL-2006002-02 (28 Oct. 2008) for CCLEAN Solid-Phase Extraction Column and Glass Fiber Filter Handling Procedures and
- KLI –CCL-2019001-01 (17 April 2019) for CCLEAN Teflon7 Sample Tubing, Silicon Peristaltic Tubing, Silicon Tubing, Teflon7 Check Valve, Stainless Steel Glass Fiber Filter Canister, and Ocean Micropump Cleaning Procedures.

These SOPs from Kinnetic Environmental, Inc. are proprietary and are available for examination at the Program Director's office in Santa Cruz, CA.

### 11.7. Responsibility and Corrective Action

If monitoring equipment fails, sampling personnel will report the problem in the comments section of their field notes and will not record data values for the variables in question. Actions will be taken to replace or repair broken equipment prior to the next field use. Under no condition will data be entered into the CEDEN database that were known to be collected with faulty equipment.

# 12. SAMPLE HANDLING AND CUSTODY

In the field, all samples will be packed in wet ice or frozen ice packs (blue ice) during shipment, so that they will be kept at approximately 4°C. Samples will be shipped in insulated containers. All caps and lids will be checked for tightness prior to shipping. All samples will be handled, prepared, transported and stored in a manner so as to minimize bulk loss, analyte loss, contamination, or biological degradation. Sample containers will be clearly labeled with an indelible marker. Where appropriate, samples may be frozen to prevent biological degradation. Water samples will be kept in Teflon™, glass, or polyethylene bottles and kept cool at a temperature of 4°C until analyzed. Maximum holding times for specific analyses are listed in Tables Table 16, Table 17, andTable 18. Ice chests are sealed with tape before shipping. Samples are placed in the ice chest with enough ice and appropriate packing material to completely fill the ice chest.

Because of the importance of program samples and analytical data, sample Chain-of-Custody (COC) must be controlled and documented in the laboratory. Sample custody and document control procedures function to identify and document tracking and handling of samples and documents. Chain-of-custody (COC) procedures require that possession of samples be traceable from the time the samples are collected until completion and submittal of analytical results. Each sampling contractor / laboratory provides its own COC. A complete COC form is to accompany the transfer of samples to the analyzing laboratory. COC forms are placed in an envelope and taped to the top of the ice chest or they may be placed in a plastic bag and taped to the inside of the ice chest lid. It is assumed that samples in tape-sealed ice chests are secure whether being transported by staff vehicle, by common carrier, or by commercial package delivery. The receiving laboratory has a sample custodian who examines the samples for correct documentation, proper preservation and holding times during the sample login process. Contract laboratories will follow sample custody procedures outlined in their QA plans. At a minimum, the login documentation will indicate the sample identification, including dates collected and received, identity of the sampler, the analyses requested, as well as the use of proper containers and preservatives. Any deviations from required sampling techniques (e.g. wrong container type, not enough sample) are noted on the sample log form. Contract laboratory QA plans are on file with the respective laboratory. All samples remaining after successful completion of analyses will be held by the analytical laboratory until authorized by the Program Director to dispose of them properly. It is the responsibility of the personnel of each analytical laboratory to ensure that all applicable regulations are followed in the disposal of samples or related chemicals.

Table 16. Sample handling and custody for CCLEAN aqueous samples.

| Parameter | Container | Volume | Initial Preservation | Holding Time |
|---|---|---|---|---|
| Total coliform, fecal coliform, Enterococcus, | 2 sterile Whirl-Pak bags per site | 125 mL | Sodium thiosulfate | 8 hrs |
| Nitrate, orthophosphate | Nalgene high-density polyethylene | 60 mL | Vacuum-filtered (0.45 μm), cool to ≤6°C | 48 hrs at ≤6 °C in the dark |
| Urea | Sterile polypropylene centrifuge tube | 50 mL | Cool to ≤6°C | 30 days frozen |
| Ammonia | I-Chem high-density polypropylene | 125 mL | Sulfuric acid | 28 days at ≤6 °C |
| Total suspended solids, dissolved silica | Nalgene high-density polypropylene | 250 mL | None | 7 days at ≤6 °C |
| Pyrethroids, fipronils, neonicotinoids, organophosphates, and phenolics | Amber glass bottle | 2 @ 1 liter | Cool to ≤6°C | 2 days at ≤6 °C |
| PFAS | High-density polyethylene | 500 mL | Cool to ≤6°C | 90 days |
| PAHs, PCBs, PBDEs, Dioxins, Furans, Pesticides | SGS AXYS stainless-steel column packed with XAD-2 resin beads and SGS AXYS glass-fiber particle filter | ≈250 liters | Cool to ≤6°C with blue ice | Keep at ≤6 °C, dark, no limits on holding time prior to extraction |

Table 17. Sample handling and custody for CCLEAN sediment samples.

| Parameter | Container | Volume | Initial Preservation | Holding Time |
|---|---|---|---|---|
| Conventional (Grain Size, TOC) | Plastic jar | 125 mL | Cool to ≤6°C, dark | Keep at ≤6 °C up to 6 months for grain size; keep at ≤6 °C up to 28 days, up to 1 year frozen for TOC |
| Benthic samples | Glass jars | Various | Relax with $MgCl_2$, fix with 10% formalin/sea water, preserve with 70% ethyl alcohol | Indefinite |
| PAHs, PCBs, PBDEs, Pesticides | Pre-cleaned, certified amber glass jar, with Teflon lid-liner | 250 mL | ≤6°C, dark | Hold at -20°C, dark, up to one year |

Table 18. Sample handling and custody for mussel samples.

| Parameter | Container | Volume | Initial Preservation | Holding Time |
|---|---|---|---|---|
| Mussels, POPs | Pre-cleaned aluminum foil bags (1/site), double wrapped in Ziploc bags | 40 mussels | Stored on blue ice | 24 hours before resection, then frozen at -20°C |
| Mussels, pathogen indicators | Pre-cleaned aluminum foil bags (1/site), double wrapped in Ziploc bags | 30 mussels | Stored on blue ice | 24 hours |

# 13. ANALYTICAL METHODS

## 13.1. Performance Based Measurement System

CCLEAN incorporates a performance-based measurement system (PBMS) approach for measurements of contaminants at low concentrations involving continuous laboratory evaluation through the use of accuracy, and precision-based materials (e.g., CRMs; OPR), laboratory matrix spikes, laboratory reagent blanks, calibration standards, laboratory-and field-duplicated blind samples, and others as appropriate. Under the performance-based CCLEAN QA program, laboratories are not required to use a single, standard analytical method for each type of analysis. Rather, they are free to choose the best or most feasible method within the constraints of cost and equipment that is suitable for meeting CCLEAN's Data Quality Objectives (DQO). Nevertheless, validated methods are used whenever possible and each laboratory will continuously demonstrate proficiency and data comparability through routine analysis of performance evaluation samples, split samples, and reference materials representing actual sample matrices. In cases where validated methods might not be available, methods from the peer-reviewed scientific literature are favored. Recommended methods for analysis of POPs are EPA methods and those used in the NOAA NS&T Program (Lauenstein and Cantillo, 1993), but equivalent methods may be used where appropriate with approval of the Program Director.

## 13.2. Method Detection Limits and Reporting Limits

Suggested methods and target method detection limits (MDLs) for non-POP constituents in ocean water, sediment, and tissue are shown in Table 19. The target MDLs are not prescriptive because it is recognized that many factors can affect the actual MDL, such as variations in sample volume and unforeseen matrix interferences. Target MDLs for non-POP constituents in influent and effluent are not specified because, while they vary widely among CCLEAN program participants, QC checks of influent and effluent data indicate that these constituents are consistently measured in all influent and effluent samples. The MDLs prescribed in this document for certain analytes may be higher than those required by SWAMP due to the analytical capabilities of the participant laboratories. While concentrations of some parameters in ocean samples may fall below the stated MDLs, there are no ocean criteria to guide selection of MDLs and efforts to lower the achievable MDLs are not warranted. Similarly, higher MDLs in this QAPP than those required by SWAMP for Enterococcus, fecal coliform, total coliform and sediment total organic carbon are sufficiently low to determine whether Ocean Plan objectives and NOAA sediment quality alert levels are being met and there is no compelling need to reduce the MDLs. Target RLs and suggested methods for POPs in water, sediment and mussel tissue are shown in Table 20.

## 13.3. Analytical Methods

There are numerous SOPs that apply to analysis of samples in this program, as follows:

SGS AXYS

- MLA-007 Rev 13.09 (11 May 2017) for analysis of PCBs and pesticides using low resolution mass spectroscopy
- MLA-210 Rev 1.05 (22 Dec 2022) for analysis of PCBs in water, soil, sediment, biosolids,

and tissue by APGC-MSMS.

- MLA-013 Rev 9.05 (02 July 2015) for analysis of polychlorinated dibenzodioxins and furans, polybrominated diphenyl ethers, PCB congeners, chlorinated pesticides and toxaphene using co-extraction techniques
- MLA-217 Rev 1.09 (08 May 2023) for analysis of polychlorinated dibenzodioxins and dibenzofurans.
- MLA-021 Rev. 12.10 (16 Oct. 2023) for analysis of PAHs
- MLA-228 Rev 01.04 (19 June 2023) for analysis of organochlorine pesticides by isotope dilution SGS AXYS MLA-228APGC-MS/MS.
- MLA-033 Rev 6.06, (26 Feb. 2019) for analysis of brominated diphenyl ethers by EPA Method 1614
- MLA-110 Rev 2.13, (Sept 2023) for analysis of Per- and Polyfluoroalkyl Substances
- SLA-011 Rev 4, (03 May 2017) for compositing samples
- SLA-013 Rev 10, (09 Feb 2018) for homogenization of solids and tissues
- SLA-015 Rev 12, (29 Oct. 2018) for moisture determination
- SLA-020 Rev 6, (29 Oct. 2018) for gravimetric lipid determination by weight of extract
- SLA-037 Rev 12, (05 May 2017) for cleaning of sample preparation equipment used for preparing metals and organic samples
- SLA-043 Rev 5, (08 Feb. 2018) for removing sample media from field sampling equipment
- SLA-048 Rev 6, (06 May 2015) for cleaning of bulk resin
- SLA-049 Rev 8, (16 May 2016) for cleaning and packing of sample columns
- MLA-110 Rev 2.V08 or later, (Sept. 2020) for analysis of Per- and Polyfluoroalkyl Substances

Physis Laboratories

- EPA 625- NCL, for fipronil and degradates, and pyrethroid pesticides
- EPA 625, for neonicotinoid pesticides
- Physis SOP for EPA Method 625- Separatory funnel liquid-liquid extraction and analysis by gas chromatography/mass spectrometry
- Physis SOP for Negative Chemical Ionization- Negative chemical ionization analysis by gas chromatography/mass spectrometry
    - Sample extractions EPA Method 8270D, EPA Method 625 or EPA Method 3510 or 3545
- Physis SOP for EPA Method 9060M – Total organic carbon by high-temperature combustion method
- Physis SOP for Standard Method 2560 – Particle size distribution by light scattering method

Moss Landing Marine Laboratories

- Nutrients are analyzed on a Lachat Quickchem 8000 flow injection analysis system using the following QuickChem procedures:
    - Urea – N, QuickChem method 10-206-001-1, compliant with EPA method RoHS-2

- Ammonia – N, QuickChem method 31-107-06-1-B, EPA method 350.1
- Nitrate -N, QuickChem method 31-107-04-1-E, EPA method 353.4
- Nitrite -N, QuickChem method 31-107-05-1-A, EPA method 353.4
- Silicate, QuickChem method 31-114-27-1-D, EPA method 366
- Orthophosphate – P, QuickChem method 31-115-01-1-I, EPA method 365.5

Coastal Conservation and Research

- Grab samples for biological analyses are washed through a 0.5mm mesh Nitex (plastic) sieves by placing small amount of sediment on the screen, immersing and sloshing in seawater within buckets of small ice chests. These containers also function as safety basins; were screen residues to be spilled, the contents could be recovered from the container. Washing suspends the sample in water minimizing physical damage to retained organisms, maintaining them at a cool temperature, and preventing desiccation. After each part of the sample is washed the screen residues are washed into jars. Pre-printed labels are included in each jar. A 7% solution of magnesium chloride (MgCl) will be added to seawater and the sample left in ambient temperature seawater for 1 hour to allow animals, primarily polychaetes, to relax. Relaxation causes taxonomically important features to become observable or more easily observable, prevents contracted and contorted specimens, prevents autotomy (self-amputation), and otherwise provides better specimens and, therefore, more accurate counts. After one hour, formaldehyde is added to the seawater to make a 4% solution which fixes the organisms. The samples are thoroughly mixed with formaldehyde to ensure proper and fast fixation. Organic stains, such as rose Bengal, are generally not used because they distort coloration and obscure characters useful in identification.
- Biological sample sorting begins with swirling the residue and decanting it with the preserving alcohol through a 0.25 mm screen. This screen residue is washed into a petri dish and the alcohol temporarily stored in a sealed jar. Subsequent swirling first in alcohol and then fresh water brings off increasingly dense residue. Most animals come off with the initial swirls, generally only shelled animals (molluscs) are swirled off later. Since swirling things by specific density, not only were most animals separated from most of the residue, but animal groups tend to separate from each other, allowing sorting to be faster and more accurate. High resolution dissecting microscopes (Nikon and Olympus) with high intensity (fiber optic) light sources are used to sort the remaining sample materials after swirling. Animals are sorted in water with fine forceps from residue into appropriate size container, mostly 1dm glass shell vials, separated into phylogenetic groups: Crustacea, Mollusca, Polychaeta, Echinodermata, and other. A label is placed into each vial and the animals stored in fresh alcohol.
- Specimens will be distributed to taxonomists experts in each group. The lab manager oversees all COC protocols to ensure that samples are properly tracked. Specimens will be identified to the lowest practical level, usually species but for cryptic groups (nematodes, oligochaetes, flatworms, etc.) to genus or family or class as appropriate. It is not feasible or necessary to identify everything to species for several reasons; many species are un-described, many cryptic groups are very time consuming to identify while yielding little interpretable information, taxonomic experts are not available for some

groups, and smaller groups are only partially retained on 0.5 mm screens (which could lead to erroneous data interpretations).

These SOPs from SGS AXYS, Physis, SLAB, and CCR are proprietary and are available for examination at the Program Director's office in Santa Cruz, CA.

### 13.4. In Situ Monitoring

Although there are no in situ instruments used for ambient monitoring, measurement probes used for in situ measurements of wastewater influent and effluent are calibrated according to approved EPA methods with SOPs on file at each wastewater treatment plant.

### 13.5. Method Failures

If failures of analytical DQOs occur, the first person to be notified will be the QA officer of each respective laboratory. If a resolution cannot be achieved internally, the problem will be discussed with the Program Director to arrive at an acceptable solution. If failures involve matrix interferences that could be resolved with method revisions, additional analyses may be approved by the Program Director with concurrence of the CCLEAN Steering Committee. All failures and corrective actions taken will be documented in the narrative analytical report submitted to the Program Director with each batch of data.

### 13.6. Sample Disposal

CCLEAN samples will be archived by the respective analytical laboratory until disposal is approved by the Program Director. Disposal of any samples will be according to applicable environmental regulations.

### 13.7. Data Delivery

Data for the period July 1 to June 30 shall be delivered to the CCLEAN Program Director no later than the following November 1.

Table 19. Methods and Target MDLs for non-POP Constituents in Ocean Water, Sediment, and Tissue.

| Analysis | Matrix | Reporting Units | Suggested Analytical Methods | MDL |
|---|---|---|---|---|
| Ammonia as N | water (dissolved) | µg/L | EPA 350.1 | 5 |
| Nitrate as N | water (dissolved) | µg/L | EPA 353.4 | 5 |
| Orthophosphate as P | water (dissolved) | µg/L | EPA 365.5 | 1 |
| Urea as N | water (dissolved) | µg/L | Mulvenna and Savidge (1992)<br>Goeyens, et al (1998) | 15 |
| PATHOGEN INDICATORS | | | | |
| Enterococcus | water | colonies/100 mL | SM 9230B, SM 9230C or Enterolert | 10 |
| Coliform, Fecal | water | MPN/100 mL | SM 9221E, SM 9222D (25-tube dilution) or Colilert | 10 |
| Coliform, Total | water | MPN/100 mL | SM 9221B, SM 9222B (25-tube dilution) or Colilert[1] | 10 |
| Silicate as Si (Silica as SiO2) | water (dissolved) | µg/L | Grasshoff and Kremling (1983) | 0.09 |
| Total Suspended Solids | water | mg/L | EPA 160.2<br>SM 2540D | 0.5 |
| Temperature | water | °C | EPA 0170.1 | 0.1 |
| pH | water | units | EPA 150.1<br>SM 4500HB | 0.1 |

| Analysis | Matrix | Reporting Units | Suggested Analytical Methods | MDL |
|---|---|---|---|---|
| SEDIMENT GRAIN SIZE ANALYSIS<br>Gravel<br>Sand<br>Silt<br>Clay | sediment (4 fractions) | %<br>(Granule 2.0 to <4.0 mm; Sand 0.0625 to <2.0 mm; Silt 0.0039 to <0.00625 mm; Clay <0.0039 mm) | SM 2560 | 0.05% |
| Total Organic Carbon | sediment | % dw (dry weight) | EPA 9060M | 0.01 |
| Moisture | sediment, mussel tissue | % ww (wet weight) | Lauenstein and Cantillo (1993) | 0.1 |
| Lipid | mussel tissue | % ww | Lauenstein and Cantillo (1993) | 0.1 |
| PATHOGEN INDICATORS<br>Enterococcus | tissue (mussels) | MPN/100 g | American Public Health Association (1970) | 20 |
| Coliform, Fecal | tissue (mussels) | MPN/100 g | American Public Health Association (1970) | 20 |
| Coliform, Total | tissue (mussels) | MPN/100 g | American Public Health Association (1970) | 20 |
| SPECIES IDENTIFICATION | Organism (benthics) | taxon | Lab SOP | N/A |

[1] = Colilert may not be used in marine water samples.

