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Southern California Bight
1998 Regional Marine Monitoring Survey
(Bight'98)

Quality Assurance Manual

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1. INTRODUCTION

The Southern California Bight (SCB; Figure I-1), an open embayment in the coast between Point Conception and Cape Colnett (south of Ensenada), Baja California, is an important and unique ecological resource. The SCB is a transitional area that is influenced by currents from cold, temperate ocean waters from the north and warm, tropical waters from the south. In addition, the SCB has a complex topography, with offshore islands, submarine canyons, ridges and basins, that provides a variety of habitats. The mixing of currents and the diverse habitats in the SCB allow for the coexistence of a broad spectrum of species, including more than 500 species of fish and 1,500 species of invertebrates. The SCB is a major migration route, with marine bird and mammal populations ranking among the most diverse in north temperate waters.

The coastal zone of the SCB is a substantial economic resource. Los Angeles/Long Beach Harbor is the largest commercial port in the United States, and San Diego Harbor is home to one of the largest US Naval facilities in the country. More than 100 million people visit southern California beaches and coastal areas annually, bringing an estimated $9B into the economy. Recreational activities include diving, swimming, surfing, and boating, with about 40,000 pleasure boats docked in 13 coastal marinas within the region (NRC 1990). Recreational fishing brings in more than $500M per year.

The SCB is one of the most densely populated coastal regions in the country, which creates stress upon its marine environment. Nearly 20 million people inhabit coastal Southern California, a number which is expected to increase another 20% by 2010 (NRC 1990). Population growth generally results in conversion of open land into non-permeable surfaces. More than 75% of southern Californian bays and estuaries have already been dredged and filled for conversion into harbors and marinas (Horn and Allen 1985). This "hardening of the coast" increases the rate of runoff and can impact water quality through addition of sediment, toxic chemicals, pathogens and nutrients to the ocean. Besides the impacts of land conversion, the SCB is already home to fifteen municipal wastewater treatment facilities, eight power generating stations, 10 industrial treatment facilities, and 18 oil platforms that discharge to the open coast.

Each year, local, state, and federal organizations spend in excess of $10M to monitor the environmental quality of natural resources in the SCB. Most of this monitoring is associated with National Pollutant Discharge Elimination System (NPDES) permits and is intended to assess compliance of waste discharge with the California Ocean Plan and the Federal Clean Water Act, which set water quality standards for effluent and receiving waters. Some of this information has played a significant role in management

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decisions in the SCB.

While these monitoring programs have provided important information, they were designed to evaluate impacts near individual discharges. Today, resource managers are being encouraged to develop management strategies for the entire SCB. To accomplish this task, they need regionally-based information to assess cumulative impacts of contaminant inputs and to evaluate relative risk among different types of stresses. It is difficult to use existing data to evaluate regional issues because the monitoring was designed to be site-specific and is limited to specific geographic areas. The monitoring provides substantial data for some areas, but there is little or no data for the areas in between. Beyond the spatial limitations, data from these programs are not easily merged to examine relative risk. The parameters measured often differ among programs. Even when the same parameters are measured, the methodologies used to collect the data often differ and interlaboratory quality assurance (QA) exercises to assess data comparability are rare.

1994 Pilot Project

To begin addressing these concerns, twelve organizations joined in a cooperative sampling effort in 1994, called the Southern California Bight Pilot Project (SCBPP). The SCBPP involved sampling 261 sites, using common methods, along the continental shelf between Point Conception and the United States/Mexico border. Assessments were made of water quality, sediment contamination, the status of biological resources and species diversity, and the presence of marine debris. The SCBPP provided a much-needed first "snapshot" of the state of the SCB.

Benefits derived from the SCBPP also included the development of new useful technical tools that could only be developed with regional data sets and participation by multiple organizations. For example, the project produced iron-normalization curves for the SCB, allowing distinction between natural and anthropogenic contributions of metals in sediments (Schiff and Weisberg 1998). A Benthic Response Index was developed that integrates complex benthic infaunal data into an easily interpreted form that describes the degree of perturbation at a site (Bergen et al. 1998). The project also produced a series of manuals containing standardized field, laboratory and data management approaches that increased comparability of data among participants, even after the SCBPP was completed.

1998 Survey

The proposed 1998 Southern California Bight Regional Monitoring Project (Bight'98) is a continuation of the successful cooperative regional-scale monitoring begun in southern California in 1994 during the SCBPP. Bight'98 builds upon the previous successes and expands on the 1994 survey by including more participants, sampling more habitats, and measuring more parameters. Sixty two organizations, including international and volunteer organizations, have agreed to participate (Table I-1).

The inclusion of new participants provides several benefits. Cooperative interactions among many organizations with different perspectives and interests, including a combination of regulators and dischargers, ensures that the most appropriate regional questions are being addressed in the study. The additional resources brought by new participants also expands the number of habitats and indicators that will be sampled. Sampling for Bight'98 will include all of the areas sampled in 1994, plus a new focus on nearshore habitats (bays, harbors and beaches) and offshore islands. Bight'98 will also coordinate with a Mexican program to characterize the condition of SCB coastal waters south of the US border. The new indicators that will be measured include shoreline microbiology, biomarkers and new chemical measures.

The Bight'98 Survey is organized into three technical components: 1) Coastal ecology, 2) Shoreline microbiology, and 3) Water quality. This document is the Quality Assurance (QA) Plan for the coastal ecology component of the program. It provides a summary of the methodologies that will be used to collect and process the samples, and the steps that will be taken to ensure data quality. It also outlines the procedures that will be used to quantify whether the project has been successful in meeting its data quality goals. The QA Plan is supported by a work plan, that provides a description of overall project design for the coastal ecology component; a field methods and logistics document that describes the procedures that will be followed by the field crews responsible for sample collection; and an information management manual that details the ways that data will be recorded, transferred among participants and stored.

II. QUALITY ASSURANCE OBJECTIVES

A. Overview

The primary goal of the QA/QC plan is to ensure that the data generated in Bight'98 are comparable among particpants. Many different organizations will be participating in the collection and analysis of samples in Bight'98; encouraging and maintaining consistency in field and laboratory operations and ensuring data comparability will be critical to success of the project.

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 project, 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 monitoring program components (e.g., field operations, water quality, sediment and tissue chemical analyses, benthic analyses, demersal fish analyses) have been established by the Bight'98 Steering Committee.

B. General Approach To Quality Assurance

The QA program for Bight'98 consists of two distinct but related activities: quality assurance and quality control. Quality assurance includes design, planning, and management activities conducted prior to implementation of the project to ensure that the appropriate kinds and quantities of data will be collected. The goals of quality assurance are to ensure that: 1) standard collection, processing, and analysis 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 project to evaluate the effectiveness of the QA activities. QC activities ensure that measurement error and bias are identified, quantified, and accounted for, 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'98 have well established monitoring programs. QA activities for Bight'98 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 pilot project 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 of the overall effectiveness of training have been identified to translate QA activities such as communication and training into QC activities such as performance audits and proficiency examinations. 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 project. Comparability of reporting units and calculations, data base management processes, and interpretative procedures must be ensured if the overall goals of the project are to be realized.

Specific MQOs for precision and accuracy, the most readily quantifiable components of data quality, have been identified for Bight'98 to ensure that the data produced by the many field crews and laboratories involved in the project will be comparable. Accuracy is defined as the difference between the measured value of an indicator and its true or expected 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- 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 the Bight'98 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, sample collection activities, and fish pathology measurements. 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. Suitable reference materials for sediment toxicity are not available.

An MQO for completeness was also defined for Bight'98. Completeness is a measure of the proportion of the expected, valid data (i.e., data not associated with some criterion of potential unacceptability) that is actually collected during a measurement process. The MQO for completeness is 90% for each measurement process. The sampling design for the project 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.

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 the Bight'98 will be achieved by:

- Developing a common field manual,

· Documenting the comparability of laboratory methods that are 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'98 QA Officer or designee

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 Field Operations Manual (1998) 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 Chief Scientist in the field operations manual).

A single laboratory manual was not developed for the project since 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 the Quality Assurance Project Plan (QAPP) which identifies performance based standards and the appropriate level of QA/QC. Procedures for benthic analyses appropriate to the Bight'98 Project are detailed in the Infaunal Sample Analyses Laboratory Procedures Manual (Appendix A).

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'98 survey will be conducted cooperatively by a number of organizations (including one or more contractors) which routinely monitor the marine environment according to their own protocol. It is important to the success of the Bight98 study that comparable data are collected by each organization.

Quality Assurance activities for field collection include:

· The development of the field operations manual which details the procedures to be used in the Bight'98 survey,

· A presurvey protocol methods calibration meetings to ensure that project participants understand the requirements outlined in the field manual, and

· A presurvey audit of new participants to demonstrate understanding and capability.

Quality Control measures for the field collection effort include:

· Specific QC requirements outlined in the QAPP, which will be the responsibility of the lead scientist of each vessel, and

· Field audits of each vessel during the sampling period.

Field operations manual

Standard field procedures are documented in the Bight'98 Field Operations Manual (1998). 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 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.

Chief 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 chief scientist of each organization is responsible for distributing the Bight'98 Field Operations Manual to all field personnel and ensuring that their staff understands and uses 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'98 will provide personnel who have extensive field experience, but not necessarily with the standard methods selected for this project. Instruction for this project, therefore, will focus on ensuring consistency in data collection among all field personnel.

Chief 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 the Bight'98 will be discussed at this meeting as well as the responsibilities of the chief scientist and boat captains during the Bight'98 survey. Each participating organization will be provided with a Workplan, Field Operations Manual and QA/QC Document for Bight'98 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 project and QA/QC activities occurring on the boat during the survey will also be discussed.

The Chief 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 Chief 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 Chief Scientist's responsibility to train their field crews, as needed, on operations to be performed. Personnel that cannot not perform an operation as required by the project will not participate in that operation.

Field audits

Field sampling capability will be established by means of field audits conducted by the Field QA Specialist prior to sampling for the Bight'98 study. These presurvey 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. The priority for conducting field audits prior to the sampling period will begin with organizations that did not participate in the 1994 Regional Survey or who have a significant number of staff members that did not participate in the 1994 Regional Survey. If resources and time are still available after all of these organizations have been audited, the organizations that participated in the Bight'98 will be subject to a field audit to confirm the capabilities that existed and were documented for the Bight'98.

A field QA/QC checklist, developed to provide comparability and consistency in this process, will be used to record the pre-cruise audit data. The Field QA Specialist will provide additional instruction when discrepancies are noted during the presurvey field QA audit. The Chief Scientist will also be notified of the audit results so that any problems can be corrected prior to sampling.

Ongoing quality control during the sample period will be established through field audits. Each vessel will be visited at least once during the survey. In addition to the information contained on the QA/QC checklist. Each vessel will also be audited by a preassigned taxonomist, who will observe species identification in the field. This data will be recorded on a Taxonomy QA/QC data sheet. If there are errors in species identification, the taxonomist will inform the Chief Scientist of the cruise to take action to correct the problem. Field personnel will be instructed regarding the appropriate identifications.

Navigation

The ability to accurately locate sampling sites is critical to the success of the survey. A minimum of a Loran-C, a radar, and a fathometer will be required for this project.

The boat captain will be responsible for calibrating the navigation equipment and maintaining a navigation log for all sampling stations. The log includes latitude and longitude coordinates, GPS coordinates (if GPS is available), depth measurements for each station, and daily calibration information. The Chief Scientist will responsible for reviewing the log as part of the daily QC check of all completed data forms. The Field QA Specialist will check basic navigation and the completeness and accuracy of the navigation logs. As position data are received at the Field Operations Center at SCCWRP, automatic-range checks will be performed on station latitude and longitude coordinates. The reported station location will be compared to the expected coordinates and flagged for further investigation if the positions differ by more than 300 m. If discrepancies are found, original data sheets will be reviewed and the Chief Scientist will be contacted to provide an explanation.

B. Laboratory Operations

Several laboratories are participating in Bight'98. Quality assurance and quality control measures are necessary to ensure that the data generated by the participating laboratories is comparable. This section addresses only general laboratory operations. The sections on each indicator (i.e., chemistry, benthic

analyses, toxicity, and biomarkers) present specific QA/QC requirements and procedures associated with the processing of specific samples.

The quality assurance measures for Bight'98 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'98 procedures and QA/QC requirements are understood.

· Apresurvey demonstration of laboratory capability

Quality control measures for laboratories participating in Bight'98 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 project

MQOs for chemical analysis are provided in Chapter 6 of this document. MQOs for benthic analysis are provide in Chapter 7 of this document. MQOs for toxicity are provided in Chapter 8 of this document. MQOs for biomarker analyses are provided in Chapter 9.

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 project. Laboratories are expected to conduct operations using good laboratory practices, including:

· A program of scheduled maintenance of analytical balances, microscopes, laboratory equipment and instrumentation.

· Routine checking of analytical balances using a set of standard reference weights (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. Chemical are disposed of properly when the expiration date has expired.

· 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'98 QAPP, the following documents and information must be current, and they must be available to all laboratory personnel participating in the project:

· 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 logbooks or laboratory notebooks.

