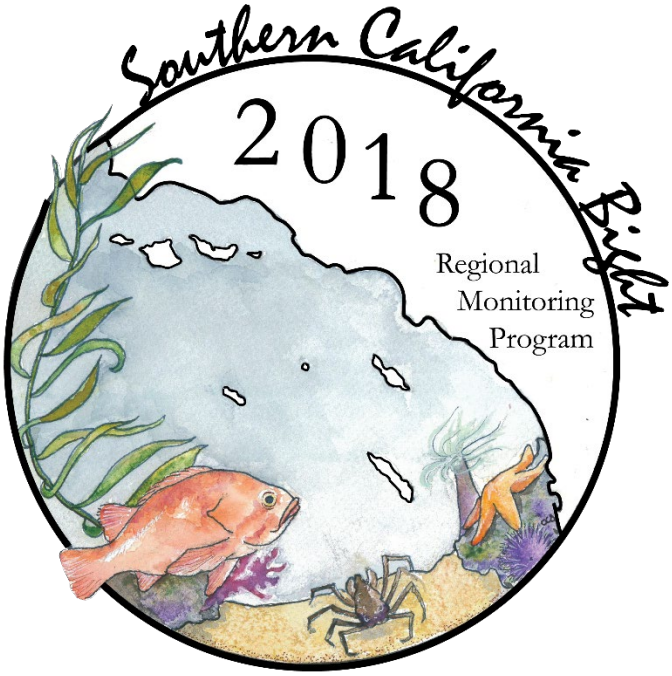
Southern California Bight
2018 Regional Marine Monitoring Program
(Bight '18)

Quality Assurance Manual



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Bight '18 Sediment Quality Planning Committee

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I. INTRODUCTION

Southern California Bight Regional Marine Monitoring Program (Bight) is a large, integrated, collaborative monitoring survey. The Bight program is conducted by dozens of agencies, collecting hundreds of samples, for a wide variety of measures including stressor (pollutant concentrations) and response variables (biological condition). Unlike most ongoing monitoring programs that are site-specific, the Bight program spans over 300 km of Southern California Bight (SCB) coastline stretching from Point Conception, north of Santa Barbara, south to the US International Border with Mexico (Figure I-1). Because of the large number of sites, wide variety of measurements, and large geographic expanse, the Bight program provides a unique perspective of environmental condition that can be achieved in no other fashion.

A total of five Bight surveys have been conducted (1994, 1998, 2003, 2008, 2013) approximately every five years. The results of the Bight surveys have been used for a variety of purposes including scientific research, regulatory compliance, and public information. All of the Bight reports, including the raw data, are publicly accessible over the internet (http://www.sccwrp.org/ResearchAreas/RegionalMonitoring/BightRegionalMonitoring.aspx). The Bight program has also helped develop a number of independent assessment tools (Benthic Response Index, Fish Response Index, Iron Normalization of Trace Metal Contamination, Sediment Quality Objectives), used frequently in examining site-specific impacts.

The Bight '18 Program is organized into five technical components: 1) Sediment Quality (formerly Contaminant Impact Assessment; Coastal Ecology); 2) Microbiology; 3) Ocean Acidification; 4) Harmful Algal Blooms; and 5) Trash. This document concerns quality assurance for the Sediment Quality component, which focuses on sediment contaminants and associated impacts on benthic infauna and demersal fish and epibenthic megainvertebrates.

The overall goal of the Sediment Quality component of Bight '18 is to assess the condition of the benthic environment and the health of the biological resources in the SCB. To accomplish this goal, Bight '18 will focus on three primary questions:

1. What is the extent and magnitude of sediment quality impacts in the Southern California Bight?
2. How does the extent and magnitude of sediment quality impacts vary over time in the Southern California Bight?
3. What is the extent and magnitude of bioaccumulation of select contaminants in seafood in the Southern California Bight?

The first two questions are similar to questions posed in previous Bight surveys, although new chemicals and new habitats will be explored in Bight'18. The third question is also similar to previous surveys, reevaluating bioaccumulation in sport fish last conducted during the 2008 survey. The details of the sampling design are captured in the Bight'18 Sediment Quality Workplan, created by the 46 participating organizations (Table I-1) in Bight'18.

The large-scale, collaborative nature of Bight places a particularly large emphasis on quality assurance (QA). Since multiple agencies participate in every facet of the monitoring,

from sampling to laboratory analysis, ensuring comparable data of the highest quality is paramount to success. The goal of this document is to serve as the vehicle to ensure the data quality and comparability among the many agencies participating in Bight, and to ensure data quality for comparisons to previous Bight surveys.

This QA Plan is only one of a series of documents produced to support Bight'18 quality and comparability for the Sediment Quality assessment. The other documents include Bight'18 Sediment Quality Workplan, Bight'18 Field Operations Manual, Bight'18 Toxicity Laboratory Manual, Bight'18 Infaunal Laboratory Manual and Bight '18 Information Management Plan. These additional documents provide details on sampling design, logistics, and laboratory analysis. Moreover, they include detailed requirements for implementation and documentation of QA for individual activities to support this QA Plan. Documentation for the additional Bight'18 elements can be found online: (http://www.sccwrp.org/ResearchAreas/RegionalMonitoring/BightRegionalMonitoring.aspx).

FIGURE I-1. Map of the Southern California Bight.

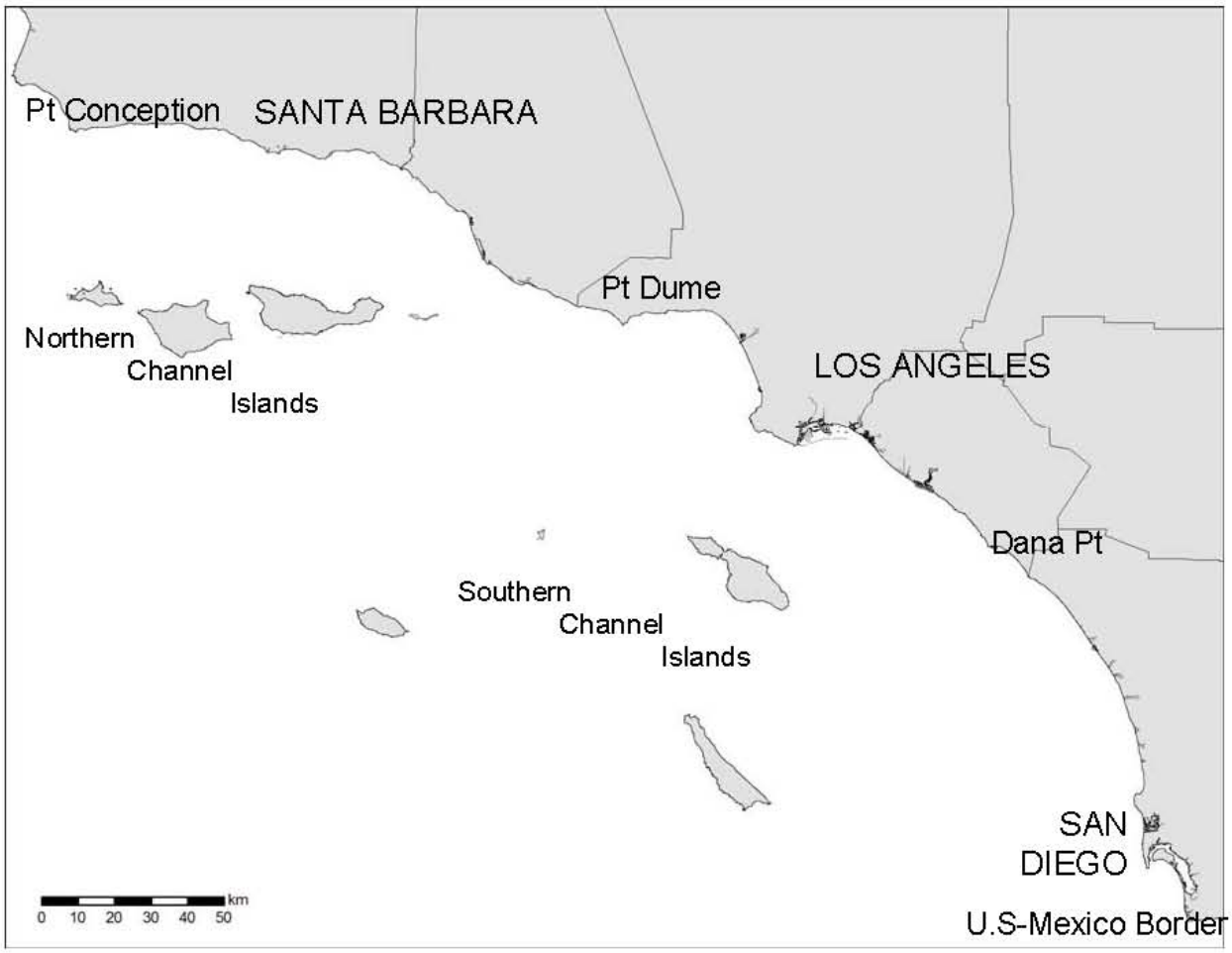

TABLE I-1. Participants in the Bight '18 Regional Monitoring Program, Sediment Quality Assessment

AES Corporation
Amec Foster Wheeler / Wood
Anchor QEA
Aquatic Bioassay and Consulting Laboratories (ABCL)
Bureau of Ocean Energy Management (BOEM)
Calscience Environmental Laboratories, Inc.
Channel Islands National Marine Sanctuary (CINMS)
Chevron USA Products Company
City of Los Angeles, Department of Water and Power (LADWP)
City of Los Angeles Environmental Monitoring Division (CLAEMD)
City of Oceanside
City of Oxnard
City of San Diego
Dominguez Channel Watershed Management Group (City of Los Angeles Watershed Protection Division, Los Angeles County Flood Control District, Los Angeles County, City of Lomita, City of Carson, City of El Segundo, City of Hawthore, City of Inglewood, City of Lawndale)
EcoAnalysts
Encina Wastewater Authority
Greater Harbor Waters Regional Monitoring Coalition
Los Angeles Regional Water Quality Control Board
Los Angeles County Department of Public Works
Los Angeles County Sanitation Districts (LACSD)
MBC Aquatic Sciences
National Oceanic and Atmospheric Administration (NOAA)
Nautilus Environmental, Inc.
Naval Facilities Engineering Command (NAVFAC) Southwest
NES Energy, Inc.
NRG Energy, Inc.
Orange County Sanitation District (OCSD)
Orange County Public Works
Pacific EcoRisk
Physis Environmental Laboratories, Inc.
Port of Long Beach
Port of Los Angeles
Riverside County Flood Control and Water Conservation District
San Diego County Dept. of Environmental Health and Municipal Co-permittees
San Diego Regional Water Quality Control Board (SDRWQCB)
San Diego Unified Port District
San Elijo Joint Powers Authority
Santa Ana Regional Water Quality Control Board
Southern California Coastal Water Research Project
Space and Naval Warfare (SPAWAR) Systems Center Pacific
State Water Resources Control Board
U.S. Fish and Wildlife Service
U.S. Geological Survey
Vantuna Research Group, Occidental College
Weck Laboratories, Inc.
Weston Solutions, Inc.

II. QUALITY ASSURANCE OBJECTIVES

A. Overview

The primary goal of the QA Plan and related quality control activities (collectively QA/QC) is to ensure that the data generated in the Bight '18 program are comparable among particpants. Many different organizations will be participating in the collection and analysis of samples in Bight '18; encouraging and maintaining consistency in field and laboratory operations and ensuring data comparability will be critical to success of the survey.

Data comparability will be achieved through a combination of standardized methods (where appropriate) and performance-based standards. Where standardized methods have been agreed upon for this survey, QA/QC measures will be used to assure that methods are applied consistently. Where performance-based standards are appropriate, QA/QC measurements will be used as a measure of performance. The appropriate QA/QC procedures for each of the Sediment Quality assessment monitoring program components (e.g., field operations, sediment and fish tissue chemical analyses, sediment toxicology, benthic analyses, demersal fish/invertebrate analyses, debris, and information management) have been established by the Bight '18 Sediment Quality Planning Committee.

B. General Approach To Quality Assurance

The QA program for Bight '18 consists of two distinct but related activities: quality assurance and quality control. Quality assurance (QA) includes design, planning, and management activities conducted prior to implementation of the survey to ensure that the appropriate kinds and quantities of data will be collected. The goals of quality assurance are to ensure: 1) field collection, processing, and laboratory analytical techniques will be applied consistently and correctly; 2) the number of lost, damaged, and uncollected samples will be minimized; 3) the integrity of the data will be maintained and documented from sample collection to entry into the data record; 4) all data will be comparable; and 5) results can be reproduced.

Quality control (QC) activities are implemented during the data collection phase of the survey to evaluate the effectiveness of the QA activities. QC activities ensure that measurement error and bias are identified, quantified, and accounted, or eliminated, if practical. QC activities include both internal and external checks. Typical internal QC checks include repeated measurements, internal test samples, use of independent methods to verify findings, and use of standard reference materials. Typical external QC checks include exchanging samples among laboratories for reprocessing to test comparability of results, independent performance audits, and periodic proficiency examinations.

Many of the organizations participating in Bight '18 have well established monitoring programs. QA activities for Bight '18 have focused on developing a common field manual and documenting the comparability of laboratory methods. Training of field and laboratory

personnel is focused on communicating goals and objectives of the survey, as well any modifications in methods or procedures that have been made to ensure data comparability. The purpose of this training is to verify that all participants will be able to implement the agreed upon procedures in a consistent manner with comparable proficiency. Quantitative measures have been developed to translate the overall effectiveness of this training on QA/QC activities. These quantitative measures are known as Measurement Quality Objectives (MQOs).