Table 20. Target RLs for POPs in Water, Sediment, and Mussel Tissue. Co-eluting PCB and PBDE congeners are indicated in this table, but they are reported individually in CEDEN submittals.

| Matrix | Parameter & Analyte | Units | Suggested Analytical Methods | Target RL |
|---|---|---|---|---|
| | PAHs | | | |
| Water | Methylnaphthalene, 1- | ng/L | SGS AXYS MLA-021 | 0.167 |
| Water | Trimethylnaphthalene, 2,3,5- | ng/L | SGS AXYS MLA-021 | 0.167 |
| Water | Dimethylnaphthalene, 2,6- | ng/L | SGS AXYS MLA-021 | 0.167 |
| Water | Methylnaphthalene, 2- | ng/L | SGS AXYS MLA-021 | 0.167 |
| Water | Biphenyl | ng/L | SGS AXYS MLA-021 | 0.167 |
| Water | Naphthalene | ng/L | SGS AXYS MLA-021 | 0.083 |
| Water | Methylphenanthrene, 1- | ng/L | SGS AXYS MLA-021 | 0.167 |
| Water | Acenaphthene | ng/L | SGS AXYS MLA-021 | 0.083 |
| Water | Acenaphthylene | ng/L | SGS AXYS MLA-021 | 0.083 |
| Water | Anthracene | ng/L | SGS AXYS MLA-021 | 0.083 |
| Water | Fluorene | ng/L | SGS AXYS MLA-021 | 0.083 |
| Water | Phenanthrene | ng/L | SGS AXYS MLA-021 | 0.083 |
| Water | Benz(a)anthracene | ng/L | SGS AXYS MLA-021 | 0.083 |
| Water | Chrysene | ng/L | SGS AXYS MLA-021 | 0.083 |
| Water | Fluoranthene | ng/L | SGS AXYS MLA-021 | 0.083 |
| Water | Pyrene | ng/L | SGS AXYS MLA-021 | 0.083 |
| Water | Benzo(a)pyrene | ng/L | SGS AXYS MLA-021 | 0.083 |
| Water | Benzo(b)fluoranthene | ng/L | SGS AXYS MLA-021 | 0.083 |
| Water | Benzo(e)pyrene | ng/L | SGS AXYS MLA-021 | 0.083 |
| Water | Benzo(k)fluoranthene | ng/L | SGS AXYS MLA-021 | 0.083 |
| Water | Dibenz(a,h)anthracene | ng/L | SGS AXYS MLA-021 | 0.167 |
| Water | Perylene | ng/L | SGS AXYS MLA-021 | 0.167 |
| Water | Benzo(g,h,i)perylene | ng/L | SGS AXYS MLA-021 | 0.167 |
| Water | Indeno(1,2,3-c,d)pyrene | ng/L | SGS AXYS MLA-021 | 0.167 |
| Water | Dibenzothiophene | ng/L | SGS AXYS MLA-021 | 0.167 |
| | Pesticides | | | |
| Water | Cyclopentadienes | | | |
| Water | Aldrin | ng/L | SGS AXYS MLA-228 | 0.003 |

| Matrix | Parameter & Analyte | Units | Suggested Analytical Methods | Target RL |
|---|---|---|---|---|
| Water | Dieldrin | ng/L | SGS AXYS MLA-228 | 0.008 |
| Water | Endrin | ng/L | SGS AXYS MLA-228 | 0.008 |
| Water | Chlordanes | | | |
| Water | Chlordane, cis- (alpha) | ng/L | SGS AXYS MLA-228 | 0.003 |
| Water | Chlordane, trans- (gamma) | ng/L | SGS AXYS MLA-228 | 0.003 |
| Water | Nonachlor, cis- (alpha) | ng/L | SGS AXYS MLA-228 | 0.003 |
| Water | Nonachlor, trans- (gamma) | ng/L | SGS AXYS MLA-228 | 0.003 |
| SGS AXYS MLA-228Water | Heptachlor | ng/L | SGS AXYS MLA-228 | 0.003 |
| Water | Heptachlor Epoxide | ng/L | SGS AXYS MLA-228 | 0.008 |
| Water | Oxychlordane | ng/L | SGS AXYS MLA-228 | 0.003 |
| SGS AXYS MLA-228 | DDTs | | | |
| Water | DDD(o,p') | ng/L | SGS AXYS MLA-228 | 0.003 |
| Water | DDE(o,p') | ng/L | SGS AXYS MLA-228 | 0.003 |
| Water | DDT(o,p') | ng/L | SGS AXYS MLA-228 | 0.003 |
| Water | DDD(p,p') | ng/L | SGS AXYS MLA-228 | 0.003 |
| Water | DDE(p,p') | ng/L | SGS AXYS MLA-228 | 0.003 |
| Water | DDT(p,p') | ng/L | SGS AXYS MLA-228 | 0.003 |
| | HCH | | | |
| Water | HCH, alpha | ng/L | SGS AXYS MLA-228 | 0.003 |
| Water | HCH, beta | ng/L | SGS AXYS MLA-228 | 0.003 |
| Water | HCH, delta | ng/L | SGS AXYS MLA-228 | 0.008 |
| Water | HCH, gamma | ng/L | SGS AXYS MLA-228 | 0.003 |
| | Other | | | |
| Water | Dacthal | ng/L | SGS AXYS MLA-228 | NA |
| Water | Endosulfan I | ng/L | SGS AXYS MLA-228 | 0.008 |
| Water | Endosulfan II | ng/L | SGS AXYS MLA-228 | 0.008 |
| Water | Endosulfan Sulfate | ng/L | SGS AXYS MLA-228 | 0.008 |

| Matrix | Parameter & Analyte | Units | Suggested Analytical Methods | Target RL |
|---|---|---|---|---|
| Water | Oxadiazon | ng/L | SGS AXYS MLA-228 | NA |
| Water | Mirex | ng/L | SGS AXYS MLA-228 | 0.003 |
| Water | Hexachlorobenzene | ng/L | SGS AXYS MLA-228 | 0.002 |
| Water | Toxaphene | ng/L | SGS AXYS MLA-228 | NA |
| Water | Hexachlorobutadiene | ng/L | SGS AXYS MLA-228 | NA |
| | Fipronil & Degradates | | | |
| Water | Fipronil | ng/L | EPA 625-NCl | 0.2 |
| Water | Fipronil Desulfinyl | ng/L | EPA 625-NCl | 0.2 |
| Water | Fipronil Sulfide | ng/L | EPA 625-NCl | 0.2 |
| Water | Fipronil Sulfone | ng/L | EPA 625-NCl | 0.2 |
| | Pyrethroid Pesticides | | | |
| Water | Allethrin | ng/L | EPA 625-NCl | 0.2 |
| Water | Bifenthrin | ng/L | EPA 625-NCl | 0.2 |
| Water | Cyfluthrin | ng/L | EPA 625-NCl | 0.2 |
| Water | Cyhalothrin, Total Lambda | ng/L | EPA 625-NCl | 0.2 |
| Water | Cypermethrin | ng/L | EPA 625-NCl | 0.2 |
| Water | Danitol (Fenpropathrin) | ng/L | EPA 625-NCl | 0.2 |
| Water | Deltamethrin/Tralomethrin | ng/L | EPA 625-NCl | 0.2 |
| Water | Esfenvalerate | ng/L | EPA 625-NCl | 0.2 |
| Water | Fenvalerate | ng/L | EPA 625-NCl | 0.2 |
| Water | Fluvalinate | ng/L | EPA 625-NCl | 0.2 |
| Water | Permethrin, cis- | ng/L | EPA 625-NCl | 0.2 |
| Water | Permethrin, trans- | ng/L | EPA 625-NCl | 0.2 |
| Water | Prallethrin | ng/L | EPA 625-NCl | 0.2 |
| Water | Tetramethrin | ng/L | EPA 625-NCl | 0.2 |
| | Neonicotinoid Pesticides | | | |
| Water | Acetamiprid | ng/L | EPA 625 | 2 |
| Water | Clothianidin | ng/L | EPA 625 | 2 |
| Water | Dinotefuran | ng/L | EPA 625 | 1.2 |
| Water | Imidacloprid | ng/L | EPA 625 | 0.4 |

| Matrix | Parameter & Analyte | Units | Suggested Analytical Methods | Target RL |
|---|---|---|---|---|
| Water | Thiacloprid | ng/L | EPA 625 | 0.4 |
| Water | Thiamethoxam | ng/L | EPA 625 | 0.4 |
| | Organophosphorus Pesticides | | | |
| Water | Azinphos-methyl | ng/L | EPA 625 | |
| Water | Bolstar (Sulprofos) | ng/L | EPA 625 | 4.0 |
| Water | Chlorpyrifos | ng/L | EPA 625 | 1.0 |
| Water | Demeton | ng/L | EPA 625 | 2.0 |
| Water | Diazinon | ng/L | EPA 625 | 1.0 |
| Water | Dichlorvos | ng/L | EPA 625 | 6.0 |
| Water | Dimethoate | ng/L | EPA 625 | 10.0 |
| Water | Disulfoton | ng/L | EPA 625 | 2.0 |
| Water | Ethoprop (Ethoprofos) | ng/L | EPA 625 | 2.0 |
| Water | Fenchlorphos (Ronnel) | ng/L | EPA 625 | 4.0 |
| Water | Fensulfothion | ng/L | EPA 625 | 2.0 |
| Water | Fenthion | ng/L | EPA 625 | 4.0 |
| Water | Malathion | ng/L | EPA 625 | 5.00 |
| Water | Methamidophos | ng/L | EPA 625 | |
| Water | Methidathion | ng/L | EPA 625 | 10.0 |
| Water | Methyl Parathion | ng/L | EPA 625 | 2.0 |
| Water | Mevinphos (Phosdrin) | ng/L | EPA 625 | 10.0 |
| Water | Phorate | ng/L | EPA 625 | 10.0 |
| Water | Phosmet | ng/L | EPA 625 | 10.0 |
| Water | Tetrachlorvinphos (Stirofos) | ng/L | EPA 625 | 4.0 |
| Water | Tokuthion (Prothiofos) | ng/L | EPA 625 | 6.0 |
| Water | Trichloronate | ng/L | EPA 625 | 2.0 |
| | Phenolics | | | |
| Water | 2,4,5-Trichlorophenol | ng/L | EPA 625 | 100 |
| Water | 2,4,6-Trichlorophenol | ng/L | EPA 625 | 100 |
| Water | 2,4-Dichlorophenol | ng/L | EPA 625 | 100 |
| Water | 2,4-Dimethylphenol | ng/L | EPA 625 | 200 |

| Matrix | Parameter & Analyte | Units | Suggested Analytical Methods | Target RL |
|---|---|---|---|---|
| Water | 2,4-Dinitrophenol | ng/L | EPA 625 | 200 |
| Water | 2-Chlorophenol | ng/L | EPA 625 | 100 |
| Water | 2-Methyl-4,6-dinitrophenol | ng/L | EPA 625 | 200 |
| Water | 2-Methylphenol | ng/L | EPA 625 | 200 |
| Water | 2-Nitrophenol | ng/L | EPA 625 | 200 |
| Water | 3-Methylphenol | ng/L | EPA 625 | 200 |
| Water | 4-Chloro-3-methylphenol | ng/L | EPA 625 | 200 |
| Water | 4-Methylphenol | ng/L | EPA 625 | 200 |
| Water | 4-Nitrophenol | ng/L | EPA 625 | 200 |
| Water | Pentachlorophenol | ng/L | EPA 625 | 100 |
| Water | Phenol | ng/L | EPA 625 | 200 |
| | Carbamates | | | |
| Water | 3-Hydroxycarbofuran | µg/L | EPA 8318 | 1 |
| Water | Aldicarb | µg/L | EPA 8318 | 1 |
| Water | Aldicarb sulfone | µg/L | EPA 8318 | 1 |
| Water | Carbaryl | µg/L | EPA 8318 | 1 |
| Water | Carbofuran | µg/L | EPA 8318 | 1 |
| Water | Methiocarb | µg/L | EPA 8318 | 1 |
| Water | Methomyl | µg/L | EPA 8318 | 1 |
| Water | Oxamyl | µg/L | EPA 8318 | 1 |
| Water | Propoxur (Baygon) | µg/L | EPA 8318 | 1 |
| | Trace Metals | | | |
| Water | Aluminum (Al) | µg/L | EPA 200.8 | 8.25 |
| Water | Antimony (Sb) | µg/L | EPA 200.8 | 0.15 |
| Water | Arsenic (As) | µg/L | EPA 200.8 | 0.16 |
| Water | Barium (Ba) | µg/L | EPA 200.8 | 0.50 |
| Water | Beryllium (Be) | µg/L | EPA 200.8 | 0.03 |
| Water | Boron (B) | µg/L | EPA 200.8 | |
| Water | Cadmium (Cd) | µg/L | EPA 200.8 | 0.02 |
| Water | Chromium (Cr) | µg/L | EPA 200.8 | 0.05 |

| Matrix | Parameter & Analyte | Units | Suggested Analytical Methods | Target RL |
|---|---|---|---|---|
| Water | Cobalt (Co) | µg/L | EPA 200.8 | 0.05 |
| Water | Copper (Cu) | µg/L | EPA 200.8 | 0.02 |
| Water | Iron (Fe) | µg/L | EPA 200.8 | 5.65 |
| Water | Lead (Pb) | µg/L | EPA 200.8 | 0.02 |
| Water | Manganese (Mn) | µg/L | EPA 200.8 | 0.01 |
| Water | Molybdenum (Mo) | µg/L | EPA 200.8 | 0.02 |
| Water | Nickel (Ni) | µg/L | EPA 200.8 | 0.04 |
| Water | Selenium (Se) | µg/L | EPA 200.8 | 0.07 |
| Water | Silver (Ag) | µg/L | EPA 200.8 | 0.02 |
| Water | Strontium (Sr) | µg/L | EPA 200.8 | 0.15 |
| Water | Thallium (Tl) | µg/L | EPA 200.8 | 0.05 |
| Water | Tin (Sn) | µg/L | EPA 200.8 | 0.30 |
| Water | Titanium (Ti) | µg/L | EPA 200.8 | 0.40 |
| Water | Vanadium (V) | µg/L | EPA 200.8 | 0.15 |
| Water | Zinc | µg/L | EPA 200.8 | 0.07 |
| Water | Water Hardness | | | |
| | PCB congeners | | | |
| Water | PCB 001 | pg/L | SGS AXYS MLA-210 | 0.008 |
| Water | PCB 002 | pg/L | SGS AXYS MLA-210 | 0.008 |
| Water | PCB 003 | pg/L | SGS AXYS MLA-210 | 0.008 |
| Water | PCB 004 | pg/L | SGS AXYS MLA-210 | 0.008 |
| Water | PCB 005 | pg/L | SGS AXYS MLA-210 | 0.008 |
| Water | PCB 006 | pg/L | SGS AXYS MLA-210 | 0.008 |
| Water | PCB 007 | pg/L | SGS AXYS MLA-210 | 0.008 |
| Water | PCB 008 | pg/L | SGS AXYS MLA-210 | 0.008 |
| Water | PCB 009 | pg/L | SGS AXYS MLA-210 | 0.008 |
| Water | PCB 010 | pg/L | SGS AXYS MLA-210 | 0.008 |
| Water | PCB 011 | pg/L | SGS AXYS MLA-210 | 0.008 |
| Water | PCB 012/13 | pg/L | SGS AXYS MLA-210 | 0.008 |
| Water | PCB 014 | pg/L | SGS AXYS MLA-210 | 0.008 |
| Water | PCB 015 | pg/L | SGS AXYS MLA-210 | 0.008 |
| Water | PCB 016 | pg/L | SGS AXYS MLA-210 | 0.008 |

| Matrix | Parameter & Analyte | Units | Suggested Analytical Methods | Target RL |
|---|---|---|---|---|
| Water | PCB 017 | pg/L | SGS AXYS MLA-210 | 0.008 |
| Water | PCB 018/30 | pg/L | SGS AXYS MLA-210 | 0.008 |
| Water | PCB 019 | pg/L | SGS AXYS MLA-210 | 0.008 |
| Water | PCB 020/28 | pg/L | SGS AXYS MLA-210 | 0.008 |
| Water | PCB 021/33 | pg/L | SGS AXYS MLA-210 | 0.008 |
| Water | PCB 022 | pg/L | SGS AXYS MLA-210 | 0.008 |
| Water | PCB 023 | pg/L | SGS AXYS MLA-210 | 0.008 |
| Water | PCB 024 | pg/L | SGS AXYS MLA-210 | 0.008 |
| Water | PCB 025 | pg/L | SGS AXYS MLA-210 | 0.008 |
| Water | PCB 026/29 | pg/L | SGS AXYS MLA-210 | 0.008 |
| Water | PCB 027 | pg/L | SGS AXYS MLA-210 | 0.008 |
| Water | PCB 031 | pg/L | SGS AXYS MLA-210 | 0.008 |
| Water | PCB 032 | pg/L | SGS AXYS MLA-210 | 0.008 |
| Water | PCB 034 | pg/L | SGS AXYS MLA-210 | 0.008 |
| Water | PCB 035 | pg/L | SGS AXYS MLA-210 | 0.008 |
| Water | PCB 036 | pg/L | SGS AXYS MLA-210 | 0.008 |
| Water | PCB 037 | pg/L | SGS AXYS MLA-210 | 0.008 |
| Water | PCB 038 | pg/L | SGS AXYS MLA-210 | 0.008 |
| Water | PCB 039 | pg/L | SGS AXYS MLA-210 | 0.008 |
| Water | PCB 040/41/71 | pg/L | SGS AXYS MLA-210 | 0.008 |
| Water | PCB 042 | pg/L | SGS AXYS MLA-210 | 0.008 |
| Water | PCB 043 | pg/L | SGS AXYS MLA-210 | 0.008 |
| Water | PCB 044/47/65 | pg/L | SGS AXYS MLA-210 | 0.008 |
| Water | PCB 045/51 | pg/L | SGS AXYS MLA-210 | 0.008 |
| Water | PCB 046 | pg/L | SGS AXYS MLA-210 | 0.008 |
| Water | PCB 048 | pg/L | SGS AXYS MLA-210 | 0.008 |
| Water | PCB 049/69 | pg/L | SGS AXYS MLA-210 | 0.008 |
| Water | PCB 050/53 | pg/L | SGS AXYS MLA-210 | 0.008 |
| Water | PCB 052 | pg/L | SGS AXYS MLA-210 | 0.008 |
| Water | PCB 054 | pg/L | SGS AXYS MLA-210 | 0.008 |
| Water | PCB 055 | pg/L | SGS AXYS MLA-210 | 0.008 |
| Water | PCB 056 | pg/L | SGS AXYS MLA-210 | 0.008 |