· Control charts - Control charts must be developed and maintained throughout the project 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'98 Steering Committee. This individual serves as the point of contact for the QA Officer or his 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 project, key laboratory personnel should participate in an orientation session conducted during an initial site visit or via communication with the QA Officer or his designee. 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'98 Field Operations Manual (1998). Procedures for benthic analyses are provided in the Infaunal Sample Analysis Laboratory Procedure (SCCWRP, 1998) which is attached as an appendix to this document. Procedures for chemistry, toxicity and biomarker analyses are referenced in the appropriate chapters.

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 QA Officer or his designee to determine if each 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'98 to evaluate the quality of data being generated by the participating laboratories relative to the MQOs developed for this project. It is the responsibility of each participating laboratory to ensure that all the Bight'98 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 QA Officer or his designee may be conducted may be conducted at any time during the project. In addition, participating laboratories are required to participate in interlaboratory comparison studies detailed in the indicator section of this document (Chemistry, Benthic Analyses, Toxicity, Biomarkers).

IV. MEASUREMENTS OF FISH AND INVERTEBRATE ASSEMBLAGES AND FISH PATHOLOGY

A. Overview

This section presents Bight'98 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'98 Field Operations Manual (1998). 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. The Bight'98 Field Operations Manual contains a list of trawl stations and their locations. The contents of the net will be examined and fish and invertebrates will be identified to species, measured for length (fish only), counted, weighed, and examined for evidence of gross external pathologies. Organisms suspected of having pathologies will be

fixed in 10% buffered formalin and shipped to SCCWRP. 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, 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 towing speed and trawl duration on the Trawl Cover Sheet. The Chief Scientist will be responsible for reviewing all trawl data sheets and the boat captain's log daily for investigating and correcting any discrepancies.

The field QA/QC auditor will monitor adherence to collection methodology during a presurvey audit of each field crew. During the audit, the field QA/QC auditor 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 QA/QC auditor will use a standardized field QA/QC checklist to ensure consistency and comparability of observations between crews. Any discrepancies will be noted and corrected during the audit.

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 4-1). All of the samples collected (except for repeat trawls for bioaccumulation samples) 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'98 Field Operations Manual (1998). The quality of fish and invertebrate identification, enumeration, biomass, and length measurements will be ensured through presurvey training, audits, and intercalibration, and in-survey and postsurvey audits.

The chief scientist of each organization will be responsible for reviewing standard sampling procedures with his/her field crew and conducting training as needed. The field QA/QC auditor will assess understanding of trawl processing protocol by each new organization during a presurvey evaluation.

During the survey, each chief scientist 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'98 Field Operations Manual. In addition, each chief scientist will recount, reweigh, and remeasure 2 fish species (with at least 10 individuals) each day during the survey to provide data for precision estimates relative to the target measurement quality objectives (MQOs).

The field QA/QC auditor will conduct at least one in-survey visit during trawl sampling per vessel during the field survey. The auditor 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'98 Field Operations Manual. He or she will also check to see that 2 fish 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 4-1). Precision objectives for counts, weights, and lengths will be 10% (Table 4-1).

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 an organization 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 passed to each organization. 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 5% errors (fish and invertebrates combined) will be asked to redo the assessment.

During the survey a project-assigned taxonomist will audit taxonomic identifications in the field in at least one visit per vessel. These taxonomists will audit at least 25% of fish and invertebrate species collected per day during a visit.

Each organization will also be asked to provide at least 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. A voucher collection of organisms collected in the Bight'98 trawls will be developed during the survey. The collection will be housed at SCCWRP along with the Bight'98 voucher collection; both will eventually be archived in a museum. In addition, each organization will be encouraged to develop its own voucher collection. Extra voucher specimens will be saved to provide a reference collection to assist training in subsequent years.

Following the survey, the original identification of voucher specimens will be checked by lead project fish and invertebrate taxonomists. 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 95% (i.e., <5% unidentified species or errors) (Table 4-1).

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 during the survey by the project-assigned taxonomists, and postsurvey voucher checks. Field crews will examine all fish and invertebrates and preserve any suspected of having a pathology. Organisms collected for pathological examination must be preserved according to the protocol described in the Bight'98 Field Operations Manual. Specimens will be returned to the laboratory with a sample identification label that notes the suspected pathology.

Because of the potential difficulty in proper field identification of pathologies, all definitive examinations will be conducted by a qualified pathologist. This pathologist will examine the organisms and provide the project-assigned taxonomist with the results.

A voucher collection of preserved specimens or photographs representing every type of pathological condition identified in the Bight'98 fish and invertebrates. Each of these examples should be verified by an external pathologist experienced with the species in question. Similarly, each organization should maintain its own reference collection of pathological fish and invertebrates. These reference collections will be used to very the diagnoses made in future years to ensure intra- and interlaboratory consistency. A reference collection will also be developed for future training purposes.

To maintain a consistent level of field crew performance, the Bight'98 program has established an overall completeness and accuracy objectives of 95% (i.e., <5% unidentified pathologies or errors) (Table 4-1).

V. ANALYSIS OF CHEMICAL CONTAMINANTS IN SEDIMENTS AND TISSUES

A. Overview

Quality assurance of chemical measurements has many diverse aspects. This section presents Bight'98 QA/QC protocols and requirements covering a wide range of activities, from sample collection and laboratory analysis to final validation of the resultant data. Much of the guidance for this section is based on USEPA SW846 and protocols developed for the EMAP-E Virginian Province, as well as those developed over many years on the National Oceanic and Atmospheric Administration's (NOAA) National Status and Trends (NS&T) Program. This guidance is applicable to low parts per billion analyses of both marine sediment and fish samples unless, otherwise noted.

The Bight'98 project will measure a variety of organic and inorganic contaminants in marine sediment and whole fish samples . (Table 5-1) Bight'98 requires that laboratories demonstrate comparability continuously through strict adherence to common QA/QC procedures, routine analysis of Certified Reference Materials, and regular participation in interlaboratory comparison exercises (round-robins). The QA/QC program adopts a "performance-based" approach to achieving quality assurance of low-level contaminants. Laboratories are not required to use a single, standard analytical method for each type of analysis. They are free to choose the best, or most feasible method within the constraints of cost and equipment, provided that the resulting data is of known and documented quality.

Each laboratory must demonstrate its capability to meet the reporting objectives for each of the target analytes. 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 an available on-going intercalibration exercise, and meet the performance criteria prior to analysis of the survey samples.

The participating laboratories must continue to review their laboratory performance 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), intercalibration samples, spiked samples, sample duplicates, and laboratory reagent blanks.

B. Sample Collection, Preservation, and Holding Time

Field personnel must strictly adhere to Bight'98 protocols to ensure the collection of representative, uncontaminated sediment and fish tissue 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 or fish trawls 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 which come in direct contact with the sample should be made of non-contaminating materials (e.g., glass, high-quality stainless steel and/or Teflon®) 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); and

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

C. Laboratory Operations Overview

Bight'98 will involve the distribution of chemistry samples to several different laboratories. Each participating laboratory will analyze samples using existing methodology and report results only for the constituents that match those listed in Table 5-1.

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

The performance-based Bight'98 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'98 program are summarized in Tables 5-3 through 5-6. The following sections discuss general aspects of the QA/QC elements.

Prior to the analysis of samples, each laboratory should calculate method detection limits 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. These results should then be used to determine when control limit criteria have not been met and corrective actions must be taken, before processing a subsequent sample batch.

To accomplish the objectives of the Bight'98 study, three criteria must be met for any analytical methods used:

· Sufficient sensitivity must be obtained to achieve the required reporting objectives for any target analytes (Table 5-7). 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 any laboratory must be consistent with that of other laboratories. Laboratories analyzing the Bight'98 samples must participate in the on-going intercalibration exercises. The acceptable performance for a specific laboratory is that concentrations of any measurable constituents obtained by this laboratory must be within 3 standard deviations of the average measured values by all the laboratories (precision criteria) or the limits set by the intercalibration groups (refer to Table 5-9).

· Analyses of certified reference materials must yield values within the specified range of the certified values (Tables 5-3 to 5-6). Due to the inherent variability in analyses near the method detection limit, control limit criteria for relative accuracy only apply to analytes having CRM true 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 analyzing the survey 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'98 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 (Tables 5-3 to 5-6).

Organics. 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 which results from quantification within the demonstrated working calibration range may be reported by the laboratory without annotation (i.e., quantification based on extrapolation outside the calibration range is not acceptable). Samples with measured concentrations outside the calibration range should be diluted as appropriate, and reanalyzed. For results below the lowest calibration point (RL), samples may be further concentrated, or results "flagged" (annotated) as MDL. 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 and ICP-MS instruments are calibrated with a calibration blank and a minimum of one calibration standard. 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

In Bight'98, 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 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. Laboratories must submit documented MDLs for each analytical method (summarized in a spreadsheet) to the Chairperson of the Chemistry Technical Committee prior to analysis of field samples. MDLs should be determined in both fish tissue and sediment, using "clean" sample matrices in order to minimize the interference of sample analytes on estimation of detection limits of target analytes.

Each laboratory is to follow the procedure specified in 40 CFR Part 136 (Federal Register, Oct. 28, 1984) to calculate nominal MDLs for 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 the reporting level (RL, see below) as a default. The amount of sample (i.e., weight 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 could be determined on a certified reference material or be calculated from a spiked clean matrix.

Reporting levels

In the Bight'98 program, RLs are used to report concentrations of target analytes (Table 5-7). As defined by the Bight'98 Chemistry Technical Committee, RLs are identical to the lowest concentration of any specific calibration range. The RL is therefore the lowest quantitative value which can be justified and reported in terms of calibration reliability. Values below the RL, but above the nominal MDL are reported when detected, but must be flagged or annotated using the footnote supplied for data reporting. Laboratories must

demonstrate their capability to achieve the required RLs by matching the lowest level of calibration standards to the reporting level and meeting the control limit criteria for the initial calibration. Table 5-7 shows the Bight 98 Reporting Levels.

Trace Metals. The maximum acceptable MDLs are set at one-fifth of the ERL. For the purpose of this study, reporting levels (RLs) are used interchangeably with maximum acceptable MDLs.

Trace Organics. The RLs for the PAH in sediment are set based on the combination of the ERL and historical data. The RLs for the chlorinated pesticide in sediments are also based on the ERL values. Sediment RLs for PCB congeners are based on total PCB ERLs. Fish tissue RLs for chlorinated hydrocarbon analytes are based on tissue residue guidelines for protection of wildlife, as recommended by Environment Canada. RLs for biomarker compounds (LABs) are based on the sensitivity of the method and what can be expected of the LAB concentrations in the SCB.

Performance criteria at the RL

The initial performance demonstration of precision near the RL can be derived from the MDL determination or separate analysis. The standard deviation of at least 7 replicates of clean matrix spiked at or near the RL should be < 0.35 times the RL. In order for test performance to be estimated for ongoing organics analyses, each sample batch should include at least one spike at or near the RL (see Reporting level spikes (organics).

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 three analytical techniques, one initial calibration verification standard is made from a source different from the source that is used for the calibration standards. The initial calibration is near the mid-range of the calibration and must be within ±10% of the true value when analyzed. ICP-AES and ICP-MS also require a second initial calibration standard of a substantially different concentration than the first initial calibration standard; the second initial calibration 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.

For trace organics measurements using full scan GC/MS, instrument tuning needs to be performed by analyzing 50 ng of decafluorotriphenylphosphine (DFTPP) prior to use of the instrument. The fragmentation profiles from this analysis have to be within the EPA-recommended criteria (see USEPA SW-846). The initial instrument calibration performed to establish calibration ranges for specific analytes is checked through the analysis of calibration verification standards (i.e., calibration standard solutions) prior to analysis of each batch of samples. Calibration verification standard solutions used for the calibration checks should contain all the analytes of interest at concentrations at or near the mid-level of a multi-point calibration range.

If the control limit for analysis of the calibration verification standard is not met (Tables 5-3 to 5-6), the analyst(s) should identify and eliminate the source(s) causing the failure and perform another calibration verification. If problems persist, preventive maintenance or corrective actions must be performed. A

calibration verification standard is injected. The results should be assessed using the calibration verification criteria (Tables 5-3 to 5-6). If the calibration verification criteria are not met, a new initial calibration must be performed. No sample analysis should begin until a satisfactory calibration verification is achieved.

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 5-3 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, the level of any analyte in the method blank must be below MDL or less than 5% of the level of the analyte in the samples. 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 should be re-analyzed within the batch if that 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. The relative percent difference (RPD) between the sample and sample duplicate results is calculated as follows:

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

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 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 (unless the unspiked sample contains more than this amount in which case 1 to 5 times the

pre-existing 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 project. However, these data may not truly reflect the magnitude of matrix interference with the analyses since spiking materials may not enter the complex matrix. 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 are only used 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 other than 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 discovered, 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.

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 and 5-9) 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.