C. Measurement Quality Objectives

MQOs establish acceptable levels of uncertainty for each measurement process. MQOs typically address the major components of data quality: representativeness, completeness, precision, accuracy and comparability. Data comparability, or "the confidence with which one data set can be compared to another" (Stanley and Verner 1985), is a primary concern in this survey because of the large number of participants. Comparability of reporting units and calculations, data base management processes, and interpretative procedures must be ensured if the overall goals of the survey are to be realized.

Specific MQOs for precision and accuracy, the most readily quantifiable components of data quality, have been identified for Bight '18 to ensure that the data produced by the many field crews and laboratories involved in the survey will be comparable. Accuracy is defined as the difference between the measured value of an indicator and its true value, which represents an estimate of systematic error or net bias (Kirchner 1983, Hunt and Wilson 1986, Taylor 1987). Precision is the degree of mutual agreement among individual measurements and represents an estimate of random error (Kirchner 1983, Hunt and Wilson 1986, Taylor 1987). Together, accuracy and precision provide an estimate of the total error or uncertainty associated with a measured value. Requiring participating field crews and laboratories to achieve standard, quantitative MQOs for accuracy and precision will help to ensure that individual data sets are free of any crew- and/or laboratory-specific bias and that the degree of random error is consistent across data sets. Accuracy and precision goals for indicators to be measured during Bight '18 are provided in Table 2-1. Accuracy and precision cannot be defined for all parameters because of the nature of the measurements. For example, accuracy measurements are not possible for toxicity testing or sample collection activities. Measurement of accuracy and precision in sediment toxicity testing would require the use of reference materials with a known level of toxicity that is stable during storage, and such reference materials are not available.

An MQO for completeness was also defined for Bight '18. Completeness is a measure of the proportion of the expected, valid data (i.e., data not associated with some criterion of unacceptability) collected during a measurement process. The MQO for completeness is 90% for each measurement process. The sampling design for the survey is sufficiently redundant to absorb the loss of up to 10% of the samples without compromising the goals of the program, provided that the lost samples are not concentrated in a single subpopulation of interest. Redundancy was incorporated at this level because monitoring programs of this size typically lose as many as 10% of samples as a result of logistical difficulties or failure to achieve quality control criteria.

TABLE 2-1. Measurement Quality Objectives for Bight '18 indicators and data. (NA – not applicable; SD – standard deviation).

Indicators	Accuracy	Precision	Completeness
Sediment Properties			
sediment grain size	NA	20%	90%
total organic carbon	15%	20%	90%
organic contaminants	30%	30%	90%
inorganic contaminants	20%	30%	90%
Benthic Infauna			
sample collection	NA	NA	90%
sorting	5%	NA	90%
taxa discriminated	10%	NA	90%
count accuracy	10%	NA	90%
identification accuracy	10%	NA	90%
Sediment Toxicity			
amphipod survival	NA	2 SD	90%
mussel development	NA	2 SD	90%
Demersal fish and macroinvertebrates			
sample collection	NA	NA	90%
counting	10%	NA	90%
identification	10%	NA	90%
length	10%	NA	90%
biomass	10%	NA	90%
external anomalies	10%	NA	90%

D. Quality Assurance And Quality Control Activities

Establishing MQOs is of little value if the proper quality assurance activities are not undertaken to ensure that such objectives will be met. Quality assurance in Bight '18 will be achieved by:

- Developing a common field manual,
- Documenting comparability of laboratory methods consistent with the MQOs, and
- Implementing training workshops to ensure that participants are familiar with the methods and are able to achieve the MQOs.

The effectiveness of quality assurance efforts will be measured by quality control activities that fall into two categories:

- Routine QC checks coordinated by each laboratory or field crew's internal QA Officer, and
- Performance audits conducted by the Bight '18 QA Officers (Technical Committee Chairs) or designees

The goal of these activities is to quantify accuracy and precision, but, most importantly, they will be used to identify problems that need to be corrected as data sets are generated and assembled.

A new Field Operations Manual has been prepared to standardize data collection efforts in the field. Each participating organization collecting samples in the field has identified a single point of contact for field operations (referred to as the Lead Scientist in the field operations manual).

A single laboratory manual was not developed for the survey because each of the participating laboratories have their own internal operating procedures. Comparability of laboratory efforts will be ensured through compliance with the requirements listed in this Quality Assurance Plan (QAP) which identifies performance-based standards and the appropriate level of QA/QC. Procedures for benthic analyses appropriate to the Bight '18 Program are detailed in the Macrobenthic (Infaunal) Sample Analysis Laboratory Manual (2018). Procedures for toxicology analyses appropriate to the Bight '18 Program are detailed in the Toxicity Laboratory Manual (2018).

The manuals and the QA/QC requirements were prepared in coordination with the appropriate personnel from each of the participating organizations. Potential problem areas identified in the preparation and review of these manuals were resolved using a consensus-based approach. Copies of these manuals have been distributed to all participants in the program. These manuals will form the basis for training workshops and provide a reference for field and laboratory personnel during sample collection and processing activities.

III. REQUIREMENTS FOR FIELD AND LABORATORY OPERATIONS

A. Field Operations

The Bight '18 Program will be conducted cooperatively by a number of organizations (including one or more contractors), which routinely monitor the marine environment according to their own protocols. It is important to the success of the Bight '18 Program that comparable data are collected by each organization during the survey.

Quality Assurance activities for field collection include:

- The development of the field operations manual which details the procedures to be used in the Bight '18 program,
- A series of presurvey methods and taxonomy protocol intercalibration meetings/exercises to ensure that survey participants understand the requirements outlined in the field manual, and
- A presurvey field audit of participants who routinely use Bight protocols in their regular monitoring activities is acceptable for demonstrating understanding and capability. This allows for more time to perform in-survey audits on new participants or organizations with significant personnel changes.

Quality control measures for the field collection effort include:

- Specific QC requirements outlined in the QAP, which will be the responsibility of the lead scientist of each vessel, and
- Field audits of each vessel.

Field operations manual

Standard field procedures are documented in the Bight '18 Field Operations Manual (2018). The field manual includes detailed descriptions of collection procedures, criteria for acceptable samples, and conditions under which samples need to be recollected. The field operations manual will provide the basis for protocol calibration exercises and a reference for field personnel during sampling activities.

The field manual provides an overview of field teams and activities and procedures related to safety, protocol calibration, navigation requirements, sampling schedule and station types, procedures for benthic sampling, procedures for trawl sampling, procedures for packaging and shipping of samples, contingency plans, and procedures for managing information collected in the field.

Lead Scientists and Boat Captains will be instructed on the field procedures to be followed during the survey and they, in turn, will instruct their field personnel on the proper procedures for the survey. The lead scientist of each organization is responsible for distributing the Bight '18 Field Operations Manual to all field personnel and ensuring that their staff understand and use the protocols detailed in the manual.

Training and protocol calibration

Proper training of field personnel is a critical aspect of quality assurance. Organizations participating in Bight '18 will provide personnel who have extensive field experience, but not necessarily with the standard methods selected for this survey. Instruction for this survey, therefore, will focus on ensuring consistency in data collection among all field personnel.

Lead Scientists and Boat Captains of all organizations participating in the survey will be required to attend a protocol calibration meeting, which will be conducted several weeks before the survey. The goals and objectives of Bight '18 will be discussed at this meeting as well as the responsibilities of the chief scientist and boat captains. Each participating organization will be provided with a Workplan, Field Operations Manual and QA/QC Document for Bight '18 and will be instructed on field procedures to be used during the survey, including proper entry of data on field data forms. The meeting will emphasize decision-making procedures for determining whether a station should be abandoned and whether a sample is acceptable. Lines of communication within the survey and QA/QC activities occurring on the boat during the survey will also be discussed.

The Lead Scientist of each organization will train their field personnel, as needed, on the field operations to be conducted during the survey. It will be the responsibility of the Lead Scientist of each organization to review the Workplan and Field Operations Manual with their field crews and to ensure that they understand the field procedures and specific field QA/QC requirements that must be followed during the survey. It is also the Lead Scientist's responsibility to train their field crews, as needed, on operations to be performed. Personnel that cannot perform an operation as required by the survey will not participate in that operation.

Field audits

Field sampling capability will be established by means of field audits conducted by the Field QA Auditor. These field audits will be conducted to assess equipment, vessels, and protocols used by participating organizations, and to instruct the crew as needed on the procedures described in the field operation manual and the QA/QC document. Organizations that did not participate in a previous Bight Program will receive the first field audits; previous Bight Program participants who have a significant number of new staff members or previous Bight Program participants that did not meet all of their Data Quality Objectives (DQOs, see below) will be the second priority.

A Field QA/QC Checklist, developed to provide comparability and consistency in this process, will be used to record the audit data. The Field QA Specialist will provide additional

instruction to the cruise leader and crew when discrepancies are noted during the field QA audit. The Lead Scientist will also be notified of audit results so that any problems can be corrected.

Ongoing quality control during the sample period will be established through an organization's internal field audits. Methods for internal audits are described in the Bight '18 Field Manual. Cruise leaders will audit their own crew during trawls at a minimum of 10% of their assigned trawl sites (or a minimum of one site, if less than ten sites are assigned) on a minimum of four species of animals (two fish and two invertebrates). Deviations from the MQO will result in retraining until performance achieves the MQO. These internal audit sheets will be collated after the survey to evaluate success. In addition, external auditors with taxonomic expertise will observe species identification in the field and record observations. These data will be recorded on a Taxonomy QA/QC data sheet. If there are errors in species identification, the auditor will inform the Lead Taxonomists of the cruise to take action to correct the problem. The Lead Taxonomists must re-train their crew or voucher these problematic species for further identification back at their laboratory.

Navigation

The ability to accurately locate sampling sites is critical to the success of the survey. At minimum, each vessel will be required to have the following instrumentation: A Global Positioning System (GPS); a radar; and a fathometer. A field computer for recording station and sampling information is strongly recommended.

The Boat Captains will be responsible for accurate occupation of the sampling sites and will assist as necessary in maintaining a record of all station occupation and sampling event information. The information required to be recorded for every station occupation and sampling event is described in the Bight '18 Information Management Manual. The Cruise Leaders are required to assure that all field-collected data are complete and accurate. The lead scientist will submit station occupation and sampling event data in electronic form to the Information Management Officer on a weekly basis. These weekly submissions will be reviewed to track the overall sampling progress, identify strata that are at risk of being under-sampled due to unanticipated rates of station abandonment, and to verify that each field team is accurately and completely sampling each station.

B. Laboratory Operations

Several laboratories are participating in Bight '18. Quality assurance and quality control measures are necessary to ensure that the data generated by the participating laboratories are comparable. This section addresses only general laboratory operations. The sections on each indicator (i.e., chemistry, benthic analyses, and toxicity) present specific QA/QC requirements and procedures associated with the processing of specific samples.

The quality assurance measures for Bight '18 include the following:

- The development of MQO's for laboratory generated data,

- The documentation of the participating laboratories general laboratory practices and internal QA/QC procedures

- Mandatory participation in meetings to calibrate laboratory protocols and training to ensure that Bight '18 procedures and QA/QC requirements are understood.

- A presurvey demonstration of laboratory capability

Quality control measures for laboratories participating in Bight '18 include the following:

- An ongoing demonstration of laboratory capability

- Development and implementation of QA/QC procedures for evaluating performance of laboratories relative to MQO's developed for the survey

MQOs for chemical analysis are provided in Chapter V of this document. MQOs for benthic analysis are provided in Chapter VI of this document. MQOs for toxicity are provided in Chapter VII of this document.

Documentation of general laboratory practices

All laboratories providing analytical support for chemical or biological analyses must have the appropriate facilities to store and prepare samples, and appropriate instrumentation and staff to provide data of the required quality within the time period dictated by the survey. Laboratories are expected to conduct operations using good laboratory practices, including:

- A program of scheduled maintenance (annually at minimum) of analytical balances, microscopes, laboratory equipment and instrumentation.

- Routine checking of analytical balances using a set of standard reference weights (ASTM Class 3, NIST Class S-1, or equivalents).

- Checking and recording the composition of fresh calibration standards against the previous lot. Acceptable comparisons are 2% of the previous value.

- Recording all analytical data in bound logbooks in ink.

- Daily monitoring and documenting the temperatures of cold storage areas and freezer units.

- Verifying the efficiency of fume hoods.

- Having a source of reagent water meeting American Society of Testing and Materials (ASTM) Type I specifications (ASTM 1984) available in sufficient quantity to

support analytical operations. The conductivity of the reagent water should not exceed 1 S/cm at 25C.

- Labeling all containers used in the laboratory with date prepared, contents, and initials of the individual who prepared the contents.

- Dating and storing all chemicals safely upon receipt. Chemicals are disposed of properly when the expiration date has passed.

- Using a laboratory information management system to track the location and status of any sample received for analysis.

Laboratories should 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. Laboratories must also be able to provide analytical data and associated QA/QC information in a format and time frame specified by the Laboratory Coordinator or the Information Management Officer.

In addition to the Bight '18 QAP, the following documents and information must be current, and they must be available to all laboratory personnel participating in the survey:

- Laboratory QA Plan: Clearly defined policies and protocols specific to a particular laboratory including personnel responsibilities, laboratory acceptance criteria for release of data, and procedures for determining the acceptability of results.

- Laboratory Standard Operating Procedures (SOPs) - Detailed instructions for performing routine laboratory procedures. In contrast to the Laboratory Methods Manual, SOPs offer step-by-step instructions describing exactly how the method is implemented in the laboratory, specific for the particular equipment or instruments on hand.