| Matrix | Parameter & Analyte | Units | Suggested Analytical Methods | Target RL |
|---|---|---|---|---|
| Water | PCB 057 | pg/L | SGS AXYS MLA-210 | 0.008 |
| Water | PCB 058 | pg/L | SGS AXYS MLA-210 | 0.008 |
| Water | PCB 059/62/75 | pg/L | SGS AXYS MLA-210 | 0.008 |
| Water | PCB 060 | pg/L | SGS AXYS MLA-210 | 0.008 |
| Water | PCB 061/70/74/76 | pg/L | SGS AXYS MLA-210 | 0.008 |
| Water | PCB 063 | pg/L | SGS AXYS MLA-210 | 0.008 |
| Water | PCB 064 | pg/L | SGS AXYS MLA-210 | 0.008 |
| Water | PCB 066 | pg/L | SGS AXYS MLA-210 | 0.008 |
| Water | PCB 067 | pg/L | SGS AXYS MLA-210 | 0.008 |
| Water | PCB 068 | pg/L | SGS AXYS MLA-210 | 0.008 |
| Water | PCB 072 | pg/L | SGS AXYS MLA-210 | 0.008 |
| Water | PCB 073 | pg/L | SGS AXYS MLA-210 | 0.008 |
| Water | PCB 077 | pg/L | SGS AXYS MLA-210 | 0.008 |
| Water | PCB 078 | pg/L | SGS AXYS MLA-210 | 0.008 |
| Water | PCB 079 | pg/L | SGS AXYS MLA-210 | 0.008 |
| Water | PCB 080 | pg/L | SGS AXYS MLA-210 | 0.008 |
| Water | PCB 081 | pg/L | SGS AXYS MLA-210 | 0.008 |
| Water | PCB 082 | pg/L | SGS AXYS MLA-210 | 0.008 |
| Water | PCB 083/99 | pg/L | SGS AXYS MLA-210 | 0.008 |
| Water | PCB 084 | pg/L | SGS AXYS MLA-210 | 0.008 |
| Water | PCB 085/116/117 | pg/L | SGS AXYS MLA-210 | 0.008 |
| Water | PCB 086/97/108/119/125 | pg/L | SGS AXYS MLA-210 | 0.008 |
| Water | PCB 088/91 | pg/L | SGS AXYS MLA-210 | 0.008 |
| Water | PCB 089 | pg/L | SGS AXYS MLA-210 | 0.008 |
| Water | PCB /90/101/113 | pg/L | SGS AXYS MLA-210 | 0.008 |
| Water | PCB 092 | pg/L | SGS AXYS MLA-210 | 0.008 |
| Water | PCB 093/95/98/100/102 | pg/L | SGS AXYS MLA-210 | 0.008 |
| Water | PCB 094 | pg/L | SGS AXYS MLA-210 | 0.008 |
| Water | PCB 096 | pg/L | SGS AXYS MLA-210 | 0.008 |
| Water | PCB 103 | pg/L | SGS AXYS MLA-210 | 0.008 |
| Water | PCB 104 | pg/L | SGS AXYS MLA-210 | 0.008 |

| Matrix | Parameter & Analyte | Units | Suggested Analytical Methods | Target RL |
|---|---|---|---|---|
| Water | PCB 105 | pg/L | SGS AXYS MLA-210 | 0.008 |
| Water | PCB 106 | pg/L | SGS AXYS MLA-210 | 0.008 |
| Water | PCB 107/124 | pg/L | SGS AXYS MLA-210 | 0.008 |
| Water | PCB 109 | pg/L | SGS AXYS MLA-210 | 0.008 |
| Water | PCB 110/115 | pg/L | SGS AXYS MLA-210 | 0.008 |
| Water | PCB 111 | pg/L | SGS AXYS MLA-210 | 0.008 |
| Water | PCB 112 | pg/L | SGS AXYS MLA-210 | 0.008 |
| Water | PCB 114 | pg/L | SGS AXYS MLA-210 | 0.008 |
| Water | PCB 118 | pg/L | SGS AXYS MLA-210 | 0.008 |
| Water | PCB 120 | pg/L | SGS AXYS MLA-210 | 0.008 |
| Water | PCB 121 | pg/L | SGS AXYS MLA-210 | 0.008 |
| Water | PCB 122 | pg/L | SGS AXYS MLA-210 | 0.008 |
| Water | PCB 123 | pg/L | SGS AXYS MLA-210 | 0.008 |
| Water | PCB 126 | pg/L | SGS AXYS MLA-210 | 0.008 |
| Water | PCB 127 | pg/L | SGS AXYS MLA-210 | 0.008 |
| Water | PCB 128/166 | pg/L | SGS AXYS MLA-210 | 0.008 |
| Water | PCB 129/138/160/163 | pg/L | SGS AXYS MLA-210 | 0.008 |
| Water | PCB 130 | pg/L | SGS AXYS MLA-210 | 0.008 |
| Water | PCB 131 | pg/L | SGS AXYS MLA-210 | 0.008 |
| Water | PCB 132 | pg/L | SGS AXYS MLA-210 | 0.008 |
| Water | PCB 133 | pg/L | SGS AXYS MLA-210 | 0.008 |
| Water | PCB 134/143 | pg/L | SGS AXYS MLA-210 | 0.008 |
| Water | PCB 135/151/154 | pg/L | SGS AXYS MLA-210 | 0.008 |
| Water | PCB 136 | pg/L | SGS AXYS MLA-210 | 0.008 |
| Water | PCB 137 | pg/L | SGS AXYS MLA-210 | 0.008 |
| Water | PCB 139/140 | pg/L | SGS AXYS MLA-210 | 0.008 |
| Water | PCB 141 | pg/L | SGS AXYS MLA-210 | 0.008 |
| Water | PCB 142 | pg/L | SGS AXYS MLA-210 | 0.008 |
| Water | PCB 144 | pg/L | SGS AXYS MLA-210 | 0.008 |
| Water | PCB 145 | pg/L | SGS AXYS MLA-210 | 0.008 |
| Water | PCB 146 | pg/L | SGS AXYS MLA-210 | 0.008 |
| Water | PCB 147/149 | pg/L | SGS AXYS MLA-210 | 0.008 |

| Matrix | Parameter & Analyte | Units | Suggested Analytical Methods | Target RL |
|---|---|---|---|---|
| Water | PCB 148 | pg/L | SGS AXYS MLA-210 | 0.008 |
| Water | PCB 150 | pg/L | SGS AXYS MLA-210 | 0.008 |
| Water | PCB 152 | pg/L | SGS AXYS MLA-210 | 0.008 |
| Water | PCB 153/168 | pg/L | SGS AXYS MLA-210 | 0.008 |
| Water | PCB 155 | pg/L | SGS AXYS MLA-210 | 0.008 |
| Water | PCB 156/157 | pg/L | SGS AXYS MLA-210 | 0.008 |
| Water | PCB 158 | pg/L | SGS AXYS MLA-210 | 0.008 |
| Water | PCB 159 | pg/L | SGS AXYS MLA-210 | 0.008 |
| Water | PCB 161 | pg/L | SGS AXYS MLA-210 | 0.008 |
| Water | PCB 162 | pg/L | SGS AXYS MLA-210 | 0.008 |
| Water | PCB 164 | pg/L | SGS AXYS MLA-210 | 0.008 |
| Water | PCB 165 | pg/L | SGS AXYS MLA-210 | 0.008 |
| Water | PCB 167 | pg/L | SGS AXYS MLA-210 | 0.008 |
| Water | PCB 169 | pg/L | SGS AXYS MLA-210 | 0.008 |
| Water | PCB 170 | pg/L | SGS AXYS MLA-210 | 0.008 |
| Water | PCB 171/173 | pg/L | SGS AXYS MLA-210 | 0.008 |
| Water | PCB 172 | pg/L | SGS AXYS MLA-210 | 0.008 |
| Water | PCB 174 | pg/L | SGS AXYS MLA-210 | 0.008 |
| Water | PCB 175 | pg/L | SGS AXYS MLA-210 | 0.008 |
| Water | PCB 176 | pg/L | SGS AXYS MLA-210 | 0.008 |
| Water | PCB 177 | pg/L | SGS AXYS MLA-210 | 0.008 |
| Water | PCB 178 | pg/L | SGS AXYS MLA-210 | 0.008 |
| Water | PCB 179 | pg/L | SGS AXYS MLA-210 | 0.008 |
| Water | PCB 180/193 | pg/L | SGS AXYS MLA-210 | 0.008 |
| Water | PCB 181 | pg/L | SGS AXYS MLA-210 | 0.008 |
| Water | PCB 182 | pg/L | SGS AXYS MLA-210 | 0.008 |
| Water | PCB 183/185 | pg/L | SGS AXYS MLA-210 | 0.008 |
| Water | PCB 184 | pg/L | SGS AXYS MLA-210 | 0.008 |
| Water | PCB 186 | pg/L | SGS AXYS MLA-210 | 0.008 |
| Water | PCB 187 | pg/L | SGS AXYS MLA-210 | 0.008 |
| Water | PCB 188 | pg/L | SGS AXYS MLA-210 | 0.008 |
| Water | PCB 189 | pg/L | SGS AXYS MLA-210 | 0.008 |

| Matrix | Parameter & Analyte | Units | Suggested Analytical Methods | Target RL |
|---|---|---|---|---|
| Water | PCB 190 | pg/L | SGS AXYS MLA-210 | 0.008 |
| Water | PCB 191 | pg/L | SGS AXYS MLA-210 | 0.008 |
| Water | PCB 192 | pg/L | SGS AXYS MLA-210 | 0.008 |
| Water | PCB 194 | pg/L | SGS AXYS MLA-210 | 0.008 |
| Water | PCB 195 | pg/L | SGS AXYS MLA-210 | 0.008 |
| Water | PCB 196 | pg/L | SGS AXYS MLA-210 | 0.008 |
| Water | PCB 197/200 | pg/L | SGS AXYS MLA-210 | 0.008 |
| Water | PCB 198/199 | pg/L | SGS AXYS MLA-210 | 0.008 |
| Water | PCB 201 | pg/L | SGS AXYS MLA-210 | 0.008 |
| Water | PCB 202 | pg/L | SGS AXYS MLA-210 | 0.008 |
| Water | PCB 203 | pg/L | SGS AXYS MLA-210 | 0.008 |
| Water | PCB 204 | pg/L | SGS AXYS MLA-210 | 0.008 |
| Water | PCB 205 | pg/L | SGS AXYS MLA-210 | 0.008 |
| Water | PCB 206 | pg/L | SGS AXYS MLA-210 | 0.008 |
| Water | PCB 207 | pg/L | SGS AXYS MLA-210 | 0.008 |
| Water | PCB 208 | pg/L | SGS AXYS MLA-210 | 0.008 |
| Water | PCB 209 | pg/L | SGS AXYS MLA-210 | 0.008 |
| Water | PBDE congeners | | | |
| Water | PBDE 007 | pg/L | EPA 1614 | 0.167 |
| Water | PBDE 88/11 | pg/L | EPA 1614 | 0.167 |
| Water | PBDE 010 | pg/L | EPA 1614 | 0.167 |
| Water | PBDR 12/13 | pg/L | EPA 1614 | 0.167 |
| Water | PBDE 015 | pg/L | EPA 1614 | 0.167 |
| Water | PBDE 017/25 | pg/L | EPA 1614 | 0.167 |
| Water | PBDE 028/33 | pg/L | EPA 1614 | 0.167 |
| Water | PBDE 030 | pg/L | EPA 1614 | 0.167 |
| Water | PBDE 032 | pg/L | EPA 1614 | 0.167 |
| Water | PBDE 035 | pg/L | EPA 1614 | 0.167 |
| Water | PBDE 037 | pg/L | EPA 1614 | 0.167 |
| Water | PBDE 047 [1] | pg/L | EPA 1614 | 0.167 |
| Water | PBDE 049 | pg/L | EPA 1614 | 0.167 |
| Water | PBDE 051 | pg/L | EPA 1614 | 0.167 |

| Matrix | Parameter & Analyte | Units | Suggested Analytical Methods | Target RL |
|---|---|---|---|---|
| Water | PBDE 066 | pg/L | EPA 1614 | 0.167 |
| Water | PBDE 071 | pg/L | EPA 1614 | 0.167 |
| Water | PBDE 075 | pg/L | EPA 1614 | 0.167 |
| Water | PBDE 077 | pg/L | EPA 1614 | 0.167 |
| Water | PBDE 079 | pg/L | EPA 1614 | 0.167 |
| Water | PBDE 085 | pg/L | EPA 1614 | 0.167 |
| Water | PBDE 099 [1] | pg/L | EPA 1614 | 0.167 |
| Water | PBDE 100 | pg/L | EPA 1614 | 0.167 |
| Water | PBDE 105 | pg/L | EPA 1614 | 0.167 |
| Water | PBDE 116 | pg/L | EPA 1614 | 0.167 |
| Water | PBDE 119/120 | pg/L | EPA 1614 | 0.167 |
| Water | PBDE 126 | pg/L | EPA 1614 | 0.167 |
| Water | PBDE 128 | pg/L | EPA 1614 | 0.167 |
| Water | PBDE 138/166 | pg/L | EPA 1614 | 0.167 |
| Water | PBDE 140 | pg/L | EPA 1614 | 0.167 |
| Water | PBDE 153 | pg/L | EPA 1614 | 0.167 |
| Water | PBDE 154 | pg/L | EPA 1614 | 0.167 |
| Water | PBDE 155 | pg/L | EPA 1614 | 0.167 |
| Water | PBDE 181 | pg/L | EPA 1614 | 0.333 |
| Water | PBDE 183 | pg/L | EPA 1614 | 0.333 |
| Water | PBDE 190 | pg/L | EPA 1614 | 0.333 |
| Water | PBDE 203 | pg/L | EPA 1614 | 0.333 |
| Water | PBDE 206 | pg/L | EPA 1614 | 1.667 |
| Water | PBDE 207 | pg/L | EPA 1614 | 1.667 |
| Water | PBDE 208 | pg/L | EPA 1614 | 1.667 |
| Water | PBDE 209 | pg/L | EPA 1614 | 3.333 |
| | Dioxins and Furans[1] | | | |
| Influent and Effluent | TCDD, 2,3,7,8- | pg/L | SGS AXYS MLA-217 | 0.008 |
| Influent and Effluent | PeCDD, 1,2,3,7,8- | pg/L | SGS AXYS MLA-217 | 0.008 |

| Matrix | Parameter & Analyte | Units | Suggested Analytical Methods | Target RL |
|---|---|---|---|---|
| Influent and Effluent | HxCDD, 1,2,3,4,7,8- | pg/L | SGS AXYS MLA-217 | 0.008 |
| Influent and Effluent | HxCDD, 1,2,3,6,7,8- | pg/L | SGS AXYS MLA-217 | 0.008 |
| Influent and Effluent | HxCDD, 1,2,3,7,8,9- | pg/L | SGS AXYS MLA-217 | 0.008 |
| Influent and Effluent | HpCDD, 1,2,3,4,6,7,8- | pg/L | SGS AXYS MLA-217 | 0.008 |
| Influent and Effluent | OCDD, 1,2,3,4,6,7,8,9- | pg/L | SGS AXYS MLA-217 | 0.008 |
| Influent and Effluent | TCDF, 2,3,7,8- | pg/L | SGS AXYS MLA-217 | 0.008 |
| Influent and Effluent | PeCDF, 1,2,3,7,8- | pg/L | SGS AXYS MLA-217 | 0.008 |
| Influent and Effluent | PeCDF, 2,3,4,7,8- | pg/L | SGS AXYS MLA-217 | 0.008 |
| Influent and Effluent | HxCDF, 1,2,3,4,7,8- | pg/L | SGS AXYS MLA-217 | 0.008 |
| Influent and Effluent | HxCDF, 1,2,3,6,7,8- | pg/L | SGS AXYS MLA-217 | 0.008 |
| Influent and Effluent | HxCDF, 1,2,3,7,8,9- | pg/L | SGS AXYS MLA-217 | 0.008 |
| Influent and Effluent | HxCDF, 2,3,4,6,7,8- | pg/L | SGS AXYS MLA-217 | 0.008 |
| Influent and Effluent | HpCDF, 1,2,3,4,6,7,8- | pg/L | SGS AXYS MLA-217 | 0.008 |
| Influent and Effluent | HpCDF, 1,2,3,4,7,8,9- | pg/L | SGS AXYS MLA-217 | 0.008 |
| Influent and Effluent | OCDF, 1,2,3,4,6,7,8,9- | pg/L | SGS AXYS MLA-217 | 0.008 |
| | PFAS | | | |
| Water | PFBA | ng/L | SGS AXYS MLA-110 | 3 |
| Water | PFPeA | ng/L | SGS AXYS MLA-110 | 2 |
| Water | PFHxA | ng/L | SGS AXYS MLA-110 | 0.8 |
| Water | PFHpA | ng/L | SGS AXYS MLA-110 | 0.8 |
| Water | PFOA | ng/L | SGS AXYS MLA-110 | 0.8 |