Reporting level spikes (organics)

Since a large number of samples are expected to contain organic analytes with concentrations near RLs, it is important to estimate the confidence of the measurements near these levels. For each batch of samples analyzed, a relatively clean matrix (clean sand or Orange Roughy) is spiked with a standard solution containing all analytes of interest at levels approximately 20% above RLs. This sample is processed and analyzed along with other field samples. Recovery data from all participating laboratories will be gathered and analyzed to yield a confidence range for each method measuring low-level target analytes.

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'98 study is presented in Table 5-8.

As an alternative, laboratory control material (LCM) may be used in replacing a CRM. A LCM is similar to a CRM in that it is a homogeneous matrix that closely matches the samples being analyzed. For the Bight'98 study, two sediment materials were collected from Santa Monica Bay and the Palos Verdes Shelf and repeatedly analyzed by a number of laboratories. Although concentrations of the analytes of interest in these materials are not certified, they can be used to assess the precision (i.e., consistency) of a single

laboratory, as well as 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. However, CRMs still must be analyzed at regular intervals (e.g., monthly or quarterly) to provide a check on accuracy.

Routine analysis of CRMs or, when available, LCMs, is a vital aspect of the "performance-based" Bight'98 QA philosophy. At least one CRM or LCM must be analyzed along with each batch of samples (Tables 5-3 to 5-6 and 5-9). 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 or LCMs (Tables 5-3 to 5-6 and 5-9). If the laboratory fails to meet either the precision or accuracy control limit criteria for a given analysis of the CRM or LCM, the data for the entire batch of samples is suspect.

Calculations and instruments should be checked; the CRM or LCM may have to be reanalyzed (i.e., reinjected) 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 quantification. 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 therefore should be carefully monitored; each laboratory must report the percent recovery of the surrogate(s) along with the target analyte data for each sample. If possible, isotopically-labeled analogs of the analytes 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 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 Project Manager or his designee to acknowledge initial receipt of the data package(s), verify that the four data evaluation steps (see below) are completed, notify the analytical laboratory of any additional information or corrective actions deemed necessary after the data evaluation, and, following satisfactory resolution of all "corrective action" issues, take final action by notifying 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., the QA Coordinator, Lab Coordinator and/or staff analytical chemists) to assist the Project Manager in technical evaluation of the submitted data packages. While the Project Manager has ultimate responsibility for maintaining official contact with the analytical laboratory and verifying that the data evaluation process is completed, it is the responsibility of the QA Coordinator to closely monitor and formally document each step in the process as it is completed. This 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 project 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 and (if holding times have been exceeded) can sometimes limit options for reanalysis. The following steps are to be followed and documented in evaluating Bight'98 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. In Bight'98, this should include the following steps:

- Project 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 computer diskettes.

- The electronic data file(s) should be parsed and entered into the Bight'98 database to verify that the correct format has been supplied.

- Once the data have been entered into the Bight'98 database, automated checks should be run to verify that results have been reported for all expected samples and all analytes.

The Project Manager 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.

<u>Assessing data quality</u>

Data validation, or the process of assessing data quality, can begin after Bight'98 personnel 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'98 laboratories are expected to submit data that has been tabulated and checked 100% 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'98 personnel upon request. The first-step validation checks performed by Bight'98 personnel will be limited to the following:

· A check to verify that all reporting units and numbers of significant figures are correct;

· 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;

· 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'98 (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 ratios are found, it may indicate a problem in the measurement or data reduction, which requires further investigation.

The second major aspect of data validation is to compare the QA/QC data against established criteria for acceptable performance (specified earlier in this plan). This will involve the following steps:

· 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 laboratory control material(s) 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.

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

· The summary results, particularly those for the laboratory control material (i.e., Certified Reference Material), should be evaluated by comparing them against the QA/QC warning and control limit criteria for accuracy, precision, and blank contamination specified in Table 5-3.

· 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'98 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 Certified Reference Material or matrix spike sample. In these situations, the QA 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'98 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:

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

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

· 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 Project Plan.

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

The chemistry QA results will be presented in the Bight'98 Annual Quality Assurance Report and will also become a permanent part of the database documentation (i.e., meta data). The QA/QC data collected by the Bight'98 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.

VI. MACROBENTHIC COMMUNITY ASSESSMENT

A. Overview

This section provides the Bight'98 QA/QC protocols and requirements for the production of biological data, from sample collection through taxonomic analysis, that will be used in the assessment of benthic infaunal communities. A laboratory procedures manual has been prepared which describes the acceptable procedures for use in Bight'98 (Appendix A). 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, abundance, and major taxa biomass. The data produced by each laboratory will be aggregated into a single data set and made available for data analysis and interpretation.

B. Sample Collection, Preservation, and Holding

Sediment samples for benthic infaunal analysis will be collected at each station using a SCCWRP-modified 0.1 m2 Van Veen grab (Stubbs et al. 1987). The participation of several different vessels and field sampling teams in Bight'98 requires that uniform procedures be followed in the field to ensure high quality samples and consistent results. Field personnel will be provided with the Field Operations Manual (1998) and instruction on sampling procedures, application of sample acceptance criteria, sample processing, and use of field data forms. 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.

Capability will be established by means of field audits by the Field QA Specialist prior to sampling for the survey. During the field audits, the QA Specialist will provide corrective instruction as necessary. The Field QA Specialist (or designee) will also conduct subsequent audits on benthic sampling procedures during the Bight'98 survey to assure that sampling is conducted in a uniform manner and all required information is recorded by all field crews.

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'98 survey is likely to result in the selection of some sites where Van Veen grab 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 and relocation procedures are provided in Section VII, and sample acceptability criteria and minimum sampling effort are stipulated in Section 9 of the Field Operations Manual. As many as nine attempts at a site must be made to meet the site acceptability criteria. Once a site has been accepted, a minimum sampling effort of four attempts to collect an acceptable sample is required at each station.

Sample acceptability criteria have been established in the Field Operations Manual (1998) 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.

In the laboratories, samples will be stored in a safe and secure manner protected from environmental extremes. Exposure to temperatures above 30C should be avoided so as to retard evaporative loss. Do not refrigerate samples containing formaldehyde as paraformaldehyde will be formed at lower temperatures. Samples are to be transferred from fixative (borate-buffered 10% formalin) to preservative (70% ethanol) after 72 hr (but within two weeks) 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, top-off the sample using 100% ethanol.

C. Laboratory Operations

The laboratory analysis of infaunal samples for the Bight'98 involves three processes: sample sorting, biomass estimation, and organism identification and enumeration. Quality assurance in the form of procedures and standardized reporting requirements are provided in the Infaunal Sample Analysis Laboratory Manual for all three processes. The QA Specialist (or designee) will conduct audits of each laboratory while sample analysis is underway to assure that the Bight'98 procedures are being followed. For the most challenging process, organism identification, additional quality assurance steps are included in order to foster comparability among the taxonomic data sets produced by the four participating laboratories. The quality assurance steps for taxonomic analysis are discussed separately below. **Sample sorting**

Quality control of sorting is essential to assure the value of all the subsequent steps in the sample analysis process. Sample material is sorted into six taxa lots: annelids, mollusks, arthropods, ophiurans, ophiuroids, miscellaneous echinoderms, and "other phyla". 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, 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%.

Two approaches are used for re-sorting. In one, a 10% aliquot of every sample processed by a sorter is resorted. In the other, 10% of the samples processed by a sorter are completely resorted. In both cases, all re-sorting is conducted by an experienced sorter other than the original sorter. For the Bight'98, either of the two approaches is acceptable. The re-sort method used is noted on the sorting form Quality Control Report section of the Sorting form along with results.

Percent sorting efficiency is:

$$\frac{\text{Number of Organisms originally sorted X 100}}{\text{\# of Organisms originally sorted + \# found in resort}}$$

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 technician 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 and, if of significant biomass, included in the sample biomass estimation.

Once all quality control criteria for sample sorting have been met, the sample debris may be is discarded.

Taxonomic analysis

The goal of taxonomic analysis for Bight'98 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'98 to provide an important element of quality assurance for this aspect of the project. 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, sharing of expertise, and monthly workshops. Results of the workshops and other information is communicated to the membership through a monthly newsletter.

SCAMIT's cooperation includes the provision of standards for nomenclature use and a mechanism for mutual assistance and exchange of information among the taxonomists involved in Bight'98. The taxonomic nomenclature used in Bight'98 follows the SCAMIT hierarchical species listing (SCAMIT 1994). This list represents a consensus for standard usage of taxa names in 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 special SCAMIT/Bight'98 workshops prior to the sampling period that focus on the taxonomy of groups requiring particular review to promote uniform treatment in the upcoming survey. Pre-survey workshops consider nemertea, platyhelminths, and other groups. The workshops provide training, pooling of regional resources, and designation of the local expert(s) to be called upon for assistance during sample analysis.

A pre-qualification exercise will be performed in order to assure comparability among laboratories identifying infaunal samples. Each organization will provide a list of taxonomists and their specialty areas. Taxonomists who were not involved in the 1994 survey will be sent two samples for ID. The results for number of taxa, number of organisms, and accuracy of the ID will be scored by a committee using procedures described in Montagne and Bergen (1997). If the results meet the minimum quality objectives (MQO), the taxonomist will be considered qualified. Otherwise, the benthic group will bring a recommendation on qualifying the taxonomist to the Steering Committee.

After sample analysis has begun, SCAMIT/Bight'98 workshops will continue at least monthly to address taxonomic problems arising during analysis of the Bight'98 samples. A process for integrating these workshops into the sample analysis process is described in the Infaunal Sample Analysis Laboratory Manual (Figure 6-1). Protocols for the erection and documentation of provisional species names, based largely upon SCAMIT recommendations (SCAMIT 1986), are provided in the Bight'98 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'98 workshops will culminate in a synoptic review 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). This review also draws upon

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the results of the quality control re-analysis of 10% of the samples analyzed by each laboratory.

While the SCAMIT/Bight'98 workshops are the primary means for exchange of information and assistance, the taxonomists participating in analysis of Bight'98 samples should maintain frequent and informal interaction throughout the process. The use of the Bight'98 bulletin board established for this purpose is encouraged.

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'98 samples analyzed in that laboratory. Procedures for the creation, maintenance and documentation of the voucher collections are provided in the 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'98 voucher collection upon completion of the survey. These collections provide material for review during SCAMIT/Bight'98 workshops and the synoptic review of the data upon completion of analysis.

The ultimate repository of the Bight'98 voucher collection and sample material has not yet been identified. This decision will have to balance the need to have the vouchers & sample material properly cared for; and the need to have the material easily available for subsequent review or re-analysis. Taxonomists involved in subsequent regional monitoring efforts will want access to the pilot project sample material. This access makes it possible for the taxonomist to re-identify taxa lots as appropriate to maintain the integrity of the original survey (see SCAMIT Comments & Recommendations to the Monitoring Sub-Committee of the Southern California Bight Review Committee, Jan 1998). SCCWRP's central role in the project as well as its central location makes it the logical repository of the sample material. This would require SCCWRP to make a long-term commitment to the maintenance of such collections, including curatorial care and management of future access. If this commitment cannot be met then other alternatives (e.g., natural history museums) will have to be explored.

Quality Control of Taxonomic Analysis

While the quality of taxonomic analysis in Bight'98 relies heavily on the measures described above, quality control is also provided by the re-identification of 10% of the samples processed by each laboratory. Re-identification will be conducted at a participating laboratory other than that which originally analyzed the samples. Samples for re-identification are selected randomly from each lab's assigned set of samples and randomly re-distributed to the other three laboratories. Results are reported on standardized re-identification sheets. The taxonomists conducting the re-identification do not have access to the original results.

The results are returned to the originating lab where the original two sets of results are compared recorded on the re-identification sheet and a standardized comparative report of results discrepancies is prepared. Discrepancies are identified and the report returned to the lab responsible for the re-identification. The two laboratories attempt to reconcile discrepancies. In the process, apparent error is discriminated from actual error and the number of each type of error recorded. Apparent errors are cases where the discrepancy is a result of a difference in the level of the identification, rather than a misidentification. For example, the discrepancy between a report of Tubulanus sp. and Tubulanus frenatus 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 a 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 a indication where assistance from other taxonomists involved in the Bight'98 is needed. Nomenclature differences are also examples of apparent error. Examples of real error are misidentifications and miscounts. In addition to characterizing analytical accuracy, this process provides information for the SCAMIT/Bight'98 synoptic review of the data compiled from the four laboratories at the end of the survey. Significant discrepancies in count are resolved by a third count.

A MQO of 10% has been established for the accuracy of taxonomic analysis of infaunal samples. After reconciliation of differences, the percent accuracy for the sample is calculated by the formula below. The calculation considers real errors only. The number of counting errors is based upon the difference between the original count and the resolved count.

Number of Organisms in QC resolved recount - Number of errors
X 100 Number of Organisms in resolved count

The following types of errors are included in the total number of errors:

· Counting errors (e.g., counting eleven individuals of a species as 10, including dead bivalves in a count); · Identification errors (e.g., identifying species X as species Y where both are present); · Unrecorded taxa errors (e.g., not identifying species X when it is present). · Recording errors (e.g., recording species X as species Y by recording on the wrong line on a pre-printed data entry sheet).