- Instrument performance study information - Information on instrument baseline noise, calibration standard response, analytical precision and bias data, detection limits, etc. This information usually is recorded in log books or laboratory notebooks.

- Control charts - Control charts must be developed and maintained throughout the survey for all appropriate analyses and measurements (see section 4.2.5).

Personnel in the laboratories should be well versed in good laboratory practices, including standard safety procedures. It is the responsibility of the laboratory manager and/or supervisor to ensure that safety training is mandatory for all laboratory personnel. The 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 should be readily available to laboratory personnel. Proper procedures for safe storage, handling and disposal of chemicals should be followed at all times; each chemical

should be treated as a potential health hazard and good laboratory practices should be implemented accordingly.

Protocol calibration and training

Each participating laboratory has a representative to the Bight '18 Sediment Quality Planning Committee. This individual serves as the point of contact for the Planning Committee Chair or his/her designee in identifying and resolving issues related to data quality.

To ensure that the samples are analyzed in a consistent manner throughout the duration of the survey, key laboratory personnel should participate in an orientation session conducted during an initial site visit or via communication with the Planning Committee Chair or Technical Committee Chairs. The purpose of the orientation session is to familiarize key laboratory personnel with the QA program requirements and procedures.

Complete and detailed procedures for processing and analysis of samples in the field are provided in the Bight '18 Field Operations Manual. Procedures for benthic analyses are provided in the Macrobenthic (Infaunal) Sample Analysis Laboratory Manual. Procedures for toxicity are provided in the Toxicology Laboratory Manual. Procedures for chemistry analysis are referenced in the appropriate chapters of this document.

Demonstration and documentation of performance

Laboratories are required to demonstrate acceptable performance before analysis of samples can proceed, as described for each indicator in subsequent sections. Initially, a QA assistance and performance audit will be performed by Technical Committee Chairs and Co-Chairs to determine if laboratory effort is in compliance with the procedures outlined in this document and to assist the laboratory where needed.

Specific QA/QC procedures have been developed for Bight '18 to evaluate the quality of data being generated by the participating laboratories relative to the MQOs developed for this survey. It is the responsibility of each participating laboratory to ensure that all the Bight '18 QA/QC procedures outlined in the subsequent chapters are followed.

Quality control of laboratory operations will be evaluated on a continuous basis through the use of internal and external performance evaluations. Technical systems audits by the Technical Committee Chairs and Co-Chairs may be conducted at any time during the survey. In addition, participating laboratories are required to participate in interlaboratory comparison studies detailed in the indicator section of this document (Chemistry, Benthic Analyses, Toxicity).

IV. MEASUREMENTS OF FISH AND INVERTEBRATE ASSEMBLAGES AND FISH PATHOLOGY

A. Overview

This section presents Bight '18 QA/QC protocols and requirements for demersal fish and invertebrate assemblage analyses, from sample collection to final validation of the resultant data. Sample collection methods are documented in the Bight '18 Field Operations Manual. The field crews will generate data on species identification, enumeration, biomass, length measurements (fish only), and gross external pathology.

Field crews will conduct a standard 10-min trawl at selected stations (5-min in bays, harbors, and marinas). The Bight '18 Field Operations Manual contains a list of trawl stations and their locations. Organisms suspected of having pathologies will have a representative fixed in 10% buffered formalin and shipped to SCCWRP. If appropriate, diseased specimens will be examined by a pathologist.

B. Field Operations

Trawling

Field crews must adhere to prescribed sampling protocols because fish and invertebrate assemblage data (species identification, enumeration, biomass, and length) are significantly influenced by the collection methods. Factors influencing the catch are gear type and condition, net deployment, trawl duration, and tow speed. All crews must have standard nets to ensure comparability of gear. The importance of maintaining the trawl duration and speed should be stressed during the presurvey protocol calibration meeting. During sampling, crews must record trawl duration on the Trawl Cover Sheet. The Cruise Leader will be responsible for reviewing all trawl data sheets and the Boat Captain's log daily for investigating and correcting any discrepancies.

The Field Auditors (Field Committee Chair/Co-Chair; or Trawl Committee Chair/Co-chair) will monitor adherence to collection methodology during an audit of field crews. During the audit, the Field Auditors will ensure that the following trawling procedures are executed correctly: 1) the net is rigged properly; 2) the trawl is deployed and retrieved properly; and 3) the trawl data sheets are accurate and complete. The Field Auditors will use a standardized field QA/QC checklist to ensure consistency and comparability of observations among crews. Any discrepancies will be noted and relayed to the Cruise Leader for corrective action.

Acceptability criteria have been established for trawl sample collection. Because some stations have rocky bottoms, the completeness objective for successful trawls will be 90% (Table 2-1). All of the samples collected will be processed, identified, counted, measured (fish only), and weighed.

Species enumeration, length, and biomass measurements

Demersal fish and invertebrate species identification, enumeration, individual lengths (fish only), and biomass will be determined in the field following protocols presented in the Bight '18 Field Operations Manual. The quality of fish and invertebrate identification, enumeration, biomass, and length will be ensured through presurvey training, intercalibration, and in-survey and postsurvey audits.

The Lead Scientist of each organization will be responsible for reviewing standard sampling procedures with his/her field crew and conducting training as needed. The Field Auditors will assess understanding of trawl processing protocol by each new organization during an evaluation.

During the survey, each Cruise Leader will check to make sure that the scales are calibrated at the start of each day, that the appropriate identification aids and processing equipment are on board, and that processing follows the protocol described in the Bight '18 Field Operations Manual. In addition, each Cruise Leader will recount, reweigh, and remeasure 4 species (two fish and two invertebrate, each with at least 10 individuals) at a minium of 10% of assigned trawl sites. These internal QA/QC checks are detailed in the Bight '18 Field Manual. Internal QA/QC checks not meeting MQOs will result in re-training.

External Field Auditors will attempt to conduct at least one in-survey visit during trawl sampling per vessel. The auditors will check to make sure that the scales are calibrated at the start of each day, that the appropriate identification aids and processing equipment are on board, and that processing follows the protocol described in the Bight '18 Field Operations Manual. He or she will also check to see that two fish species and 2 invertebrate species are recounted, reweighed, and remeasured during the visit.

Completeness objectives for fish and invertebrate counts and weights, and fish lengths will be 90% (Table 2-1). Precision objectives for counts, weights, and lengths will be 10% (Table 2-1).

Species identification

The taxonomic identification of demersal fish and invertebrate species will be ensured by a presurvey training and intercalibration, in-survey audits, and postsurvey voucher checks.

Presurvey QA activities include a taxonomic information transfer meeting, an in-field training/intercalibration exercise, and an intercalibration exercise assessing organizational fish and invertebrate identification abilities. The taxonomic information transfer meeting will provide literature lists, taxonomic keys, and discussions on how to identify species expected on the survey. The in-field training/intercalibration exercise will provide training for individuals less familiar with the fauna and intercalibration for those with more experience. It will be conducted on a participating organization's vessel with lead taxonomists from all participating organizations. Trawls will be conducted at different depths and ways to identify the species will

be discussed. The taxonomic assessment exercise will assess the probability that identification errors will be made in the field. Each organization will identify specimens of representative fish and invertebrate species in buckets that will be transferred among participating organizations. The assessment will focus on estimating irretrievable error rates (i.e., incorrect identifications in the field with specimens not returned to the laboratory). Thus correct identifications and "return for further identification" are acceptable but identification errors are not. An organization with greater than 10% errors (fish and invertebrates separately) will be asked to redo the assessment.

During the survey a survey-assigned taxonomist will audit taxonomic identifications in the field at least once per vessel. These taxonomists will observe and audit fish and invertebrate species collected during each visit.

Each organization will also be asked to provide at least one voucher specimen of each species identified in the field. Prior to the survey, each field crew will be given a list of fish and invertebrate species likely to be encountered in the survey to facilitate tracking of specimens collected. The voucher organisms collected in the Bight '18 trawls will be submitted to SCCWRP. The collection will be temporarily housed at SCCWRP until taxanomic validation is finalized by SCAMIT and SCAITE taxonomists. The collection will eventually be archived at the Natural History Museum of Los Angeles County (NHMLAC). All erroneous identifications for an organization will be corrected in the database. To maintain a consistent level of field crew performance, overall completeness and accuracy objectives will be 90% (i.e., <10% taxonomic errors) (Table 2-1).

C. Gross External Pathology

The field crew must examine all demersal fish and invertebrates collected for evidence of external gross pathologies. Fish will be examined for the following anomalies: fin erosion, tumors, external parasites, color anomalies, skeletal deformities, and lesions. Invertebrates will be examined for burn spots and other anomalies. The quality of gross pathology determinations will be ensured principally through information provided prior to the survey, checks conducted in the field by internal and external auditors, and postsurvey voucher checks. Field crews will examine all fish and invertebrates and preserve a representative of any species suspected of having a pathology. Organisms collected for pathological examination must be preserved according to the protocol described in the Bight '18 Field Operations Manual. Specimens will be submitted to SCCWRP with a sample identification label that notes the suspected pathology.

Because of the potential difficulty in proper field identification of pathologies, some definitive examinations may be required and will be conducted by a qualified pathologist. This pathologist will examine the organisms and provide descriptive assessments to SCCWRP.

A voucher collection of preserved specimens or photographs representing every type of pathological condition identified in the Bight '18 fish and invertebrates. Each of these examples may be verified by an external pathologist, if deemed necessary. Similarly, each organization should maintain its own reference collection of pathological fish and invertebrates. These

reference collections will be used to verify the diagnoses made in future years and to ensure intra- and interlaboratory consistency.

To maintain a consistent level of field crew performance, the Bight '18 program has established an overall completeness and accuracy objectives of 90% (i.e., <10% unidentified pathologies or errors) (Table 2-1).

V. ANALYSIS OF CHEMICAL CONTAMINANTS IN SEDIMENTS

A. Overview

There are many aspects to assuring the quality of chemical measurements. This section presents Bight '18 QA/QC protocols and requirements covering a wide range of activities, from sample collection and laboratory analysis, to the final validation of the resultant data. There have been five previous Bight surveys (1994, 1998, 2003, 2008, 2013) and each subsequent Quality Assurance Manual has been built upon the previous manual. Guidance for the original Quality Assurance Manual was based on USEPA SW846 and protocols developed for the EMAP-E Virginian Province, as well as those developed over many years by the National Oceanic and Atmospheric Administration's (NOAA) National Status and Trends (NS&T) Program. The protocols described herein are applicable to low parts-per-billion analyses of marine sediment samples unless, otherwise noted.

The Bight '18 Program will measure a variety of organic and inorganic contaminants in marine sediment samples (Table 5-1). In addition, this survey requires that the participating analytical laboratories demonstrate comparability continuously through strict adherence to common QA/QC procedures, routine analysis of Certified Reference Materials (CRMs), and regular participation in interlaboratory comparison exercises (round-robin analyses). The QA/QC program has adopted a "performance-based" approach to achieving quality assurance of low-level contaminants. Laboratories are not required to use the same analytical methods for each type of analysis. Instead, each laboratory is free to choose the best, or most feasible method available within the constraints of cost and equipment, and provided that the resulting data meets all of the specified QA/QC criteria for accuracy, precision, and sensitivity.

Each laboratory must demonstrate its capability to meet the stated measurement quality objectives (MQOs) for each of the target analytes, in each respective matrix. Initially, each laboratory should establish a method detection limit (MDL) for each target analyte following the MDL protocol cited in 40 CFR Part 136. Laboratories must participate in any available on-going intercalibration exercises, and meet the performance criteria prior to analysis of the survey samples.

The participating laboratories must review their laboratory performance on a continuous basis and make corrections if QA/QC criteria are not met. The comparability in performance among laboratories is continuously evaluated based on analysis of certified reference materials (CRMs), selected intercalibration samples, spiked samples, sample duplicates, and laboratory reagent blanks.

B. Sample Collection, Preservation, and Holding Time

Field personnel must strictly adhere to Bight '18 protocols to insure the collection of representative, uncontaminated sediment chemistry samples. These sample collection protocols

are described in detail in the Field Operations Manual. Briefly, the key aspects of quality control associated with chemistry sample collection are as follows:

- Field personnel must be thoroughly trained in the proper use of sample collection gear, and must be able to distinguish acceptable versus unacceptable sediment grab samples in accordance with pre-established criteria.

- Field personnel must be thoroughly trained to recognize and avoid potential sources of sample contamination (e.g., engine exhaust, winch wires, deck surfaces, ice used for cooling).

- Samplers and utensils that come in direct contact with the sample should be made of non-contaminating materials (high-quality stainless steel only) and should be thoroughly cleaned between sampling stations.

- Sample containers should be of the recommended type (Table 5-2) and must be free of contaminants (i.e., carefully pre-cleaned).

- Conditions for sample collection, preservation and holding times should be followed (Table 5-2).

C. Laboratory Operations

Overview

The Bight '18 program will involve the distribution of sediment and tissue chemistry samples among several different laboratories. Each participating laboratory will analyze samples using existing methodology and report results for the constituents listed in Table 5-1.

The QA/QC requirements presented in the following sections are intended to provide a common foundation for the protocols used by each laboratory. The resultant QA/QC data will facilitate assessment of the comparability of results among the different laboratories and for the different analytical procedures. The QA/QC requirements specified in this plan represent the minimum requirements for any given analytical method. Additional method-specific requirements should always be followed, as long as the minimum requirements presented in this document have been met.