| Matrix | Parameter & Analyte | Units | Suggested Analytical Methods | Target RL |
|---|---|---|---|---|
| Water | PFNA | ng/L | SGS AXYS MLA-110 | 0.8 |
| Water | PFDA | ng/L | SGS AXYS MLA-110 | 0.8 |
| Water | PFUnA | ng/L | SGS AXYS MLA-110 | 0.8 |
| Water | PFDoA | ng/L | SGS AXYS MLA-110 | 0.8 |
| Water | PFTrDA | ng/L | SGS AXYS MLA-110 | 0.8 |
| Water | PFTeDA | ng/L | SGS AXYS MLA-110 | 0.8 |
| Water | PFBS | ng/L | SGS AXYS MLA-110 | 0.8 |
| Water | PFPeS | ng/L | SGS AXYS MLA-110 | 0.8 |
| Water | PFHxS | ng/L | SGS AXYS MLA-110 | 0.8 |
| Water | PFHpS | ng/L | SGS AXYS MLA-110 | 0.8 |
| Water | PFOS | ng/L | SGS AXYS MLA-110 | 0.8 |
| Water | PFNS | ng/L | SGS AXYS MLA-110 | 0.8 |
| Water | PFDS | ng/L | SGS AXYS MLA-110 | 0.8 |
| Water | PFDoS | ng/L | SGS AXYS MLA-110 | 0.8 |
| Water | 4:2 FTS | ng/L | SGS AXYS MLA-110 | 3 |
| Water | 6:2 FTS | ng/L | SGS AXYS MLA-110 | 3 |
| Water | 8:2 FTS | ng/L | SGS AXYS MLA-110 | 3 |
| Water | N-MeFOSA | ng/L | SGS AXYS MLA-110 | 0.8 |
| Water | N-EtFOSA | ng/L | SGS AXYS MLA-110 | 0.8 |
| Water | PFOSA | ng/L | SGS AXYS MLA-110 | 0.8 |
| Water | N-MeFOSAA | ng/L | SGS AXYS MLA-110 | 0.8 |
| Water | N-EtFOSAA | ng/L | SGS AXYS MLA-110 | 0.8 |
| Water | N-MeFOSE | ng/L | SGS AXYS MLA-110 | 8 |
| Water | N-EtFOSE | ng/L | SGS AXYS MLA-110 | 8 |
| Water | HFPO-DA | ng/L | SGS AXYS MLA-110 | 3 |
| Water | ADONA | ng/L | SGS AXYS MLA-110 | 3 |
| Water | 9Cl-PF3ONS | ng/L | SGS AXYS MLA-110 | 3 |
| Water | 11Cl-PF3OUdS | ng/L | SGS AXYS MLA-110 | 3 |
| | PAHs | | | |
| Sediment[2] | Methylnaphthalene, 1- | µg/kg | SGS AXYS MLA-021 | 1.0 |
| Sediment[2] | Trimethylnaphthalene, 2,3,5- | µg/kg | SGS AXYS MLA-021 | 1.0 |

| Matrix | Parameter & Analyte | Units | Suggested Analytical Methods | Target RL |
|---|---|---|---|---|
| Sediment[2] | Dimethylnaphthalene, 2,6- | µg/kg | SGS AXYS MLA-021 | 1.0 |
| Sediment[2] | Methylnaphthalene, 2- | µg/kg | SGS AXYS MLA-021 | 1.0 |
| Sediment[2] | Biphenyl | µg/kg | SGS AXYS MLA-021 | 1.0 |
| Sediment[2] | Naphthalene | µg/kg | SGS AXYS MLA-021 | 0.5 |
| Sediment[2] | Methylphenanthrene, 1- | µg/kg | SGS AXYS MLA-021 | 1.0 |
| Sediment[2] | Acenaphthene | µg/kg | SGS AXYS MLA-021 | 0.5 |
| Sediment[2] | Acenaphthylene | µg/kg | SGS AXYS MLA-021 | 0.5 |
| Sediment[2] | Anthracene | µg/kg | SGS AXYS MLA-021 | 0.5 |
| Sediment[2] | Fluorene | µg/kg | SGS AXYS MLA-021 | 0.5 |
| Sediment[2] | Phenanthrene | µg/kg | SGS AXYS MLA-021 | 0.5 |
| Sediment[2] | Benzo(a)anthracene | µg/kg | SGS AXYS MLA-021 | 0.5 |
| Sediment[2] | Chrysene | µg/kg | SGS AXYS MLA-021 | 0.5 |
| Sediment[2] | Fluoranthene | µg/kg | SGS AXYS MLA-021 | 0.5 |
| Sediment[2] | Pyrene | µg/kg | SGS AXYS MLA-021 | 0.5 |
| Sediment[2] | Benzo(a)pyrene | µg/kg | SGS AXYS MLA-021 | 0.5 |
| Sediment[2] | Benzo(b)fluoranthene | µg/kg | SGS AXYS MLA-021 | 0.5 |
| Sediment[2] | Benzo(e)pyrene | µg/kg | SGS AXYS MLA-021 | 0.5 |
| Sediment[2] | Benzo(j,k)fluoranthenes | µg/kg | SGS AXYS MLA-021 | 0.5 |
| Sediment[2] | Dibenz(a,h)anthracene | µg/kg | SGS AXYS MLA-021 | 1.0 |
| Sediment[2] | Perylene | µg/kg | SGS AXYS MLA-021 | 1.0 |
| Sediment[2] | Benzo(g,h,i)perylene | µg/kg | SGS AXYS MLA-021 | 1.0 |
| Sediment[2] | Indeno(1,2,3-c,d)pyrene | µg/kg | SGS AXYS MLA-021 | 1.0 |
| Sediment[2] | Dibenzothiophene | µg/kg | SGS AXYS MLA-021 | 1.0 |
| | Pesticides | | | |
| | Cyclopentadienes | | | |
| Sediment[2] | Aldrin | µg/kg | SGS AXYS MLA-228 | 0.5 |
| Sediment[2] | Dieldrin | µg/kg | SGS AXYS MLA-228 | 0.1 |
| Sediment[2] | Endrin | µg/kg | SGS AXYS MLA-228 | 0.1 |
| | Chlordanes | | | |
| Sediment[2] | Chlordane, cis- | µg/kg | SGS AXYS MLA-228 | 0.1 |

| Matrix | Parameter & Analyte | Units | Suggested Analytical Methods | Target RL |
|---|---|---|---|---|
| Sediment[2] | Nonachlor, cis- | µg/kg | SGS AXYS MLA-228 | 0.1 |
| Sediment[2] | Chlordane, trans | µg/kg | SGS AXYS MLA-228 | 0.1 |
| Sediment[2] | Heptachlor | µg/kg | SGS AXYS MLA-228 | 0.5 |
| Sediment[2] | Heptachlor Epoxide | µg/kg | SGS AXYS MLA-228 | 0.1 |
| Sediment[2] | Oxychlordane | µg/kg | SGS AXYS MLA-228 | 0.5 |
| Sediment[2] | Nonachlor, trans- | µg/kg | SGS AXYS MLA-228 | 0.1 |
| | DDTs | | | |
| Sediment[2] | DDD(o,p') | µg/kg | SGS AXYS MLA-228 | 0.1 |
| Sediment[2] | DDE(o,p') | µg/kg | SGS AXYS MLA-228 | 0.1 |
| Sediment[2] | DDT(o,p') | µg/kg | SGS AXYS MLA-228 | 0.1 |
| Sediment[2] | DDD(p,p') | µg/kg | SGS AXYS MLA-228 | 0.1 |
| Sediment[2] | DDE(p,p') | µg/kg | SGS AXYS MLA-228 | 0.1 |
| Sediment[2] | DDT(p,p') | µg/kg | SGS AXYS MLA-228 | 0.1 |
| | HCH | | | |
| Sediment[2] | HCH, alpha | µg/kg | SGS AXYS MLA-228 | 0.2 |
| Sediment[2] | HCH, beta | µg/kg | SGS AXYS MLA-228 | 0.2 |
| Sediment[2] | HCH, delta | µg/kg | SGS AXYS MLA-228 | 0.1 |
| Sediment[2] | HCH, gamma | µg/kg | SGS AXYS MLA-228 | 0.2 |
| Sediment[2] | Dacthal | µg/kg | SGS AXYS MLA-228 | NA |
| | Other | | | |
| Sediment[2] | Endosulfan I | µg/kg | SGS AXYS MLA-228 | 0.1 |
| Sediment[2] | Endosulfan II | µg/kg | SGS AXYS MLA-228 | 0.1 |
| Sediment[2] | Endosulfan Sulfate | µg/kg | SGS AXYS MLA-228 | 0.1 |
| Sediment[2] | Mirex | µg/kg | SGS AXYS MLA-228 | 0.1 |
| Sediment[2] | Oxadiazon | µg/kg | SGS AXYS MLA-228 | NA |
| Sediment[2] | Hexachlorobenzene | µg/kg | SGS AXYS MLA-228 | 0.1 |
| Sediment[2] | Toxaphene | µg/kg | SGS AXYS MLA-228 | NA |
| Sediment[2] | Hexachlorobutadiene | µg/kg | SGS AXYS MLA-228 | NA |
| | PCB congeners | | | |
| Sediment[2] | PCB 005/8 | µg/kg | SGS AXYS MLA-210 | 0.5 |
| Sediment[2] | PCB 018 | µg/kg | SGS AXYS MLA-210 | 0.1 |

| Matrix | Parameter & Analyte | Units | Suggested Analytical Methods | Target RL |
|---|---|---|---|---|
| Sediment[2] | PCB 028 | µg/kg | SGS AXYS MLA-210 | 0.1 |
| Sediment[2] | PCB 031 | µg/kg | SGS AXYS MLA-210 | 0.1 |
| Sediment[2] | PCB 33/20/21 | µg/kg | SGS AXYS MLA-210 | 0.1 |
| Sediment[2] | PCB 044 | µg/kg | SGS AXYS MLA-210 | 0.1 |
| Sediment[2] | PCB 49/43 | µg/kg | SGS AXYS MLA-210 | 0.1 |
| Sediment[2] | PCB 52/73 | µg/kg | SGS AXYS MLA-210 | 0.1 |
| Sediment[2] | PCB 056/60 | µg/kg | SGS AXYS MLA-210 | 0.1 |
| Sediment[2] | PCB 66/80 | µg/kg | SGS AXYS MLA-210 | 0.1 |
| Sediment[2] | PCB 70/76 | µg/kg | SGS AXYS MLA-210 | 0.1 |
| Sediment[2] | PCB 061/74 | µg/kg | SGS AXYS MLA-210 | 0.1 |
| Sediment[2] | PCB 87/115/116 | µg/kg | SGS AXYS MLA-210 | 0.1 |
| Sediment[2] | PCB 093/95 | µg/kg | SGS AXYS MLA-210 | 0.1 |
| Sediment[2] | PCB 086/97 | µg/kg | SGS AXYS MLA-210 | 0.1 |
| Sediment[2] | PCB 099 | µg/kg | SGS AXYS MLA-210 | 0.1 |
| Sediment[2] | PCB 90/101/89 | µg/kg | SGS AXYS MLA-210 | 0.1 |
| Sediment[2] | PCB 105/127 | µg/kg | SGS AXYS MLA-210 | 0.1 |
| Sediment[2] | PCB 110 | µg/kg | SGS AXYS MLA-210 | 0.1 |
| Sediment[2] | PCB 118/106 | µg/kg | SGS AXYS MLA-210 | 0.1 |
| Sediment[2] | PCB 128 | µg/kg | SGS AXYS MLA-210 | 0.1 |
| Sediment[2] | PCB 132/168 | µg/kg | SGS AXYS MLA-210 | 0.1 |
| Sediment[2] | PCB 138/163/164 | µg/kg | SGS AXYS MLA-210 | 0.1 |
| Sediment[2] | PCB 141 | µg/kg | SGS AXYS MLA-210 | 0.1 |
| Sediment[2] | PCB 149/139 | µg/kg | SGS AXYS MLA-210 | 0.1 |
| Sediment[2] | PCB 151 | µg/kg | SGS AXYS MLA-210 | 0.1 |
| Sediment[2] | PCB 153 | µg/kg | SGS AXYS MLA-210 | 0.1 |
| Sediment[2] | PCB 156 | µg/kg | SGS AXYS MLA-210 | 0.1 |
| Sediment[2] | PCB 158/160 | µg/kg | SGS AXYS MLA-210 | 0.1 |
| Sediment[2] | PCB 170/190 | µg/kg | SGS AXYS MLA-210 | 0.1 |
| Sediment[2] | PCB 174/181 | µg/kg | SGS AXYS MLA-210 | 0.1 |
| Sediment[2] | PCB 177 | µg/kg | SGS AXYS MLA-210 | 0.1 |
| Sediment[2] | PCB 180 | µg/kg | SGS AXYS MLA-210 | 0.1 |

| Matrix | Parameter & Analyte | Units | Suggested Analytical Methods | Target RL |
|---|---|---|---|---|
| Sediment[2] | PCB 183 | µg/kg | SGS AXYS MLA-210 | 0.1 |
| Sediment[2] | PCB 187/182 | µg/kg | SGS AXYS MLA-210 | 0.1 |
| Sediment[2] | PCB 194 | µg/kg | SGS AXYS MLA-210 | 0.1 |
| Sediment[2] | PCB 195 | µg/kg | SGS AXYS MLA-210 | 0.1 |
| Sediment[2] | PCB 201 | µg/kg | SGS AXYS MLA-210 | 0.1 |
| Sediment[2] | PCB 196/203 | µg/kg | SGS AXYS MLA-210 | 0.1 |
| | PBDE congeners | | | |
| Sediment[2] | PBDE 007 | ng/kg | EPA 1614 | 1.0 |
| Sediment[2] | PBDE 8/11 | ng/kg | EPA 1614 | 1.0 |
| Sediment[2] | PBDE 010 | ng/kg | EPA 1614 | 1.0 |
| Sediment[2] | PBDE 12/13 | ng/kg | EPA 1614 | 1.0 |
| Sediment[2] | PBDE 015 | ng/kg | EPA 1614 | 1.0 |
| Sediment[2] | PBDE 017/25 | ng/kg | EPA 1614 | 1.0 |
| Sediment[2] | PBDE 028/33 | ng/kg | EPA 1614 | 1.0 |
| Sediment[2] | PBDE 030 | ng/kg | EPA 1614 | 1.0 |
| Sediment[2] | PBDE 032 | ng/kg | EPA 1614 | 1.0 |
| Sediment[2] | PBDE 035 | ng/kg | EPA 1614 | 1.0 |
| Sediment[2] | PBDE 037 | ng/kg | EPA 1614 | 1.0 |
| Sediment[2] | PBDE 047 | ng/kg | EPA 1614 | 1.0 |
| Sediment[2] | PBDE 049 | ng/kg | EPA 1614 | 1.0 |
| Sediment[2] | PBDE 051 | ng/kg | EPA 1614 | 1.0 |
| Sediment[2] | PBDE 066 | ng/kg | EPA 1614 | 1.0 |
| Sediment[2] | PBDE 071 | ng/kg | EPA 1614 | 1.0 |
| Sediment[2] | PBDE 075 | ng/kg | EPA 1614 | 1.0 |
| Sediment[2] | PBDE 077 | ng/kg | EPA 1614 | 1.0 |
| Sediment[2] | PBDE 079 | ng/kg | EPA 1614 | 1.0 |
| Sediment[2] | PBDE 085 | ng/kg | EPA 1614 | 1.0 |
| Sediment[2] | PBDE 099 | ng/kg | EPA 1614 | 1.0 |
| Sediment[2] | PBDE 100 | ng/kg | EPA 1614 | 1.0 |
| Sediment[2] | PBDE 105 | ng/kg | EPA 1614 | 1.0 |
| Sediment[2] | PBDE 116 | ng/kg | EPA 1614 | 1.0 |
| Sediment[2] | PBDE 119/120 | ng/kg | EPA 1614 | 1.0 |