Each contributing laboratory must maintain an identification and enumeration accuracy of 90% or greater. If accuracy falls below this level, the taxa lot(s) contributing most to the error are singled out. These taxa lots in the preceding or next five samples analyzed by that laboratory (or taxonomist) must be re-analyzed. If the errors are found to be systematic, those taxa lots in all samples processed by that laboratory (or taxonomist) must be re-analyzed. The taxa lot(s) in which substantial error is found must be re-identified in all samples analyzed by the original laboratory. The calculated accuracy is reported on the Quality Control Accuracy Report, as well as any actions required. The completed report is signed by the responsible supervisor.

D. Information Management

Sample tracking

Each Laboratory will provide a means of sample tracking within their laboratory. The sample tracking process must include documentation of receipt of samples, assurance that sample storage procedures are followed and that required tracking information is transmitted to the Information Management Officer.

Record keeping and reporting

Each laboratory must be 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 Bight'98, certain standard forms of notation are employed with the taxonomist's bench sheet that assure that all labs collect the required formation in a uniform fashion. Standardized forms are used for sorting, biomass estimation, and all QC checks. Each participating laboratory will retain its taxonomic bench sheets and voucher sheets.

All QC reports will to be submitted with the analytical results. Copies of all these documents are to be retained by the individual laboratories. Copies of all quality control reports are to be provided to the

Quality Assurance Coordinator.

The laboratory manager supervisor is responsible for assuring that all steps in the process of analyzing infaunal samples follow Bight'98 procedures and that all QC steps are completed and documented. The manager supervisor must implement any specified corrective actions resulting from QC protocols. He or she is 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.

VII. SEDIMENT TOXICITY TESTING

A. Overview

This section describes QA/QC procedures that will be used for the assessment of sediment toxicity during the Bight 98 Survey. Four methods will be used to assess sediment toxicity during the survey. The toxicity of whole sediment will be measured using an amphipod (Eohaustorius estuarius) survival test. Samples of interstitial water will be evaluated for effects on bacterial luminescence using the Microtox test system. Sediment elutriates will be evaluated for toxicity to a dinoflagellate using the QwikLite luminescence test. The presence of organic compounds with the ability of to induce the CYP1A1 gene in mammalian cells will be measured in extracts of the sediment using the P450 RGS test.

B. Laboratory Capability

Prior to participating in the Bight 98 survey, the test laboratory must document their ability to conduct the tests with the selected test species. This should consist of a record of at least three prior tests in which test acceptability was attained. In addition, the laboratory should have conducted at least three prior reference toxicant tests so that a control chart can be constructed. A written description of the test method used must also be provided to the Steering Committee prior to the analysis of samples.

C. Interlaboratory Comparability

All laboratories conducting the amphipod survival tests must participate in an interlaboratory comparison exercise prior to sample testing. This exercise will include the analysis of field collected sediments and a reference toxicant test. Successful completion of this exercise by a laboratory will be evaluated using two criteria: 1) attainment of test acceptability criteria, and 2) agreement of results between laboratories. The criteria for establishing agreement of results will be determined 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.

Documentation of interlaboratory comparability is not needed for the Microtox, Quiklite, and P450 RGS methods, since these measurements will be conducted by single laboratories

D. Sample Collection

Methods for collection of sediment are described in the Field Operations Manual. Surface sediment (top 2 cm) will be collected from Van Veen grabs and stored in precleaned polyethylene jars. Samples may be

stored in the dark at 4 -C for up to two weeks before testing. Sediment samples should be homogenized in the laboratory before testing.

All samples shall be accompanied by chain of custody and sample tracking forms. These forms should include dates of receipt, homogenization, and testing of each sample, as well as storage conditions.

E. Amphipod Survival Test

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

Quality of test organisms

All test organisms will be obtained from a common source during the survey. 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 10 days or less.

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 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 cadmium dissolved in seawater. Cadmium concentrations will be selected to provide an estimate of the LC50 and will be verified by chemical analysis of the stock solution. Reference toxicant test results that fall outside of control chart limits (2 sd of mean) will trigger a review of test procedures and a possible retest of the corresponding sediment samples.

Test conditions

Water quality of the overlying water and pore water will be measured for each sample type at the beginning of the exposure. Overlying water quality will also be measured at the end of exposure. Temperature will be measured continuously in the exposure room. Instruments will be calibrated daily. Deviations in water quality will be noted on the data files.

Test acceptability

This toxicity test procedure is considered unacceptable if amphipod survival in "home sediment" is less than 90%, or if survival in any control replicate is less than 80%. Reference toxicant results must 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 EPA (1994) guidance.

F. Qwiklite (Qwiksed) Test

The QwikLite toxicity test measures the light output from bioluminescent dinoflagellates for assessment of toxic effects when exposed to many chemicals, either individually or in compounds, effluents, and antifoulant coatings. Successful bioassays have provided data on acute response as well as chronic effects

(from 3 hours up to 11 days) on two species of dinoflagellates, Gonyaulax polyedra and Pyrocystis lunula (QwikLite Basics 1996; ASTM 1997). The basis of detection is to measure a light reduction from bioluminescent dinoflagellates following exposure to a toxicant. Bioluminescence is the production of light by living organisms due to an enzyme-catalyzed chemical reaction. Upon exposure to a toxicant, the dinoflagellates may shed an outer cell membrane called a theca and form a cyst. Consequently, light production decreases from bioluminescent dinoflagellates. Encystment is a normal response by dinoflagellates to an unfavorable or stressful environment. Twenty-four hour toxicity tests will be conducted on leachates made from collected sediments.

Leachates are diluted to 6.25% of the original concentration for testing. Solutions of leachates are prepared with dinoflagellates at a concentration of approximately 200 cells/mL and dispensed into 5 replicates for each test concentration and controls. Bioluminescence measurements are conducted 24 hr following setup by measuring light output with a photomultiplier tube. The IC50 (concentration of the tested material which reduces or inhibits bioluminescence by 50% when compared to control cells) is calculated. A lower IC50 value indicates greater toxicity, since less leachate water is required to be mixed with clean seawater to produce the effect.

Quality of Test Organisms

All cultures were obtained from university culture collections and are maintained on a standard seawater media. Enriched seawater media (ESM) is routinely used for cell culture and maintenance (ASTM 1990). Subcultures of the dinoflagellates are routinely subsampled and counted for assuring a constant cell density in culture. Control aliquots of the cells are always tested with experimental cells to ensure a minimum bioluminescence is being emitted.

Accuracy and precision

Suitable reference materials are not available to determine the absolute accuracy of the QwikSed test. There is a strong positive correlation between QwikSed and the sea urchin development tests ($r=0.812$; $p<0.001$), indicating that the QuikSed test produces data comparable to other test methods. Five replicates of each test concentration will be tested, providing documentation of measurement precision. Between-test precision will be assessed using reference toxicants. Copper sulfate and cadmium chloride solutions will be periodically used throughout the test series as a positive control to check the sensitivity of the dinoflagellate cell stocks and compared to earlier IC50s. Reference toxicant results that fall outside of established control limits will trigger corrective action (assessment of test methods and possible reanalysis of samples).

Test conditions

Cultures of dinoflagellates are maintained in sterile ESM under 40-watt cool-white fluorescent bulbs on a 12:12hr (light:dark) cycle at 19-20-C. Cells are cultured in ~600 mL ESM in borosilicate Erlenmeyer flasks at 2000-3000 cells/mL. Bioluminescent dinoflagellates are most stimulable and produce maximum light during the dark phase.

Sediment leachates are prepared by mixing sediments with filtered seawater in a 1:4 ratio for 1.5 hours (COE and EPA, 1991). Total ammonia and pH in each sample is measured either with the HACH spectrophotometer or an Orion Ammonia electrode. IC50's from the QwikSed tests are compared with total ammonia measured in the leachates to detect confounding toxicity influences.

Test acceptability

Test acceptability includes the following: the culture and test temperature was not greater than 20-C nor less than 18-C; incident light on the cultures for maintenance and testing was approximately 4000 lux; mean control bioluminescence was not less than 1e6 PMT counts accumulated while stirring each cuvette for 30 sec after 24 of test setup; the pH of dilution replicates was within the range of 7.7-8.3.

G. Microtox^{TM} Test

A Microtox^{TM} test will be conducted on sediment pore water samples according to Microbics Corp. (1992) guidelines. The pore water will be obtained from centrifugation of sediment samples collected using a Van Veen grab. This test consists of exposing luminescent bacteria (Vibrio fisheri) to serial dilutions of test material and measuring the change in light production over time. a decrease in light production is considered a toxic response to the test material. The Microtox^{TM} software package calculates an EC50 (effective concentration of test material to produce a 50% decrease in light production) as the test end-point.

Quality of test organisms

All test organisms are obtained frozen from Azure Environmental (Carlsbad, CA). The test organisms are kept frozen until use. The quality of each vial of organisms is tested using a basic test protocol on a 100 mg/l phenol standard solution. An EC50 within a range of 13-26 mg/l indicates the acceptability of the vial. If the EC50 does not fall within the defined range, the vial will be discarded and a new vial tested.

Accuracy and precision

The relative sensitivity of the test organisms and test precision will be documented with the phenol standard reference toxicant test.

Test conditions

With the exception of turbid samples, water quality measurements will not be taken. Sample temperature is held constant (15 -C) during testing by the MicrotoxTM M500 analyzer. Turbid samples will be centrifuged to remove the turbidity before performing the test (Microbics Corp. 1992).

Test acceptability

Microtox^{TM} software calculates the EC50 and determines if the data meet test acceptability criteria. Most test rejections are due to pipetting errors. If a test is rejected, the sample will be retested.

H. P450 Reporter Gene System (RGS)

Analysis of sample extracts using the P450 Reporter Gene System (RGS) will follow methodology described elsewhere (Anderson et al, 1995; APHA, 1996, ASTM, 1997). The P450 Reporter Gene System (RGS) utilizes a human cell line (101L) stably transfected with a plasmid containing firefly luciferase linked to the human CYP1A1 promoter. Extracts of soil, sediment, or tissue (using EPA Method 3550) are applied at volumes of 2-20 mL to replicate wells in 6-well plates. In the presence of compounds which induce CYP1A1, including high molecular weight PAHs, coplanar PCBs, dioxins, and furans, luciferase is produced by these cells. Following the addition of the substrate luciferin, an enzymatic reaction produces light, which is measured in relative light units (RLU) using a luminometer. Fold induction (over solvent

blank) is then calculated for each sample and used to determine equivalency values.

Accuracy and precision A reference inducer is included during testing of each batch of environmental samples. The reference inducer, 2,3,7,8-Tetrachlorodibenzo-p-dioxin, (TCDD) at a concentration of 1 ng/mL, and a solvent blank, typically dichloromethane (DCM), are applied to replicate wells. The fold induction response, defined as the mean RLU of the test substance divided by the mean RLU of the solvent blank, is evaluated compared to a long-term Quality Control chart. Fold induction produced by 1 ng/mL TCDD on each test date must fall within two standard deviations of the mean fold induction from the QC chart. Calibration of the luminometer is performed monthly, using a luciferase control kit purchased from Analytical Luminescence (Cockeysville, MD).

Test acceptability

Environmental extracts are applied to three replicate wells, and the coefficient of variation (% CV) is evaluated for each sample. A CV that is >20% is unacceptable, and that extract must be retested. In addition, any extract that produces a fold induction response >100 must be diluted and retested. Typically, an extract is diluted 1:10 in DCM, and applied to three replicate wells.

VIII. FISH BIOMARKERS

A. Overview

This section describes QA/QC procedures that will be used for the assessment of contaminant exposure and effect biomarkers responses in flatfish. PAH exposure in fish will be estimated by measuring PAH metabolites in fish bile (bile FACs). PAH metabolites are detected based on their ability to fluoresce. Contaminant-induced effects in field exposed organisms will be estimated by measuring DNA damage in fish blood cells. The single cell comet assay will be used to measure DNA damage.

B. Sample Collection

The fish trawl, selection, and dissection protocols are described in detail in the 1998 Survey Field Operations Manual. Briefly, fish will be collected by otter trawl and dissected on board by trained personnel. The appropriate fish tissues will be immediately frozen on dry ice. In the laboratory, fish bile will be composited, aliquoted into amber vials, and stored at -80-C. Blood samples will be stored in liquid nitrogen until analyzed.

C. Bile FACs Measurement

Accuracy and precision

The concentration of fluorescent aromatic compounds (FACs) in fish bile will be measured according to Krahn et al. (1986) using HPLC and fluorescence detection. A five point calibration curve is established using PAH standards that encompass the expected range of sample concentrations. Method quality assurance includes analysis of calibration standards, method blanks, replicates, and a bile reference pool prior to sample analysis.

A method blank is analyzed at the beginning of each set of samples. If the fluorescence response of the blank appears stable, it is used to correct the background noise of the calibration standards, the bile

reference pool, and all samples. If the fluorescence response exceeds the usual background noise, corrective maintenance is performed.