The performance-based Bight '18 QA program for analytical chemistry laboratories is based on an initial demonstration of laboratory capability (e.g., performance evaluation) and an ongoing demonstration of capability. Control limit criteria and recommended frequency of analysis for each QA/QC element or sample type required in the Bight '18 program are summarized in Tables 5-3 to 5-6. The following sections discuss general aspects of the QA/QC elements.

Prior to the analysis of samples, each laboratory should calculate MDLs for each analyte using the acceptable analytical method, establish a Reporting Limit (RL) for each analyte, establish an initial calibration curve for all analytes, and demonstrate acceptable performance on a known or blind accuracy-based material. Following a successful first phase, the laboratory must demonstrate its continued capabilities by participating in an on-going series of interlaboratory comparison exercises, repeated analysis of certified reference materials (CRMs), laboratory control standards, and analysis of laboratory method blanks and spiked samples. These steps are detailed in the following sections.

The results for the various QA/QC samples should be reviewed by laboratory personnel immediately following the analysis of each sample batch. The results should then be used to determine whether any control limit criteria have not been met, and if corrective actions must be taken before any further sample analyses.

To accomplish the objectives of the Bight '18 study, three criteria must be met for any analytical methods used:

- Sufficient sensitivity must be obtained to achieve the required data reporting objectives for any target analytes (Table 5-1). The confidence of these reporting requirements is estimated by assessing the analytical variation resulting from repeated analyses of spiked samples close to these levels (sensitivity criteria).

- Performance of each laboratory must be consistent with that of the other laboratories. Laboratories analyzing the Bight '18 samples must participate in the on-going intercalibration exercises. The results must be within specified limits agreed upon by the chemistry committee.

- Analyses of certified reference materials must yield values within the specified range of the certified values. However, due to the inherent variability in analyses near the method detection limit, control limit criteria for relative accuracy will only apply to analytes having certified values that are >10 times the MDL established by the laboratory (accuracy criteria).

The on-going intercalibration exercises are used to provide an initial check on the performance of the participating laboratories against these criteria. Any laboratory that fails to meet these criteria should repeat analyses of the intercalibration samples before commencing analyses of actual Bight '18 program samples.

Continuous performance evaluation against these criteria can be achieved by analyses of sample duplicates, spiked blanks, matrix spikes, reporting level spikes, laboratory control standards, and certified reference materials. The data quality requirements for the Bight '18 study are summarized in Tables 5-3 to 5-6. Discussion of each component is detailed below.

Initial calibration

Equipment should be calibrated prior to the analysis of each sample batch, after each major equipment disruption, and whenever on-going continuing calibration checks do not meet recommended control limit criteria (Table 5-1).

Organics. The calibration range must be established for each constituent from a minimum of five analytical standards of increasing concentration. The calibration range should be well characterized and must be established prior to the analysis of samples. Only data resulting from quantification within the demonstrated working calibration range may be reported by a laboratory without annotation (i.e., quantification based on extrapolation outside the calibration range is not acceptable). Samples with measured concentrations above the calibration range should be diluted as appropriate, and reanalyzed. For results below the lowest calibration point or reporting limit (RL), samples may be further concentrated, or the results must be "flagged" (annotated) as <RL. The latter is acceptable only if: (1) sample extraction/concentration steps were sufficient to meet the target analyte RL goals of the study, or (2) matrix problems have required sample dilution.

Trace metals. ICP/AES instruments are calibrated with a calibration blank and a minimum of one calibration standard. ICP/MS and the atomic absorption spectrometers including flame atomic absorption (FAA), graphite furnace (GFAA), hydride generation, and cold vapor are calibrated using a minimum of 1 blank and three calibration standards. The linear coefficient of the calibration curve must be at least 0.995 to be acceptable.

Initial Documentation of Method Detection Limits (MDLs)

In the Bight '18 program, the MDL will be used to demonstrate the capability of a laboratory to reach the sensitivity required to measure a specific constituent and demonstrate acceptable precision. The MDL represents a quantitative estimate of low-level response detected at the maximum sensitivity of a method. The Code of Federal Regulations (40 CFR Part 136) gives the following rigorous definition: *"The MDL is the minimum concentration of a substance that can be measured and reported with 99% confidence that the analyte concentration is greater than zero and is determined from analysis of a sample in a given matrix containing the analyte."* The calculated MDL is a function of method precision at low analyte concentrations.

Each laboratory is to follow the procedure specified in 40 CFR Part 136 (Federal Register, Oct. 28, 1984) to calculate nominal MDLs for each target analyte and each analytical method employed. Briefly, at least seven replicates of each representative matrix should be spiked at a concentration between one and five times the estimated detection limit (except for certain trace metals; see below for details), or at RL as a default. The amount of sample (i.e., mass of sediment or tissue) used in calculating the MDL should match, as closely as possible, the amount of sample typically used. The mean and standard deviation of the replicates are used to compute the MDL by multiplying the standard deviation by the Student t value for the 99% confidence interval (for n=7, t=3.143).

Trace metals. The MDLs for aluminum, antimony, arsenic, barium, beryllium, cadmium, chromium, copper, iron, lead, mercury, nickel, selenium, silver, and zinc should be determined on a certified reference material or be calculated from a spiked clean matrix.

Reporting levels

In the Bight '18 program, each laboratory will report results down to at least their established reporting limits. Each laboratory RL must be at or below the concentrations listed in Table 5-1. Results should be flagged if the they are between the RL and the MDL.

Calibration verification

An initial calibration verification standard is analyzed at the beginning of each analysis following the calibration procedure to check the accuracy of the calibration. For all the analytical techniques, one initial calibration verification standard is required from a source different from the source that is used for the calibration standards. The initial calibration verification standard is near the mid-range of the calibration and must be within ±10% of the true value when analyzed. ICP/AES also requires a second initial calibration check standard of a substantially different concentration than the first initial calibration check standard; the second initial calibration check standard must also be within ±10% of the true value when analyzed.

For continuing trace metal measurements, the continuing calibration verification (CCV) verifies that the instrument stays in calibration throughout the analysis. The CCV is prepared in the same acid matrix as the calibration standard. It is analyzed after every ten samples and at the end of the run. The CCV can come from any source that is near the mid-range of the calibration and must be within the ranges specified in Table 5-3.

Calibration blanks (trace metals)

Laboratories need to analyze calibration blanks (pure matrix used to prepare calibration standard solutions) prior to analysis of samples to ensure that the instrument is free of contamination. Concentrations of all target analytes obtained from analysis of the calibration blanks should be below MDLs.

Method blanks

Method blanks (also called procedural blanks) are used to assess laboratory contamination during all stages of sample preparation and analysis. For both organic and inorganic analyses, one laboratory reagent blank should be run in every sample batch. The method blank should be processed through the entire analytical procedure in a manner identical to the samples. Control limits for blanks (Tables 5-4 to 5-6) are based on the laboratory's maximum acceptable method detection limits (trace metals) or reporting levels (trace organics and TOC) as documented prior to the analysis of samples. For trace metals, it is preferable that the level of any analyte in the method blank be below the MDL. Alternatively, the concentration of any target analyte must be less than 5% of the ERL for those constituents that have an ERL established, or less than 5% of the concentration of the analyte in the sample for those analytes without established ERL values. A reagent blank concentration equal to or greater than three times the MDL for one or more of the analytes of interest requires definitive corrective action to identify and eliminate the source(s) of contamination before proceeding with sample analysis.

For trace organics, if the method blank contains any analyte with a measured concentration greater than RL, all samples for that batch should be re-analyzed if the analyte is detected in samples. Concentrations lower than RL should be reported, but not used to correct concentrations in the field samples.

Sample duplicates

Analysis of sample duplicates is used to assess the precision of an analytical method in quantifying target analytes and not required for all methods. Samples collected in the field have the potential to be highly heterogeneous. It is incumbent on the laboratory to make a reasonable effort to homogenize the samples prior to analysis but it is still possible for sample homogeneity to have a large effect on the variability of the results. The relative percent difference (RPD) between the sample and sample duplicate results is calculated as follows:

$$RPD = \frac{(C1 - C2)}{(C1 + C2)/2} \times 100$$

Where: C1 = the larger of the duplicate results for a given analyte, and
C2 = the smaller of the duplicate results for a given analyte.

The data from this process are typically used to establish a statistical range with which the precision of subsequent analyses can be assessed.

Matrix spikes and matrix spike duplicates

A laboratory spiked sample matrix (commonly called a matrix spike or MS) and a laboratory spiked sample matrix duplicate (commonly called a matrix spike duplicate or MSD) will be used both to evaluate the effect of the sample matrix on the recovery of the compound(s) of interest and to provide an estimate of analytical precision. A minimum of one MS/MSD should be analyzed for 10% of samples. The matrix spike solution should contain all the analytes of interest. The final spiked concentration of each analyte in the sample should be between 10 and 100 times the MDL for that analyte, as previously calculated by the laboratory. If the unspiked sample contains more than this amount, then the sample should be spiked with one to five times the preexisting concentration in the sample.

Recovery data for the fortified compounds ultimately are intended to provide a basis for determining the prevalence of matrix effects in the samples analyzed during the survey. However, these data may not reflect the true magnitude of matrix interference with the analyses since recently spiked analytes often do not permeate the sample matrix to the same extent as in field contaminated sediments. This is particularly true for measurements of trace organics in complex matrices. Therefore, it is recommended that recovery data from analyses of MS and MSD samples be used only as an evaluation tool for methods measuring trace organics.

For trace metals, the spike control limits are presented in Table 5-3 for all elements except iron and aluminum due to their high concentrations. If the percent recovery for any

analyte in the MS or MSD is lower than the control limits, the raw data quantitation reports should be reviewed. If the reason for a low percent recovery value is not identified, the instrument response may be checked using a calibration standard. Low matrix spike recoveries may be a result of matrix interference and further instrument response checks may not be warranted, especially 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 should be given in the cover letter accompanying the data package. Corrective actions taken and verification of acceptable instrument response must be included. These corrective actions can include re-analysis of the samples associated with the MS/MSD.

Analysis of the MS/MSD also is useful for assessing laboratory precision. The RPD between the MS and MSD results should be within the control limits (Tables 5-3 to 5-6) for at least one result per batch. If results for any analytes do not meet the control limit criteria, calculations and instruments should be checked. A repeat analysis may be required to confirm the results.

Certified Reference Materials

Certified Reference Materials (CRMs) generally are the most useful QC samples for assessing the accuracy of a given analysis (i.e., closeness of a measurement to the "true" value). CRMs can be used to assess accuracy because they have "certified" concentrations of the analytes of interest, as determined through replicate analyses by a reputable certifying organization using two independent measurement techniques for verification. In addition, the certifying organization may provide "non-certified" or "informational" values for other analytes of interest. Such values are determined using a single measurement technique, which may introduce unrecognized bias. Therefore, non-certified values must be used with caution in evaluating the performance of a laboratory using a method which differs from the one used by the certifying organization. A list of reference materials used for the Bight '18 study is presented in Table 5-7.

A Laboratory Control Material (LCM) may be used in addition to, but not as a replacement for, CRMs. A LCM is similar to a CRM in that it is a homogeneous matrix that closely matches the samples being analyzed. Although the concentrations of the target analytes in these materials are not certified, they can be used to assess the precision (i.e., consistency) of a single laboratory, and to determine the degree of comparability among different laboratories. In practice, LCMs may be preferred for routine (i.e., day to day) analysis because CRMs are relatively expensive. Moreover, as-collected (i.e., wet) LCMs from the study area are more representative of the types of samples that will be delivered to the laboratories during the actual study. However, for the Bight '18 study the specified CRMs must be analyzed with every sample batch to provide a check on analytical performance.

Routine analysis of CRMs and LCMs is a vital aspect of the "performance-based" Bight '18 QA philosophy. For the organic analyses, one CRM (NIST 1944) must be analyzed along with each batch of samples. For the metals analyses, CRM 540 must be analyzed with each batch of samples. However, only one of these CRM (540) will be used for determination data

acceptability criteria. For CRMs, both the certified and non-certified concentrations of the target analytes should be known to the analyst(s) and should be used to provide an immediate check on performance before proceeding with a subsequent sample batch. Performance criteria for both precision and accuracy have been established for analysis of CRMs and LCMs (Tables 5-3 to 5-6).

If the laboratory fails to meet either the precision or accuracy control limit criteria for a given analysis of the CRM, the data for the entire batch of samples is suspect. Calculations and instruments should be checked; the CRM may have to be reanalyzed to confirm the results. If the values are still outside the control limits in the repeat analysis, the laboratory is required to find and eliminate the source(s) of the problem and repeat the analysis of that batch of samples until control limits are met, before continuing with further sample processing. The results of the CRM or LCM analysis should never be used by the laboratory to "correct" the data for a given sample batch.

Surrogate standards

Recovery surrogates are compounds chosen to simulate the analytes of interest in organic analyses. The recovery surrogate represents a reference analyte against which the signal from the analytes of interest is compared directly for the purpose of determining extraction efficiency. Recovery surrogates must be added to each sample, including QA/QC samples, prior to extraction. The reported concentration of each analyte should NOT be adjusted to correct for the recovery of the surrogate standards. The surrogate recovery data should be 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 should be used as recovery surrogates for GC/MS analyses.

Control limit criteria for surrogate recoveries are provided in Tables 5-4 to 5-5. Each laboratory should set its own control limit criteria based on the experience and best professional judgment of the analyst(s). It is the responsibility of the analyst(s) 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).