| Matrix | Parameter & Analyte | Units | Suggested Analytical Methods | Target RL |
|---|---|---|---|---|
| Sediment[2] | PBDE 126 | ng/kg | EPA 1614 | 1.0 |
| Sediment[2] | PBDE 128 | ng/kg | EPA 1614 | 1.0 |
| Sediment[2] | PBDE 138/166 | ng/kg | EPA 1614 | 1.0 |
| Sediment[2] | PBDE 140 | ng/kg | EPA 1614 | 1.0 |
| Sediment[2] | PBDE 153 | ng/kg | EPA 1614 | 1.0 |
| Sediment[2] | PBDE 154 | ng/kg | EPA 1614 | 1.0 |
| Sediment[2] | PBDE 155 | ng/kg | EPA 1614 | 2 |
| Sediment[2] | PBDE 181 | ng/kg | EPA 1614 | 2 |
| Sediment[2] | PBDE 183 | ng/kg | EPA 1614 | 2 |
| Sediment[2] | PBDE 190 | ng/kg | EPA 1614 | 2 |
| Sediment[2] | PBDE 203 | ng/kg | EPA 1614 | 2 |
| Sediment[2] | PBDE 206 | ng/kg | EPA 1614 | 10 |
| Sediment[2] | PBDE 207 | ng/kg | EPA 1614 | 10 |
| Sediment[2] | PBDE 208 | ng/kg | EPA 1614 | 10 |
| Sediment[2] | PBDE 209 | ng/kg | EPA 1614 | 20 |
| | Pesticides | | | |
| | Cyclopentadienes | | | |
| Mussel Tissue[2] | Aldrin | µg/kg | SGS AXYS MLA-228 | 0.5 |
| Mussel Tissue[2] | Dieldrin | µg/kg | SGS AXYS MLA-228 | 0.1 |
| Mussel Tissue[2] | Endrin | µg/kg | SGS AXYS MLA-228 | 0.1 |
| | Chlordanes | | | |
| Mussel Tissue[2] | Chlordane, cis- | µg/kg | SGS AXYS MLA-228 | 0.1 |
| Mussel Tissue[2] | Nonachlor, cis- | µg/kg | SGS AXYS MLA-228 | 0.1 |
| Mussel Tissue[2] | Chlordane, trans | µg/kg | SGS AXYS MLA-228 | 0.1 |
| Mussel Tissue[2] | Heptachlor | µg/kg | SGS AXYS MLA-228 | 0.5 |
| Mussel Tissue[2] | Heptachlor Epoxide | µg/kg | SGS AXYS MLA-228 | 0.1 |

| Matrix | Parameter & Analyte | Units | Suggested Analytical Methods | Target RL |
|---|---|---|---|---|
| Mussel Tissue[2] | Oxychlordane | µg/kg | SGS AXYS MLA-228 | 0.5 |
| Mussel Tissue[2] | Nonachlor, trans- | µg/kg | SGS AXYS MLA-228 | 0.1 |
| | DDTs | | | |
| Mussel Tissue[2] | DDD(o,p') | µg/kg | SGS AXYS MLA-228 | 0.1 |
| Mussel Tissue[2] | DDE(o,p') | µg/kg | SGS AXYS MLA-228 | 0.1 |
| Mussel Tissue[2] | DDT(o,p') | µg/kg | SGS AXYS MLA-228 | 0.1 |
| Mussel Tissue[2] | DDD(p,p') | µg/kg | SGS AXYS MLA-228 | 0.1 |
| Mussel Tissue[2] | DDE(p,p') | µg/kg | SGS AXYS MLA-228 | 0.1 |
| Mussel Tissue[2] | DDT(p,p') | µg/kg | SGS AXYS MLA-228 | 0.1 |
| | HCH | | | |
| Mussel Tissue[2] | HCH, alpha | µg/kg | SGS AXYS MLA-228 | 0.2 |
| Mussel Tissue[2] | HCH, beta | µg/kg | SGS AXYS MLA-228 | 0.2 |
| Mussel Tissue[2] | HCH, delta | µg/kg | SGS AXYS MLA-228 | 0.1 |
| Mussel Tissue[2] | HCH, gamma | µg/kg | SGS AXYS MLA-228 | 0.2 |
| | Other | | | |
| Mussel Tissue[2] | Dacthal | µg/kg | SGS AXYS MLA-228 | NA |
| Mussel Tissue[2] | Endosulfan I | µg/kg | SGS AXYS MLA-228 | 0.1 |
| Mussel Tissue[2] | Endosulfan II | µg/kg | SGS AXYS MLA-228 | 0.1 |
| Mussel Tissue[2] | Endosulfan Sulfate | µg/kg | SGS AXYS MLA-228 | 0.1 |

| Matrix | Parameter & Analyte | Units | Suggested Analytical Methods | Target RL |
|---|---|---|---|---|
| Mussel Tissue[2] | Mirex | µg/kg | SGS AXYS MLA-228 | 0.1 |
| Mussel Tissue[2] | Oxadiazon | µg/kg | SGS AXYS MLA-228 | NA |
| Mussel Tissue[2] | Hexachlorobenzene | µg/kg | SGS AXYS MLA-228 | 0.1 |
| Mussel Tissue[2] | Toxaphene | µg/kg | SGS AXYS MLA-228 | NA |
| Mussel Tissue[2] | Hexachlorobutadiene | µg/kg | SGS AXYS MLA-228 | NA |
| | PCB congeners | | | |
| Mussel Tissue[2] | PCB 005/8 | µg/kg | SGS AXYS MLA-210 | 0.5 |
| Mussel Tissue[2] | PCB 018 | µg/kg | SGS AXYS MLA-210 | 0.1 |
| Mussel Tissue[2] | PCB 028 | µg/kg | SGS AXYS MLA-210 | 0.1 |
| Mussel Tissue[2] | PCB 031 | µg/kg | SGS AXYS MLA-210 | 0.1 |
| Mussel Tissue[2] | PCB 33/20/21 | µg/kg | SGS AXYS MLA-210 | 0.1 |
| Mussel Tissue[2] | PCB 044 | µg/kg | SGS AXYS MLA-210 | 0.1 |
| Mussel Tissue[2] | PCB 49/43 | µg/kg | SGS AXYS MLA-210 | 0.1 |
| Mussel Tissue[2] | PCB 52/73 | µg/kg | SGS AXYS MLA-210 | 0.1 |
| Mussel Tissue[2] | PCB 056/60 | µg/kg | SGS AXYS MLA-210 | 0.1 |
| Mussel Tissue[2] | PCB 66/80 | µg/kg | SGS AXYS MLA-210 | 0.1 |
| Mussel Tissue[2] | PCB 70/76 | µg/kg | SGS AXYS MLA-210 | 0.1 |
| Mussel Tissue[2] | PCB 061/74 | µg/kg | SGS AXYS MLA-210 | 0.1 |

| Matrix | Parameter & Analyte | Units | Suggested Analytical Methods | Target RL |
|---|---|---|---|---|
| Mussel Tissue[2] | PCB 87/115/116 | µg/kg | SGS AXYS MLA-210 | 0.1 |
| Mussel Tissue[2] | PCB 093/95 | µg/kg | SGS AXYS MLA-210 | 0.1 |
| Mussel Tissue[2] | PCB 086/97 | µg/kg | SGS AXYS MLA-210 | 0.1 |
| Mussel Tissue[2] | PCB 099 | µg/kg | SGS AXYS MLA-210 | 0.1 |
| Mussel Tissue[2] | PCB 90/101/89 | µg/kg | SGS AXYS MLA-210 | 0.1 |
| Mussel Tissue[2] | PCB 105/127 | µg/kg | SGS AXYS MLA-210 | 0.1 |
| Mussel Tissue[2] | PCB 110 | µg/kg | SGS AXYS MLA-210 | 0.1 |
| Mussel Tissue[2] | PCB 118/106 | µg/kg | SGS AXYS MLA-210 | 0.1 |
| Mussel Tissue[2] | PCB 128 | µg/kg | SGS AXYS MLA-210 | 0.1 |
| Mussel Tissue[2] | PCB 132/168 | µg/kg | SGS AXYS MLA-210 | 0.1 |
| Mussel Tissue[2] | PCB 138/163/164 | µg/kg | SGS AXYS MLA-210 | 0.1 |
| Mussel Tissue[2] | PCB 141 | µg/kg | SGS AXYS MLA-210 | 0.1 |
| Mussel Tissue[2] | PCB 149/139 | µg/kg | SGS AXYS MLA-210 | 0.1 |
| Mussel Tissue[2] | PCB 151 | µg/kg | SGS AXYS MLA-210 | 0.1 |
| Mussel Tissue[2] | PCB 153 | µg/kg | SGS AXYS MLA-210 | 0.1 |
| Mussel Tissue[2] | PCB 156 | µg/kg | SGS AXYS MLA-210 | 0.1 |
| Mussel Tissue[2] | PCB 158/160 | µg/kg | SGS AXYS MLA-210 | 0.1 |
| Mussel Tissue[2] | PCB 170/190 | µg/kg | SGS AXYS MLA-210 | 0.1 |

| Matrix | Parameter & Analyte | Units | Suggested Analytical Methods | Target RL |
|---|---|---|---|---|
| Mussel Tissue[2] | PCB 174/181 | µg/kg | SGS AXYS MLA-210 | 0.1 |
| Mussel Tissue[2] | PCB 177 | µg/kg | SGS AXYS MLA-210 | 0.1 |
| Mussel Tissue[2] | PCB 180 | µg/kg | SGS AXYS MLA-210 | 0.1 |
| Mussel Tissue[2] | PCB 183 | µg/kg | SGS AXYS MLA-210 | 0.1 |
| Mussel Tissue[2] | PCB 187/182 | µg/kg | SGS AXYS MLA-210 | 0.1 |
| Mussel Tissue[2] | PCB 194 | µg/kg | SGS AXYS MLA-210 | 0.1 |
| Mussel Tissue[2] | PCB 195 | µg/kg | SGS AXYS MLA-210 | 0.1 |
| Mussel Tissue[2] | PCB 201 | µg/kg | SGS AXYS MLA-210 | 0.1 |
| Mussel Tissue[2] | PCB 196/203 | µg/kg | SGS AXYS MLA-210 | 0.1 |
| | PBDE congeners | | | |
| Mussel Tissue[2] | PBDE 007 | ng/kg | EPA 1614 | 1.0 |
| Mussel Tissue[2] | PBDE 8/11 | ng/kg | EPA 1614 | 1.0 |
| Mussel Tissue[2] | PBDE 010 | ng/kg | EPA 1614 | 1.0 |
| Mussel Tissue[2] | PBDE 12/13 | ng/kg | EPA 1614 | 1.0 |
| Mussel Tissue[2] | PBDE 015 | ng/kg | EPA 1614 | 1.0 |
| Mussel Tissue[2] | PBDE 017/25 | ng/kg | EPA 1614 | 1.0 |
| Mussel Tissue[2] | PBDE 028/33 | ng/kg | EPA 1614 | 1.0 |
| Mussel Tissue[2] | PBDE 030 | ng/kg | EPA 1614 | 1.0 |

| Matrix | Parameter & Analyte | Units | Suggested Analytical Methods | Target RL |
|---|---|---|---|---|
| Mussel Tissue[2] | PBDE 032 | ng/kg | EPA 1614 | 1.0 |
| Mussel Tissue[2] | PBDE 035 | ng/kg | EPA 1614 | 1.0 |
| Mussel Tissue[2] | PBDE 037 | ng/kg | EPA 1614 | 1.0 |
| Mussel Tissue[2] | PBDE 047 | ng/kg | EPA 1614 | 1.0 |
| Mussel Tissue[2] | PBDE 049 | ng/kg | EPA 1614 | 1.0 |
| Mussel Tissue[2] | PBDE 051 | ng/kg | EPA 1614 | 1.0 |
| Mussel Tissue[2] | PBDE 066 | ng/kg | EPA 1614 | 1.0 |
| Mussel Tissue[2] | PBDE 071 | ng/kg | EPA 1614 | 1.0 |
| Mussel Tissue[2] | PBDE 075 | ng/kg | EPA 1614 | 1.0 |
| Mussel Tissue[2] | PBDE 077 | ng/kg | EPA 1614 | 1.0 |
| Mussel Tissue[2] | PBDE 079 | ng/kg | EPA 1614 | 1.0 |
| Mussel Tissue[2] | PBDE 085 | ng/kg | EPA 1614 | 1.0 |
| Mussel Tissue[2] | PBDE 099 | ng/kg | EPA 1614 | 1.0 |
| Mussel Tissue[2] | PBDE 100 | ng/kg | EPA 1614 | 1.0 |
| Mussel Tissue[2] | PBDE 105 | ng/kg | EPA 1614 | 1.0 |
| Mussel Tissue[2] | PBDE 116 | ng/kg | EPA 1614 | 1.0 |
| Mussel Tissue[2] | PBDE 119/120 | ng/kg | EPA 1614 | 1.0 |
| Mussel Tissue[2] | PBDE 126 | ng/kg | EPA 1614 | 1.0 |

| Matrix | Parameter & Analyte | Units | Suggested Analytical Methods | Target RL |
|---|---|---|---|---|
| Mussel Tissue[2] | PBDE 128 | ng/kg | EPA 1614 | 1.0 |
| Mussel Tissue[2] | PBDE 138/166 | ng/kg | EPA 1614 | 1.0 |
| Mussel Tissue[2] | PBDE 140 | ng/kg | EPA 1614 | 1.0 |
| Mussel Tissue[2] | PBDE 153 | ng/kg | EPA 1614 | 1.0 |
| Mussel Tissue[2] | PBDE 154 | ng/kg | EPA 1614 | 1.0 |
| Mussel Tissue[2] | PBDE 155 | ng/kg | EPA 1614 | 2.0 |
| Mussel Tissue[2] | PBDE 181 | ng/kg | EPA 1614 | 2.0 |
| Mussel Tissue[2] | PBDE 183 | ng/kg | EPA 1614 | 2.0 |
| Mussel Tissue[2] | PBDE 190 | ng/kg | EPA 1614 | 2.0 |
| Mussel Tissue[2] | PBDE 203 | ng/kg | EPA 1614 | 2.0 |
| Mussel Tissue[2] | PBDE 206 | ng/kg | EPA 1614 | 10 |
| Mussel Tissue[2] | PBDE 207 | ng/kg | EPA 1614 | 10 |
| Mussel Tissue[2] | PBDE 208 | ng/kg | EPA 1614 | 10 |
| Mussel Tissue[2] | PBDE 209 | ng/kg | EPA 1614 | 20 |

[1] = Analyzed in influent/effluent only.

[2] = Sediment and mussel tissue persistent organic pollutants are reported on a dry-weight basis.

Table 21. California Ocean Plan Table 3 constituents not measured in influent and effluent by CCLEAN.

| For the Protection of Marine Aquatic Life |
|---|
| Arsenic |
| Cadmium |
| Chromium (Hexavalent) |
| Copper |
| Lead |
| Mercury |
| Nickel |
| Selenium |
| Silver |
| Zinc |
| Cyanide |
| Total Chlorine Residual |
| Ammonia (expressed as nitrogen) |
| Chronic Toxicity |
| Phenolic Compounds (non-chlorinated) |
| Chlorinated Phenolics |
| Radioactivity |
| |
| For the Protection of Human Health - Noncarcinogens |
| acrolein |
| antimony |
| bis(2-chloroethoxy) methane |
| bis(2-chloroisopropyl) ether |
| chlorobenzene |
| chromium (III) |
| di-n-butyl phthalate |
| dichlorobenzenes |
| 1,1-dichloroethylene |
| diethyl phthalate |
| dimethyl phthalate |
| 4,6-dinitro-2-methylphenol |
| 2,4-dinitrophenol |
| ethylbenzene |
| hexachlorocyclopentadiene |
| isophorone |
| nitrobenzene |
| thallium |
| toluene |
| 1,1,2,2-tetrachloroethane |

| |
|---|
| tributyltin |
| 1,1,1-trichloroethane |
| 1,1,2-trichloroethane |

| For Protection of Human Health - Carcinogens |
|---|
| acrylonitrile |
| benzene |
| benzidine |
| beryllium |
| bis(2-chloroethyl) ether |
| bis(2-ethylhexyl) phthalate |
| carbon tetrachloride |
| chloroform |
| 1,4-dichlorobenzene |
| 3,3í-dichlorobenzidine |
| 1,2-dichloroethane |
| dichloromethane |
| 1,3-dichloropropene |
| 2,4-dinitrotoluene |
| 1,2-diphenylhydrazine |
| halomethanes |
| hexachloroethane |
| N-nitrosodimethylamine |
| N-nitrosodiphenylamine |
| tetrachloroethylene |
| toxaphene |
| trichloroethylene |
| 2,4,6-trichlorophenol |
| vinyl chloride |

# 14. QUALITY CONTROL

Concentrations of pollutants in environmental samples are often low. Therefore, a quality-assurance program for the chemical analysis of samples requires stringent laboratory conditions and careful control over all aspects of the sampling and analyses. Each step in the analytical process is a potential source of contamination and must be consistently monitored to ensure that the final measurement is not adversely affected by any processing steps. A general discussion of Quality Control and various aspects of the CCLEAN quality control program are summarized below.

### 14.1. General Laboratory Quality Control for Non-Biological Data

Laboratories providing analytical support to CCLEAN will have the appropriate facilities to store, prepare, and process samples in an ultra-clean environment, and will have appropriate instrumentation and staff to perform analyses and provide data of the required quality within the time period dictated by the Program. The laboratories are expected to satisfy the following:

- Demonstrate capability through pertinent certification and satisfactory performance in inter-laboratory comparison exercises.
- Provide qualification statements regarding their facility and personnel.
- Maintain a program of scheduled maintenance of analytical balances, laboratory equipment and instrumentation.
- Conduct routine checking of analytical balances using a set of standard reference weights (American Society of Testing and Materials Class 3, NIST Class S-1, or equivalents). Analytical balances are serviced at six-month intervals or when test weight values are not within the manufacturer's instrument specifications, whichever occurs first.
- Conduct routine checking and recording the composition of fresh calibration standards against the previous lot. Acceptable comparisons are within 2% of the previous value.
- Record all analytical data in bound (where possible) logbooks, with all entries in ink, or electronically.
- Monitor and document the temperatures of cold storage areas and freezer units on a continuous basis.
- Verify the efficiency of fume/exhaust hoods.
- Have a source of reagent water meeting specifications described in Section 8.0 available in sufficient quantity to support analytical operations.
- Label all containers used in the laboratory with date prepared, contents, initials of the individual who prepared the contents, and other information as appropriate.
- Date and safely store all chemicals upon receipt. Proper disposal of chemicals when the expiration date has passed.
- Have QAPP, SOPs, analytical methods manuals, and safety plans readily available to staff.
- Have raw analytical data readily accessible so that they are available upon request.