Before analyzing samples, a concentration of the calibration standard is analyzed three times to assess instrument stability. The performance of the HPLC is considered stable if the relative standard deviation (RSD) of the standard replicates is within 10%. If the RSD exceeds 10%, corrective maintenance is performed. The calibration standard is again measured after every 7 samples.

Accuracy and precision of the measurements is assessed by conducting replicate measurements of a bile reference sample containing elevated FACs. Reference samples are analyzed at a frequency corresponding to 10% of the samples in a batch. If the FACs value exceeds 30% of the expected value, corrective maintenance will be performed.

Data acceptability

Sample measurements should fall within the instrument calibration curve. If the fluorescence response exceeds the calibration curve, the sample is further diluted and re-analyzed, or smaller injection volumes of bile are used.

D. Comet Assay (DNA Damage)

Increases in cellular DNA damage have been found to coincide with decreased health status in numerous organisms. The integrity of fish blood cell DNA will be examined by measuring DNA single-strand breaks using the Comet assay. Twenty microliters of fresh fish blood will be transferred to 500 -l of cryo-storage buffer (phosphate buffered saline, pH 7.4 (PBS), 10% DMSO, and 50 -g/ml Protease (Sigma, proteinase K, P-0390)) and frozen in liquid nitrogen until time of analysis. The Comet assay procedures are identical those of Steinert et al. (1998) with the substitution of PBS in all buffer solutions. From thawed fish blood samples 3000-10,000 cells will be immobilized on a microscope slide in agarose. The immobilized cells will be lysed and the DNA denatured under alkaline conditions to express strand breaks. An electric field is applied across the slides allowing relaxed or broken strands of DNA to migrate away from the immobilized nuclei. The DNA is stained with ethidium bromide and analyzed using an epifluorescent microscope and image analysis software (Kinetics, Ltd, Komet, V. 3.1,) to determine the amount and distance of DNA migration from the nucleus of 25-100 cells per slide.

Accuracy and precision

Accuracy and precision of the measurements is assessed by conducting replicate measurements of known reference samples, human white blood cell reference samples, negative (no damage) control cells and positive (hydrogen peroxide treated) control cells. Twenty-one slides are processed through each denaturation/electrophoresis cycle, of these 21 samples (batch), 3 will be negative control slides and 3 positive control slides.

Slides with cell densities too high to perform image analysis will be diluted and re-run. The mean damage levels of the controls must be within the 95% confidence limits previously determined for the reference samples. Reference values outside the expected limits will require a review of the procedures used for that batch and repeat analysis of that batch of samples.

Data acceptability

Only DNA damage values from batches where positive and negative controls fall within the expected

limits will be acceptable.

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X. APPENDIX A.
BENTHIC LABORATORY PROCEDURES

INTRODUCTION

This document describes laboratory procedures to be followed in the analysis of infaunal samples collected for the Southern California Bight 1998 Regional Marine Monitoring Survey (Bight'98). The procedures described are based upon existing practices utilized in POTW monitoring programs within the region and those employed during the 1994 Southern California Bight Pilot Project (SCBPP). Some modifications have been made to assure data comparability and to facilitate the coordination of the quality control steps required for the Bight'98 infaunal survey. It is the responsibility of each participating laboratory's supervisor to assure 1) these procedures are followed during sample processing and analysis, 2) all quality control steps are implemented, and 3) copies of all records, forms, and documents generated in the process are securely maintained on file until all aspects of the survey and resulting reports are completed.

In overview, the process of sample analysis consists of four steps after receipt of the sample in the laboratory; 1) the sample is washed and transferred to preservative, 2) All organisms are removed from the

debris contained in the sample and sorted into major taxa groupings, 3) the biomass is estimated for these major taxa groupings, and 4) all specimens in the sample are identified and enumerated. Quality control activities are required for the steps 2 and 4. These include repeating the procedures at each of these steps for a sub-set of samples. Results of this process are used to determine whether the measurement quality objectives (MQOs) established for each of these steps are met.

In addition, taxonomists must participate in a series of workshops jointly sponsored by Bight'98 and the Southern California Association of Marine Invertebrate Taxonomists (SCAMIT) which will focus on taxonomic problems arising during analysis of the Bight'98 samples. These workshops culminate in a synoptic review of the data set compiled from all participating laboratories.

Copies of this manual are available on the web site of the Southern California Coastal Water Research Project (http://sccwrp.org).

1. SAMPLE TREATMENT AND STORAGE

1.1 Upon receipt in the laboratory, samples will be in formalin fixative and must be washed and transferred to preservative. The removal of formalin is necessary for two reasons. Formaldehyde becomes increasingly acidic over time and prolonged exposure damages organisms with calcareous structures (e.g., shelled mollusks). Also, formaldehyde is a noxious, potentially dangerous chemical; its replacement with ethanol makes subsequent handling of the sample safer. Other benefits of the washing process are the removal of excess silt from mudballs that may have broken down during fixation and, in some cases, the opportunity to separate the bulk of organisms in a sample from the inorganic debris through the application of an elutriation process.

1.2 The samples are to remain in buffered fixative for at least 72 hours. No sample should remain in fixative for longer than two weeks.

1.3 The preservative to be used for infaunal samples is a 70% solution of ethanol. It is recommended that the preservative be buffered with marble chips, especially if the ethanol used is produced by industrial distillation rather than fermentation.

1.4 Procedure

1.4.1 Working under a fume hood and with eye protection, decant fixative through a 0.5mm or finer mesh sieve.

1.4.2 After decanting the formalin, refill the sample container with water, agitate gently by swirling, and wash the entire sample into the sieve.

1.4.3 Gently wash the sample with a low-pressure stream of water to remove any fine silt.

1.4.4 Using a spatula and wash bottle containing preservative, transfer the sample back to the sample container, top the sample with preservative, and tightly affix the lid.

1.4.5 Place an internal label in each sample container bearing the station name, sampling date, split number (if more than one container is used. Labels are to be written in pencil or indelible ink on 100% rag-paper, poly-paper, or other paper suitable for permanent wet labels.

1.4.6 After each sample is washed, closely examine the sieve to assure that all organisms have

been removed to avoid cross contamination of subsequent samples.

1.4.7 Elutriation. If a sample is primarily coarse sand, subsequent sorting can be greatly facilitated if inorganic material in the sample is separated from the lighter organic debris and organisms by the following elutriation process.

1.4.8 After washing the formalin from the sample, spread the sample material out in a shallow pan and cover with water.

1.4.9 Gently agitate the sample by hand to allow the lighter fraction of debris and organisms to separate from the heavier material.

1.4.10 Decant the water off with the lighter material through the sieve. Repeat the process several times until no more material is observed being carried off in the decanted water.

1.4.11 Collect the material carried off in the decanted water into a small sample container, top with preservative, and return to the original sample container along with the balance of the sample material. Fill the container with preservative and tightly affix the lid. Be sure that both the containers are properly labeled with internal labels.

1.5 Store infaunal samples in a safe and secure manner protected from environmental extremes. Avoid temperatures above 30°C as high temperatures will lead to evaporative loss of preservative.

1.6 Routinely inspect all samples to assure that the container closure is tight and the preservative level adequate. If evaporative loss of preservative is evident, top-off the sample using 100% ethanol. The use of 70% ethanol for this purpose will lead to dilution of the sample preservative because of the different evaporation rates of ethanol and water.

2. SAMPLE SORTING

2.1 Sorting is the process by which organisms (that were alive at time of collection) in a benthic sample are removed from the organic and inorganic residues that compose the sample and sorted into broad taxonomic categories for subsequent taxonomic analysis. Sorting must be accurate and complete to assure the value of all the subsequent steps in the sample analysis process.

2.2 Procedure

2.2.1 All laboratories participating in the Bight'98 infaunal survey have established sorting procedures that are compatible with the aims of this survey. The following points stipulate those elements essential to the process or unique to the Bight'98.

2.2.2 Begin the sorting process by filling out a Bight'98 Sorting Record form with the sample name, date, sorter's name, and date sorting begins. If the sample consists of more than a single jar, they are to be treated together as a single station. Make sure you have all jars composing the sample.

2.2.3 Sort the sample under a stereo microscope. It is recommended that the sample be sorted in small volume increments.

2.2.4 The entire sample is to be sorted. If an unusual sample is encountered for which sorting

of an aliquot may be a reasonable alternative, the laboratory supervisor is to contact the Bight'98 Benthic Specialist. The decision whether to allow sorting by aliquot will be made by the Benthic Specialist.

2.2.5 All sorting must be done in 70% ethanol, with care taken to assure that the sample being sorted is always fully covered with alcohol.

2.2.6 The organisms removed from the sample are sorted into the lots for which biomass will be estimated. These are:

Other Phyla is a single collective lot containing all other phyla.

2.2.7 Remove all individual organisms (including nematodes) and fragments from the sample with the exception of foraminiferans and planktonic species or life stages. All fragments, such as decapod chelae and legs, should be placed in their respective taxa lots. Sorters are to be instructed "If in doubt, pick it out". 2.2.8 Note on the Sorting Record form the number of taxa lots composing the sorted sample, the number of containers used if sample is split, and the time (to the nearest ½ hour) required to sort the sample. 2.2.9 Aggregate the taxa lots into one or more sample containers. Each taxa lot should be internally labeled with the station name (a four digit number). Place an internal label in each sample container bearing the station name, sampling date, split number (if more than one container is used). Labels are to be written in pencil or indelible ink on 100% rag-paper, poly-paper, or other paper suitable for permanent wet labels.

3. BIOMASS ESTIMATION

3.1 An estimation of biomass is determined, based upon wet-weights of the six taxonomic categories into which the organisms were sorted. Biomass is reported to the nearest 0.1 gram (wet weight).

3.2 Procedure

3.2.1 All laboratories participating in the Bight'98 survey have established wet-weight biomass procedures that are compatible with the aims of this survey. The following points are intended to stipulate those elements essential to the process or unique to the Bight 98 survey. Either of the two methods used by participating laboratories for removing excess preservative prior to weighing may be used: draining organisms on a fine sieve, followed by air-drying for a measured 5 minutes on absorbent paper; or pouring the sample into a funnel fitted with a fenestrated plate, followed by the application of gentle vacuum to pass air through the sample until liquid is no longer visible in the funnel stem. Because biomass is being estimated as wet weight, both techniques are considered to yield equivalent results.

3.2.2 Biomass estimations are required for each of the six taxa lot created in the sorting process. These are:

Annelida	Mollusca	Misc. Echinodermata
Arthropoda	Ophiuroidea	

3.2.3 All taxa lots should be inspected by a taxonomist prior to weighing to assure that all individuals and fragments have been properly grouped and that foraminifera and plankton have

not been included in the sample. The mollusk lot should also be inspected to assure that empty mollusk shells are not included in the biomass estimation.

3.2.4 An electronic balance capable of reading to 0.01 gram is to be used for biomass estimation. The balance must be calibrated prior to conducting the analysis.

3.2.5 Begin the biomass estimation process by filling out the Bight'98 Biomass Estimation Record with the sample log number, station, date, technician's name, and date of biomass estimation. If more than one container comprises the sample, make sure you have all containers for the sample.

3.2.6 Remove hermit crabs from shells prior to weighing.

3.2.7 To avoid biasing the biomass data, very large organisms are to be weighed separately. For example, the chance capture of a megafaunal animal such as an Allocentrotus fragilis, would typically result in an echinoderm biomass tens or hundreds of times that contributed by all other echinoderms in the sample. In this case, separate biomass estimations are to be determined for the Allocentrotus and for the remaining specimens comprising the taxa lot. If a technician is uncertain whether an organisms should be treated in this manner, the laboratory supervisor should be consulted.

3.2.8 The measured net biomass is to be recorded to the nearest 0.01 gram (wet weight). Record the report biomass of each taxa lot (and any large individuals) to the nearest 0.1 gram (wet weight) on the Biomass Estimation Record. The gross, tare and net weights of each measurement must be recorded.

4. TAXONOMIC ANALYSIS AND ENUMERATION

4.1 The object of taxonomic analysis is to accurately identify all organisms contained within each sample to the lowest possible taxonomic category and to provide an accurate count of the organisms in each identified taxon.

4.2 The goal of the Bight'98 infaunal survey is to provide species level identifications whenever possible. However, because of difficulties in the taxonomy and the lack of expertise within the participating laboratories the following exceptions are made:

- Nematodes are identified to phylum Nematoda
- Kinorhynchs are identified to phylum Kinorhyncha
- Oligochaete annelids are identified to class Oligochaeta
- Hirudinean annelids are identified to class Hirudinea
- Podocopid ostracods are identified to order Podocopida
- Harpacticoid copepods are identified to order Harpacticoida

4.3 The number of organisms reported must account for all organisms in a sample alive at the time of collection. Care must be taken to avoid reporting empty mollusk shells or crustacean molts in the data. Fragments of bilaterally symmetrical organisms will be identified and counted only if the fragment includes the anterior end of the organism. For radially symmetrical organisms (e.g., ophiuroids, anthozoans) only fragments bearing the majority of the oral disk will be identified and counted.