Internal standards (organics)

Internal standards are added to each sample extract just prior to instrumental analysis to enable optimal quantification, particularly of complex extracts subject to matrix effects or retention time shifts relative to the analysis of standards. Internal standards are essential if the actual recovery of the surrogates added prior to extraction is to be calculated. The internal standards also can be used to detect and correct for problems in the instrument. The elements or compounds used as internal standards must be different from those already used as recovery surrogates. The analyst(s) should monitor internal standard retention times and recoveries to determine if instrument maintenance or repair, or changes in analytical procedures, are indicated. Corrective action should be initiated based on the experience of the analyst(s) and not solely because warning or control limits are exceeded. Instrument problems that may have affected the

data or resulted in the reanalysis of the sample should be documented properly in logbooks and internal data reports and used by the laboratory personnel to take appropriate corrective action.

D. Data Evaluation Procedures

It is the responsibility of the Chemistry Technical Committee Chair/Co-chair or his/her designee to acknowledge initial receipt of the data package(s), verify that the four data evaluation steps (see below) are completed. The analytical laboratory must be notified of any additional information or corrective actions deemed necessary after the data evaluation. Following satisfactory resolution of all "corrective action" issues, the final action is to notify the laboratory in writing that the submitted results have been officially accepted as complete. It may be necessary or desirable for a team of individuals (e.g., members of the Chemistry Technical Committee, Lab Coordinator and/or staff analytical chemists) to assist the Chemistry Technical Committee Chair/Co-Chair in technical evaluation of the submitted data packages. While the Chemistry Technical Committee Chair/Co-Chair has ultimate responsibility for maintaining official contact with the analytical laboratory and verifying that the data evaluation process is completed and to formally document each step in the process as it is completed, it is the responsibility of the Sediment Quality Planning Committee Chair to monitor the process to ensure completeness. Formal documentation should be in the form of a data evaluation tracking form or checklist that is filled in as each step is completed. This checklist should be supplemented with detailed memos to the survey file outlining any concerns with data omissions, analysis problems, or descriptions of questionable data identified by the laboratory.

Evaluation of the data package should begin as soon as possible following its receipt, since delays increase the chance that information may be misplaced or forgotten. In addition, if holding times have been exceeded, options for reanalysis may be limited. The following steps are to be followed and documented in evaluating Bight '18 chemistry data:

- Checking data completeness (verification)
- Assessing data quality (validation)
- Assigning data qualifier codes
- Taking final actions

<u>Checking data completeness</u>

The first part of data evaluation is to verify that all required information has been provided in the data package. For the Bight '18 program, this should include the following steps:

- Survey personnel should verify that the package contains the narrative explanations signed by the laboratory manager, hard copies of all results (including QA/QC results), and accompanying CDs or electronic deliverables.

- The electronic data file(s) should be parsed and entered into the Bight '18 chemistry database to verify that the correct format has been supplied.

- Once the data have been entered into the appropriate Bight '18 database, automated checks should be performed to verify that results have been reported for all expected samples and all analytes.

The Chemistry Technical Committee Chair should contact the laboratory and request any missing information as soon as possible after receipt of the data package. If information was omitted because required analyses were not completed, the laboratory should provide and implement a plan to correct the deficiency. This plan may include submittal of a revised data package and possible reanalysis of samples.

Assessing data quality

Data validation, or the process of assessing data quality, can begin after Bight '18 personnel (supervised by the Chemistry Technical Committee Chair/co-Chair) have determined that the data package is complete. Normally, the first major part of validation involves checking 100% of the data for any possible errors resulting from transcription of tabulated results, misidentification or miscalculations. However, Bight '18 laboratories are expected to submit data that has been tabulated and checked thoroughly for accuracy; the raw data reports needed to perform these checks (e.g., chromatograms, original quantitation reports) are not submitted as part of the data package. The laboratory is required to maintain this raw data in an orderly manner and to have these records available for review by Bight '18 personnel upon request. The first-step validation checks performed by Bight '18 personnel will be limited to the following:

1. A check to verify that all reporting units and numbers of significant figures are correct.

2. A check to verify that all of the laboratory's calculated percent recovery values (for calibration check samples, Laboratory Control Materials, and matrix spikes) and relative percent difference values (for duplicates) are correct.

3. A check to verify that the reported concentrations for each analyte fall within "environmentally-realistic" ranges, determined from previous studies and expert judgment. In addition, past studies indicate that the different compounds in each class of chemicals being measured on Bight '18 (e.g., PAHs, PCBs, DDTs and other chlorinated pesticides) typically occur in the environment in more or less fixed ratios to one another. For example, the DDT breakdown products p,p-DDD and p,p-DDE typically occur at higher concentrations than p,p-DDT in marine sediments in off Southern California. If anomalous departures from expected relative concentrations are found, it may indicate a problem in the measurement or data reduction, which in turn warrants further investigation.

The second major aspect of data validation is to compare the QA/QC data against established criteria for acceptable performance. This will involve the following steps:

1. Results for QA/QC samples should be tabulated, summarized and evaluated. A set of summary tables should be prepared from the database showing the percent recovery values and relative percent difference values (where applicable) for the CRMs, LCMs

and matrix spike/matrix spike duplicate samples. The tables should indicate the percent recovery values for each individual batch of samples, as well as the average, standard deviation, coefficient of variation, and range for all batches combined.

2. Similar summary tables should be prepared for the laboratory reagent blank QA/QC samples.

3. The summary results, particularly those for the CRMs and/or LCMs should be evaluated by comparing them against the QA/QC warning and control limit criteria for accuracy, precision, and blank contamination.

4. Method detection limits reported by the laboratory for each analyte should be tabulated.

There are several possible courses of action to be taken if the reported data are deficient (i.e., warning and/or control limits exceeded) during the assessment of data quality. The laboratory's cover letter (narrative explanation) should be consulted to determine if the problems were satisfactorily addressed. If only warning limits were exceeded, then it is appropriate for the laboratory to report the results. Violation of control limits, however, will result in one of the following courses of action. Either all associated results will be qualified in the database as estimated values (explained in the following section), or the data will be rejected and deleted from the database because the analysis was judged to be out of control (based on the professional judgment of the reviewer).

Assigning data qualifier codes

Data qualifier codes are notations used by laboratories and data reviewers to briefly describe, or qualify, data and the systems producing data. Bight '18 data reviewers will assign data qualifier codes in situations where there are violations of control limit criteria. The most typical situation is when a laboratory fails to meet the accuracy control limit criteria for a particular analyte in a CRM or matrix spike sample. In these situations, the reviewer should verify that the laboratory did meet the control limit criteria for precision. If the lack of accuracy is found to be consistent (i.e., control limit criteria for precision were met), then it is likely that the laboratory experienced a true bias for that particular analyte. In these situations, all reported values for that particular analyte will be qualified with a code that has the following meaning: *"The reported concentration is considered an estimate because control limits for this analyte were exceeded in one or more quality control samples."*

Because some degree of expert judgment and subjectivity typically is necessary to evaluate chemistry QA/QC results and assign data qualifier codes, data validation will be conducted only by qualified personnel. It is the philosophy of the Bight '18 that data which are qualified as estimates because of minor violation of a control limit in a QA/QC sample are still usable for most assessment and reporting purposes. However, it is important to note that all QA/QC data will be readily available in the database along with the results data, so that interested data users can make their own estimation of data quality.

Taking final action

Upon completion of the above steps, a report summarizing the QA review of the data package should be prepared, samples should be properly stored or disposed of, and laboratory data and accompanying explanatory narratives should be archived both in a storage file and in the database. Technical interpretation of the data begins after the QA review has been completed.

Reports documenting the results of the QA review of a data package should summarize all conclusions concerning data acceptability and should note significant quality assurance problems that were found. These reports are useful in providing data users with a written record on data concerns and a documented rationale for why certain data were accepted as estimates or were rejected. The following items should be addressed in the QA report:

1. Summary of overall data quality, including a description of data that were qualified.

2. Brief descriptions of analytical methods and the method(s) used to determine detection limits.

3. Description of data reporting, including any corrections made for transcription or other reporting errors, and description of data completeness relative to objectives stated in the QA Plan.

4. Descriptions of initial and ongoing calibration results, blank contamination, and precision and bias relative to QA plan objectives (including tabulated summary results for CRMs, LCMs and matrix spike/matrix spike duplicates).

The chemistry QA results will be presented in the appropriate Bight '18 technical reports, and will also become a permanent part of the database documentation (i.e., meta data). The QA/QC data collected by Bight '18 will be used not only to assess the accuracy and precision of individual laboratory measurements, but ultimately to assess the comparability of data generated by multiple laboratories.

Table 5-1. Bight '18 Marine Monitoring Program Target Analyte List and Reporting Limits For Sediments

Metals	Reporting Limit (ng/g dry wt)
Aluminum	NA
Antimony	10,000
Arsenic	1,600
Barium	NA
Beryllium	200
Cadmium	90
Chromium	16,000
Copper	7,000
Iron	NA
Lead	9,300
Mercury	30
Nickel	4,200
Selenium	1,000
Silver	200
Zinc	30,000

PBDEs	Reporting Limit (ng/g dry wt)
BDE-17	0.1
BDE-28	0.1
BDE-47	0.1
BDE-49	0.1
BDE-66	0.1
BDE-85	0.1
BDE-99	0.1
BDE-100	0.1
BDE-138	0.1
BDE-153	0.1
BDE-154	0.1
BDE-183	0.1
BDE-190	0.1

Pesticides	Reporting Limit (ng/g dry wt)
4,4'-DDT	0.5
2,4'-DDT	0.5
4,4'-DDD	0.5
2,4'-DDD	0.5
4,4'-DDE	0.5
2,4'-DDE	0.5
4,4'-DDMU	0.5
alpha-Chlordane	0.5
gamma-Chlordane	0.5
cis-nonachlor	0.5
trans-nonachlor	0.5
oxychlordane	0.5

Pyrethroids	Reporting Limit (ng/g dry wt)
Bifenthrin	0.5
Cyfluthrin (total)	0.5
Cypermethrin (total)	0.5
lambda-Cyhalothrin (total)	0.5
cis-Permethrin	0.5
trans-Permethrin	0.5
Deltamethrin	0.5
Esfenvalerate	0.5

Fipronils	Reporting Limit (ng/g dry wt)
Fipronil	0.5
Fipronil Desulfinyl	0.5
Fipronil Sulfide	0.5
Fipronil Sulfone	0.5

Table 5-1 (Cont.). Bight '18 Marine Monitoring Program Target Analyte List and Reporting Limits For Sediments

PCBs	Reporting Limit (ng/g dry wt)
PCB-8	3
PCB-18	3
PCB-28	3
PCB-37	3
PCB-44	3
PCB-49	3
PCB-52	3
PCB-66	3
PCB-70	3
PCB-74	3
PCB-77	3
PCB-81	3
PCB-87	3
PCB-99	3
PCB-101	3
PCB-105	3
PCB-110	3
PCB-114	3
PCB-118	3
PCB-119	3
PCB-123	3
PCB-126	3
PCB-128	3
PCB-138	3
PCB-149	3
PCB-151	3
PCB-153	3
PCB-156	3
PCB-157	3
PCB-158	3
PCB-167	3
PCB-168	3
PCB-169	3
PCB-170	3
PCB-177	3
PCB-180	3
PCB-183	3
PCB-187	3
PCB-189	3
PCB-194	3
PCB-195	3
PCB-201	3
PCB-206	3

PAHs	Reporting Limit (ng/g dry wt)
1,6,7-Trimethylnaphthalene	20
1-Methylnapthalene	20
1-Methylphenanthrene	20
2,6-Dimethylnaphthalene	20
2-Methylnapthalene	20
Acenaphthene	20
Acenaphthylene	20
Anthracene	20
Benz[a]anthracene	80
Benzo[a]pyrene	80
Benzo[b]fluoranthene	80
Benzo[e]pyrene	80
Benzo[g,h,i]perylene	80
Benzo[k]fluoranthene	80
Biphenyl	20
Chrysene	80
Dibenz[a,h]anthracene	80
Fluoranthene	80
Fluorene	20
Indeno[1,2,3-c,d]pyrene	80
Naphthalene	20
Perylene	80
Phenanthrene	20
Pyrene	80

Table 5-2. Summary of chemistry sample collection and holding time conditions. (Maximum holding time for mercury is 6 months.)

Parameter	Container Type	Container Size (mL)	Preservation Requirements	Maximum Holding Time[a]	Data Submittal Time
Sediment Grain Size	plastic	125 (80% full)	cold (4 °C)	6 months	6 months
Sediment Total Organic Carbon	amber glass	250 (80% full)	frozen (-20 °C)	1 year	6 months
Trace Metals	amber glass	250 (80% full)	frozen (-20 °C)	1 year	6 months
Trace Organics	amber glass	2 x 125 (80% full)	frozen (-20 °C)	1 year	6 months

a Holding time starts from sampling date for sediment and from compositing date for tissue.