In addition, laboratories involved in CCLEAN are required to demonstrate capability continuously through the following protocols:

- Strict adherence to routine QA/QC procedures.
- Routine analysis of CRMs, if available.
- Regular participation in annual certification programs.
- Satisfactory performance at least annually in the analysis of blind Performance Evaluation Samples and/or participation in inter-laboratory comparison exercises.

Laboratory QC samples must satisfy SWAMP measurement quality objectives (MQOs) and frequency requirements. DQOs are specified in Section 7. Frequency requirements are provided on an analytical batch level. CCLEAN defines an analytical batch as 20 or fewer samples and associated quality control that are processed by the same instrument within a 24-hour period (unless otherwise specified by method). Details regarding sample preparation are method- or laboratory SOP-specific, and may consist of extraction, digestion, or other techniques.

### 14.2. General Laboratory Quality Control for Biological Data

Sorting efficiency is used to quantify the sorting accuracy of the laboratory. Once samples are sorted, a second technician will sort the residues of 10% of the samples originally sorted to recover organisms missed by the primary sorter and to assess sorting accuracy. If a second sorting technician is not available and a taxonomist performs sorting activities, the same taxonomist may re-sort the remnant for evaluating sorting accuracy.

Recount accuracy is used to quantify the sorting accuracy of the laboratory. Previously a subset of samples (approximately 10%) that have been sorted and identified were sent to a reference laboratory. However, sending specimens out to be re-identified is no longer recommended. In recent years the number of benthic taxonomists has been shrinking through attrition and lack of incentives for new practitioners. Now more than ever the old axiom applies that “a species is what the best taxonomist says it is”. In taxonomy there are quite often legitimate disputes over names both within and between bio-regions, and by sticking to taxonomists familiar with the local fauna results can be compared to each other over time. The approach of the laboratory used is to have samples identified by the best regional people available, and rely on their judgment as to the identifications.

### 14.3. Field Performance Measurements, General

Following is a list of definitions of field performance measurements that are frequently included in sampling protocols. Some of these measurements only need to be taken when an established procedure is changed, while others should be taken at various intervals throughout the sampling process.

Source Solution Blanks - account for any pre-existing contamination in the water or preservatives used to prepare the sample containers as well as the field or travel blanks.

Bottle Blanks - account for contamination in sampling containers, in addition to any contamination due to the source solution.

Reference Performance Spikes - spiked onto XAD-2 resin to determine retention of POPs during field sampling.

Travel Blanks - account for contaminants introduced during the transport process between the laboratory and field site, in addition to any contamination from the source solution and container.

Equipment Blank - account for contamination introduced by the field sampling equipment.

Field Duplicates - account for variability in the field and laboratory.

Field Blanks - account for all of the above sources of contamination that might be introduced to a sample as well as that which would be due to the sampling equipment and the immediate field environment. Field blanks are generated under actual field conditions and are subjected to the same aspects of sample collection, field processing, preservation, transport, and laboratory handling as the environmental samples. Field blanks for water generally consist of ultra-pure water and those for sediment analyses generally consist of ultra-pure sand. True field blanks for biological tissue samples do not exist.

### 14.4. Field Performance Measurements Used by CCLEAN

Routine preparation, collection, and analysis of all the blanks and duplicates mentioned above would be redundant and inefficient. Since POPs in influent and effluent and environmental water samples are orders of magnitude lower than in sediments or tissues, extreme care must be taken in the handling and analysis of influent and effluent or water samples. Ultra-pure solvents and materials will be used in all aspects of cleaning, storage, and analysis. The solid-phase extraction columns and pre-filters will be cleaned and the cleaning process will be verified by analytical results of final solvent rinses. Contamination of solvents and source solutions will be routinely checked, and corrective steps taken whenever contamination is indicated. Certified clean borosilicate glass containers will be used for sediment and tissue samples.

Although travel blanks are not routinely used for water, sediment, or tissue samples, they may be implemented in the future. In the meantime, the possibility of contamination during the transport between the laboratory and field site will be mitigated by the measures taken to keep the sample bottles in an enclosed clean environment.

Deuterated compounds are spiked onto the XAD-2 resin beads before deployment for sampling. These compounds are analyzed in the laboratory to determine retention of captured contaminants during field sampling. Low recoveries of these deuterated compounds could indicate losses during the sampling period.

An equipment blank for POP water samples is collected once per sampling effort from a randomly selected sampling apparatus. Two-hundred liters of Milli-Q water (or equivalent) will be pumped through the sample tubing connected to solid-phase extraction (SPE) columns and filters. The sample will be exposed to the interior of the sampler tubing and all fittings, all of which will have been rigorously cleaned with ultra-pure solvents. Sediments will be collected with grab sampler coated with a chemically-inert coating, but equipment blanks will not be taken. Since bivalves will be hand collected, equipment blanks are not relevant for tissue samples.

Field duplicates will be collected for mussel sampling. Duplicate samples will be used to evaluate sampling precision and environmental variability.

True field blanks are not routinely collected in this field and are not routinely reported in the literature. Instead, samples will be collected and handled in ways that minimize contamination. For POP sampling, containers will be routinely checked for contamination, and plastic material for storage, transport, and protection of samples will be avoided. Only ultra-pure solvents will be used in the preparation of the XAD resin and filters. The XAD resin and filters will remain enclosed and inaccessible to aerial contamination until deployed for sampling.

Collection of true sediment field blanks also has been deemed unnecessary due to use of precautions that minimize contamination of the samples. All surfaces of sediment sampling and processing instruments coming into contact with the sample will be made of inert materials, such as Teflon ® or stainless steel coated with Dykon ® (or equivalent), and will be thoroughly cleaned prior to field use. Equipment also will be cleaned with Alconox (or equivalent) detergent between stations and rinsed with hydrochloric acid, followed by methanol, to avoid any carryover contamination from one station to another. Sampling will be conducted on board ship with gloved hands and the sample will be placed into pre-cleaned certified glass jars with Teflon ® -lined lids for POP analyses.

Bivalves will be handled in the field according to established protocols of the California State Mussel Watch Program designed to minimize sample contamination. Bivalves destined for POP analysis will be wrapped in aluminum foil, placed on dry ice, and kept frozen until homogenization and analysis.

### 14.5. Laboratory Performance Measurements

Laboratory performance measurements are designed to determine whether data quality criteria are met, as defined below. These types of samples serve to check if errors are introduced during the analysis process and at what step(s) and at what magnitude(s).

Method Blanks (also called laboratory reagent blanks or preparation blanks). These account for contaminants present in the solvents, preservatives, and analytical solutions used during the quantification of the parameter.

Injection Internal Standards - account for error introduced by the analytical instrument.

Replicate Samples - replicates of the raw material that are extracted and analyzed to measure laboratory precision.

Laboratory Replicate Samples - replicates of extracted material that measure the measurement precision.

Matrix Spike Samples (MS) - field samples to which a known amount of contaminant is added and measured to determine potential analytical interference present in the field sample.

Matrix Spike Replicate Samples (MSR or MSD) - used to assess both measurement precision and accuracy. They are especially useful when field samples may not contain many of the target compounds because measuring non-detects in replicate does not allow the data reviewer to measure the precision or the accuracy of the data in an analytical batch.

Certified Reference Materials (CRMs) - method of determining measurement accuracy, especially if a CRM contains a certified value at concentrations similar to those expected in the samples to be analyzed.

## 14.6. CCLEAN Laboratory Quality Control Procedures

The performance-based protocols utilized in CCLEAN for analytical chemistry laboratories consist of several elements, as follows:

### *14.6.1. Precision Criteria*

Precision is the reproducibility of an analytical method. Each laboratory is expected to maintain control charts for use by analysts in monitoring the overall precision of the CRM or LCM. Upper and lower control chart limits (e.g., warning limits and control limits) will be continually updated; control limits based on 99% confidence intervals around the mean are recommended. The relative standard deviation (RSD) will be calculated for each analyte of interest in the CRM based on the last seven (7) CRM analyses. Acceptable precision targets for various analyses are listed in Section 7.

### *14.6.2. Laboratory Replicates for Precision*

A minimum of one field sample per batch of CCLEAN samples submitted to the laboratory will be processed and analyzed in duplicate or more for precision. The relative percent difference between two replicate samples or the relative standard deviation between more than two replicate samples (RPD or RSD respectively) will be less than the DQOs listed in Section 7 for each analyte of interest. Following are the calculations:

RPD = ABS([X1 - X2] / [(X1 + X2) / 2])

Where: X1 = the first sample result
X2 = the duplicate sample result.

RSD = [stdev (X,,X2 ,..XN)] / [average (X,, X2 , ..XN)]

Where: X1 = the first sample result
XN = each successive sample result

ABS — absolute value
STDEV — standard deviation

If results for any analytes do not meet the DQO for the RPD or RSD, calculations and instruments will be checked. A repeat analysis may be required to confirm the results. Results that repeatedly fail to meet the objectives indicate sample inhomogeneity, unusually high concentrations of analytes or poor laboratory precision. In this case, the laboratory is obligated to halt the analysis of samples and eliminate the source of the imprecision before proceeding.

### *14.6.3. Accuracy Criteria*

The "absolute" accuracy of an analytical method can be assessed using CRMs only when certified values are provided for the analytes of interest. Nevertheless, the concentrations of many analytes of interest to CCLEAN may be provided only as non-certified values in some of the more commonly used CRMs. Therefore, control limit criteria are based on "relative accuracy", which is evaluated for each analysis of the CRM or LCM by comparison of a given laboratory's values to the "true" or "accepted" values. In the case of CRMs, this includes only certified values. The "true" values are defined as the 95% confidence intervals of the mean.

Based on typical results attained by experienced analysts in the past, accuracy control limits have been established both for individual compounds and combined groups of compounds (Section 7). There are three combined groups of compounds for the purpose of evaluating relative accuracy for organic analyses: PAHs, PCBs, and pesticides. For each group of analytes, 70% of the individual analytes must be within 35% of the certified 95% confidence interval. No individual analyte value shall exceed ±30% of the 95% confidence interval more than once in consecutive analyses without appropriate documentation and consultation with CCLEAN staff. Due to the inherent variability in analyses near the method detection limit, control limit criteria for relative accuracy only apply to analytes with true values that are >3 times the method detection limit (MDL) established by the laboratory.

### *14.6.4. Continuing Calibration Checks*

Calibration-check solutions traceable to a recognized organization must be inserted as part of the sample stream. The source of the calibration check solution shall be independent from the standards used for the calibration. Calibration check solutions used for the continuing calibration checks will contain all the analytes of interest. The frequency of these checks is dependent on the type of instrumentation used and, therefore, requires considerable professional judgment. All organic analyses shall be bracketed by an acceptable calibration check. A calibration check standard shall be run every 12 hours at a minimum.

If the control limits for analysis of the calibration check solution (set by the laboratories) are not met, the initial calibration must be repeated. The calibration check for 90% of the analytes shall not deviate more than ±25% from the known value for PAHs and ±20% for PCBs and pesticides. If possible, the samples analyzed before the calibration check solution that failed the DQOs will be reanalyzed following recalibration. The laboratory will begin by reanalyzing the last sample analyzed before the calibration check solution that failed. If the RPD between the results of this reanalysis and the original analysis exceeds precision DQOs (Section 7) the instrument is assumed to have been out of control during the original analysis. If possible, reanalysis of samples will progress in reverse order until it is determined that the RPD between initial and reanalysis results are within DQOs (MP Section 7). Only the re-analysis results will be reported by the laboratory. If it is not possible or feasible to perform reanalysis of samples, all earlier data (i.e., since the last successful calibration control check) are suspect. In this case, the laboratory will prepare a narrative explanation to accompany the submitted data.

#### *14.6.5. Laboratory Reagent Blank*

For POP analyses, one laboratory reagent blank will be run in every sample batch. The reagent blank will be processed through the entire analytical procedure in a manner identical to the samples. Reagent blanks should be less than the MDL or not exceed a concentration greater than 10% of the lowest reported sample concentration. A reagent blank concentration > 2x the MDL or > 10% of the lowest reported sample concentration for one or more of the analytes of interest will require corrective action to identify and eliminate the source(s) of contamination before proceeding with sample analysis. If eliminating the blank contamination is not possible, all impacted analytes in the analytical batch shall be flagged. In addition, a detailed description of the contamination source and the steps taken to eliminate or minimize the contamination shall be included in the transmittal letter. Subtracting method blank results from sample results is not permitted.

#### *14.6.6. Injection Internal Standards*

The usage of the terms injection internal standard, surrogate, and internal standard varies considerably among laboratories. Surrogates are compounds chosen to simulate the analytes of interest in POP analyses. These are used to estimate analyte losses during the extraction and clean-up process and must be added to each sample, including QA/QC samples, prior to extraction. The reported concentration of each analyte is adjusted to correct for the recovery of the surrogate compound, as done in the NOAA NS&T Program. The surrogate recovery data will be carefully monitored; each laboratory must report the percent recovery of the surrogate(s) along with the target analyte data for each sample. If possible, isotopically-labeled analogs of the analytes will be used as surrogates.

Each laboratory will set its own warning limit criteria based on the experience and best professional judgment of the analyst. It is the responsibility of the analyst to demonstrate that the analytical process is always “in control” (i.e., highly variable surrogate recoveries are not acceptable for repeat analyses of the same certified reference material and for the matrix spike/matrix spike duplicate). The warning limit criteria used by the laboratory will be provided in the standard operating procedures submitted to CCLEAN.

### *14.6.7. Dual-Column Confirmation*

Dual-column chromatography is required for analyses using GC-ECD due to the high probability of false positives arising from single-column analyses. This requirement does not apply to high resolution methods for XAD samples.

### *14.6.8. Matrix Spikes and Matrix Spike Duplicates*

When required, a laboratory-fortified sample matrix (a matrix spike, or MS) and a laboratory fortified sample matrix duplicate (a matrix spike duplicate, or MSD) will be used both to evaluate the effect of the sample matrix on the recovery of the compounds of interest and to provide an estimate of analytical precision. A minimum of 5% of the total number of samples submitted to the laboratory in a given year will be selected at random for analysis as matrix spikes and matrix spike duplicates. A field sample is first homogenized and then split into three subsamples. Two of these subsamples are fortified with the matrix spike solution and the third subsample is analyzed to provide a background concentration for each analyte of interest. The matrix spike solution should contain as many representative analytes from the CCLEAN POP analyte list as feasible. The final spiked concentration of each analyte in the sample will be at least 10 times the MDL for that analyte, as previously calculated by the laboratory. Additionally, the total number of spikes should cover the range of expected concentrations. Recovery is the accuracy of an analytical test measured against a known analyte addition to a sample. Recovery is calculated as follows:

$$\text{Recovery} = \frac{(\text{Matrix plus spike result} - \text{Matrix result}) \times 100}{\text{spike}}$$

Recovery data for the fortified compounds ultimately will provide a basis for determining the prevalence of matrix effects in the samples analyzed during the project. If the percent recovery for any analyte in the MS or MSD is less than the recommended warning limit of 50 percent, the chromatograms and raw data quantitation reports will be reviewed. If an explanation for a low percent-recovery value is not discovered, the instrument response may be checked using a calibration standard. Low recoveries of matrix spikes may result from matrix interferences and further instrument response checks may not be warranted. This is especially true if the low recovery occurs in both the MS and MSD, and the other QC samples in the batch indicate that the analysis was “in control”. An explanation for low percent-recovery values for MS/MSD results will be discussed in a cover letter accompanying the data package. Corrective actions taken and verification of acceptable instrument response will be included. Analysis of the MS/MSD is also useful for assessing laboratory precision. The RPD between the MS and MSD results should be less than the target criterion listed in Section 7 for each analyte of interest.

### *14.6.9. Field Replicates and Field Split Samples*

As part of the quality assurance program of CCLEAN, duplicate or split samples will be collected for sediment and mussels samples for subsequent chemical analysis. Field duplicates or splits will be submitted as blind samples to the analytical laboratory. Field splits also will be collected and sent blind to additional laboratories selected to participate in the split sample analysis. One

field duplicate or field split will be collected for interlaboratory analysis from each sample matrix each year. The analysis of field replicates and field splits can provide an assessment of both inter-and intra-laboratory precision and variance in the sample matrix at the field site. Splits also may be made of laboratory extracts for analysis of POPs. Analysis of these splits can be used to determine variation within and between laboratories in the actual measurement of POPs.

# 15. INSTRUMENT/EQUIPMENT TESTING, INSPECTION, AND MAINTENANCE

## 15.1. Field Equipment

Field measurement equipment will be checked for operation in accordance with the manufacturer's specifications. This includes battery checks, routine replacement of disposable parts, and cleaning as required. All equipment will be inspected for damage at a minimum when first installed / used and when returned from use. Contractors performing sampling operations will be responsible for ensuring that all equipment in their use is maintained properly. Spares parts for all field equipment are stored at the respective field sampling contractor facilities. Any equipment deficiencies that occur during sampling will be corrected immediately by trained field personnel. Impairments of samples due to equipment problems will be reported to the Program Director as soon as possible and solutions agreed upon. All such sampling problems will be reported in the Sampling Report.