4.4 Epibiotic (fouling) organisms are noted as present but not quantified. These data are not included in the

final survey data. The level to which epibiotic organisms are identified is left to the discretion of each laboratory.

4.5 Parasites are noted as present but not quantified. Ectoparasites of fish such as Livoneca, which may be temporary members of the benthic community, are counted.

4.6 Each participating laboratory will use their own taxonomy bench sheets for recording the identifications and counts.

4.7 Nomenclature and orthography follows that used in the Edition 3 of the Southern California Association of Marine Invertebrate Taxonomists' taxonomic listing (SCAMIT 1998). This list represents a consensus for standard usage of taxa names in POTW monitoring programs in the Southern California Bight.

4.8 Taxonomists are to employ two standard notations (Voucher and Exclude) for the annotation of their data sheets While other non-standard notation may also be used, the use of these standard notations is required where applicable. In addition, the Exclude code will be included as part of the electronic data record. See the Bight'98 Information Management Plan for the proper form for this field for data submission.

4.9 Voucher Notation

4.9.1 Form: The annotation employed for this purpose is the letter V followed by the number of specimens removed from the sample (i.e., V-3)

4.9.2 Purpose: To note the removal of specimens from a sample for use as vouchers. Use of this notation is essential to the process of quality control and assessment. Removal of organisms without annotation confuses the resolution of discrepancies during quality control re-analysis, and leads to overstatement of error rates.

4.9.3 Rule of Use: Removal of any specimens from a sample to the voucher collection is clearly noted on the bench sheet by means of the Voucher notation..

4.10 Exclude Notation

4.10.1 Form: The letters EX written on the row of the bench sheet containing the data record for the taxon to be excluded

4.10.2 Purpose: Provides an aid to data analysis when calculating metrics using the number of taxa present (e.g., diversity, species richness). This field in the final data set represents the taxonomist's recommendation that the reported taxon be excluded from counts of the number of taxa reported in the sample.

4.10.3 Rule of Use: The Exclude annotation is made on the bench sheet whenever a taxon should be excluded from counts of the number of taxa reported in the sample. This annotation is employed when three conditions co-exist:

The identification is not at the species-level (e.g., Pleustidae or Polydora sp).

And

The reported taxon is represented in the sample by other members of its taxon, which have been identified at lower levels.

And

The taxonomist cannot determine if the specimen is distinct from the other members of its taxon represented in the sample.

4.10.4 It is necessary that the taxonomists make this evaluation during sample analysis (i.e., by annotation of the bench sheet). It cannot be effectively applied after the fact, as there is no way of determining later whether the third criterion for use was met.

4.10.5 The EXCLUDE notation will be included as part of the electronic data record submitted by each laboratory. 4.10.6 Examples of Use:

Both Dipolydora sp and Dipolydora socialis are reported in a sample and the taxonomist cannot determine if the specimen reported as D. sp is distinct from D. socialis. Exclude (annotate record with **EX**)

An unidentifiable onuphid polychaete is reported as Onuphidae. It is the only member of its family present in the sample. **Do Not Exclude**

Both Modiolus sp and Modiolus capax are reported in a sample. However, the taxonomist is confident that the specimen identified at the genus-level is not M. capax. **Do Not** Exclude

4.11 Temporary "In-House" provisional names are erected for those specimens that a taxonomist considers to be distinctive but cannot match with an existing description. These provisional names act as markers for these taxa, allowing them to be consistently discriminated in the samples for which the taxonomist is responsible. In-house provisional names are supported by a written differential diagnosis (and figures if necessary) sufficient to allow taxonomists in the other participating laboratories to recognize the species. These diagnoses are sent to other taxonomists participating in the survey. The provisional name is formed from the lowest taxon name in which the specimen may be placed with certainty followed by a composite name containing the laboratory's Bight'98 code and a number; for example, Rhachotropis LA2.

4.12 Timely and frequent communication among the taxonomists analyzing the samples will improve the data produced in the survey. An e-mail list-server will be established that will facilitate this communication. All (and only) taxonomists involved in the Bight 98 survey will be members of the list. Messages posted to the list will automatically post to all members, assuring wide and uniform distribution of the contents.

4.13 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.

4.14 Messages posted to the list-server should always include in the subject line the taxon (if any) to which the posting refers. The body should always begin with the originators name, followed (if appropriate) by the Phylum, Class, Family of topic, then the remainder of the text.

4.15 Following identification and enumeration, all the specimens are retained in taxa lots within the sample. Minimally, the material must be segregated into the following 17 taxa lots:

This level of separation facilitates the quality control process and eases both the burden of re-analysis resulting from failure of a laboratory to meet the measurement quality objective and the recovery of material during the end-of-survey synoptic review.

Further segregation of all polychaetes at the family level has been found useful in some POTW monitoring surveys and is recommended.

4.16 All taxa lots within a sample are provided an internal label with the taxa lot name and station name. These taxa lots are contained in vials and all the lots in a sample aggregated into one or more sample containers. If a taxa lot includes bulky specimens, they may be placed loose in the sample container along with the shell vials containing the remainder of that and other taxa lots. An internal label is placed in each sample container bearing the station name, sampling date, split number (if more than one container is used; e.g., 1of 2). Labels are written in pencil or indelible ink on 100% rag-paper, poly-paper, or other paper suitable for permanent wet labels.

5. QUALITY CONTROL

5.1 The laboratory analysis of infaunal samples for Bight'98 involves four processes: sample washing and preservation, sample sorting, biomass estimation, and organism identification and enumeration. Quality assurance in the form of procedures and standardized reporting requirements are provided in this document for all four processes. Quality control exercises will be implemented at stages for which MQOs have been established (sample sorting, identification and enumeration). These exercises include repeating the procedures at each of these stages for a sub-set of samples. The results will be used to determine achievement of the MQOs established for each stage.

5.2 The approach employed to estimate infaunal biomass (measurement of the wet-weight of alcohol-preserved collective taxa lots) does not lend itself to meaningful quality control re-weighing. This variability is a result of the inability to achieve a stable and repeatable amount of preservative within a taxa lot between successive weighings. In addition, there is a tendency for material held in alcohol to lose weight over time. Toleration of the deficiencies of the technique is necessary in order to obtain an estimate of biomass while assuring the preservation of the specimens in a condition that will allow their subsequent identification.

5.3 For the most challenging process, organism identification, additional quality control steps are included in order to foster comparability among the taxonomic data sets produced by the participating laboratories and taxonomists

5.4 In addition, the Benthic Specialist (or designee) may conduct audits of each laboratory while sample analysis is underway to assure that the Bight'98 procedures are being followed.

5.5 Sample Sorting

5.5.1 Quality control of sorting is essential to assure the value of all the subsequent steps in the sample analysis process. An accuracy MQO of 5% (equivalent to 95% removal efficiency) has been set for this stage of the sample analysis. Achievement of this MQO will be determined by re-sorting of 10% of the residue remaining from the original sort.

5.5.2 A standard sorting form is used for tracking the sample. It includes the name of the technician responsible, time required for sorting, comments, and re-sorting results. Re-sorting

of samples is employed for quality control of sorting.

5.5.3 A minimum of 10% of all material in Bight'98 samples will be re-sorted to monitor sorter performance and to determine achievement of the MQO of 5%.

5.5.4 Two alternative approaches (described below) are used for re-sorting; the Aliquot method, or the Whole Sample method. The method chosen is at the option of the laboratory. However, a single method must be employed for all samples for which a laboratory provides sorting. The re-sort method used must be noted on the sorting form along with results.

5.5.5 *Aliquot Method:* A representative aliquot of at least 10% of the sample volume of every sample processed by each sorter is re-sorted.

5.5.6 *Whole Sample Method:* At least 10% of the samples processed by each sorter are completely re-sorted.

5.5.7 Regardless of the method employed, all re-sorting is conducted by an experienced sorter other than the original sorter.

5.5.8 The responsible supervisor of each participating laboratory is responsible for selection of the method to be used for re-sorting and the unbiased selection of samples and method of obtaining a sample aliquot.

5.5.9 The re-sorting process is to follow the procedures given in §2 of this document.

5.5.10 Percent sorting efficiency is calculated as follows:

Whole Sample Method: $\%_{\text{Efficiency}} = 100 * [\#\text{Orgs}_{\text{Orig sorted}}$ divided by $(\#\text{Orgs}_{\text{Orig sorted}} + \#\text{Orgs}_{\text{from Re-sort}})]$

Aliquot Method: $\%_{\text{Efficiency}} = 100 * [\#\text{Orgs}_{\text{Orig sorted}}$ divided by $(\#\text{Orgs}_{\text{Orig sorted}} + \#\text{Orgs}_{\text{from Re-sort}} * \%_{\text{aliquot}})]$

5.5.11 If sorting efficiency is greater than 95%, no action is required. Sorting efficiencies below 95% will require continuous monitoring of that technician until efficiency is improved. If the Whole Sample Method is employed, failure to achieve 95 % sorting efficiency will require re-sorting of all samples previously sorted by that technician.

5.5.12 Organisms found in the re-sort should be included in the results from the sample.

5.5.13 The calculated sorting efficiency is recorded on the Sorting Form for each sample for which QC re-sorting is conducted.

5.5.14 Sample debris left after sorting must be retained by the laboratory responsible for the sorting. It is to be properly labeled and preserved with 70% ethanol. Upon completion of all quality control and assessment steps for the survey, the Benthic Specialist will notify each participating laboratory that the sample debris may be discarded.

5.6 Quality Control of Taxonomic Analysis

5.6.1 The goal of taxonomic analysis for the Bight'98 infaunal survey is species level identification of all macrobenthic organisms collected and an accurate count of each species. This task is complicated by the participation of multiple laboratories and taxonomists in the analysis. Two approaches are taken for providing data quality control. The first is an assessment of each laboratory's accuracy by re-analysis of a subset of samples from each laboratory. The procedures for sample re-analysis are based upon those developed and employed in the Southern California Bight Pilot Project (Montagne & Bergen 1997). The second focuses on ensuring consistent and comparable results among the participating taxonomists through cooperative activities with SCAMIT.

5.6.2 Quality control is provided by the re-identification of 10% of the samples processed by each laboratory. Samples for re-identification are selected randomly from each lab's assigned set of samples by the Bight'98 Benthic Specialist and re-distributed to the other laboratories.

5.6.3 The re-identification will be conducted at participating laboratories and by taxonomists other than those who originally analyzed the samples. The taxonomists conducting the re-identification do not have access to the original results.

5.6.4 Each laboratory's supervisor will be informed by the Benthic Specialist as to which samples are to be re-identified. The laboratory supervisor is responsible for assuring that these samples are made available to the laboratory responsible for re-identification.

5.6.5 The specimens in each sample will be re-identified and enumerated using the procedures given in §4 of this document. Results are reported on the re-analytical laboratory's bench sheet. Upon completion of the re-analysis, the results and original analytical results are exchanged between laboratories.

5.6.6 The supervisors of the laboratories involved compare the original results to those of the re-analysis. All differences in results are listed on the Discrepancy Report. Only discrepancies are reported on this form. A copy of this report is sent to the laboratory responsible for the original analysis.

5.6.7 The two laboratories attempt to reconcile discrepancies. To facilitate this process, two to four SCAMIT/Bight'98 workshops will be scheduled in which taxonomists will jointly meet for discrepancy resolution. Significant discrepancies in count (±5% of original count) are resolved by a third count performed by the re-analytical lab.

5.6.8 The cause and resolution of discrepancies is reported on the Discrepancy Resolution Report. While completion of this report is the responsibility of the re-analytical laboratory, both labs must work together to reach agreement. If agreement cannot be reached, arguments are presented to the Benthic Specialist for a decision. The Benthic Specialist may seek assistance from SCAMIT members or other experienced taxonomists in reaching a decision.

5.6.9 Once resolution and explanation of all discrepancies has been completed, the Discrepancy Resolution report is sent to the Benthic Specialist along with copies of both laboratory's bench sheets and the Discrepancy Report. Copies of all reports and bench sheets are to be retained by both laboratories.

5.6.10 The Benthic Specialist reviews the results submitted, discusses with the laboratories

any issues needing clarification or arbitration.

5.6.11 The Benthic Specialist is responsible for completing the rest of the form, applying the Discrepancy classifications and Resolution codes (see foot of Discrepancy Resolution Report form), and determining the effect of the resolution (increase, decrease, or no change) on the number of taxa and the organism count reported in the original results.

5.6.12 These results are then used to calculate the % error of the original laboratory's analysis. Percent error will be calculated for three aspects of sample analysis; number of taxa discriminated (%Err# Tax), total organism count (%Err# Orgs), and identification accuracy (%ErrID).

5.6.13 The error rates are calculated as follows:

$\%Err_{\# Tax} = 100 * [(\# Taxa_{Resolved} - \# Taxa_{Original})$ divided by $\# Taxa_{Resolved}]$

$\%Err_{\# Orgs} = 100 * [(\# Organisms_{Resolved} - \# Organisms_{Original})$ divided by $\# Organisms_{Resolved}]$

$\%Err_{ID} = 100 * (\# Taxa_{MisID}$ divided by $\# Taxa_{Resolved})$

The first two aspects provide measures of data quality as relates to parameters such as species richness, abundance, and diversity. The third aspect, identification 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 calculations only consider errors in the original analysis. The results of these calculations are reported on the Infaunal ID & Enumeration Accuracy Report.