Table 5-3
Summary of Data Quality Objectives for the
Trace Metal Measurements

MEASUREMENT	FREQUENCY	CONTROL LIMIT
Method Blank	1/batch	<MDL or <5% of the measured concentration in samples
Certified Reference Materials ERA Soil #540 LOT#099	1/batch	Within PT performance acceptance limits of certified values for all 15 analytes
ICP-AES		
Calibration	Initial setup	Minimum 1 blank and one calibration standard
Interference check	1/run	±20% true value
Initial calibration verification (ICV)	2 points/batch	±10% true value
Continuing calibration verification (CCV)	10%	±10% true value
Matrix spike	10%	At least one matrix spike per batch must be within 30% true value. Should all spiked sample recoveries be outside 30% of true value, add a post-digestion spike to the unspiked sample and analyze. If all spike recoveries are outside 30% of true value, note matrix caused poor spike recovery. If all spike recoveries are within 30% of true value, repeat digestion. Spike duplicate results must have an RPD ≤ 20% if MSD is analyzed.
Spiked blank	1/batch	±25% true value
Duplicate sample or matrix spike sample		10% Statistical process control analyses (within 3σ)
ICP-MS		
Calibration	Initial setup	Minimum 1 blank and three calibration standards
Initial calibration verification (ICV)	1 point/batch	±10% true value
Continuing calibration verification (CCV)	10%	±10% true value
Calibration Blank	10%	<MDL. If > MDL, run two more times, the average must be <MDL. If average > MDL, reanalyze.
Matrix spike	10%	At least one matrix spike per batch must be within 30% true value; ≤ 30% RPD for over 10 times MDL. If ≥ 30% RPD and

Table 5-3
Summary of Data Quality Objectives for
Trace Metal Measurements (Cont.)

		post-digestion spike recovery is > 25% note matrix problem. If > 20% RPD and post-digesiton spike recovery is ≤ 25% repeat digestion and analysis
Spiked blank	1/batch	±25% true value
Duplicate sample or matrix spike sample	10%	Within ±30% RPD
<u>Atomic Absorption (AA, GFAA, Hydride Generation, Cold Vapor)</u>		
Calibration	Initial setup	Minimum 1 blank and three calibration standards; linear coefficient ≥ 0.995
Initial calibration verification (ICV)	1/batch	±10% true value
Continuing calibration verification (CCV)	10%	±20% true value
Calibration Blank	10%	<MDL. If > MDL, run two more times, the average must be <MDL. If average > MDL, reanalyze.
Matrix spike	10%	At least one matrix spike per batch must be within 30% true value. If all matrix spike analyses are ≥ 20%, interference test must be conducted
Spiked blank	1/batch	15% true value
Duplicate sample or matrix spike sample	10%	Within ±30% RPD
Interference check	As required	(a) Dilution test: Select typical sample with concentration 25 times the MDL. Dilute sample 5 times. The concentration of the undiluted sample and 5 times the concentration of the diluted sample must be within 10%. If > 10% or all samples are below 10 times the MDL, then proceed to (b). (b) Post-digestion spike: Spike sample to bring concentration to 2 to 5 times the original concentration or 20 times the MDL. The recovery must be within 15%. If not, perform the standard addition procedure described in USEPA SW846

Table 5-4
Summary of Data Quality Objectives for
Polynuclear Aromatic Hydrocarbon[a] Measurements

MEASUREMENT	FREQUENCY	CONTROL LIMIT
Initial calibration		Relative standard deviation (RSD) of the response factor within ± 25% for 80% of the analytes. Or correlation coefficient ($r^2 > 0.990$) for linear and non-linear curves. First or second order curves allowed.
Initial calibration verification	1/batch	Initial calibration verification should be performed immediately following the initial calibration. Relative percent difference (RPD) compared to initial calibration should be less than 30% of all analytes. Second source of calibration standards is used.
Cont. calibration verification	1 set/batch	Continued calibration verification should be performed at the beginning and end of each batch. The one in the middle is optional for long batches or long run times. Relative percent difference (RPD) compared to intial calibration should be less than 20% for 80% of the analytes. The same source of standards to initial calibration is used.
Method Blank	1/batch	< 10 times the MDL for all analytes
Matrix spikes/MS duplicates	1/batch	60-140% recovery of spiked mass for >80% of analytes; RPD <40% for >70% of analytes
Certified reference material	1/batch	Within ± 40% of specified value for ≥ 80% of analytes selected by and agreed to by the Chemistry Technical Subcommittee
Surrogate spikes	1/sample	Laboratories develop their own control limits; all surrogate recovery data should be reported
Internal standards (Optional)	1/sample	Laboratories develop their own

[a]maximum of 20 samples per extraction batch and a reasonable number of sample extracts per instrument batch.

Table 5-5
Summary of the Data Quality Objectives for
PCBs, Chlorinated Pesticides, Pyrethroids, PBDEs, and Fipronil Measurements[a]

MEASUREMENT	FREQUENCY	CONTROL LIMIT
Initial calibration		Relative standard deviation (RSD) of the response factor within ± 25% for 80% of the analytes. Or correlation coefficient ($r^2 > 0.990$) for linear and non-linear curves. First or second order curves allowed.
Initial calibration verification	1/batch	Initial calibration verification should be performed immediately following the initial calibration. Relative percent difference (RPD) compared to initial calibration should be less than 30% of all analytes. Second source of calibration standards is used.
Cont. calibration verification	1 set/batch	Calibration verification should be performed at the beginning and end of each batch. The one in the middle is optional for long batches or long run times. Relative percent difference (RPD) compared to intial calibration should be less than 25% for 80% of the analytes. The same source of standards to initial calibration is used.
Method Blank	1/batch	< 10 times the MDL for all analytes and also < RL
Certified reference material[b]	1/batch	Within ±40% of the certified value for ≥ 70% of the analytes selected by and agreed to by the Chemistry Technical Subcommitee
Matrix spikes/duplicates (MS/MSD)	1 set/batch	60-140% recovery of spiked mass for > 70% of analyte within each class; RPD <40% for >70% of analyte
Surrogate spikes	1/sample	Laboratories develop their own control limits
Internal standards (Optional)	1/sample	Laboratories develop their own

[a] maximum of 20 samples per extraction batch and a reasonable number of sample extracts per instrument batch.
[b] pertains to analytes where certified values are available and documented

Table 5-6
Summary of the Data Quality Objectives for Total Organic Carbon[a] Measurements

MEASUREMENT	FREQUENCY	CONTROL LIMIT
Initial calibration		RSD < 20%
Calibration verification	1/batch	RPD compared to inital calibraiton should be less than 20%
Calibration blank	1/batch	Below MDLs
Method blank	1/batch	< 10 Times the MDL
Sample duplicates	1/batch	RPD < 30%
Certified reference material	1/batch	Within ±20% of certified value

[a] maximum of 20 samples per extraction batch and a reasonable number of sample extracts per instrument batch.

Table 5-7
Certified Reference Materials Recommended by the Chemistry Technical Committee

<u>Calibration solution</u>	
SRM 1491	Aromatic hydrocarbons in hexane/toluene
SRM 1492	Chlorinated pesticides in hexane
SRM 1493	Polychlorinated biphenyl congeners in 2,2,4-trimethylpentane
<u>Environmental matrix (Organics)</u>	
CRM-SRM 1944 (NIST)	PCBs, PAHs, chlorinated hydrocarbons, and PBDEs in marine sediment
FRMs (Field Sediments)	Marine Sediment from Port of Los Angeles for pyrethroids and fipronils; Marine Sediment from Palos Verdes for PCBs, PAHs, CHCs, and PBDEs
CRM-SRM 1946 (NIST)	PCBs, Chlorinated hydrocarbons, and PBDEs in Lake Superior fish tissue
FRMs (Field Tissue)	PCBs, Chlorinated hydrocarbons, and PBDEs in Palos Verdes Shelf fish tissue
<u>Environmental matrix (Trace Metals)</u>	
CRM-ERA 540	Priority Pollutant Soil Certified Standard
FRMs (Field Sediment)	Marine Sediment from Palos Verdes
CRM-DORM-4	Metals in fish protein (NRC Canada)
FRMs (Field Tissue)	PCBs, Chlorinated hydrocarbons, and PBDEs in Palos Verdes Shelf fish tissue
<u>Environmental matrix (total organic carbon)</u>	
SRM 1944 or PACS-2 (NRC Canada)	TOC in marine sediment

VI. ANALYSIS OF CHEMICAL CONTAMINANTS IN FISH

A. Overview

This section presents Bight '18 quality assurance/quality control (QA/QC) protocols and requirements for bioaccumulation assessment covering sample composite and laboratory analysis. There has been one previous fish bioaccumulation survey (2008) and current QA manual has been built upon the previous manual. The protocols described herein are applicable to low parts-per-billion analyses of fish tissue samples unless, otherwise noted.

The Bight '18 program will measure several organic and inorganic contaminants in fish tissue samples (Table 6-1). In addition, this survey requires that the participating laboratories demonstrate comparability through strict adherence to common QA/QC procedures and participation in the intercalibration exercise prior to start of field sample analysis. The QA/QC program has adopted a similar "performance-based" approach to assess chemical contaminants in sediments to achieving quality assurance of low-level contaminants in fish tissue.

B. Tissue composite samples

Upon collection, each fish will be tagged with a unique identification number and measured for total length (longest length from tip of tail fin to tip of nose/mouth), fork length (longest length from fork to tip of nose/mouth), and weight. During dissection, each fish will be sexed and the weight of fillet is recorded.

Dissection and compositing of muscle tissue samples will be performed following U.S. EPA guidance (U.S. EPA 2000). There will be a total of three composite samples per species per zone, 27 zones total. A total of five specimens will be collected per composite sample. Specimens of legal size or larger is preferred, though not required. If more than five specimens are collected, then the middle 75% of the length distribution will be used for the composite. Specimens from this interquartile range will be selected at random for inclusion in each composite.

Fillet muscle tissue with the skin off will be used for analysis. Muscle fillets are recommended by the U.S. EPA (U.S. EPA 2000). Skin removal has been repeatedly used in past California monitoring including the Toxic Substances Monitoring Program, the Coastal Fish Contamination Program, and most southern California NPDES monitoring programs. If some species are too small to be filleted, fish are processed whole but with head, tail, and viscera removed.

C. Contaminants

Tissue samples will be analyzed for PCB congeners, DDTs, PBDEs, mercury, and lipid content (Table 6-1). Reporting levels should be equivalent to or below Office of Environmental Health Hazard Assessment advisory tissue levels (OEHHA ATLs) for comparative purposes.

Quality assurance activities shall focus on accuracy, precision, sensitivity, and comparability (Table 6-2 and 6-3).

Table 6-1. List of constituents and reporting limit in tissue

Analyte	Reporting Limit (ng/wet g)
Total PCB [a]	1
Total DDT [b]	1
PBDEs	0.6
Mercury [c]	20
Lipids	0.1%

a Congeners 8, 18, 28, 37, 44, 49, 52, 66, 70, 74, 77, 81, 87, 99, 101, 105, 110, 114, 118, 119, 123, 126, 128, 138, 149, 151, 153, 156, 157, 158, 167, 168, 169, 170, 177, 180, 183, 187, 189, 194, 195, 201, 206
b o,p'- and p,p'- isomers of DDT, DDE, and DDD, plus p,p'-DDMU
d Can be measured as total Hg

Table 6-2
Summary of Data Quality Objectives for the
Metal (mercury, selenium, and arsenic) Measurements in Tissue

MEASUREMENT	FREQUENCY	CONTROL LIMIT
Method Blank	1/batch	<MDL or < 5% of measured concentration in the sample
Certified Reference Materials DORM-4	1/batch	Within ±30% of certified value for all analytes
Calibration	Initial setup	
Initial calibration verification (ICV)	1 points/batch	±20% true value
Continuing calibration verification (CCV)	1/10 samples	±20% true value
Matrix spike/MS duplicate	1/batch	RPD ≤ 25%, 75-125% recovery
Spiked blank	1/batch	±25% true value
Duplicate sample	1/batch	Within ±25% RPD

Table 6-3
Summary of the Data Quality Objectives for
PCBs, Chlorinated Pesticides, and PBDEs Measurements[a] in Tissue

MEASUREMENT	FREQUENCY	CONTROL LIMIT
SRM 1946	1/batch	Within ±50% of the certified value for ≥ 70% of the analytes selected by and agreed to by the Chemistry Technical Subcommitee
Initial calibration		Relative standard deviation (RSD) of the response factor within ± 25% for 80% of the analytes. Or correlation coefficient ($r^2 > 0.990$) for linear and non-linear curves. First or second order curves allowed.
Initial calibration verification	1/batch	Initial calibration verification should be performed immediately following the initial calibration. Relative percent difference (RPD) compared to initial calibration should be less than 30% of all analytes. Second source of calibration standards is used.
Cont. calibration verification	1 set/batch	Calibration verification should be performed at the beginning and end of each batch. Relative percent difference (RPD) compared to intial calibration should be less than 25% for 80% of the analytes
Method blank	1/batch	< 10 times the MDL for all analytes
Matrix spikes/MS duplicates (MS/MSD)	1 set/batch	50-150% recovery of spiked mass for >70% of analyte within each class; RPD <50% for >70% of analyte
Surrogate spikes	1/sample	Laboratories develop their own control limits
Internal standards (Optional)	1/sample	Laboratories develop their own

[a] maximum of 20 samples per extraction batch and a reasonable number of sample extracts per instrument batch.

VII. MACROBENTHIC COMMUNITY ASSESSMENT

A. Overview

This section provides the Bight '18 QA/QC protocols and requirements for production of biological data about macrobenthic (infaunal) communities, from sample collection through taxonomic analysis. Field and laboratory manuals describing acceptable Bight '18 procedures have been prepared and distributed. Single benthic samples are collected at each station in the survey. Each sample is screened and fixed in the field, returned to one of the participating laboratories, and analyzed for species composition and abundance. The data produced by each laboratory will be aggregated into a single data set and made available for data analysis and interpretation.

B. Field Operations

Sediment samples for infaunal analysis will be collected at each station using a modified 0.1 m^2 Van Veen grab in coastal and estuary sites (Stubbs et al. 1987) or from a 4-inch (inner diameter) sediment core in brackish estuaries. The participation of several different vessels and field sampling teams in Bight '18 requires that uniform procedures be followed in the field to ensure high quality samples and consistent results. Field personnel will be provided with the 2018 Field Operations Manual and instruction on sampling procedures, application of sample acceptance criteria, sample processing, and the collection of required sampling event information. All personnel are expected to understand and properly carry out all steps in the collection, screening, relaxation, and fixation of infaunal samples, and the subsampling and handling of sediment chemistry and toxicity samples.