## 15.2. Laboratory Equipment

All laboratories providing analytical support for chemical or biological analyses will have the appropriate facilities to store, prepare, and process samples. Moreover, appropriate instrumentation and staff are necessary to provide data of the required quality within the schedule required by the program. Laboratory operations must include the following procedures:

- A program of scheduled maintenance of analytical balances, microscopes, laboratory equipment, and instrumentation.
- Routine checking of analytical balances using a set of standard reference weights (American Society of Testing and Materials (ASTM) Class 3, NIST Class S-1, or equivalents).
- Checking and recording the composition of fresh calibration standards against the previous lot, wherever possible. Acceptable comparisons are < 2% of the previous value.
- Recording all analytical data in bound (where possible) logbooks, with all entries in ink, or electronic format.
- Monitoring and documenting the temperatures of cold storage areas and freezer units once per week.
- Verifying the efficiency of fume hoods.
- Having a source of reagent water meeting ASTM Type I specifications (ASTM, 1984) available in sufficient quantity to support analytical operations. The conductivity of the reagent water will not exceed 18 megaohms at 25°C. Alternately, the resistivity of the reagent water will exceed 10 mmhos/cm.
- Labeling all containers used in the laboratory with date prepared, contents, initials of the individual who prepared the contents, and other information, as appropriate.
- Dating and safely storing all chemicals upon receipt. Proper disposal of chemicals when the expiration date has passed.
- Having QAPP, SOPs, analytical methods manuals, and safety plans readily available to staff.

- Having raw analytical data, such as chromatograms, accessible so that they are available upon request.

Laboratories will maintain appropriate equipment per the requirements of individual laboratory SOPs and will be able to provide information documenting their ability to conduct the analyses with the required level of data quality. Such information might include results from interlaboratory comparison studies, control charts and summary data of internal QA/QC checks, and results from certified reference material analyses. Spares parts for all laboratory equipment are stored at the respective analytical laboratories. Any equipment deficiencies that occur during analyses will be corrected immediately by trained personnel. Impairments of analytical results due to equipment problems will be reported to the Program Director as soon as possible and solutions agreed upon. All such equipment problems will be reported in the narrative data report.

## 16. INSTRUMENT/EQUIPMENT CALIBRATION AND FREQUENCY

Pumps used for collecting water samples are calibrated by collecting water discharged from the sampling instrumentation and for direct measurement of volume.

All project laboratories maintain calibration practices as part of the method SOPs. Individual laboratory QA officers are responsible for ensuring that calibration practices are performed as required by SOPs. Records of all calibration measurements will be maintained by each individual laboratory. Any equipment deficiencies that occur will be corrected immediately by trained personnel. Impairments of samples due to equipment problems will be reported to the Program Director as soon as possible and solutions agreed upon. All such sampling problems will be reported in the Sampling Report.

# 17. INSPECTION/ACCEPTANCE OF SUPPLIES AND CONSUMABLES

Field sampling contractors and analytical laboratories are responsible for inspection / acceptance of all project-related materials. Contractors and laboratories will perform inspections per the acceptance criteria within their respective SOPs.

# 18. Non-Direct Measurements (Existing Data)

Three types of non-direct data are used in the CCLEAN program, as follows:

- Flow data are obtained for wastewater treatment plants from treatment plant flow meters, which also provide electronic signals to the automated sampling equipment. Flow data are used to estimate the loads of water constituents using the concentration data measured directly by the program (i.e., load = concentration x flow volume). It is assumed that functional flow meters and access points for sampling influent and effluent are necessary for this program.
- Data on concentrations of ocean chlorophyll are obtained from satellite images provided by NASA for assessment of the effects of nutrient discharges to Monterey Bay. CCLEAN does not apply any measures of data quality to the satellite imagery and associated chlorophyll concentrations.
- National Status and Trends (NS&T) Mussel Watch data on concentrations of POPs in mussels are an important part of CCLEAN, as they cover areas of Monterey Bay not sampled by CCLEAN. NS&T QA procedures are very stringent and have been the basis for procedures used to collect and analyze shellfish for POPs nationwide.

# 19. DATA MANAGEMENT

CCLEAN monitoring data will be maintained as established in Section 9 above. Hard copies of all field logs, COCs, and other data sheets will be maintained by contractors conducting field sampling operations. Hard copies of lab reports will be stored at the Program Director's office as well as with the responsible laboratories. Supporting documentation for laboratory reports will be maintained by individual laboratories per their respective SOPs.

A summary of specific data management aspects is provided below:

### 19.1. Field Data Management

All field data will be reviewed for legibility and errors as soon as possible after the conclusion of sampling. All field data that is entered electronically will be hand-checked at a rate of 10% of entries as a check on data entry. Any corrective actions required will be documented in correspondence to the QAO.

### 19.2. Laboratory Data Management

Record keeping of CCLEAN analytical data will employ standard record-keeping and tracking practices. All laboratory analytical data will be entered into electronic files by the instrumentation being used or, if data are manually recorded, then it will be entered by the analyst in charge of the analyses, per laboratory standard procedures. All analytical data will conform to CEDEN requirements that it contain unique identification numbers for tracking.

The management of water quality data will be initiated with the use of field and laboratory data sheets. Data handling equipment and procedures for laboratory analytical data will be consistent with laboratory standard procedures. Laboratory analytical data that will be recorded using various analytical instruments will be formatted consistent with CEDEN data management rules. Backup copies of all data files will be made at the laboratory at the end of every day and stored electronically consistent with standard laboratory procedures. All laboratory data entry will conform to the standardized list available via CEDEN (http://www.ceden.us/Metadata/ControlledVocab.php), so that the data can be loaded into the CEDEN-comparable Project Database with minimal effort.

Following the completion of internal laboratory quality control checks, analytical results will be forwarded electronically to the Program Director. The analytical laboratories will provide data in electronic format, encompassing both a narrative and electronic data deliverable (EDD). The required form of electronic submittals, including CEDEN-comparable Microsoft Excel® templates, will be provided to the laboratories to ensure the files can be imported into the Project database with a minimum of editing. The data will be managed in a manner to expedite efficient upload into the CEDEN database. Data will be screened for the following major items:

- Conformity check between electronic data provided by the laboratory and the narrative reports
- Conformity check between the Chain-of-Custody Forms and laboratory reports

- A check for laboratory data report completeness
- A check for typographical errors on the laboratory reports
- A check for suspect values

Checked data will be delivered to the Central Coast Regional Water Quality Control Board via the online web-checking tool by January 31 each year for the program year ending the previous June 30.

# Group C: Assessment and Oversight

## 20. Assessments & Response Actions

The Project Director and project managers for each contractor will ensure that qualified personnel are employed in all phases of project implementation and that all personnel receive appropriate training to complete assigned tasks consistent with the CCLEAN Monitoring Plan (CCLEAN 2024).

### 20.1. Field Audits

Periodic audits may be conducted of field sampling procedures to ensure adherence to the CCLEAN QAPP. However, before any field sampling is conducted, the Project Manager for each subcontractor will verify that proper equipment is available for all field personnel. This includes sampling equipment, safety equipment, and field measurement equipment (if appropriate). It will also be verified that all personnel involved in field activities have received sufficient training and are able to properly use the equipment and follow procedures. The Project Manager or Field Program Manager may also verify the application of procedures and equipment periodically. If the Project Manager or Field Program Manager finds any deficiencies, corrective actions will be put in place and reported, and follow-on inspections will be performed to ensure the deficiencies have been addressed. Information from field audits will be included in the annual QA Audit report submitted to the CCLEAN Steering Committee and the Regional Board by November 31 each year.

### 20.2. Laboratory Data Reviews

The QAO will be responsible for reviewing the laboratory's data for completeness and accuracy. The data will also be checked to make sure that the appropriate methods were used and that all required QC data was provided with the sample analytical results. Laboratory data reviews will be conducted following receipt of each data package from a laboratory in order to ensure that all information is complete and any deviations from planned methodologies are either corrected or the reasons for change are documented. Any laboratory data that is discovered to be incorrect or missing will immediately be reported to both the laboratory and Program Director. The laboratory's QA manual details the procedures that will be followed by laboratory personnel to correct any invalid or missing data. The Program Director has the authority to request re-testing if a review of any of the laboratory data are found to be invalid or if it would compromise the quality of the data and resulting conclusions from the proposed project.

### 20.3. Laboratory Performance Audits

Initially, a QA performance audit may be performed by CCLEAN Program Director to determine if each laboratory is in compliance with the procedures outlined in the QAPP and to assist the laboratory where needed. Reviews will be conducted at least once every five years during the duration of the program. Results will be reviewed with laboratory staff and corrective action recommended and implemented where necessary. Moreover, laboratory performance will be assessed on a continuous basis through the use of laboratory intercomparison studies, such as EPA and NIST round-robins, and analysis of split samples by contract laboratories. An annual QA

Audit report will be submitted by the Program Director to the CCLEAN Steering Committee and the Regional Board by November 31 each year.

### 20.4. Corrective Actions

If an audit of any field sampling or laboratory operation discovers any discrepancy, the Program Director will discuss the observed discrepancy with the appropriate person responsible for the activity (see organization chart). The discussion will begin with whether the information collected is accurate, what were the cause(s) leading to the deviation, how the deviation might impact data quality, and what corrective actions might be considered. The Program Director and QA Officer have the power to halt all sampling and analytical work if the deviation(s) noted are considered detrimental to data quality.

# 21. REPORTS TO MANAGEMENT

## 21.1. CCLEAN Reports

CCLEAN requires an Annual Report (Table 22) to be submitted to the Central Coast Regional Water Quality Control Board by March 31 each year. The report includes the following items:

- a description of the study design,
- locations of sampling sites,
- a summary of sampling methods,
- highlights of temporal trends and spatial variation in data,
- comparison to water quality objectives and other applicable standards or alert levels, as described in Section 7
- synthesis of results relating data from different measurements to each other, and
- any recommended program changes.

Data are submitted to the Water Board electronically and are available to interested parties by contacting the CCLEAN Program Director.

The goal of the CCLEAN Annual Report is to provide a summary of results that addresses each program question and is understandable to informed lay people. Core management and scientific questions are stated first, followed by a concise summary of the major findings and the degree of confidence associated with these. Figures and maps are the main mode of presenting findings, and a summary of sampling effort is included. Statements about patterns in the monitoring results are accompanied by interpretations that discuss the implications of the results. More detailed data summaries, information on sampling and analysis methods, and discussion of QA/QC issues are presented in appendices.

## 21.2. CCLEAN QAPP

As the CCLEAN program programmatic documents are revised, the CCLEAN QAPP will be updated accordingly. Draft and final QAPP documents are submitted on the schedule shown in Table 22.

Table 22. Project Reporting Schedule.

| Type of Report | Frequency (daily, weekly, monthly, quarterly, annually, etc.) | Projected Delivery Dates(s) | Person(s) Responsible for Report Preparation | Report Recipients |
|---|---|---|---|---|
| Draft CCLEAN Annual Report | Annually | Jan 31 | Program Director | CCLEAN Steering Committee and Water Board |
| Final CCLEAN Annual Report | Annually | Mar 31 | Program Director | Water Board |
| CCLEAN electronic data deliverable | Annually | Jan 31 | Program Director | Water Board |
| CCLEAN QA Audit | Annually | Dec 31 | Program Director and QAO | CCLEAN Steering Committee |
| CCLEAN Monitoring Plan | Annually | July 1 | Program Director | CCLEAN Steering Committee and Water Board |
| Draft Revisions to CCLEAN QAPP | As necessary | TBD | Program Director and QAO | CCLEAN Steering Committee and Water Board |
| Final Revisions to CCLEAN QAPP | As necessary | TBD | Program Director | Water Board |

# Group D: Data Validation and Usability

## 22. Data Review, Verification, and Validation Requirements

Data verification is the process of evaluating the completeness, correctness, and conformance / compliance of a specific data set against the method, procedural, or contractual specifications. The QAO will conduct data verification, as described in Section 14 on Quality Control, in order to ensure that it is SWAMP-comparable with respect to completeness, correctness, and conformance with minimum requirements.

Data validation is an analyte- and sample-specific process that evaluates the information after the verification process (i.e., determination of method, procedural, or contractual compliance) to determine analytical quality and any limitations. The QAO will conduct data validation in order to ensure that the data are SWAMP-comparable with respect to its end use as described in Section 7. Data generated by project activities will be reviewed against the data quality objectives cited in Section 7 and the quality assurance/quality control practices cited in Section 14, 15, 16, and 17. Data will be separated into three categories: data meeting all data quality objectives, data failing precision or recovery criteria, and data failing to meet accuracy criteria. Data meeting all data quality objectives, but with failures of quality assurance/quality control practices will be set aside until the impact of the failure on data quality is determined. Once determined, the data will be moved into either the first category or the last category.

Data falling in the first category is considered usable by the project. Data falling in the last category is considered not usable. Data falling in the second category will have all aspects assessed. If sufficient evidence is found supporting data quality for use in this project, the data will be moved to the first category, but will be flagged consistent with lookup tables provided by the SWAMP data checker.

# 23. VERIFICATION AND VALIDATION METHODS

Each laboratory's QA Officer will be responsible for performing internal checks for all data per laboratory quality assurance procedures prior to submission to the Program Director. Once received by the Program Director, all data records will be checked visually and recorded as checked by initials and dates.

Any data that is discovered to be incorrect or missing during the CCLEAN verification or validation process will immediately be reported to the Program Director. If errors involve laboratory data, then this information will also be reported to the laboratory's QAO. Each laboratory's QA manual details the procedures that will be followed by laboratory personnel to correct any invalid or missing data. The QAO will be responsible for reporting and correcting any errors that are found in the data during the verification and validation process.

If there are any data quality problems identified, the QAO will try to identify whether the problem is a result of project design issues, sampling issues, analytical methodology issues, or QA/QC issues (from laboratory or non-laboratory sources). If the source of the problems can be traced to one or more of these basic activities, then the person or people in charge of the areas where the issues lie will be contacted and efforts will be made to immediately resolve the problem. If the issues are too broad or severe to be easily corrected then reconciliation and correction will be done by a committee composed of the CCLEAN Program Director, CCLEAN QAO, and the respective laboratory's Manager or QA Officer.

# 24. RECONCILIATION WITH USER REQUIREMENTS

As CCLEAN's studies include designs to estimate sources, loads, trends and effects of pollutants, any appropriate data that achieve the data quality objectives will contribute to the program's ability to answer its core questions. Such data may include those from other programs, such as the CCAMP and National Status and Trends Mussel Watch programs. The project needs sufficient numbers of data points, as represented by the completeness data quality objective in order to do trend analyses and determine the trends and effects of POPs on the prioritized beneficial uses. The ability of the project to determine trends will increase with each subsequent year of data. Trend analysis is performed with linear regression analysis or Seasonal Kendall Test to determine the relationship between data values and times or with analysis of variance for differences among years or locations. The CCLEAN Steering Committee annually reviews project results and this review helps ensure that the project is satisfying the program objectives. Moreover, program findings are regularly presented to regulatory agencies and the scientific community for peer review. Any limitations affecting the ability of the data to be used to meet original project objectives will be noted in annual reports.

The users of CCLEAN data have various requirements for data and information. The current program participants need data and information to inform decisions about achievement of NPDES permit influent and effluent limits, control of contaminant sources, wastewater plant performance, the effects of their discharges on beneficial uses and ways of reducing those effects. In order to support regulatory stakeholders, CCLEAN data will be delivered to SWAMP/CEDEN and to Region 3 to be included in 303(d) /305(b) assessments. Other stakeholders, such as the Monterey Bay National Marine Sanctuary and California Department of Fish and Game, use the data to assess the condition of marine water quality and establish priorities for management or remedial actions to improve the quality of marine habitats, especially for threatened species. Consequently, CCLEAN must adapt to the changing interests and priorities of program participants.

Regardless of the questions or priorities of participants, CCLEAN should provide the data necessary for testing hypotheses associated with program questions as efficiently as possible as well as to inform management actions. In order to base management actions on program results, it is necessary to know the sources and relative amounts of error in program data and variables derived from the data. Data for each of the program questions is discussed in this context below.

*What are the status and long-term trends in the quality of ocean waters, sediments, and associated beneficial uses?*

This question is answered by analyzing samples of water, tissue and sediment, comparing the results to regulatory and other criteria and testing them for trends. The main sources of error in these data are natural differences associated with small-scale variation in field samples and laboratory analytical error. Analysis of field duplicates of mussel samples provides an estimate of error that incorporates both sampling and analytical error. Analysis of field duplicates for

dieldrin over the life of the CCLEAN program has yielded an average difference between field duplicates of 23.4%. We can get a more accurate estimate of analytical error from the analysis of Certified Reference Materials (CRMs). The average difference between certified concentrations of dieldrin in the CRM NIST 1588a) analyzed by the laboratory (SGS AXYS) has been 20.6%. By taking a conservative approach and propagating the error through both sources (square root of ($23.4\%^2$ + $20.6\%^2$) we estimate the true value to be the reported value ±31.2%. We do not have data for field duplicates of sediment samples, but analysis of CRM (NIST 1944) in the CCLEAN program indicates an average difference between the reported value and the certified value for 4,4-DDT is 19.3%, which is very similar to the 20.6% error for dieldrin in mussels.

There are not applicable CRMs for water, but experiments performed by SGS AXYS, in which known amounts of contaminants were added to a large volume of water that was sampled with the SGS AXYS XAD-2 resin, provided data for estimating sampling efficiency (i.e., percent retention x percent recovery) for this method. Percent retention was calculated by passing a known amount of a pollutant through a column and determining the amount retained by analysis of the input and the output:

$$\text{Retention Efficiency} = \frac{\text{Input} - \text{Output}}{\text{Input}}$$

Recovery efficiency was calculated by eluting a retained pollutant from a column and analyzing the eluate:

$$\text{Recovery Efficiency} = \frac{\text{Amount recovered}}{\text{Amount on column prior to elution}}$$

The sampling efficiency for dieldrin was 81.8%±6.6 (retention = 100±1; recovery = 81.8±6.6). This equates to a sampling error of 19.2%. Sampling efficiencies for other compounds are presented in the SGS AXYS Infiltrex 300 User's Manual, included in Appendix B.