5.6.14 Based upon the results of data quality assessment for the SCBPP, an MQO of 10%, representing the maximum allowable deviation from the "true" value, has been established for number of taxa, total number of organisms, and identification accuracy. Each contributing laboratory must strive to avoid exceeding this level of error. The results of this assessment process will provide a measure of the quality of Bight'98 infaunal data, and add to the SCBPP baseline for selection of MQOs in future regional surveys based upon the SCBPP/Bight'98 model.

5.6.15 In addition to providing for an assessment of analytical accuracy, this process provides information for the end-of-survey SCAMIT/Bight'98 synoptic review of the data set compiled from the participating laboratories.

5.6.16 Each participating laboratory must create a voucher collection of all species identified in Bight'98 samples analyzed in that laboratory. These collections are separate from the laboratories' existing voucher collections and will be the source of material from which is drawn a common Bight'98 voucher collection upon completion of the survey. These collections provide material for review during SCAMIT/Bight'98 workshops and the synoptic review of the data upon completion of analysis.

5.6.17 The voucher collections are to contain specimen lots of one or more individuals of each reported taxon. The specimens are to be representative of the taxon. At the taxonomist's

discretion, more than one specimen lot may be added to the collection. This is particularly appropriate when differences in specimen maturity, or within-taxon variability need representation. Only those taxa discriminated to the species-level (or stipulated higher level e.g., Oligochaeta) are to be included in the collection. Species-level identification is considered to include provisional species and conditional taxa. Tentative identifications, as indicated by "?" are not to be represented. See the SCAMIT Newsletter (SCAMIT 1986) for protocols and recommendations on provisional and open nomenclature.

5.6.18 Only glass containers are used for the storage of the voucher material, unless specimens are inappropriate for wet storage. Each voucher container should contain an internal label bearing the complete taxon name, author and date. Within the voucher container each specimen lot should be contained within a shell vial closed with cotton or other stopper. Specimens too large to be contained in shell vials may be stored in jars. Each lot is to be accompanied by an internal label bearing the taxon name, station name of sample from which the specimen(s) was removed, a count of the number of specimens in the lot, the analytical laboratory's designation (OC, HY, etc.), and the identifying taxonomist's initials. The use of shell vials for all specimens other than large species will facilitate the consolidation of the voucher collections upon completion of the survey.

5.6.19 Labels are written in pencil or indelible ink on 100% rag-paper, poly-paper, or other paper suitable for permanent wet labels.

5.6.20 Taxonomists from the participating laboratories are required to participate in special SCAMIT/Bight'98 workshops. Workshops prior to the sampling period focus on the taxonomy of groups requiring particular review to promote uniform treatment in the upcoming survey. The workshops provide training, pooling of regional resources, and designation of the local expert(s) to be called upon for assistance during sample analysis.

5.6.21 Based upon these workshops and the results of the SCBPP quality control results, a limited number of taxa may be selected for special treatment. These are groups for which prior experience leads us to believe consistent identification will not be possible unless all the collected material is identified by a single taxonomist or small team of taxonomists. During regular sample analysis, all members of a taxon selected for this specialized treatment will be identified at a standard collective level (e.g., class or other high-level category), counted and segregated into a lot for subsequent processing by the specialist(s). Details of this process will be developed during the SCAMIT/BIGHT98 workshops.

5.6.22 After sample analysis has begun, SCAMIT/Bight'98 workshops continue at least monthly to address taxonomic problems arising during analysis of the Bight'98 samples. At these meetings, diagnoses of any "in-house" provisional taxa erected by any of the laboratories will be distributed to the other participants and assistance sought to resolve their identity. SCAMIT provisional species names will be provided for those found to be or suspected of being new species.

5.6.23 The series of SCAMIT/Bight'98 workshops culminates in a synoptic review of the data set compiled from all participating laboratories, and investigation of possible inconsistencies revealed in that process (including examination of voucher specimens or sample lots as needed for resolution). This review also draws upon the results of the quality control re-analysis of 10% of the samples analyzed by each laboratory.

6. RECORD KEEPING AND PROCEDURAL RESPONSIBILITY

6.1 Each laboratory must be 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'98 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. All QC reports are to be submitted to the Benthic Specialist upon completion of sample analysis. Copies of all these documents are to be retained by the individual laboratories. Analytical results are to be transmitted to the Information Management officer.

6.2 The laboratory supervisor is responsible for assuring that all steps in the process of analyzing infaunal samples follow Bight'98 procedures and that all QC steps are completed and documented. The supervisor must implement any specified corrective actions resulting from QC protocols. He or she is 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.

7. REFERENCES

Montagne, D. E. & M. Bergen. 1997. Quality Control and Assessment of Infaunal Identification and Enumeration: The SCBPP Experience. Southern California Research Project Annual Report 1996. Westminster, CA. pp 147-154.

SCAMIT. 1986. Protocols and Recommendations for the Use of Open Nomenclature. SCAMIT Newsletter, May 1986, vol. 5 No. 2.

SCAMIT. 1998. A Taxonomic Listing of Soft Bottom Macro- and Megainvertebrates from Infaunal and Epibenthic Monitoring Programs in the Southern California Bight. Edition 3. Southern California Assoc. of Marine Invertebrate Taxonomists, San Pedro, CA. 167 pp.

8. DATA FORMS

This section includes examples of the data forms used for the laboratory analysis and QC of Bight'98 infaunal samples. They are:

Infaunal Sorting Sheet and Sorting Quality Control Report

Infaunal Biomass Sheet

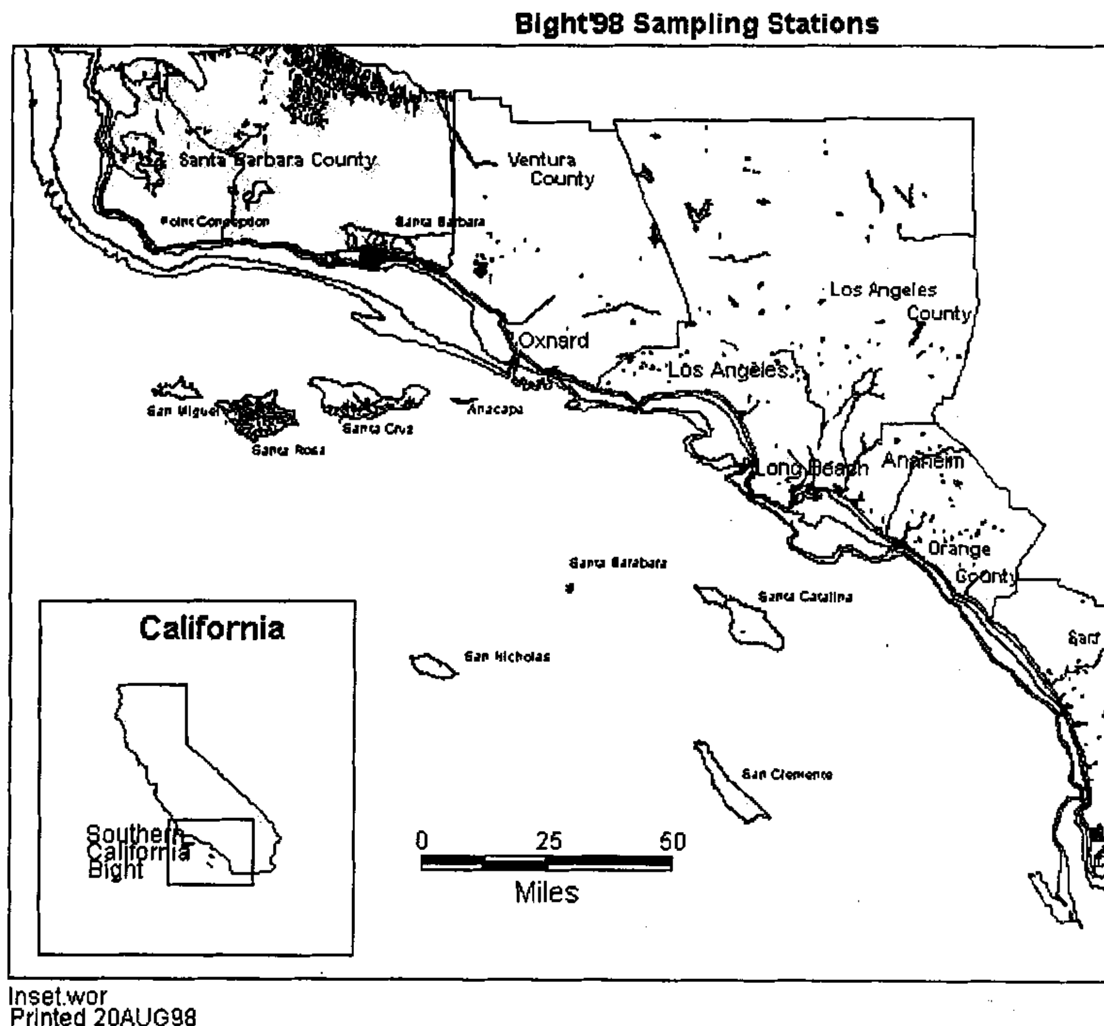
Infaunal Analysis QC Discrepancy Report (a multi-page form)

Infaunal Analysis QC Discrepancy Resolution Report (a multi-page form)

Infaunal Id & Enumeration Accuracy Report

FIGURES

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



TABLES

TABLE I-1. Participants in the Bight'98 Regional Monitoring Program. Participants in the coastal ecology component are asterisked.

AES Corporation*
Algalita Marine Research Foundation*
Aliso Water Management Authority (AWMA)*
Aquatic Bioassay and Consulting Laboratories (ABCL)*
Center for Environmental Cooperation (CEC)*
Central Coast Regional Water Quality Control Board*
Channel Islands National Marine Sanctuary (CINMS)*

Chevron USA Products Company*
City of Long Beach
City of Los Angeles Environmental Monitoring Division (CLAEMD)*
City of Los Angeles Stormwater Division
City of Oceanside*
City of Oxnard*
City of San Diego*
City of Santa Barbara
City of Ventura
Columbia Analytical Services*
Divers Involved Voluntarily in Environmental Rehabilitation & Safety (DIVERS)
Encina Wastewater Authority*
Goleta Sanitation District
Granite Canyon Marine Pollution Studies Lab*
Houston Industries, Inc.*
Instituto de Investigacione, Oceanologicas (UABC)
Los Angeles Department of Water and Power (LADWP)*
Los Angeles County Dept. of Beaches & Harbors*
Los Angeles County Dept. of Health Services
Los Angeles Regional Water Quality Control Board*
Los Angeles County Sanitation Districts (LACSD)*
Marine Corps Base - Camp Pendleton
National Fisheries Institute of Mexico (SEMARNAP)*
NOAA International Programs Office
NRG Energy, Inc.*
Orange County Environmental Health Division
Orange County Public Facilities and Resources (OCPFRD)*
Orange County Sanitation District (OCSD)*
San Diego County Dept. of Environmental Health
San Diego Interagency Water Quality Panel (Bay Panel)*
San Diego Regional Water Quality Control Board (SDRWQCB)*
San Elijo Joint Powers Authority*
Santa Ana Regional Water Quality Conrol Board*
Santa Barbara Health Care Services
Santa Monica Bay Restoration Project*
Secretaria de Marina (Mexican Navy)
Southeast Regional Reclamation Authority (SERRA)*
Southern California Coastal Water Research Project (SCCWRP)*
Southern California Edison (SCE)*
Southern California Marine Institute (SCMI)
State Water Resources Control Board (SWRCB)*
Surfrider Foundation
University of California, Santa Barbara
USC Wrigley Institute for Environmental Studies (WIES)*
US EPA Region IX*
US EPA Office of Research and Development*
US Geological Survey
US Navy, Space & Naval Warfare Systems Center, San Diego (USN)*

TABLE 2-1. Measurement Quality Objectives for Bight'98 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%
counting	10%	NA	90%
identification	10%	NA	90%
Sediment Toxicity			
amphipod survival	NA	2 SD	90%
Microtox	NA	2 SD	90%
QwikLite	NA	2 SD	90%
P450 RGS	NA	2 SD	90%
Biomarkers			
bile FACs	30%	30%	90%
DNA damage	NA	2 SD	90%
Demersal fish and macroinvertebrates			
sample collection	NA	NA	90%
counting	NA	10%	90%
identification	5%	NA	90%
length	NA	10%	90%
biomass	NA	10%	90%
ross pathology	5%	NA	90%
Contaminants in fish	30%	30%	90%

Table 5-1. Constituents that will be measured in marine sediments and whole fish by laboratories participating in the Southern California Bight Pilot Project.