As described in the 2018 Field Operations Manual, pre-survey field audits conducted by the Field Technical Committee Chair/Co-chair will be used to establish the capability of field sampling teams. During the field audits, the auditor will provide corrective instruction as necessary. Field audits will also be conducted during the Bight '18 program to assure that sampling is done in a uniform manner and field crews record all required information.

A Measurement Quality Objective (MQO) of 90% has been established for completeness of the field collection of benthic samples. This completeness goal was established in an attempt to derive the maximum statistical power of the sampling design. The MQO was not set at 100% in recognition that the randomized selection of sampling sites employed in the Bight '18 program is likely to result in the selection of some sites where sampling will be difficult or impossible. Nevertheless, field crews are expected to strive to meet or exceed this MQO. To this end, site acceptability criteria are provided in the Field Operations Manual.

Sample acceptability criteria have been established in the Field Operations Manual based on sample condition and depth of penetration of the grab. An acceptable grab is characterized by an even surface with minimal disturbance and little or no leakage of overlying water, and a penetration depth of at least 5 cm, if the target depth of 8 cm cannot be achieved. Samples not

meeting these criteria are rejected.

C. Laboratory Operations

The laboratory analysis of infaunal samples for Bight '18 involves three processes: sample treatment and storage, sample sorting, and organism identification and enumeration. Quality assurance in the form of procedures and standardized reporting requirements are provided in the Macrobenthic (Infaunal) Sample Analysis Laboratory Manual for all three processes. For the most challenging process, organism identification, additional quality control and quality assessment steps are included in order to foster comparability among the taxonomic data sets produced by the participating laboratories. The quality assessment steps for taxonomic analysis are discussed separately below.

Sample treatment and storage

In the laboratories, samples will be stored in a secure location that is protected from environmental extremes. Exposure to temperatures above 30°C should be avoided so as to retard evaporative loss. Do not refrigerate samples containing formaldehyde, as paraformaldehyde will be formed at low temperatures. Samples are to be transferred from fixative (borate-buffered 10% formalin) to preservative (70% non-denatured ethanol) after a minimum of 48 hours, but no more than 120 hours of collection. When transferring, thoroughly wash the fixative from the sample, using a 0.5 mm (or smaller) mesh screen to avoid specimen loss. Stored samples must be periodically inspected to assure that the closure is tight and the preservative level adequate. If evaporative loss of preservative is evident, then top off with straight, undiluted (95%) EtOH.

Sample sorting

Sorting must be accurate and complete to assure the value of all the subsequent steps in the sample analysis process. Organisms, are removed from the organic and inorganic residues (grunge) that compose the sample. They are then sorted into broad taxonomic categories for subsequent taxonomic analysis. A standard sorting form is used for tracking the sample. It includes the name of the laboratory and technician responsible, time required for sorting, number of taxa lots and sample containers, percent aliquot re-sorted for QA/QC, and comments.

Re-sorting of samples is employed for quality control of sorting. Each laboratory participating in the survey has an existing re-sorting protocol for this purpose. All share a minimum re-sorting effort of 10% of the material sorted with a minimum acceptable removal efficiency of 95%, the equivalent of an accuracy MQO of 5%.

Sorting QA is to follow the Aliquot method. In this approach, a 10% (minimum) aliquot of every sample processed by a sorter is resorted. Individual labs may elect to re-sort a larger aliquot as they see fit, but the percent aliquot should be reported along with the efficiency results. Percent sorting efficiency is:

$$\frac{\text{Number of Organisms originally sorted X 100}}{\text{\# of Organisms originally sorted + (\# found in resort / aliquot percentile as decimal)}}$$

If sorting efficiency is greater than 95%, no action is required. Sorting efficiencies below 95% will require re-sorting of all samples sorted by that technician and continuous monitoring of that individual until efficiency is improved. Actions taken are to be described on the Quality Control Report section of the Sorting form and the report signed by the responsible supervisor. Organisms found in the resort should be added to the original data sheet. Upon completion of all quality control and assessment steps for the survey, the Benthic Committee Chairperson (or designee) will notify each participating laboratory that the sample grunge is to be discharged to Dr. Susan Kidwell of the University of Chicago.

Taxonomic analysis

The goal of taxonomic analysis for Bight '18 is species level identification of all macrobenthic organisms collected and an accurate count of each species. This task is complicated by the participation of several laboratories in this analysis. The challenge of achieving accurate and consistent results inherent in a large survey of infaunal organisms is compounded by differences in expertise, experience, and opinion of the many taxonomists involved in the analysis.

The Southern California Association of Marine Invertebrate Taxonomists (SCAMIT) is cooperating with Bight '18 to provide an important element of quality assurance for this aspect of the survey. SCAMIT is a regional organization of taxonomists, many of whom are primarily involved in infaunal monitoring studies of wastewater impacts within the Southern California Bight. SCAMIT was founded in 1982 with the goals of promoting the study of marine invertebrate taxonomy and developing a regionally standardized taxonomy for use in environmental monitoring studies. Activities center on cooperation and communication among the region's taxonomists, the sharing of expertise, and on participation in monthly workshops. Results of the workshops and other information are communicated to the membership through a bimonthly newsletter.

SCAMIT's cooperation includes the provision of standards for nomenclature and a mechanism for mutual assistance and exchange of information among the taxonomists involved in Bight '18. The taxonomic nomenclature used in Bight '18 follows *A Taxonomic Listing of Soft Bottom Macro- and Megainvertebrates from Infaunal and Epibenthic Monitoring Programs in the Southern California Bight*, Edition 12 (SCAMIT, 2018). An electronic version of a species list derived from that publication will be made available to the participating organizations submitting data. The full Edition 12 document will be available at the SCAMIT website (www.SCAMIT.org). This list represents a consensus for standard usage of taxon names in Publicly Owned Treatment Works (POTW) monitoring programs in the Bight. In addition, SCAMIT protocols for the use of open nomenclature (SCAMIT 1986) are followed. Taxonomists from the participating laboratories are required to participate in SCAMIT/Bight '18 workshops during the duration of the survey that focus on the taxonomy of groups requiring particular review to promote uniform treatment of problematic taxa. The workshops provide training, pooling of regional resources, and designation of local expert(s) to be called upon for

assistance during sample analysis.

In order to assure that the data produced by the Bight '18 program meet the standards set during the previous regional surveys, it is essential that all participating taxonomists have the expertise and experience necessary to produce data of comparable quality. Qualification criteria have been established to assure that the taxonomists participating in the Bight '18 are capable of meeting that standard. Each organization will provide a list of taxonomists and their specialty areas. Agencies or their contractors employing taxonomists who did not perform analysis of infaunal samples for previous Bight Surveys are required to ensure the Benthic Committee that their taxonomists meet the qualifying criteria prior to participation in the Bight '18 Macrobenthic survey. The two criteria are:

- Candidate taxonomists who will be working under the direct oversight, guidance or mentorship of an experienced taxonomist who analyzed samples in previous Bight surveys are considered to meet the standard for Bight '18.

- Candidate taxonomists who will be not be working under the direct supervision, guidance, and mentorship of an experienced taxonomist who analyzed samples in previous Bight Surveys must complete and pass a qualification exercise prior to acceptance as a taxonomist for Bight '18.

In summary, the exercise is based upon that used as quality control and assessment in the previous Bight Surveys (Montagne & Bergen 1997, Ranasinghe et al. 2003, Ranasinghe et al. 2007, Ranasinghe et al 2012, Gillet et al. 2017). All exercises will be coordinated by the chair/co-chair of the Benthic Committee.

The candidate taxonomist(s) will identify two lots of specimens from samples from each stratum they are expected to process in the upcoming survey. These taxa lots should be part of samples collected during the most recent Bight Program (e.g., Bight '13 samples for new taxonomists participating in Bight '18) or similar survey from the Southern California Bight. Test samples are selected randomly from each stratum from the previous survey and will exclude QC samples. Candidate taxonomists will identify and count all organisms in the samples to the appropriate, targeted taxonomic level for the survey they originated from and those data will be transmitted to the Benthic Committee Chair and Co-chair.

The results of the analysis are compared to those of the original taxonomist and the discrepancies noted. Each discrepancy will be addressed in a reconciliation meeting between the original taxonomist(s) and the candidate taxonomist(s), where possible. This meeting should be facilitated by someone with the appropriate taxonomic background and familiarity with Southern California Bight taxa, but not the original taxonomists for the test samples). Discrepancies found to be the result of error on the part of the candidate taxonomist will be tallied and percent error rates for the number of taxa, organism count, and the accuracy of identification will be calculated using the taxonomic QA/QC. The candidate taxonomist must be able to meet the measurement quality objective (MQO) of 90% for each of the parameters.

Based upon the performance of the candidate taxonomist, the Benthic Committee Chair,

Co-chair and a group of Southern California taxonomists will evaluate the ability of the candidate to participate in the forthcoming Bight Program. Depending upon performance results, a candidate taxonomist may have no restrictions, may be limited to identifying taxa only from certain strata, or may not be asked to participate in the forthcoming survey at all. Opportunity should be provided to the candidate taxonomist to undertake corrective action(s) to improve any deficiencies and a subsequent re-testing, if all parties are willing to do so.

After sample analysis has begun, SCAMIT/Bight '18 workshops will continue at least monthly to address taxonomic problems arising during analysis of the Bight '18 samples. Protocols for the erection and documentation of provisional species names, based largely upon SCAMIT recommendations (SCAMIT 1986), are provided in the 2018 Macrobenthic (Infaunal) Sample Analysis Laboratory Manual. These protocols are intended to assure that adequate documentation is created for any provisional name erected and that the information is quickly and efficiently communicated to all participating taxonomists.

The series of SCAMIT/Bight '18 workshops will culminate in a synoptic data review (SDR) of the data set compiled from all laboratories, and investigation of possible inconsistencies revealed in that process (including examination of voucher specimens or sample lots as needed for resolution). The SDR also draws upon the results of the quality control re-analysis of 10% of the samples analyzed by each laboratory (described below).

While the SCAMIT/Bight '18 workshops are the primary means for exchange of information and assistance, the taxonomists participating in analysis of Bight '18 samples should maintain frequent interaction throughout the process. Timely and frequent communication among the taxonomists analyzing the samples will improve the data produced in the survey. The SCAMIT e-mail list-server (general_topics@discussion.list.scamit.org) will be used to facilitate this communication. All taxonomists involved in the Bight '18 Program will be members of SCAMIT and therefore have access to the list. Messages posted to the list will automatically post to all members, assuring wide and uniform distribution of the contents as well as archiving all discussion for future reference. Appropriate uses of the list server are informing the other members of unusual or newly encountered species, the erection of in-house provisionals, and requests for information or assistance.

The creation and maintenance of voucher collections is an essential element of the QA/QC process. A voucher collection is an invaluable tool during the course of the study, when access to voucher specimens greatly assists the taxonomists in avoiding inconsistent identifications. Upon completion of the study, voucher collections provide other workers the means to determine the identity of species as understood by the original taxonomist. Each participating laboratory must create a voucher collection of all species identified in Bight '18 samples analyzed in that laboratory. Procedures for the creation, maintenance and documentation of the voucher collections are provided in the Macrobenthic (Infaunal) Sample Analysis Laboratory Manual. These collections are separate from the laboratories' existing voucher collections and will be the source of material from which is drawn a common Bight '18 voucher collection upon completion of the survey. These collections provide material for review during SCAMIT/Bight '18 workshops and the synoptic review of the data upon completion of analysis.

The ultimate repository of the Bight '18 voucher collection will be the Natural History Museum of LA County (NHMLAC) invertebrate collection. Upon completion of the survey and analysis of all data, the chair of the benthic committee will notify all taxonomists and facilitate the transmission of all voucher material to the Museum.

Quality assessment of taxonomic analysis

While the quality of taxonomic analysis in Bight '18 relies heavily on the measures described above, quality control and assessment is also provided by the re-identification of a random 10% of the samples processed by each team of taxonomists. Re-identification of all material other than that originally identified will be conducted by Los Angeles County Sanitation District (LACSD) taxonomists. Material originally identified by LACSD taxonomists will be re-identified by taxonomists from the City of San Diego. The taxonomists conducting the re-identification do not have access to the original results. The results of the re-analysis are compared to the original results and a standardized comparative report of discrepancies is prepared. The taxonomists responsible for the original and re-analytical results reconcile the discrepancies. In the process, errors made by the original taxonomists are classified and the number of each type of error recorded. Examples of errors are misidentifications, miscounts, overlooked specimens, or misapplication of counting rules. Errors are discriminated from discrepancies resulting from differences in levels of identification. For example, the discrepancy between a report of *Photis* sp and *Photis lacia* does not represent an error, but rather a decision by one taxonomist to identify the specimen only to genus level. This decision may be based on the taxonomist's judgment that the specimen's condition is too poor for species identification, or may reflect his or her lack of expertise in this particular group of organisms. In the latter case, the difference in treatment provides an indication where assistance from other taxonomists involved in the Bight '18 is needed. Nomenclature differences are also examples of discrepancies that are not classified as error, but that still require corrective action. In addition to assessing analytical accuracy and the survey data quality relative to the MQOs, this process provides information for the SDR performed at the end of the survey as the last step in compiling a final survey data set.