Sampling error and natural variation also affect our ability to detect trends. This error consists of the natural and sampling-related variation in the measured variable at each point in time, as well as the variation between times. A consideration of such variation can inform the redesign of CCLEAN where trend detection might be the primary objective of sampling and high inherent variability allows a lower sampling frequency without substantially reducing the time required to detect a significant trend.

*Do ocean waters and sediments comply with California Ocean Plan and associated NPDES permits?*

This question is answered by comparing measured concentrations of contaminants to the California Ocean Plan NPDES permit influent and effluent limits and other sediment criteria. The same sources of error apply as for the question above.

*What are the major sources of contaminants to ocean waters?*

The same errors associated with sampling water, as described above, apply to this question. Moreover, there is error associated with the estimates of flow. Loads estimates previously made for rivers were based upon the average of the daily loads calculated for each sampling period, which were multiplied by 365. The average flow rates during the sampling periods varied from the overall daily average flow by an average of 130%. Consequently, when the sampling and analytical error are combined with the error in flow estimates, the error in load estimates for rivers could be as high as 133%. Because flows of wastewater effluent vary much less than rivers throughout the year, averages from the 30-day sampling periods are more similar to the annual average and associated errors in load estimates are much smaller. Calculations for wastewater reveal an average error in the flow estimate of 6.6%, resulting in an error of 20.3% in load estimates.

*What are the effects of wastewater discharges in ocean waters?*

Hypothesis testing associated with this question involves both measures of association between load estimates and ambient ecological variables, as well as the screening of effluent for reproductive endocrine disruption in the fish assays. We are not aware of methods for estimating the error of these methods.

Other user requirements could lead to future changes in the CCLEAN program. For example, changes from the current method of high-volume water sampling could be made in response to changes in the contaminants of concern. Increased interest in the environmental effects of pharmaceuticals and personal care products could result in broader application of POCIS to sample these polar compounds.

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# 26. APPENDIX A

Table A1. Control Limits for surrogates per analytical method.

| Analytical Method | Compound | Surrogate | Percent Recovery Limits | Comments |
|---|---|---|---|---|
| MLA-217 REV. 01 | Dioxins/Furans | $^{13}C_{12}$-2,3,7,8-TCDD | 25-130 | |
| MLA-217 REV. 01 | Dioxins/Furans | $^{13}C_{12}$-2,3,7,8-TCDF | 24-130 | |
| MLA-217 REV. 01 | Dioxins/Furans | $^{13}C_{12}$-1,2,3,7,8-PeCDD | 25-130 | |
| MLA-217 REV. 01 | Dioxins/Furans | $^{13}C_{12}$-1,2,3,7,8-PeCDF | 24-130 | |
| MLA-217 REV. 01 | Dioxins/Furans | $^{13}C_{12}$-2,3,4,7,8-PeCDF | 21-130 | |
| MLA-217 REV. 01 | Dioxins/Furans | $^{13}C_{12}$-1,2,3,4,7,8-HxCDD | 32-130 | |
| MLA-217 REV. 01 | Dioxins/Furans | $^{13}C_{12}$-1,2,3,6,7,8-HxCDD | 28-130 | |
| MLA-217 REV. 01 | Dioxins/Furans | $^{13}C_{12}$-1,2,3,4,7,8-HxCDF | 26-130 | |
| MLA-217 REV. 01 | Dioxins/Furans | $^{13}C_{12}$-1,2,3,6,7,8-HxCDF | 26-123 | |
| MLA-217 REV. 01 | Dioxins/Furans | $^{13}C_{12}$-1,2,3,7,8,9-HxCDF | 29-130 | |
| MLA-217 REV. 01 | Dioxins/Furans | $^{13}C_{12}$-2,3,4,6,7,8-HxCDF | 28-130 | |
| MLA-217 REV. 01 | Dioxins/Furans | $^{13}C_{12}$-1,2,3,4,6,7,8-HpCDD | 23-130 | |
| MLA-217 REV. 01 | Dioxins/Furans | $^{13}C_{12}$-1,2,3,4,6,7,8-HpCDF | 28-130 | |
| MLA-217 REV. 01 | Dioxins/Furans | $^{13}C_{12}$-1,2,3,4,7,8,9-HpCDF | 26-130 | |

| | | | | |
|---|---|---|---|---|
| MLA-217 REV. 01 | Dioxins/Furans | 13C12-OCDD | 17-130 | |
| MLA-210 REV. 01 | PCBs | $^{13}C_{12}$-3,3',4,4'-TCB | 10-145 | |
| MLA-210 REV. 01 | PCBs | $^{13}C_{12}$-3,4,4',5-TeCB | 10-145 | |
| MLA-210 REV. 01 | PCBs | $^{13}C_{12}$-2,2',4,6,6'-PeCB | 10-145 | |
| MLA-210 REV. 01 | PCBs | $^{13}C_{12}$-2,3,3',4,4'-PeCB | 10-145 | |
| MLA-210 REV. 01 | PCBs | $^{13}C_{12}$-2,3,4,4',5-PeCB | 10-145 | |
| MLA-210 REV. 01 | PCBs | $^{13}C_{12}$-2,3',4,4',5-PeCB | 10-145 | |
| MLA-210 REV. 01 | PCBs | $^{13}C_{12}$-2',3,4,4',5-PeCB | 10-145 | |
| MLA-210 REV. 01 | PCBs | $^{13}C_{12}$-3,3',4,4',5-PeCB | 10-145 | |
| MLA-210 REV. 01 | PCBs | $^{13}C_{12}$-2,2',4,4',6,6'-HxCB | 10-145 | |
| MLA-210 REV. 01 | PCBs | $^{13}C_{12}$-2,3,3',4,4',5-HxCB [3] | 10-145 | |
| MLA-210 REV. 01 | PCBs | $^{13}C_{12}$-2,3,3',4,4',5'-HxCB [3] | 10-145 | |
| MLA-210 REV. 01 | PCBs | $^{13}C_{12}$-2,3',4,4',5,5'-HxCB | 10-145 | |
| MLA-210 REV. 01 | PCBs | $^{13}C_{12}$-3,3',4,4',5,5'-HxCB | 10-145 | |
| MLA-210 REV. 01 | PCBs | $^{13}C_{12}$-2,2',3,3',4,4',5-HpCB | 10-145 | |
| MLA-210 REV. 01 | PCBs | $^{13}C_{12}$-2,2',3,4,4',5,5'-HpCB | 10-145 | |

| | | | | |
|---|---|---|---|---|
| MLA-210 REV. 01 | PCBs | $^{13}C_{12}$-2,2',3,4',5,6,6'-HpCB | 10-145 | |
| MLA-210 REV. 01 | PCBs | $^{13}C_{12}$-2',3,3',4,4',5,5'-HpCB | 10-145 | |
| MLA-210 REV. 01 | PCBs | $^{13}C_{12}$-2,2',3,3',5,5',6,6'-OcCB | 10-145 | |
| MLA-210 REV. 01 | PCBs | $^{13}C_{12}$-2,3,3',4,4',5,5',6-OcCB | 10-145 | |
| MLA-210 REV. 01 | PCBs | $^{13}C_{12}$-2,2',3,3',4,4',5,5',6-NoCB | 10-145 | |
| MLA-210 REV. 01 | PCBs | $^{13}C_{12}$-2,2',3,3',4,5,5',6,6'-NoCB | 10-145 | |
| MLA-210 REV. 01 | PCBs | $^{13}C_{12}$-2,2',3,3',4,4',5,5',6,6'-DeCB | 10-145 | |
| MLA-110 REV. 02 | PFAS | $^{13}C_4$-PFBA | 5-150 | |
| MLA-110 REV. 02 | PFAS | $^{13}C_5$-PFPeA | 15-150 | |
| MLA-110 REV. 02 | PFAS | 13C5-PFHxA | 15-150 | |
| MLA-110 REV. 02 | PFAS | $^{13}C_4$-PFHpA | 20-150 | |
| MLA-110 REV. 02 | PFAS | $^{13}C_8$-PFOA | 15-150 | |
| MLA-110 REV. 02 | PFAS | 13C9-PFNA | 20-150 | |
| MLA-110 REV. 02 | PFAS | 13C6-PFDA | 40-150 | |
| MLA-110 REV. 02 | PFAS | 13C7-PFUnA | 50-150 | |
| MLA-110 REV. 02 | PFAS | 13C2-PFDoA | 50-150 | |
| MLA-110 REV. 02 | PFAS | $^{13}C_2$-PFTeDA | 50-150 | |
| MLA-110 REV. 02 | PFAS | 13C3-PFBS | 40-150 | |
| MLA-110 REV. 02 | PFAS | 13C3-PFHxS | 50-150 | |
| MLA-110 REV. 02 | PFAS | 13C8-PFOS | 50-150 | |

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| MLA-110 REV. 02 | PFAS | $^{13}C_2$-4:2 FTS | 10-150 | |
| MLA-110 REV. 02 | PFAS | $^{13}C_2$-6:2 FTS | 15-150 | |
| MLA-110 REV. 02 | PFAS | $^{13}C_2$-8:2 FTS | 15-150 | |
| MLA-110 REV. 02 | PFAS | 13C8-PFOSA | 40-150 | |
| MLA-110 REV. 02 | PFAS | $D_3$-NMeFOSA | 5-150 | |
| MLA-110 REV. 02 | PFAS | $D_5$-NEtFOSA | 5-150 | |
| MLA-110 REV. 02 | PFAS | $D_3$-NMeFOSAA | 15-170 | |
| MLA-110 REV. 02 | PFAS | $D_5$-NEtFOSAA | 40-210 | |
| MLA-110 REV. 02 | PFAS | $D_7$-NMeFOSE | 1-150 | |
| MLA-110 REV. 02 | PFAS | $D_9$-NEtFOSE | 1-150 | |
| MLA-110 REV. 02 | PFAS | $^{13}C_3$-HFPO-DA | 10-150 | |
| MLA-228 REV 01 | Organochlorine Pesticides | $^{13}C_6$-1,4-dichlorobenzene | ** | |
| MLA-228 REV 01 | Organochlorine Pesticides | $^{13}C_6$-1,2,3-trichlorobenzene | ** | |
| MLA-228 REV 01 | Organochlorine Pesticides | $^{13}C_6$-1,2,3,4-tetrachlorobenzene | ** | |
| MLA-228 REV 01 | Organochlorine Pesticides | $^{13}C_6$-Pentachlorobenzene | 20-150 | |
| MLA-228 REV 01 | Organochlorine Pesticides | $^{13}C_6$-Hexachlorobenzene | 20-150 | |
| MLA-228 REV 01 | Organochlorine Pesticides | $^{13}C_6$-alpha-HCH | 30-150 | |
| MLA-228 REV 01 | Organochlorine Pesticides | $^{13}C_6$-beta-HCH | 30-150 | |
| MLA-228 REV 01 | Organochlorine Pesticides | $^{13}C_6$-gamma-HCH | 30-150 | |
| MLA-228 REV 01 | Organochlorine Pesticides | $^{13}C_{10}$-Heptachlor | 30-150 | |
| MLA-228 REV 01 | Organochlorine Pesticides | $^{13}C_{12}$-Aldrin | 30-150 | |

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| MLA-228 REV 01 | Organochlorine Pesticides | $^{13}C_{10}$-Oxychlordane | 30-200 | |
| MLA-228 REV 01 | Organochlorine Pesticides | $^{13}C_{10}$-trans-Chlordane | 30-200 | |
| MLA-228 REV 01 | Organochlorine Pesticides | $^{13}C_{12}$-o,p'-DDE | 40-150 | |
| MLA-228 REV 01 | Organochlorine Pesticides | $^{13}C_{12}$-p,p'-DDE | 40-150 | |
| MLA-228 REV 01 | Organochlorine Pesticides | $^{13}C_{10}$-trans-Nonachlor | 30-150 | |
| MLA-228 REV 01 | Organochlorine Pesticides | $^{13}C_{10}$-cis-Nonachlor | 30-150 | |
| MLA-228 REV 01 | Organochlorine Pesticides | $^{13}C_{12}$-o,p'-DDD | 40-150 | |
| MLA-228 REV 01 | Organochlorine Pesticides | $^{13}C_{12}$-p,p'-DDD | 40-150 | |
| MLA-228 REV 01 | Organochlorine Pesticides | $^{13}C_{12}$-o,p'-DDT | 40-150 | |
| MLA-228 REV 01 | Organochlorine Pesticides | $^{13}C_{12}$-p,p'-DDT | 40-150 | |
| MLA-228 REV 01 | Organochlorine Pesticides | $^{13}C_{10}$-Mirex | 30-150 | |
| MLA-228 REV 01 | Organochlorine Pesticides | $^{13}C_{6}$-delta-HCH | 30-150 | |
| MLA-228 REV 01 | Organochlorine Pesticides | $^{13}C_{12}$-Dieldrin | 30-150 | |
| MLA-228 REV 01 | Organochlorine Pesticides | $^{13}C_{12}$-Endrin | 30-150 | |
| MLA-228 REV 01 | Organochlorine Pesticides | $^{13}C_{12}$-Endrin aldehyde | 30-150 | |
| MLA-228 REV 01 | Organochlorine Pesticides | $^{13}C_{10}$-cis-Heptachlor epoxide | 30-150 | |
| MLA-228 REV 01 | Organochlorine Pesticides | $^{13}C_{12}$-Endrin ketone | 30-150 | |
| MLA-228 REV 01 | Organochlorine Pesticides | $^{13}C_{9}$-Endosulfan sulfate | 30-150 | |

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| MLA-228 REV 01 | Organochlorine Pesticides | $^{13}C_{12}$-Methoxychlor | 30-150 | |
| MLA-228 REV 01 | Organochlorine Pesticides | $^{13}C_{9}$-alpha-Endosulfan | 30-150 | |
| MLA-228 REV 01 | Organochlorine Pesticides | $^{13}C_{9}$-beta-Endosulfan | 30-150 | |
| MLA-228 REV 01 | Organochlorine Pesticides | $^{13}C_{12}$-PCB 159 | 40-130 | (only when technical toxaphene is analyzed) |
| MLA-021 REV. 12 | PAHs | $d_8$-naphthalene | 15 – 130 | |
| MLA-021 REV. 12 | PAHs | $d_8$-acenaphthylene | 20 – 130 | |
| MLA-021 REV. 12 | PAHs | $d_{10}$-phenanthrene | 30 – 130 | |
| MLA-021 REV. 12 | PAHs | $d_{10}$-fluoranthene * | 30 – 130 | |
| MLA-021 REV. 12 | PAHs | $d_{12}$-benz[a]anthracene | 30 – 130 | |
| MLA-021 REV. 12 | PAHs | $d_{12}$-chrysene | 30 – 130 | |
| MLA-021 REV. 12 | PAHs | $d_{12}$-benzo[b]fluoranthene | 30 – 130 | |
| MLA-021 REV. 12 | PAHs | $d_{12}$-benzo[k]fluoranthene | 30 – 130 | |
| MLA-021 REV. 12 | PAHs | $d_{12}$-benzo[a]pyrene | 30 – 130 | |
| MLA-021 REV. 12 | PAHs | $d_{12}$-perylene | 30 – 130 | |
| MLA-021 REV. 12 | PAHs | $d_{14}$-dibenz[ah]anthracene * | 30 – 130 | |

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| MLA-021 REV. 12 | PAHs | $d_{12}$-indeno[1,2,3-cd]pyrene | 30 – 130 | |
| MLA-021 REV. 12 | PAHs | $d_{12}$-benzo[ghi]perylene | 30 – 130 | |
| MLA-021 REV. 12 | PAHs | $d_{10}$-2-methylnaphthalene | 20 – 130 | |
| MLA-021 REV. 12 | PAHs | $d_{12}$-2,6-dimethylnaphthalene | 20 – 130 | |
| MLA-021 REV. 12 | PAHs | $d_{10}$-biphenyl | 15 – 130 | |
| MLA-021 REV. 12 | PAHs | $d_8$-dibenzothiophene | 30 – 130 | |
| EPA 625.1-NCI | Fipronil & Degradates | (13C-4-Fipronil) | 50 - 150% | |
| EPA 625.1-MRM | Neonicotinoid Compounds | (d3-Thiamethoxam) | 25 - 150% | |
| EPA 625.1-MRM | Neonicotinoid Compounds | (d3-Clothianidin) | 25 - 150% | |
| EPA 625.1 | Organophosphorus Pesticides | (PCB030) | 52 - 124% | |
| EPA 625.1 | Organophosphorus Pesticides | (PCB112) | 49 - 133% | |
| EPA 625.1 | Organophosphorus Pesticides | (PCB198) | 60 - 129% | |
| EPA 625.1 | Organophosphorus Pesticides | (TCMX) | 6 - 124% | |
| EPA 625.1 | Acid Extractable Compounds | (d5-Phenol) | 0 - 85% | |
| EPA 625.1 | Acid Extractable Compounds | (2,4,6-Tribromophenol) | 31 - 143% | |
| EPA 625.1-MRM | Pyrethroids | (d5-Bifenthrin) | 50 - 150% | |

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| EPA 625.1-MRM | Pyrethroids | (d5-Fenvalerate) | 50 - 150% | |

** Recovery of dichlorobenzenes, trichlorobenzenes and tetrachlorobenzenes may be low due to loss through volatilization during the analytical work-up. These compounds may be reported only when recoveries are judged adequate for quantification. Formal recovery acceptance limits have not been established.