	Sediment	Fish		Sediment	Fish
Aluminum	Yes	No	PCB Congenersa	Yes	Yes
Antimony	Yes	No	4,4'-DDT	Yes	Yes
Arsenic	Yes	No	2,4'-DDT	Yes	Yes
Barium	Yes	No	4,4'-DDD	Yes	Yes
Beryllium	Yes	No	2,4'-DDD	Yes	Yes

Cadmium	Yes	No	4,4'-DDE	Yes	Yes
Chromium	Yes	No	2,4'-DDE	Yes	Yes
Copper	Yes	No	a-Chlordane	Yes	Yes
Iron	Yes	No	g-Chlordane	Yes	Yes
Lead	Yes	No	5-phenyldecane	Yes	No
Mercury	Yes	No	4-phenyldecane	Yes	No
Nickel	Yes	No	3-phenyldecane	Yes	No
Selenium	Yes	No	2-phenyldecane	Yes	No
Silver	Yes	No	6-phenylundecane	Yes	No
Zinc	Yes	No	5-phenylundecane	Yes	No
Acenaphthene	Yes	No	4-phenylundecane	Yes	No
Acenaphthylene	Yes	No	3-phenylundecane	Yes	No
Anthracene	Yes	No	2-phenylundecane	Yes	No
Benz[a]anthracene	Yes	No	6-phenyldodecane	Yes	No
Benzo[a]pyrene	Yes	No	5-phenyldodecane	Yes	No
Benzo[b]fluoranthene	Yes	No	4-phenyldodecane	Yes	No
Benzo[e]pyrene	Yes	No	3-phenyldodecane	Yes	No
Benzo[g,h,i]perylene	Yes	No	2-phenyldodecane	Yes	No
Benzo[k]fluoranthene	Yes	No	7&6-phenyltridecane	Yes	No
Biphenyl	Yes	No	5-phenyltridecane	Yes	No
Chrysene	Yes	No	4-phenyltridecane	Yes	No
Dibenz[a,h]anthracene	Yes	No	3-phenyltridecane	Yes	No
Fluoranthene	Yes	No	2-phenyltridecane	Yes	No
Fluorene	Yes	No	7-phenyltetradecane	Yes	No
Indeno(1,2,3-c,d)pyrene	Yes	No	6-phenyltetradecane	Yes	No
Naphthalene	Yes	No	5-phenyltetradecane	Yes	No
Perylene	Yes	No	4-phenyltetradecane	Yes	No
Phenanthrene	Yes	No	3-phenyltetradecane	Yes	No
Pyrene	Yes	No	2-phenyltetradecane	Yes	No
2,6-Dimethylnaphthalene	Yes	No	Total organic carbon	Yes	No
1-Methylnapthalene	Yes	No	Lipid	No	Yes
2-Methylnapthalene	Yes	No	Sediment grain size	Yes	No
1-Methylphenanthrene	Yes	No	Acid volatile sulfide	Yes	No
1,6,7-Trimethylnaphthalene	Yes	No			

[a]Congeners 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, 201, 206.

Table 5-2. Summary of chemistry sample collection and holding time conditions for the Bight'98

Sediment Parameter	Containera Type	Sample size Size (g)	Preservation Requirements	Maximum Holding Time
Sediment grain size	plastic or glass	100 (80% full)	cool (4°C)	28 days
Sediment total organic carbon	glass	200 (80% full)	frozen (-20°C)	6 months
Sediment trace metals	glass/plastic	200 (80% full)	frozen (-20°C)	6 months
				(2 months for Hg)
Sediment trace organics	glass	2′200 or 1′500 (80% full)	frozen (-20°C)	6 months
Sediment AVS-SEM	polycarbonate	200 (250 ml)	cool (4oC)	6 months
Fish trace organics	Container	Sample	Preservation	Maximum

Table 5-3. Data quality requirements for the Bight'98 trace metal measurements.

Measurement	Frequency	Control Limit
Method Blank	1/batch	concentration in samples
Certified Reference Blanks		
PRA PPS-46, Priority Pollutant Soil certified standard, Lot#237 and CRM 016-500, Lot#516	1/batch	See Table 5.9
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 25% true value. Should all spiked sample recoveries be greater than 25% of true value, add a post-digestion spike to the unspiked sample and analyze. If all spike recoveries are greater than 25% of true value, note matrix caused poor spike recovery. If all

		spike recoveries are less than 25%, 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 3s)
ICP-MS		
Tuning solution	4 at start of run	RPD < 5%
Calibration	Initial setup	Minimum 1 blank and one calibration standard
Initial calibration verification (ICV)		2 points/batch ±10% true value
Continuing calibration verification (CCV)	10%	±10% true value
Calibration Blank	10%	MDL, run two more times, the average must be MDL, reanalyze.
Matrix spike	10%	At least one matrix spike per batch must be within 25% true value; £ 20% RPD for over 10 times MDL. If ³ 20% RPD and post-digestion spike recovery is > 25% note matrix problem. If > 20% RPD and post-digesiton spike recovery is less than or equal to 25% repeat digestion and analysis
Spiked blank	1/batch	±25% true value
Duplicate sample or matrix spike sample	10%	Statistical process control analyses (within 3s)
Intensity standard	During run	Intensity between 30 and 120% of the internal standard
Atomic Absorption (AA, GFAA, Hydride Generation, Cold Vapor)		
Calibration	Initial setup	Minimum 1 blank and three calibration standards; linear coefficient greater than or equal to 0.995
Initial calibration verification (ICV)	1/batch	±10% true value
Continuing calibration verification (CCV)	10%	±20% true value
Calibraiton Blank	10%	MDL, run two more times, the average must be MDL, reanalyze.
Matrix spike	10%	At least one matrix spike per batch must be within 15% true value. If all matrix spike analyses are greater than or equal to 15%, interference test must be conducted
Spiked blank	1/batch	15% true value
Duplicate sample or matrix spike sample	10%	Statistical process control analyses (within 3s)
		(a) Dilution test: Select typical sample with concentration 25 times the MDL. Dilute sample 5 times. The concentration of the

Interference check	As required	undiluted sample and 5 times the concentration od thediluted 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 the data quality requirements for measurements of polycyclic aromatic hydrocarbons and linear alkylbenzenes[a]

Measurement	Frequency	Control Limit
Initial calibration		Relative standard deviation (RSD) < 25% for all analytes
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 20% for 80% of the analytes
Method Blank	1/batch	Below reporting levels for all analytes
Matrix spikes/MS duplicates	1/batch	For evaluaiton onlu as part of the on-going QA/QC efforts
Reporting level spikes	1/batch	For evaluaiton onlu as part of the on-going QA/QC efforts
Certified reference material	1/batch	For PAHs only[b]
Surrogate spikes	1/sample	Laboratories develop their own control limits
Internal standards (Optional)	1/sample	Laboratories develop their own

[a]There should be 20 samples or less in each extraction batch and a reasonable number of samples in one instrument batch.

[b]Certified values were obtained by a different analytical procedure from what the participating laboratories are employing; therefore, direct comparison is impossible. The performance criteria agreed by the group is AVERAGE ± 3'STANDARD DEVIATION.

Table 5-5. Data quality requirements for measurements of chlorinated hydrocarbons[a].

Measurement	Frequency	Control Limit
Initial calibration		Relative standard deviation (RSD) within ±15% for 80% of the analytes
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	Below reporting levels for all analytes
Sample duplicates	1/batch	RPD < 30%
Reporting level spikes	1/batch	For evaluaiton onlu as part of the on-going QA/QC efforts (performed on clean sediment or tissue)
Certified reference material	1/batch	Within ±30% of the true value for 80% of the analytes
Surrogate spikes	1/sample	Laboratories develop their own control limits
Internal standards (Optional)	1/sample	Laboratories develop their own

Table 5-6. Data quality requirements for measurements of total organic carbon.[a]

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	Below reporting levels for all analytes
Sample duplicates	1/batch	RPD < 30%
Certified reference material	1/batch	Within ±20% of certified value
[a]There should be 20 samples or less in each extraction batch and a reasonable number of samples in one instrument batch.		

Table 5-7. Reporting objectives for Bight '98.

	Sediment (ng/g dry)	Fish (ng/g wet)	Sediment (ng/g dry)	Fish (ng/g wet)	
Aluminum	a	NA	PCB Congeners[b]	7.5	20[c]
Antimony	10,000	NA	4,4'-DDT	1	10
Arsenic	1,600	NA	2,4'-DDT	1	10
Barium	a	NA	4,4'-DDD	1	10
Beryllium	200	NA	2,4'-DDD	1	10
Cadmium	200	NA	4,4'-DDE	1	10
Chromium	16,000	NA	2,4'-DDE	1	10
Copper	7,000	NA	a-Chlordane	d	f
Iron	a	NA	g-Chlordane	d	f

Lead	9,300	NA	5-phenyldecane	50	NA
Mercury	30	NA	4-phenyldecane	50	NA
Nickel	4,200	NA	3-phenyldecane	50	NA
Selenium	1,000	NA	2-phenyldecane	50	NA
Silver	200	NA	6-phenylundecane	50	NA
Zinc	30,000	NA	5-phenylundecane	50	NA
Acenaphthene	50	NA	4-phenylundecane	50	NA
Acenaphthylene	50	NA	3-phenylundecane	50	NA
Anthracene	50	NA	2-phenylundecane	50	NA
Benz[a]anthracene	50	NA	6-phenyldodecane	50	NA
Benzo[a]pyrene	50	NA	5-phenyldodecane	50	NA
Benzo[b]fluoranthene	50	NA	4-phenyldodecane	50	NA
Benzo[e]pyrene	50	NA	3-phenyldodecane	50	NA
Benzo[g,h,i]perylene	100	NA	2-phenyldodecane	50	NA
Benzo[k]fluoranthene	50	NA	7&6-phenyltridecane	50	NA
Biphenyl	50	NA	5-phenyltridecane	50	NA
Chrysene	50	NA	4-phenyltridecane	50	NA
Dibenz[a,h]anthracene	100	NA	3-phenyltridecane	50	NA
Fluoranthene	50	NA	2-phenyltridecane	50	NA
Fluorene	50	NA	7-phenyltetradecane	50	NA
Indeno(1,2,3-c,d)pyrene	100	NA	6-phenyltetradecane	50	NA
Naphthalene	50	NA	5-phenyltetradecane	50	NA
Perylene	50	NA	4-phenyltetradecane	50	NA
Phenanthrene	50	NA	3-phenyltetradecane	50	NA
Pyrene	50	NA	2-phenyltetradecane	50	NA
2,6-Dimethylnaphthalene	50	NA	Total organic carbon	a	NA
1-Methylnapthalene	50	NA	Lipid	NA	a
2-Methylnapthalene	50	NA	Sediment grain size	a	NA
1-Methylphenanthrene	50	NA	Acid volatile sulfide	NA	NA
1,6,7-Trimethylnaphthalene	50	NA			

aReport value.
[b]Congeners 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, 201, 206.
[c]GC/MS method has a reporting level of 40 ng/g of fish homogenate (1:1 fish:water) and samples containing undetectable PCBs will be re-analyzed with a reporting level of 20 ng/g of fish homogenate.
[d]Will be determined later based on biological thresholds.

Table 5-8. Certified reference materials recommended by the Bight'98 Chemistry Technical Committee. SRMs are available from NIST (301/975-6776); all other reference materials are available from NRC (613/993-2359).

Calibration solution	
SRM 1491	Aromatic hydrocarbons in hexane/toluene
SRM 1492	Chlorinated pesticides in hexane
SRM 1493	Polychlorinated biphenyl congeners in 2,2,4-trimethylpentane
Environmental matrix (Organics)	
LCM	Aromatic hydrocarbons and chlorinated hydrocarbons in marine sediment from Santa Monica Bay and the Palos Verdes Shelf; acceptance ranges are determined by participating laboratories
CARP-1	Chlorinated hydrocarbons in whole fish
Environmental matrix (Trace Metals)	
CRM-016-050	Metals on sediment, Lot L516 (aluminum, arsenic, barium, beryllium, cadmium, chromium, copper, iron, lead, mercury, nickel, and zinc in sediment)
ERA PPS-46	Priority Pollutant Soil Certified Standard, L 237 (antimony, selenium, and silver)
Environmental matrix (total organic carbon)	
PACS-1	TOC in marine sediment

Table 5-9. Limit ranges used for performance evaluation of trace metal measurements

(CRM-016-050, Metals on Sediment, Lot 516)		
Constituent	**True Value, Mg/Kg**	**Specified Limits, Mg/Kg**
Arsenic	6.48	0.8 - 12.2
Barium	79.3	56.9 - 101.8
Beryllium	0.49	ND - 1.1
Cadmium	0.47	ND - 1.2
Copper	15.5	11.9 - 19.1
Iron	16,800	11,700 - 22,000
Lead	14.1	7.7 - 20.6
Mercury	0.11	0.02 - 0.21
Nickel	16.7	11.9 - 21.6
Zinc	69.7	49.1 - 90.3
(ERA PPS-46, Priority Pollutant soil certified standard, Lot 237)		
Antimony	34.4	ND - 100
Chromium	88.2	70.5 - 106
Selenium	51.5	33.6 - 69.3
Silver	70.6	52.6 - 88.7