Based upon the results of data quality assessment previous Bight Surveys, an MQO of 90% accuracy has been established for number of taxa, total number of organisms, and identification accuracy. Accuracy is calculated as the percent error in the original results. Percent error will be calculated for three aspects of sample analysis; number of taxa discriminated (richness), total organism count (abundance), and identification accuracy (taxonomic accuracy).

The error rates are calculated as follows:

1. Taxa Discriminated = $1 - \{[\ ABS(\#\ Taxa_{Resolved} - \#\ Taxa_{Original})\ ]/\ \#\ Taxa_{Resolved}\} * 100$
2. Count Accuracy = $1 - \{[ABS(\#\ Individuals_{Resolved} - \#\ Individuals_{Original})]/\ \#\ Individuals_{Resolved}\} * 100$.
3. Identification Accuracy = $[1 - (\#\ Individuals_{Mis\text{-}ID'd} / \#\ Individuals_{Resolved})] * 100$

The first two aspects provide measures of data quality relates to abundance and taxonomic richness of a sample. The third aspect, taxonomic accuracy, is expressed as percent error in identification of individual taxa. It provides a measure of data quality as a representation of community composition. The results of this assessment process will provide a measure of the quality of Bight '18 infaunal data.

D. Information Management

Record keeping and reporting

Each laboratory is responsible for maintaining thorough and complete records through all stages of the sample analysis and QC procedures. Each laboratory will employ its own bench sheet for taxonomic analysis. For the Bight '18 infaunal survey, certain standard forms of notation are employed with the taxonomist's bench sheet that assure that all labs collect the required information in uniform fashion. Standardized forms are used for sorting and all QC checks. Each participating laboratory will retain its taxonomic bench sheets and voucher sheets in electronic and/or paper form. All QC reports are to be submitted to the Benthic Committee Chairperson (or designee) upon completion of sample analysis. To insure against loss of documents, copies of all these documents are to be retained by the individual laboratories.

The laboratory supervisor is responsible for assuring that all steps in the process of analyzing infaunal samples follow Bight '18 procedures and that all QC steps are completed and documented. The supervisor or laboratory manager must implement any specified corrective actions resulting from QC protocols. They are also responsible for preparing their data and documents for transmission to the Information Management Officer in the proper form. All data entry must be subject to the established transcription error checking procedures within the originating laboratory. Analytical results are to be transmitted to the Information Management officer in electronic data files that conform to Bight '18 data submission formats and standards as described in the Information Management Plan. It is the submitting laboratory's responsibility to see that these standards are met.

VIII. SEDIMENT TOXICITY TESTING

A. Overview

This section describes QA/QC procedures that will be used for the assessment of sediment toxicity during the Bight '18 Program. There will be two toxicity tests that will be employed for assessment of the sediment. The toxicity of whole sediment will be analyzed using an amphipod (*Eohaustorius estuarius*) 10-day survival test at both offshore and embayment stations. Toxicity at the sediment-water interface will be evaluated with a 48-hour mussel (*Mytilus galloprovincialis*) embryo development test only at embayment stations.

B. Laboratory Capability

Prior to participating in the Bight'18 program, each testing laboratory must document their ability to conduct the test(s) using the selected methods. This documentation should consist of a record of at least three prior reference toxicant tests that have met test acceptability criteria. The laboratory should have constructed a control chart from these tests which can serve as the documentation.

C. Interlaboratory Comparability

All laboratories conducting toxicity tests must participate in the interlaboratory comparison exercise prior to sample testing, for each method that they will be performing during the survey. The 2018 Toxicity Laboratory Manual describes the details of this intercalibration exercise including test methods and standard operating procedures, acceptable test requirements, test MQOs, and test scoring and evaluation. The intercalibration exercise will include the analysis of field collected sediments and a reference toxicant. The field samples will be distributed blindly to the participating laboratories. Successful completion of this exercise by a laboratory will be evaluated based on two criteria: 1) attainment of test acceptability criteria, and 2) comparability among laboratories.

Comparability of the labs in the intercalibration exercise will be based on four factors: the percentage difference from the mean for each sample, a comparison of the toxicity category for each sample, relative percent difference (RPD) for duplicate samples, and results from the reference toxicant test. Test scoring is a function of the difference between the laboratory mean sample results of the field collected samples delivered blind to the laboratories and the grand mean of all participating laboratories. Toxicity category is a function of the difference between the laboratory mean categorical classification of the blind samples and the category assigned to the grand mean of all laboratories. Toxicity category classification corresponds to the categories defined by the California State Water Resources Control Board's Sediment Quality Objectives. The RPD is a function of laboratory precision based on difference of results from blind duplicates of the field collected samples. Reference toxicant scoring is a function of the difference between the laboratory reference toxicant mean sample results and the grand mean of

reference toxicity for all participating laboratories. These four factors are combined into a single score with cutoffs for acceptable performance based on comparability established by the Toxicology Committee. Laboratories unable to successfully complete the interlaboratory comparison exercise will be asked to examine their test procedures, make suggested changes, and retest the comparison samples. Failure to meet the interlaboratory comparison criteria will result in the addition of a cautionary data qualifier flag to that laboratory's data or exclusion from testing during the monitoring program.

In additon to the intercalibration before sample testing, split samples will be tested by the laboratories during the survey. These samples will be used to verify that the results remain comparable during the course of testing. The results of these additional samples will be for informational purposes only.

D. Sample Handling

Detailed methods for collection of sediment are described in the Field Operations Manual. Surface sediment (top 2 cm from offshore stations and top 5 cm from embayment stations) will be collected from Van Veen grabs and stored in Teflon-lined bags. A second Teflon bag will be provided to split the sediment sample (3L for the mussel test and 2.5-3L for the amphipod test). If one laboratory is conducting both toxicity tests and their field crew is collecting the sample, they may choose to not split the sample into two Teflon bags. A target sediment holding time of no more than two weeks from sample collection has been established in order to minimize the potential alteration of the sediment toxicity due to storage; this time period is not a criterion for judging test acceptability. The goal for the survey is to initiate tests within 10 days of sampling to allow time if retesting is necessary. Tests on samples that are stored from more than two weeks up to four weeks will also be considered valid, but a data qualifier will be attached to the record to indicate that the desired storage time was exceeded. Samples stored for more than four weeks before the start of toxicity testing will be considered unacceptable for testing and the data will not be included in the survey database.

All samples shall be accompanied by chain of custody forms. These forms should include date of sampling and date of receipt.

Prior to the day of test set-up, a small sample of sediment must be tested for pore water ammonia, salinity and pH. This will be achieved by mixing the water into the sediment and removing a sediment sample. The sediment sample for water quality should be centrifuged at 3000 x g for 30 minutes at 15°C to separate sediment particles from the water. This analysis will be used to determine whether the overlying water salinity will need to be adjusted for the amphipod test. Records of this water quality measurement must be submitted with the other toxicity data at the end of the survey.

E. Amphipod Survival Test

An amphipod survival test will be conducted according to USEPA (1994). This test consists of a 10-day exposure of *Eohaustorius estuarius* to sediment under static conditions. Amphipods are placed in glass chambers containing a 2 cm layer of sediment overlain with seawater. The number of surviving amphipods is determined at the end of the test and used to calculate the percentage survival.

Quality of test organisms

Species identification should be verified through consultation with a taxonomist, if necessary. Individuals selected for testing should be visually inspected to confirm that they are the proper size and in good condition (i.e., no external damage). Holding time prior to testing should be no shorter than 2 days and no greater than 10 days.

Accuracy and precision

The accuracy of sediment toxicity tests of field samples cannot be determined since a reference material of known toxicity is not available. A water only reference toxicant test will be run with every batch of test samples in order to document amphipod relative sensitivity and test precision. This test will consist of a 96-hour exposure to five different concentrations of ammonia dissolved in seawater. Ammonia concentrations will be selected to provide an estimate of the concentration at which the mortality of test organisms is 50%, or LC50, and will be verified by analysis of a sample from each of the exposure concentrations. Reference toxicant test results that fall outside of control chart limits (2 standard deviations of the mean) will trigger a review of test procedures and a possible retest of the corresponding sediment samples. A negative control consisting of amphipod home sediment will be analyzed with each test batch.

Test conditions

Water quality of the overlying water will be measured every other day for each sample type. Water quality measurements on the pore water will be measured only at the beginning of the exposure. Temperature will be measured at least daily in a surrogate test chamber. Water quality measurement instruments will be calibrated daily. Deviations in water quality will be noted in the data files.

Interference by ammonia

The presence of high concentrations of ammonia in pore water may be a confounding factor for sediment toxicity tests with *E. estuarius*. Laboratories will be required to measure and record the concentration of un-ionized ammonia in the pore water from each station, after receipt of the sediment sample in the laboratory, but before test set-up and again at test initiation. If the pore water concentration exceeds the limit of 0.8 mg/L un-ionized ammonia for any station within a batch, the laboratory will be required to extend the 4-day ammonia reference toxicant to 10 days. The decision on extending the test will be based on the sample from test initiation. The data will be recorded for both time points. The results of the 10-day ammonia reference test will be compared to the concentrations of ammonia in the test samples to determine if the levels are high enough to account for any observed toxicity in the sediment samples.

Salinity adjustment

Samples will be tested at salinities close to the ambient sample salinity. The salinity of each test sample will be determined by measuring the salinity of the pore water after receipt of the sample in the laboratory, but before test set-up. For each salinity range tested in a batch, an appropriate salinity control for each must be included. Salinity test ranges are as follows:

- If 0-4 ppt in initial pore water, then 2 ± 2 ppt
- If 5-9 ppt in initial pore water, then 7 ± 2 ppt
- If 10-14 ppt in initial pore water, then 12 ± 2 ppt
- If 15-19 ppt in initial pore water, then 17 ± 2 ppt
- If 20-24 ppt in initial pore water, then 22 ± 2 ppt
- If 25-29 ppt in initial pore water, then 27 ± 2 ppt
- If ≥ 30 ppt in initial pore water, then 32 ± 2 ppt

Test acceptability

The *Eohaustorius* test procedure is considered unacceptable if mean survival in the negative control is less than 90%, or if the coefficient of variation among the control replicates is > 11.9%. If a test batch with ≥ 90% control survival has a CV > 11.9%, any sample in that batch with a mean survival ≥ 90% does not need to be retested. Reference toxicant results should also be within two standard deviations of the mean response specific to the laboratory. Water quality parameters (salinity, temperature, pH, and ammonia) should also be within the tolerance range of the test organism, as specified in U.S. EPA (1994) guidance.

F. Sediment-Water Interface Test with Mussel Embryos

A sediment-water interface test using mussel (*Mytilus galloprovincialis*) embryos will be conducted on a subset of stations following a modification of published methods (USEPA 1995, Anderson *et al.* 1996). This modified method consists of adding screened and homogenized sediment to a glass chamber to a depth of 5 cm and overlain with ambient salinity seawater. A screen tube is then rested on the sediment surface to which the fertilized mussel eggs are added. After 48 hr, the screen tube is removed and the developed embryos are rinsed into a vial and preserved. The embryos are then examined microscopically to determine normal development. The number of normally developed embryos is compared to the number of embryos added at the start to determine the endpoint of %normal-alive.

Quality of test organisms

The animals may either be collected by the testing laboratories or purchased commercially. Fresh animals will be acquired as needed throughout the survey. It is recommended that a group of test organisms will be obtained from beginning of the survey and held under conditions conducive to keeping them in spawning condition throughout the survey (USEPA 1995). This group of animals will be used in the event that fresh animals in spawning

condition are not available during the survey. Previous experience has indicated that there can be difficultly purchasing mussels in spawning condition during the summer.

Accuracy and precision

The accuracy of sediment toxicity tests of field samples cannot be determined since a reference material of known toxicity is not available. A water only reference toxicant test will be run with the test samples in order to document amphipod relative sensitivity and test precision. This test will consist of a 48-hour exposure to five different concentrations of ammonia dissolved in seawater. Ammonia concentrations will be verified by analysis of a sample from each of the exposure concentrations. Ammonia concentrations will be selected to provide an estimate of the LC50 and will be verified by analysis of a sample from each of the exposure concentrations. Reference toxicant test results that fall outside of control chart limits (2 standard deviations of the mean) will trigger a review of test procedures and a possible retest of the corresponding sediment samples. A negative control consisting of a test chamber with a screen tube and laboratory seawater, but no sediment will be analyzed with each test batch. This control will test for the presence of any toxicity caused by the exposure system. A second negative control consisting of a shell vial with laboratory seawater will also be analyzed with each test batch to ensure the health of the organisms. The control from the simultaneously tested reference toxicant exposure can serve this purpose.

Test conditions

Water quality of the overlying water will be measured every other day for each sample type. Samples for water quality will be drawn from outside the screen tube or from a surrogate chamber to prevent loss of embryos. Temperature will be measured at least daily in a surrogate test chamber. Water quality measurement instruments will be calibrated daily. Deviations in water quality will be noted in the data files.

Interference by ammonia

The presence of high concentrations of ammonia in the overlying water may be a confounding factor for sediment-water interface toxicity tests with *M. galloprovincialis*. The no effect concentration results from the 48-hr ammonia reference test will be compared to the concentrations of ammonia in the test samples to determine if the levels can account for any observed toxicity in the sediment samples. The Toxicology Committee will review the information and decide on the necessity for data qualifiers.

Test acceptability

The *M. galloprovincialis* sediment-water interface test procedure is considered unacceptable if mean survival in the screen tube control has a %normal-alive value of less than 70%. Reference toxicant results should also be within two standard deviations of the mean response specific to the laboratory. Water quality parameters (salinity, temperature, pH, and ammonia) should also be with the tolerance range of the test organism, as specified in U.S. EPA (1995) guidance.

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